Final Scientific/Technical Report

"Develop Improved Materials to Support the Hydrogen Economy"

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Working Partners:	See Individual Project Reports
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DOE Managers: Technical Project Officer: Reginald Tyler
Contracting Officer: Stephanie Carabajal

Executive Summary:

Project Objective and Background: The Edison Materials Technology Center (EMTEC) solicited and funded hydrogen infrastructure related projects that have a near term potential for commercialization. The subject technology of each project is related to the US Department of Energy hydrogen economy goals as outlined in the multi-year plan titled, "Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan." Preference was given to cross cutting materials development projects that might lead to the establishment of manufacturing capability and job creation.

The Edison Materials Technology Center (EMTEC) used the US Department of Energy hydrogen economy goals to find and fund projects with near term commercialization potential. An RFP process aligned with this plan required performance based objectives with go/no-go technology based milestones. Protocols established for this program consisted of a 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 specified proposals/projects for hydrogen production, hydrogen storage or hydrogen infrastructure processing which may include sensor, separator, compression, maintenance, or delivery technologies. EMTEC was especially alert for projects in the appropriate subject area that have cross cutting materials technology with near term manufacturing and commercialization opportunities.

Accomplishments: EMTEC funded and provided oversight to forty-six subcontract projects that advanced over thirty technologies. The program provided phased additional funding to the more successful projects, which allowed many of the projects to access additional funding from the Ohio Third Frontier Program, DoE or industrial investment.

At a DoE meeting in 2005 for congressionally directed projects we were told that no such project had ever won and R&D100 award and challenged to do so. Working with Makel and Argonne this program won an R&D100 award in 2006.

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Catacel – Hydrogen Production

Novel Stackable Structural Reactor (SSRTM) for Low-cost Hydrogen Production
EFC-H1-09-1A; EFC-H1-09-2A; EFC-H3-07-1; EFC –H4-09A

Project Overview: The Catacel SSR® is intended to be a drop-in replacement for conventional ceramic catalyst media in the stationary steam reforming process that has been used for many years to produce hydrogen from natural gas. Current technology requires that the ceramic catalyst bed be replaced every three to five years due to mechanical degradation of the media. The Catacel SSR will eliminate the periodic replacement that is required for ceramic packed beds. SSR is expected to provide a significant capacity increase (~30%) or a significant energy savings (~20%) in most reformers that use this technology. In an existing plant, simple replacement of catalyst with SSR is expected to show a 1-year payback on investment with energy savings.

In the Phase II project, catalyst formulations were synthesized and verified, manufacturing processes were devised and implemented, lab durability work was performed, and installation methods were devised. This project provided convincing evidence that SSR would show benefit in a real plant with minimal risk to the plant operator.

Subsequent to the Phase II project, Catacel reached an agreement with Hydro-Chem (division of Linde) to advocate and assist with installation of SSR in a new 250m3/hr hydrogen plant for Borcelik Steel in Gemlick, Turkey. Borcelik agreed to the installation. Using internal funds, Catacel manufactured 8ft3 of SSR product and numerous installation tools. Catacel personnel installed SSR in the plant on July 14-15, 2008. The installation process was extremely smooth. The plant started up on July 20, and reached full capacity on July 24. Since then, it has been continuously operating at full capacity.

Catacel expected to obtain operational data from the Borcelik plant that would illuminate SSR performance advantages. Unfortunately, it does not appear that this data will be meaningfully attainable. In the Borcelik plant design, Hydro-Chem incorporated two new technologies in addition to SSR. The burner in the reformer was changed from a standard Hydro-Chem design to a Flox forced-air design. The PSA was changed from a standard Hydro-Chem design to a Quest-Air design. Each of these technologies can have significant impact on plant operation parameters (positive or negative). This makes it difficult if not impossible to distinguish performance benefits resulting from individual technologies.

At the moment, the plant is operating with similar output and efficiency as compared to a typical plant with ceramic catalyst installed. There have been some operational issues with the Flox burner. It operates correctly in direct fire mode, but behaves erratically in the intended “Flox” mode when PSA off-gas is mixed in the fuel. We suspect that problems with the burner are masking potential benefits that could be attributed to SSR.

One potential SSR problem has been observed at Borcelik. Visual inspection of the catalyst tubes through a peep-hole in the furnace while operating shows red bands that seem to be near the end of each individual SSR. Hydro-Chem has expressed some concern that these bands are hot spots that could lead to eventual tube failure. They have not, as yet, been concerned enough to require action. At this point, nobody knows exactly how hot the bands are, or if in fact they are a problem. Catacel strongly suspects these bands represent areas of reduced heat transfer, which could negatively impact the overall performance of SSR in some way. In any path forward, Catacel must develop an understanding of the cause and affect of these bands and implement any necessary corrective action.

Very positive upside potential to the SSR is being demonstrated by the Borcelik installation. To date the plant has been running for over four years. Installation went very smoothly, demonstrating that there is little or no risk to installing SSR in other plants. And there is clear reward to other plants if the expected performance benefits can be realized.

Project Objectives and Work Plan: The Phase III plan for SSR will identify and work out this potential final bug in the technology, and demonstrate once and for all that performance benefits can be realized. We must identify the root cause, determine if corrective action is necessary, and then take that corrective action to fix the red rings. We are concerned that this is impacting performance in some way, and we know that our customer is concerned, which in itself is enough reason to necessitate corrective action.

And, we still must prove the benefits of SSR in a plant. Although there has been significant learning from the existing Borcelik plant, we will never be able to clearly prove benefits at this installation because of the other technology influences. We plan to find another small plant that is currently operating with ceramic catalyst, and convince the operator to allow us to replace that ceramic catalyst with Catacel SSR technology. We have already demonstrated that there is little risk in making this change. We can also show that there is potential benefit from fuel savings based on lab experiments and computer simulations. Operating parameters from this plant would be carefully documented before it is shut down for catalyst change. After startup, the new parameters can be directly compared with the old so that SSR performance advantages can be documented. This new plant could be a second unit at Borcelik that is already operating on ceramic. Preferably it will be in the U.S. for easier Catacel access. We expect to begin work on this Phase III project on September 1, 2008, and complete it in six months.

Task 1 – Understand and Correct the Red Rings – At this moment, we can identify several possible root causes for the rings: 1) unusual turbulence or flow path at the interface between SSR parts, 2) parts have slipped away from each other in the tube, 3) cone or backing collapse in local areas, such that the fins are not in contact with the tube, or 4) some areas of catalyst have become ineffective, either by attrition, poisoning, or other mechanism. Our engineering team will intellectually explore and rank all root causes using standard quality system tools. Lab experiments or engineering analysis may be needed to rule in or rule out one or more of the root causes. A Borcelik on-site action plan will be devised based on that activity. This plan will likely involve certain measurements to be made during operation, and methods for SSR removal and inspection that will allow verification of the root cause(s). The plan will also provide for quick replacement or repairs to the Borcelik unit based on findings. Appropriate changes will be made to the baseline SSR design which will eliminate the red ring issue.

Task 2 – Select a Reference Plant – Discussions have already been initiated with Linde, Hydro-Chem, Praxair, and Air Liquide to locate a small operating plant in the 2-400m3/hr size range. This could be the older Borcelik plant, or preferably it is will be an existing plant in the U.S., which will permit easier access for Catacel to measure ongoing operating results. Commitments would be obtained from the operator to allow Catacel to monitor the plant as is, install SSR, and monitor the new configuration. No other significant changes would be permitted during this change-out process. Catacel will also require a commitment from the operator to be able to use the information derived as a sales tool for SSR.

Task 3 – Document Key Operating Parameters of Reference Plant – Depending on the plant design, this might be a simple inspection and compilation of existing performance records. Or, it could involve field measurements using portable equipment. Plant rates, temperatures, pressures, reformate quality, and fuel balance/consumption will be the parameters of interest.

Task 4 – Manufacture and Install SSR in Reference Plant – Similar to the recent Borcelik activity, Catacel will manufacture catalyst for the reference plant. This new catalyst will have a modified design based on the output of Task 1 to prevent the red rings. Working with the plant operator, Catacel will remove and store ceramic catalyst from the reference plant, and install SSR using known methods.

Task 5 – Document Improvements in Performance – After the plant is started up and is lined-out, additional measurements will be made to compare to those in Task 2. These comparisons will be documented and incorporated into SSR sales tools. Payback analysis will be performed that will demonstrate to potential buyers the advantages of SSR.

Conclusions: In May 2012, Catacel installed a new version of SSR into a plant that produces hydrogen to support food ingredients manufacturing in Guadalajara, Mexico. The install was completed with minimal downtime by plant staff supervised by Catacel. A custom installation tool was devised to install 3-ft. long sections of SSR into the 8 tubes, which expand the metal foil to conform to the internal geometry of each tube.

Initial reporting at 100% plant rate indicated a 13.5% reduction in natural gas consumption by the reformer burners over the values of previous operation. In July 2012, it was observed that at a 55% plant rate, a 22% reduction in fuel to the burner can be obtained while maintaining the desired hydrogen levels in the syngas. It is projected that by reducing the reformer furnace fuel feed and temperature at which tubes are operated, reformer tube life can be expected to last beyond design life. Additionally, return on investment for the Catacel system in this plant based on natural gas pricing in Mexico, is expected to be within 2 years.

Multiple discussions are underway with potential customers. We anticipate very soon commercial catalyst orders for two additional plants, to be installed yet in 2012.

Faraday Technology, Inc. – Electrodeposition

Nanocatalyst Development Employing Electrically Mediated Processing for Hydrogen Generation: EFC-H1-01-1B; EFC-H1-01-2A

Project Objective: The overall objective of the program is to develop a low-cost, mass fabrication technology for catalyzation of MEAs for PEM electrolyzers and regenerative fuel cells; a technology which directly supports the development of the hydrogen economy. This program meets the mission of EMTEC, and is synergistic with ongoing DoE efforts in hydrogen production via electrolysis, and will meet the challenge of reducing electrolyzer cost by developing lower cost materials with improved manufacturing capability.

Background: The focus of the program is to develop nanomaterials to support the hydrogen economy. This project addresses needs in hydrogen production. A major obstacle to the commercialization of fuel cell vehicles based on PEM fuel cells, is the ability to generate and store hydrogen in such a way as to support the current transportation fuel infrastructure. PEM electrolyzers and regenerative fuel cells are technologies which could be used to produce hydrogen for the transportation market.

PEM electrolyzers consume electricity and water to produce oxygen at the cathode and hydrogen at the anode. The kinetics of the reactions within the electrolyzer must be improved to lower the power requirement and improve the efficiency of the electrolyzer, to lower the overall capital and operating costs. To enable commercialization of these technologies, the performance of those components that directly affect electrolyzer performance, *e.g.*, loading, dispersion and utilization of catalyst, must be improved using methods amenable to low cost manufacturing. In this program, Faraday will develop a sophisticated electrochemical process for catalyzation of membrane electrode assemblies (MEA) for PEM electrolyzers or regenerative fuel cells. Faraday will develop a low-cost manufacturing solution for this process, using reel-to-reel electrodeposition. Faraday will also engage in commercialization efforts to promote this technology in the marketplace. The technology is protected by a U.S. Patent No. 6,080,504 (Taylor, E.J. and Inman, M.E. (2000), Electrodeposition of Catalytic Metals Using Pulsed Electric Fields).

Progress and Status: Our novel manufacturing process eliminates the aqueous deposition/chemical reduction batch process currently used for catalyst preparation. Specifically, the proposed process utilizes sophisticated, high frequency asymmetrical waveforms to electrodeposit optimized loadings of highly dispersed catalyst particles onto support structures, only in regions with access to all the reaction components (high utilization), for incorporation into a MEA. Use of the asymmetric waveform will favor nucleation of additional small catalyst particles on the catalyst support, as opposed to the particle growth observed in the previous attempts to increase catalyst loadings using low frequency, pulse current electrodeposition. In addition, the reel-to-reel electrodeposition approach is amenable to scale-up for low-cost mass fabrication.

Makel Engineering, Inc. - Hydrogen Sensor

"Low Cost MEMS Hydrogen Sensor for Transportation Safety"
EFC-H1-15; EFC-H1-15-2; EFC-H4-15A MEI-EMT02-FR

Infrastructure and Vehicle Monitoring Safety Sensors: The objective of this program was the development of an advanced hydrogen sensor system prototype for hydrogen powered transportation applications. The use of hydrogen fuel in transportation applications requires the development of compact, reliable, low cost instrumentation for safe operation and process control in vehicles and in supporting (fuel delivery) infrastructure. The use of micromachined sensor structures and nanomaterials provides the means for low cost, compact, low power consumption, and miniaturized systems, suitable for mass production.

Personal and Device Monitoring: The objective of this program was the development of a companion product for our low-cost hydrogen sensing system for hydrogen powered Low Cost MEMS Hydrogen Sensor for Transportation Safety transportation applications. This device will be a low cost component for personal safety and for integration with low-cost fuel cell systems. This common, low cost platform will address two product niches: the Personal Hydrogen Monitor (PHM) and Integrated Hydrogen Safety Component (IHSC). The PHM will be a wearable personal safety device incorporating nanomaterial technology (hydrogen sensitive nanowires and/or nanoclusters). This device will be targeted at end-users, researchers, and maintenance personnel of fuel cell or other hydrogen powered vehicles and systems. The IHSC will serve as a standard component in lower cost, portable fuel cell devices, such as battery replacement units. This product will allow us to address a full range of market opportunities for hydrogen safety sensors from vehicles to personal electronics.

MEI partnered with the University of Wisconsin – Stevens Point for continued development of nanocluster sensor technology and with cost share partner Mahindra & Mahindra, an Indian automotive manufacturer, for commercial product development and testing. Sensors were fabricated and tested for inclusion in prototype system packages, and a manufacturing process was established. The design and fabrication of the control electronics was completed and MEI has performed final mounting of hardware, implementation of board-level operating firmware, and initial testing. MEI is well positioned to meet current and long-term market needs with these low-cost, high performance hydrogen safety sensor technologies.

Commercialization of this technology was awarded a R&D100 award in 2006: "Ultrafast Nanostructured Hydrogen Sensor" (Argonne National Lab, Makel Engineering, Edison Materials Technology Center)

Midwest Optoelectronics, LLC. (MWOE) – Hydrogen Production

Development of Improved Materials for Integrated Photovoltaic-Electrolysis Hydrogen Generation Systems

EFC-H1-16-1; EFC-H1-16-2A

Project Objectives: The objective of this two-year, two-phased program is to develop and produce integrated photovoltaic-electrolysis (IPE) hydrogen generation systems (superstrate-type photoelectrochemical systems, or PEC systems), based on the high-efficiency, multiple-junction amorphous silicon (a-Si) photoelectrode, with 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. A specific objective of the program is to achieve a small-scale production of 8ft^2 IPE hydrogen generation panels with a throughput of 20 panels per week at the end of the program.

Background: The project is directed toward the goal of producing renewable hydrogen at low-cost for the future energy demand of our nation. In MWOE's proprietary integrated photovoltaic-electrolysis process, hydrogen is generated by splitting water using sunlight and the generation process does not emit carbon dioxide, a pollutant that causes global warming.

The focus of this project is on the development of hydrogen generation panels, superstrate-type IPE panels (also referred in the following as PEC panels), that produce hydrogen cost effectively, as well as the development of a manufacturing process that could be used to produce these IPE panels in volume.

The project consists of two phases. During Phase 1, three tasks, described below, have been completed.

- Task 1: Fabrication of small-area, high-efficiency, triple-junction PEC cells
- Task 2: Fabrication of 1ft^2 and 8ft^2 PEC Systems
- Task 3: Development of detailed commercialization plan

During Phase 2, four additional tasks were performed.

- Task 4: Fabrication of 1ft^2, high-efficiency, triple-junction PEC systems
- Task 5: Small-scale pilot production of 8ft^2 PEC systems
- Task 6: Design and analysis of a large-scale production process
- Task 7: Marketing and business development

Progress and Status: Highlights of research accomplishments for this reporting period:

MWOE has:

- Maintained and monitored the operation of 11 full size PVH8A-type Photoelectrochemical systems (PEC systems) in the outdoor testing station. These panels have been under outdoor testing for over a year.
- Supplemented the outdoor testing with indoor measurement of the PEC panel performances when the weather was not collaborating.
- Have experimented with several alternative methods to apply the electric connections to the back of the PV panels to solve the performance degradation problem as discussed in

the last quarterly report. Have fabricated 35 PVH1A-type PV panels with different methods and placed them in the environmental testing chamber for accelerated testing.

- Have developed a new type of electrolysis units with the HDPE material to solve the leaking problem. The new type electrolysis units were welded together and initial results indicate excellent performance.
- Tested various configurations between the PV panel and the electrolysis units in an effort to reduce the number or size of the electrolysis units, initial results indicate the possibility to reduce the material and labor cost.

Patents:

April 10, 2006
PCT Patent Application
Title of Patent: Integrated Photovoltaic-electrolysis cell
Inventors: Malabala Adiga, Xunming Deng, Aarohi Vijh, and Liwei Xu
Filing No: PCT/2006/013222
Corresp. to Ser. No. 60/670,177 filed April 11, 2005.

May 16, 2006
The following PCT patent application has entered into National Phase.
Country selected: US
Title of Patent: Interconnected Photoelectrochemical Cells
PCT No. US2005/005121
Priority based on US Ser. No. 60/545,892
Inventors: X. Deng and L. Xu

IAP Research Inc. – Solid State Welding

Commercialization of EM Solid State Welding for High Pressure Hydrogen Storage
EFC-H1-11-1A

Project Objective: We propose to establish the feasibility of using electromagnetic solid state welding for pressure sealing applications that are highly relative to the DOE Hydrogen Multi-Year Plan for compressed hydrogen storage while at the same time have excellent near term commercialization potential in HVAC, chemical processing and power industries.

Summary and Conclusions: The Phase I effort was successful from a technical standpoint. We were able to demonstrate that if the basic requirements for EM Solid State Welding are known for a given pair of dissimilar materials, a design process utilizing FEA modeling of the process can be developed that will enable the development of a variety of applications. Ultilizing an understanding of the solid state welding requirements for copper to steel and aluminum to steel developed by Botros et. al [1], we were able to model the EM Solid State Welding Process for a baseline geometry utilizing Finite Element Analysis (FEA). The process was validated through experiments, which yielded solid state welding at the conditions predicted by the FEA analysis. However, the Phase I effort was not successful from a commercialization standpoint. We were not successful in moving forward the commercialization of EM solid state welding into a near term commercial application. The withdrawal of ETT from the Phase I effort greatly impacted the near term commercialization effort. Much of the early scope development for near term applications focused on the use of EM Solid State Welding for tube-to-tubesheet joining. ETT was critical to the commercialization of this application. After ETT withdrew from the Phase I effort, we were not able to identify an alternative application and a commercialization partner willing to make the appropriate investment in a Phase II effort. The remaining team members for the Phase I effort, IAP Research and Advanced Computational & Engineering Services (ACES), are still committed to continuing work on the development of the EM Solid State Welding process for near term applications.

PowderMet INC. – High Strength Microballoons

High Strength, Low Cost Microballoons for Hydrogen Storage

EFC-H2-12-1A; EFC-H2-12-2A; EFC-H4-12

Project Objectives: The goal of this program is to prove the concept and validate a system that delivers >>4 wt% hydrogen stored in balloons, including all balance of plant components and attachment specifications to a current fuel cell stack. This goal has two components: (a) storing at least 6 wt% hydrogen in coated balloons, and (b) mechanical analysis, balance of plant components and construction of a 4 wt% "black box" prototype hydrogen delivery system.

Introduction/Approach: Powdermet is investigating alternative methods to "lock in" hydrogen to already commercially available high strength glass and silica microballoons. Powdermet's evaporative powder coatings should take advantage of existing glass microballoon's already high strength ability to store hydrogen and augment its ability to prevent the hydrogen from leaking over time. Existing glass and silicon microballoons have been shown to hold 10% hydrogen by weight, however, hydrogen leaking is a major problem.

Results: Although the first part of the Phase II Program failed to produce large quantities of balloons that held substantial quantities of hydrogen, breakthroughs in microballoon development essential to other Powdermet products were achieved and a prototype delivery device was fabricated. Table 1 below shows the potential for these microballoons to be a very effective weight based storing solution for hydrogen although their volumetric potential is limited certain applications such as aircraft and personal portable power require weight savings over volumetric savings.

Storage Parameter	Units	DOE 2010 Target	Theoretical Limit Powdermet CVD Coated Carbon Microballoon @ 20,000psi	Theoretical with Designed 1.5 Safety Factor (practical limit) @ 14,000psi
Specific Energy	kWh/kg	2	5.49	3.67
Energy Density	kWh/L	1.5	1.32 (Assumes monosized spheres.)	0.84 (Assumes monosized spheres.)

Table 1. Theoretical Analysis Meeting DOE On-Board Hydrogen Storage Targets

Conclusions and Future Directions: Powdermet we can take the state of the art hydrogen filled glass balloon technology and improve on this by eliminating the hydrogen leaking potential by using nanometer controlled evaporative coatings on the glass microballoons. This technology will enable Powdermet to reach its goal of microballoon hydrogen storage of 10% by weight and augment many other internal Powdermet programs allowing aluminum and other metal deposition on materials at "cold" substrate temperatures without the need for Chemical Vapor Deposition (CVD).

Future Directions:

- Assembly and operation of evaporative Aluminum system for coating Powders.
- Hydrogen Storage in Glass Microballoons and Leak Rate Measurements. Coating Hydrogen filled Microballoons and Leak Rate Measurements.
- Scale up of process and Prototype Demonstration.

Accomplishments: Demonstrated that high strength coating using chemical vapor deposition in a fluidized bed, very thin, high strength coatings (1-20um) can be applied to the ultra lightweight (0.05g/cc) carbon microballoon “scaffolds” in a very cost-effective manner, leaving no residual gas inside the spheres that potentially reduce capacity. In the Phase I Program coatings on microballoons with a thickness of 1-15μm were deposited.

University of Dayton Research Institute (UDRI) - SOEC

Regenerative Solid Oxide Fuel Cell
EFC-H2-20-1A; EFC-H2-20-2A

Project Objective: The overall objective of the Phase II effort was to select anode, cathode, and electrolyte materials; generate microstructures using pore formers; fabricate solid oxide electrolysis cells (SOECs) using the selected materials, and model the SOEC performance.

Background: Considerable efforts have been devoted towards the development of solid oxide fuel cells (SOFCs), particularly on the characterization and selection of materials for SOFC components. The operation of SOFCs in the reverse mode, i.e., in the electrolyzer mode (SOECs) presents numerous challenges. The production of hydrogen on a commercial scale at a reasonable cost per pound presents an interesting opportunity to develop SOECs. Successful demonstration of commercially viable SOECs depends upon the identification of cost effective, thermally stable, and highly reliable cell component materials. These attributes will facilitate the development of SOECs with high efficiency for hydrogen production.

Summary: A NiO-YSZ/YSZ/LSM-YSZ anode-supported cell without pore formers was fabricated using the powder processing technique. These powders are commercially available. 10ScSZ was introduced as an electrolyte because it has higher ionic conductivity than YSZ. The NiO-YSZ/10ScSZ/LSM-YSZ anode-supported cell without pore formers was fabricated using the powder processing technique. 10ScSZ was purchased from NexTech, Inc. The cell was sintered at 1450ºC, which was higher than YSZ electrolyte based anode-supported cells by 50ºC. The cell was tested at 750ºC under H_2-3% H_2O equilibrium. An open circuit voltage (OCV) was varied between 0.62 and 0.57 V. This OCV is very low relative to the theoretical cell voltage (1.1V) indicating some cracking or pinholes across the electrolyte or platinum migration through the electrolyte. A two-dimensional model of a solid fuel cell operating at a nominal temperature of 800°C was built for finite element thermal stress analysis

Delphi Electronics and Safety – Hydrogen Storage

Hydrogen Storage using Porous Silicon

EFC-H2-3-1C

The Phase I objective was met, that of demonstrating technical feasibility to store hydrogen in a porous silicon matrix. Storage rates as high as 4.9% by weight have been demonstrated. Storage of 6.6% was demonstrated by another research team mid-way through our Phase I work. That value agrees exactly with our theoretical predictions at the outset of this project, and is a factor of 2 higher than state-of-the-art solid-state storage systems for hydrogen.

Our diverse team involved specialists in Chemistry, Mechanical Engineering, Semiconductor Processing and Electrical Engineering. Our work output speaks to wide range of practical, empirical and theoretical issues related to the commercial development of a hydrogen storage system based on porous silicon. A substantial body of information and understanding has been created with Phase I funding.

This 7 month project opened up an entirely new means for hydrogen storage. In addition to the progress in storage rate, we have laid out key technical challenges and issues with this novel material. At the completion of Phase I, we have a clear direction to follow for Phase II. Silicon is a well-characterized material, and we have a reasonable expectation that we can drive it to the point of a practical system faster than other competing storage approaches.

Our entire team is grateful to EMTEC for this opportunity and to Delphi for affording several of us the time to work on this project. Without their support, this exciting new idea may have lain dormant, and we would never had the benefit of all the fascinating results covered in this report.

Silicon is nice material to use for hydrogen storage. Compared to other media under study, the advantages of silicon fall into five categories. **Benign**. Silicon in small doses is good for the human body, being a component of our skeleton. While large doses can be harmful (as is true for almost every substance), any reasonably-expected dosage level will not be health concern. **Abundant**. Silicon makes up 27.6% of the Earth's crust. Semiconductor silicon comes from pure sand (quartz). In fact, the US is home to the world's premier source of quartz for the semiconductor industry: the Spruce Pine District of North Carolina. **Crashworthy**. Nanostructured materials are relatively insensitive to the bumps and jars experienced by a vehicle under normal operation (+/- 15 gs). Further, we expect that since the hydrogen is bound strongly to the silicon, requiring an endothermic release, that the charged storage material will be non-pyrophoric, greatly reducing the risk of fire in a catastrophic vehicle crash. **Empty/Full Indication**. Our Phase I team developed a means for determining the fill-level of the hydrogen tank. This is a clever method not available with most other solid-state storage media. **Smart**. The potential to up-integrate hydrogen storage with control electronics is an advantage unique to silicon, and provides long-term growth in feature & function of storage systems.

NexTech Materials, Ltd. – SOEC

High Temperature Electrochemical Cells for Hydrogen Production and Regenerative Fuel Cell System

EFC-H2-15-1A

Project Objectives: The overall objective of this project is to develop high-temperature, solid oxide electrolysis cell (SOEC) technology for the efficient production of H2 gas from water vapor (steam). SOECs show significant potential for low-cost and efficient production of H2 gas when combined with renewable or nuclear energy (and steam) sources. The successful implementation of SOEC stacks is limited by material and structural challenges, with the largest problems being:

1) High ohmic losses due to thick electrolyte membranes
2) High ohmic losses due to resistive electrolyte composition
3) High electrode/electrolyte interfacial resistances due to low reaction kinetics
4) Chemical or structural instability of cell and stack sealing during operation

The specific innovation to be pursued in this Phase I is a compact, lightweight ScSZ electrolytesupported, tubular SOEC which directly addresses all four of these limiting challenges. The proposed SOEC tube will be composed of highly conductive scandia-doped zirconia materials and with membrane thickness ranging between 30 and 50 µm. The electrodes on the tubular SOEC cell will consist of composite compositions exhibiting mixed-conductive behavior and electrochemically active interfaces designed to minimize interfacial polarization. The tubular geometry of the proposed SOEC will allow for ‘cold-end’ sealing of the cells with various metal brazes, glasses, and high-temperature polymers. The primary objective of the Phase I was to prove feasibility of the novel SOEC design, by demonstrating the manufacturability of the SOEC components and verifying cell performance.

In this Phase I Program, NexTech Materials has demonstrated the applicability of two solid oxide fuel cell components for solid oxide electrolysis operation. Two novel cell components have been demonstrated to date on this program, a flexible laminate composite electrolyte supported cell with an effective electrolyte thickness of 120 microns (Figure 1a), and a thin-film electrolyte (20 microns) hybrid cell architecture (Figure 1b). In this program, both cell platforms were for hydrogen generation by electrolysis, with the hybrid cell architecture demonstrating significant advantages in cell performance at low temperature. In Phase II, we will optimize the cells for operation in short stacks operating at 750°C.

GFS Chemical, Inc. – Hydrogen Storage

Development of Complex Metal Hydrides for Hydrogen Storage Applications

EFC-H2-11-1A

Project Objective: The project is staged among the three partnering entities. GFS Chemicals is to acquire and manufacture materials to be tested for their ability to reproducibly and efficiently desorb and adsorb hydrogen. These materials, including variously doped formulations, will be based upon the novel chemistry of lithium hexahydridoalanate (LHA –Li_3AlH_6), of which GFS is presently the sole known non-military supplier. All materials successfully made by GFS are to be submitted to UDRI for physical and chemical characterization that is needed to qualify selected substances for testing of hydrogen absorption-desorption kinetics and storage properties. These qualified materials are then to be submitted to Hy-Energy for advanced kinetics testing that is consistent with the type of testing currently being reported in the literature for similar hydrogen-rich complex salts, especially alanates.

Background: The project is justified by the need for new materials to support government efforts to establish and sustain a Hydrogen Fuel Economy in a timely manner. Current materials research has not formulated chemical compositions that allow efficient cycling of hydrogen under storage and operational conditions of temperature and pressure that would support this technology and reduce U.S. dependence on foreign petroleum sources. It is proposed that variations on the aluminum and hydrogen rich formulations derived from Li_3AlH_6 in this project will exhibit the desired superior performance.

A project overview was presented by GFS and UDRI personnel at the EMTEC Hydrogen Program Review Meeting held February 8, 2006 at the Bergamo Center in Dayton, OH. Within that time frame, it was discovered that the necessary sub-contract between UDRI and GFS to govern the partnered work defined by UDRI proposal No. R-14941-A had not been put in place, and several weeks were required to generate and edit that document to final, signed form in force as of April 1, 2006. GFS has now been billed for initial work done at UDRI so copies of the Request for Cash Reimbursement form HP-CF and the Cost Share Reporting form HP-CS are being submitted with this Report. The project is now proceeding according to projected milestones.

Progress and Status: Samples of LHA mechanically doped with aluminum carbide, titanium aluminum, and titanium aluminide provided opportunity for the evaluation at UDRI of two of three aluminum-rich mixtures conforming to the formulation $Li_3Al_2M_xH_6$ [when M = C, x = 0.75; when M = Ti, x will be 1 for TiAl and 0.33 for $TiAl_3$]. TGA and DSC data for the titanium-doped mixtures are attached; data for the aluminum carbide-doped sample will be included in the August report.

The most recent enclosed data show only a very minor change in the temperatures at which key thermal events occur. Weight loss due to hydrogen release is relatively consistent between undoped LHA samples and those doped with either of the titanium alloys. Yet to be determined is the effect of more energetic mixing of the respective samples, possibly by self-impact agitation. The original samples had been prepared in an inert glove box by persistent manual grinding over 15 minutes using an agate mortar and pestle.

Attempts to incorporate iron dopant into the LHA framework began in July and will continue. It was determined experimentally that iron pentacarbonyl (ICP), $Fe(CO)_5$, reacts with both butyllithium (BuLi) and lithium aluminum hydride (LAH), the two starting materials used in the synthesis of LHA. However, the reactions appear to follow different pathways that may allow some exploitation of the chemistry as intended in our objectives. The cleaner of the two reactions was between IPC and BuLi, which resulted in a dark solution in which the iron species was either colloidal or partially dissolved.

With this in mind, a hexane solution of BuLi containing 10 mole % iron (based on theoretical LHA yield) was added to the ether solution of LAH in the standard fashion. The reaction appeared to proceed normally, producing an off-white precipitate and a nearly colorless filtrate (LHA is a fine, white powder). This doped product filtered much more slowly than usual, was more difficult to wash and, as a result, was rendered more susceptible to ignition from the low levels of oxygen (air) that are typically present – but which have not otherwise precluded the successful isolation of pure, stable LHA via this "standard" method.

To address the greater level of material sensitivity that we are experiencing, we are implementing use of a pressure filter that should allow optimization of the "inerting" of the reaction system. This allows us to maintain the nitrogen atmosphere on the product with application of filtration pressures of possibly greater than 50 psi to facilitate the isolation of clean material – LHA doped with iron nano-particles.

Precision Energy and Technology, LLC (PET) – Reel to reel processing

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

EFC-H2-23-1A; EFC-H2-23-2B; EFC-H4-23A

The basis of our concept is to resolve the problem of thermal pressing catalyst film onto the Nafion-type membrane. While this is currently accomplished by several techniques in batch processing, the transitioning from a low volume, high cost batch process to a high volume, low cost continuous process must be overcome. What is needed is a "reel to reel" processing that will resolve these issues in order to bring commercialization to hydrogen producing membrane electrolyte assemblies. Unfortunately, reel to reel processing is not directly analogous to thermal pressing in a plane.

Our process will entail modifying a Western Technologies Associates, reel to reel processing equipment to adapt the continuous thermal processing capabilities. The approach will be to design, manufacture, assemble, and install a pair of thermally controlled rollers that act to capture the catalyst layer film with the Nafion-type membrane. These rollers must maintain a limited temperature range and apply pressure to the processing films with a small tolerance in order to be successful. Both thermal and pressure tolerances will be critical to maintaining a high sigma quality and thus a repeatable product.

Initial process setup will be based upon previous data (Chan Lim & John Davey). Additional changes in the process will be evaluated for optimization of the baseline materials processed (catalyst film and Nafion-Type membrane). This proposal does not intend to evaluate all the combinations of catalyst film and membrane thickness, but is meant to prove out the control and repeatability of the manufacturing process to make this a commercially viable process.

This project will be cost shared with a concurrent project, Ohio's Third Frontier Fuel Cell Program. That program, "Enabling Low Cost MEA Manufacturing of PEM Fuel Cells in Ohio" will also utilize the WTA "reel to reel" production process to provide a high volume, low cost approach to MEAs.

The success of this project will result in driving closer to meeting the DoE Hydrogen Multi-Year Plan by reducing cost to both reversible Hydrogen production and fuel cell cost goals of less than $30/Kw. In addition, the project will meet EMTEC's goal by making MEAs more cost effective and providing the marketplace multiple MEA products at the lowest production cost.

Hy-Energy, LLC. – Hydrogen Storage

Novel Materials for Reversible Hydrogen Storage

EFC-H2-18

Project Objectives: Hy-Energy proposes to develop lightweight reversible hydrogen storage materials based on novel alkali/transition-metal-amides. Materials will be prepared and tested for viability as a practical means of storing hydrogen for use with a variety of fuel cell applications. The technical objectives will be to prepare 5 gram samples by mechanical mixing of pre-cursor compounds under an inert atmosphere. These samples will then be tested for hydrogen uptake and release using a gas sorption analyzer (PCTPro-2000) to determine:

1) Activation properties (sorption rates and capacities vs. temperature and pressure in the first few cycles)
2) Reversible hydrogen storage capacity
3) Rates of hydrogen uptake and release
4) Thermodynamic stability (plateau pressure vs. temperature to give enthalpy of reaction)
5) Activation energies associated with uptake and release (rate vs. temperature measurements)
6) Cycle-life properties (capacity and sorption rates as a function of cycling)

Background: Hydrogen storage presents one of the most significant barriers to the establishment of a large-scale hydrogen economy in the future. The technical area that Hy-Energy will address is the development of advanced materials for efficient and cost-effective hydrogen storage. Hydrogen-powered transportation is the application with the most stringent storage requirements. Therefore, our focus will be on the development of light-weight high-energy-density materials needed for mobile energy storage applications.

Progress and Status: Two different samples of mixed amide/alanate were prepared in our newly installed Spex milling station. The samples were prepared for milling and finally loaded into sample holders for testing using the recently installed Argon glove box. Following preparation, the samples were tested for hydrogen uptake and release on the PCTPro-2000 hydrogen sorption analyzer. The first sample showed a good capacity (3.5 wt%) and a rapid release of hydrogen at a relatively low sample temperature (200°C). However the second desorption demonstrated that little hydrogen uptake occurred on recharging. Work has been completed on the construction of a residual gas analysis system (RGA) for analyzing impurities that may exist in the hydrogen released from our advanced storage materials. Initial investigations of the second amide/alanate sample showed the presence of ammonia as a desorption species. However, this occurred when the sample was subjected to extreme conditions of rapid decomposition at 300°C.

NexTech Materials, Ltd. – Hydrogen Sensor

Commercialization of Novel Ceramic Hydrogen Sensors for Fuel Cell Applications
EFC-H2-21-1B; EFC-H2-21-2A; EFC-H4-21A

Commercialization of hydrogen powered fuel cell and distributed hydrogen production systems will require sensors to monitor the presence of hydrogen in the ambient surroundings for safety purposes. Problems with commercially available H2 sensors include lack of sensitivity, cross-sensitivity to other contaminants (like carbon monoxide, hydrocarbons, and volatile organic compounds), slow response and recovery times or any combination of these issues. Additionally, the cost of the few H2 sensors that actually meet the application-specific requirements of fuel cell applications is prohibitive for many of these applications. NexTech Materials has developed a unique hydrogen sensor technology that overcomes many of these gaps left by competitive technologies and is aggressively pursuing commercialization.

In Phases I and II, NexTech established a tubular sensor design and optimized material set for its hydrogen safety sensor. Using this design and material platform, Phase III efforts focused on optimizing the design features for efficient manufacturing, developing control electronics and software algorithms for the sensor device, and optimizing the sensor packaging and assembly process for a robust sensor product. Among these, two specific activities required the most development and attention: development of the electrode design and deposition process, and optimization of the sensor design and control parameters for humidity insensitivity. In the pre-prototype design established in Phase II, electrodes were hand-deposited onto each side of porous thick film layer of the active sensing material. The humidity insensitivity represents a key technical value proposition for NexTech's sensor device, validated through successful field testing on fuel cell powered forklift trucks.

Identifying suppliers capable of meeting NexTech's specifications and volumes was a critical step toward commercialization of the hydrogen sensor device. Through Phase I and II activities, NexTech had identified prototype sources for several of its components including the alumina tubular substrate, T-08 header, and protective cap. All other components required identification and sourcing of capable suppliers. Additionally, through make versus buy analysis, NexTech identified several unit operations to outsource, including electrode deposition and resistance welding operations. NecTech developed a bill of materials for the hydrogen sensor and suppliers qualified in this Phase III project.

To support customer communications and prepare test partners for installation of NexTech's prototype hydrogen sensor device, a detailed outline drawing was developed. Key information provided in the outline drawing includes overall device dimensions, element position, connector pinout, and location of mounting holes. Mechanical samples were also fabricated to further aid customers in preparation of sensor installation and testing, prior to availability of working sensor devices. A prototype datasheet was prepared to accompany the prototype samples, providing key specifications, installation instructions and guidelines, and input/output requirements.

The provisional patent application has been completed for the hydrogen sensor.

National Fuel Cell Research Center (NFCRC) - SOEC

Novel Intermediate-Temperature Reversible SOFC for a Renewable Energy System that can Co-Produce Power and Hydrogen

EFC-H2-16-1A; EFC-H2-16-1C; EFC-H4-16A

Background and Project Objectives: A reversible solid oxide fuel cell (R-SOFC) is an energy conversion/storage device that could well complement the development of a hydrogen economy based on renewable energy sources. Compared to traditional yttria-stabilized zirconia (YSZ) electrolyte-based materials sets for an R-SOFC, the current effort advances Sr- and Mg-doped lanthanum gallate (LSGM: $La_{1-x}Sr_xGa_{1-y}Mg_yO_{3-\delta}$) based materials sets. These types of materials can bring down the operating temperature of an R-SOFC to the intermediate temperature range (700-800°C), while keeping the performance at a cost-effective and robust level. An optimized composition of LSGM with cobalt doping at the Ga sites, $La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.115}Co_{0.085}O_{3-\delta}$ (LSGMC), is the primary electrolyte material of interest. Use of yttria-doped strontium titanate (SYT) in the anode and strontium-doped samarium cobaltite (SSC) and lanthanum-doped barium cobaltite (BLC) in the cathode are additional novel aspects of the current study.

It is likely that the novel materials set being advanced could produce an R-SOFC with superior power density and robust operation and lifetime even though exposed to the cycling between fuel cell and electrolysis modes. As a result, this type of intermediate temperature materials set could be applied to more traditional SOFC applications as well, such as natural gas or other fueled power generation, auxiliary power units, etc.

Status: The major successful accomplishments have been in the following areas:

1. Finished the LSGMC composition matrix test and determined the optimal LSGMC composition;
2. Optimized the fabrication process for dry-pressing LSGMC button cells;
3. Corrected temperature-independence errors of SSC composite cathode/LSGMC electrolyte interface during the DC polarization tests;
4. Successfully fabricated and tested highly active SSC composite cathodes;
5. Successfully synthesized the SYT powder using a sol-gel process;
6. Determined by powder x-ray diffraction (XRD) analysis that the SYT is in the correct physical phase, and by transmission electron microscope (TEM)/scanning electron microscope (SEM) that the SYT powder and the sintered SYT pellet is in the desired morphology and microstructure, respectively

Ohio University – Hydrogen Production

ELECTROCHEMICAL COAL GASIFICATION WITH NOVEL ELECTRODES
EFC-H2-08-1B

PROJECT OBJECTIVES: The main objective of this project is to evaluate the feasibility of electrolyzing coal slurries at high temperatures. Within this context, the specific objectives for this application are 1. Redesign the coal electrolytic cell to work at high temperatures, and 2. Evaluate the performance of a bench-scale coal electrolytic cell to provide a scale-up design for alternative applications for the production of hydrogen.

Task 1.0- Redesign of electrolytic cell and electrode shape
The objective of this task is to minimize the internal resistance (IR) of the high temperature coal electrolytic cell (HiCEC) and to redesign the electrolytic cell to operate at high temperatures. Because the electrolysis of coal takes place in the presence of H_2SO_4, a Nafion membrane is not necessary for the electrolysis. The use of Nafion limits the operating temperature of the cell and also increases the cost of the cell.

Task 2.0- Construction of bench-scale HiCEC
The new design of the coal electrolytic cell (specified in Task 1.0) will be built at Ohio University for further testing and evaluation as described in Task 3. The electrodes of the cell will be built by electro-deposition of nobel metals on carbon fibers according to the procedures described in the literature.[2,3]

Task 3.0- Evaluation of cell performance
The objective of this task is to evaluate the performance of the cell to achieve the highest current density at the lowest cell voltage. It is expected that the changes implemented in the design of the cell would allow the following operating conditions: current densities of at least 100 mA/cm^2 at a maximum operating voltage of 0.7 V to guarantee the economic feasibility of the technology for the production of hydrogen for distributed power.

Task 4.0-. Economics Analysis
The experimental data was used to estimate the scale-up cost of the technology for different hydrogen production rates: forecourt size (up to 480 kg/H_2 day), distributed size (up to 1000 kg H_2/day), midsize (up to 100,000 kg H_2/day) and central station size (up to 2,500,000 kg H_2/day). An economic analysis of the technology and its comparison to current H_2 production technologies was performed.

The University of Toledo – Hydrogen Storage

Fabrication of Metal-Carbon-Nanostructure Composites

EFC-H2-22

Project Objectives: The main objective of the proposed research is to fabricate self-standing carbon nanostructures on a catalyst preform and incorporate metal species 'into' and 'onto' the carbon nanostructures via electrophoresis, so as to design a compact storage device for hydrogen.

Background: Giving due cognizance to the practical challenges in the fabrication of high purity carbon nanostructures with benign physical characteristics, it is proposed to explore the development, testing and demonstration of a radically new scheme for the large-scale production of CNS of uniform physical attributes. Since the fabrication of carbon nanotubes, graphite nanofibers and filaments invariably involves the presence of metallic precursor as a catalyst, an inexpensive and commonly available transition metal/alloy preform will be used as the support system for the formation of CNS. Carburization in the presence of hydrocarbons or carbon monoxide at high temperatures would lead to the formation and growth of carbon nanostructures (tubes or fibers or filaments) on the catalytic support. Atoms or nanoclusters of carefully selected metal(s) will then be incorporated either into or onto the CNS via electrophoresis. The pre-selected metal/alloy support provides the nucleation and growth site for CNS, as well as acts at the working electrode for the subsequent electrophoresis. Thus, this approach addresses the need for developing novel and compact systems based on metallized - carbon nanostructures with enhanced hydrogen uptake that meets or exceeds the 2010 target of DOE for automotive applications based on PEMFCs.

Conclusions: A large number of material processing iterations were carried out to produce uniformly distributed carbon nanostructures on thin Ni-bearing commercial wires. It was envisaged that if it was possible to metallize these nanostructures with a potential hydrogen-storage metal, then the composite can have an overall higher hydrogen storage capacity.
The commercial wire that was chosen for this purpose was chromel (18 gauge – 125 μm thick, gross nominal composition 95% Ni-5%Cr). Several surface treatment methodologies were adopted to create active sites for the formation of CNTs upon carburizaton; these included, grinding with 100 grit SiC paper, and treatment with dilute mineral acids. Pre-treatment also included a sweep in H2-N2 mixture to remove the adherent oxide surface layer. Several methods of coating the chromel wire with a thin layer of active Ni or Fe were employed so as to make the CNT formation and growth facile. Carburization was carried out in the temperature range of 650-900°C for duration 1 to 2h, using carbon monoxide or methane as the carbon source. While CNTs were formed in many cases, the best results in terms of quality and quantity were obtained in the case of methane mediated carburization of Ni-coated chromel wires at 850°C/ 1h.
This project demonstrated that self-supported CNTs can be formed on a metallic preform after optimization of surface treatment and coating. Another unique aspect of this research was that it totally obviated the entrapment of any metallic impurities within the CNTs whose removal is very laborious, tedious and time consuming.

Praxair Inc. - Hydrogen Liquefaction

Improved Hydrogen Liquefaction Process
EFC-H2-2

Project Objectives: The primary objective of this program is to develop advanced materials to reduce the cost of hydrogen liquefaction by improving management of ortho-para conversion in both existing and new plants. This proprietary materials development effort (detailed in referenced proposal) is expected to substantially reduce hydrogen liquefaction costs. Another objective of this program is to develop a conceptual design for a process that will use these advanced materials. Another objective of this program is to estimate the impact of the new process on reducing the cost and increasing the availability of liquid hydrogen for consumers. The technology developed in this Phase I project will be improved and commercialized in existing and new liquid hydrogen facilities.

If successful, this project will reduce hydrogen liquefaction costs for any size unit, increase the capacity of existing liquefaction facilities, and allow for smaller hydrogen liquefaction plants in more locations throughout the U.S. All of these would be important steps toward increasing the availability of hydrogen to a wide range of consumers and applications, making long-term implementation of the hydrogen economy more likely. Liquid hydrogen is likely to be particularly important in the transition phase to a hydrogen economy because it has a higher density than compressed gaseous hydrogen. This allows more hydrogen to be transported by a single truck, which significantly reduces distribution costs, even after allowing for the additional energy consumed by liquefaction and maintaining cryogenic temperatures.

The cost of liquid hydrogen from existing facilities would be reduced because the energy cost required to liquefy hydrogen would be reduced. In some cases, the reduced refrigeration load might allow for existing facilities to produce more liquid hydrogen if they are limited by cooling capacity or ortho-para conversion. Another key development would be to reduce the cost of smaller hydrogen liquefiers. Currently, there are only seven liquid hydrogen plants in the U.S. in only six states. This means that several consumers are more than 1000 miles away from the nearest liquid hydrogen facility. If smaller liquefiers could be built cost effectively, it would be possible to build them throughout the U.S. to reduce the distance between the liquefier and the consumer, which would further reduce hydrogen delivery costs, and ultimately, significantly reduce the cost of hydrogen to many consumers while increasing its availability. The current hydrogen industry and market are based on a few large producers located near a few large consumers. Advancing to a hydrogen economy will require a shift in the hydrogen market because it will need to focus on millions of smaller consumers. Being able to supply smaller quantities of hydrogen at multiple locations will allow the suppliers to serve these smaller consumers at a lower cost and reduce the cost of hydrogen to the consumer.

Chemsultants, Inc. – Roll-to roll processing

An Innovative and Cost-Effective Micro-Process for Roll-to-Roll Solution Casting of Multi-Layer Proton Exchange Membranes with Superior Performance, Transport and Mechanical Properties in High Temperature / Low RH Operating Environments
EFC-H3-5-1A

Project Objectives
Prove the feasibility of a new manufacturing process for roll-to-roll production of multi-layer PEMs based on interspersed, discrete layers of hydrophilic Zirconium particles and recast Nafion® polymer developed by CWRU that will be solution cast in a layered structure via a novel, advanced process to manufacture thin caliper (12 – 20 µm) membranes.

1) Optimization of the preparation of hydrophilic particle:
Zirconium phosphate and mixed phosphate / sulphophenylphosphonate nano-particles will be synthesized and exfoliated in a suitable solvent. The research team at CWRU has prior experience with this synthesis.

2) Identification and evaluation of process methodologies for casting of thin, multi-layer membranes in a roll-to-roll configuration:
Candidate casting processes including die curtain, slot die and micro-gravure will be investigated and evaluated. The Nafion® solids concentration in the primary casting solution (rheology) and the solvent evaporation and polymer annealing conditions will be optimized.

3) Develop a process for creating discrete, suitable multi-layer membranes:
Alternating layers of Nafion® and a platelet suspension of zirconium phosphate/phosphonate will be deposited onto a suitable support carrier to form multi-layer membrane structures. The membrane thickness, total loading of zirconia particles and number of layers will be varied systematically by proper experimental design. Membranes will be characterized with respect to proton conductivity and water uptake as a function of temperature and relative humidity. Mechanical tests will also be performed.

4) Develop and optimize a membrane structure:
The optimum morphologies will be further investigated as membrane-electrode-assemblies in a hydrogen/air PEM fuel cell operated at differing temperature and humidity conditions.

5) Optimize a continuous multi-layer membrane casting process:
Complete evaluation and development of a pilot process for producing suitable membranes in terms of uniformity of membrane properties and performance.

A provisional patent application was filed on September 4, 2007

EWI - Reformation

Stamping Process Development and Laser Welding for Reformer Material

EFC-H3-13-1A

The generation of affordable hydrogen is an essential element in any fuel cell future. In many application scenarios hydrogen must be derived from a more conveniently available fuel, such as commercial diesel fuel or JP8 military fuel. In these cases a reforming technology is required, and many reformer designs have emerged. Among these designs is the planar reformer, which would be fabricated from thin sheets of stamped, high-temperature, sheet metal alloys. Such a reformer design has been put forth by Delphi for possible use in their Solid State Energy Conversion Alliance (SECA)-supported project, which seeks to provide transportable power for a multitude of uses.

In Delphi’s early efforts to develop a planar reformer solution a problem with metal stamping technology was discovered. Specifically, the selected high-temperature sheet metal appeared to lack the formability necessary to achieve the desired reformer element geometry. Additionally, there were concerns about whether the selected metal could be welded without introducing excessive distortion or other welding defects.

To advance the fabrication technologies necessary for planar reformer manufacturing Delphi worked with EMTEC and EWI to identify a project plan that fit within the framework of the U.S. Department of Energy's hydrogen generation and storage program. EWI’s responsibilities under the resulting project were initially intended to include a detailed review and advancement of Delphi’s initial stamping efforts as well as development of laser welding solutions for the production of sample components, which could then be tested by Delphi.

After the project had been initiated, a funding reduction of 50% required that EWI focus on producing a reduced number of “best effort” deliverables that demonstrated overall feasibility for planar reformer fabrication. Fortunately, EWI was able to fabricate stamped and laser-welded “racetrack” coupons (see report cover) that met the required geometry and hermetic sealing that a planar reformer would require. This foreshortened effort clearly illustrated the value of metal forming analysis in the development of thin sheet metal stampings as well as the robustness of high-speed laser welding for thin sheet components.

Faraday Technology, Inc – Integrated bipolar plate sensor

Bipolar Plate Fabrication for PEM Fuel Cells with Integrated Sensors/Shunts

EFC-H3-18-1A

Project Objective: The overall objective of this project is to demonstrate the technical and economic feasibility of utilizing printed circuit board manufacturing technologies to produce lightweight, low-cost, durable bipolar plates for PEM fuel cells and electrolyzers. This technology will enable low-cost manufacturing of PEM fuel cells, via printed circuit board bipolar plate design and manufacture. The project deliverables include pilot-scale manufacturing technologies for fabrication of bipolar plates.

Background: This project addresses the challenge of low-cost manufacturing of bipolar plates for PEM fuel cells and electrolyzers, and also addresses the need for *in-situ* monitoring of the fuel cell stack by embedded sensors. Bipolar plates are typically fabricated from individual plates of graphite or passive materials, such as stainless steels, and are typically fabricated as a single unit. The proposed technology would allow large numbers of bipolar plates to be made in a printed circuit board (PCB) manufacturing facility. The board would then be cut to produce individual bipolar plates. This method will lower fabrication time and costs associated with having to rack or fixture individual plates for processing.

The proposed technology will address two of the major technical barriers outlined in the Hydrogen, Fuel Cells & Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan: 1) the need for low-cost manufacturing processes for fuel cell components, specifically MEAs, and 2) the need for integrated sensors that are robust, low-cost, and enable improved performance and durability of the fuel cell stack. Sensing and allowing the bypass of defective cells within the stack can increase the lifetime of the stack. The anticipated benefits of this program will be the development of a lean manufacturing approach to PEM fuel cell stack fabrication. The use of PCB manufacturing technologies will lower stack weight and cost. This project addresses design for manufacturing criteria. This project incorporates PCB manufacturing knowledge into the emerging fuel cell manufacturing industry, lowering the learning curve for fuel cell component manufacturers. If successful, implementation of this technology could help to revive the PCB industry in the United States.

Progress and Status: In order to reduce the cost and weight requirements of PEM fuel cell stacks, specifically bipolar plates, Faraday is developing a novel fabrication process that utilizes PCB technology. The proposed process would not only provide lower cost and weight requirements of the bipolar plates but also would provide a means to test the integrity of individual cells within the stack and the consequent by-pass of any faulty cells. This novel approach is anticipated to eliminate the use of conventional graphite or stainless steel bipolar plates, relaxing weight and fabrication inefficiency, as these plates are typically fabricated as a single unit. The proposed technology would allow the fabrication of multiple plates per single PCB. Furthermore, metallization of the novel bipolar plates will be enhanced with the *Faradayic* Process, which will utilize sophisticated, high frequency asymmetrical waveforms for electrodeposition of copper and gold. Faraday has extensive experience in copper metallization of PCBs using the *Faradayic* Process; the copper electrodeposits will be capped with a low porosity gold deposit for corrosion resistance. Physical properties of the electrodeposits, such as porosity, will be fine-tuned and optimized using the *Faradayic* Process.

Faraday conducted polarization test studies. This was done since the polarization curves run at 60°C didn't yield a clear, flat limiting current region. Tests with slower scan rates and lower scan increments were run. In addition to polarization curves, Faraday also ran potentiostatic tests. For tests run at 100 rpm, the potential was held constant at –500 mV (versus open circuit) and for tests run at 400 rpm, the potential was held constant at –640 mV (versus open circuit).

The deposits obtained from the potentiostatic curves were a bright shiny gold, but flaked off the substrate upon rinsing. From this, it is clear that an appropriate pre-treatment process is needed. The pre-treatment process was investigated in the current reporting period; a literature search was conducted to determine the most appropriate cleaning procedure for copper substrates intended for gold deposition. A non-cyanide process was selected.[1] The solutions for the process include a cathodic alkaline cleaner and an anodic alkaline cleaner, both applied at 10-30 ASF at a temperature of 60°C. Appropriate exposure time and operating current were investigated for each, as the reference provides ranges. In addition, the electrochemical steps are concluded with a bright acid dip, at room temperature for 5-10 sec. Faraday experimented with these process parameters by following each pre-treatment scheme with a potentiostatic test. Thus far, the pre-treatment schemes investigated have not yielded deposits with improved adhesion, but these investigations will be continued.

[1] Metals Handbook, Volume 2: Heat Treating, Cleaning and Finishing, 8th Edition, American Society of Metals, Metals Park, OH, 1964, page 641

Faraday Technology, Inc. – TDLAS sensor

TDLAS SENSOR FOR IN-LINE CONTINUOUS MONITORING OF PEM FUEL CELLS & ELECTROLYZERS

EFC-H4-01A

Program Objectives: This program built upon work on two Hydrogen Commercialization Program projects: 1) Nanoscale Catalyst for Hydrogen Generation, and 2) Bipolar Plate Fabrication for PEM Fuel Cells with Integrated Sensors/Shunts. In each project, identification of the need for close monitoring of the environmental conditions inside the electrolyzer or fuel cell bipolar plate lead Faraday to an opportunity for collaboration with its parent company, Physical Sciences, Inc. (PSI, Andover, MA). This collaboration has established the feasibility and conceptual design of a current PSI technology, a Tunable Diode Laser Absorption Spectrometer, for in-situ measurement of the moisture and oxygen content within the channels of a bipolar plate. The fuel cell/electrolyzer industry is in desperate need of rapid, in-line monitoring of fuel cell and electrolyzer performance. This program introduces a compact, flexible solution to this problem.

The overall objective for the proposed program was to demonstrate the technical and economic feasibility of utilizing PSI's Tunable Diode Laser Absorption Spectrometer (TDLAS) for application to fuel cell and electrolyzer stacks, as an in-line diagnostic tool. This program demonstrated an in-line configuration and produced sufficient data to demonstrate the feasibility of this technology to potential commercial clients. This demonstration was done for the moisture content within channels analogous to a bipolar plate used in a single cell configuration. This limitation was based on the availability of a moisture sensor at PSI that could be redesigned for application to the program. Extension to both moisture and oxygen sensing, while previously demonstrated in a different configuration, was beyond the time and cost constraints of this Phase III program and was not required to establish technical feasibility.

The second part of the program was to provide a conceptual design of an in-line TDLAS sensor that could be utilized in a fuel cell stack, across multiple cells in a sequencing format. This design could be adapted in such a way as to be presented with the data to potential strategic alliance partners and licensees. Faraday and PSI seek to work closely with EMTEC to utilize EMTEC's wide range of commercial connections, including the Fuel Cell Supply Chain Database, to present opportunities for alliances.

The specific objectives of the proposed EMTEC project were:

1. Design and build of single cell bipolar plates with simple channels and transparent ports parallel to each channel allowing introduction of the diagnostic laser.
2. Redesign/adaptation of the TDLAS for sensing down the length of a channel.
3. Demonstration of the TDLAS for moisture or oxygen sensing down the length of a channel in a bipolar plate.
4. Conceptual design of a TDLAS capable of continuous, sequenced in-line sensing of a fuel cell or electrolyzer stack.
5. Estimation of the capital and operating cost of the TDLAS for in-line fuel cell or electrolyzer stack monitoring.

In addition, both Faraday and PSI have identified near-term applications beyond the fuel cell/electrolyzer market, improving the chance of successful technology commercialization. In particular, Faraday's work with microfluidic sensors lends itself to integration with the proposed technology for more immediate commercialization opportunities in on-line process sensing as well as monitoring chemical reactions in microchannel reactors. PSI has identified a wide range of applications for the TDLAS including improving efficiency of gas leak detection surveying, monitoring and controlling trace gases in chemical and pharmaceutical processing, and monitoring emissions in energy production plants. Furthermore, Faraday had a recent Department of Energy SBIR program developing low-cost manufacturing methods specifically for fabricating channels in stainless steel bipolar plates. As part of that program, a subcontract to Prof. Shimpalee at the University of South Carolina was aimed towards computational fluid dynamics modeling of the environment within the bipolar plate channels, and the effect of various channel designs on fuel cell performance. These two efforts aligned with this program, to provide data for enhancing the modeling effort and therefore improving the performance of the PEM fuel cell bipolar plates, enabling a better cost-performance ratio that could approach Department of Energy stack cost targets.

Conclusions

1. *Design and build of single cell bipolar plates with simple channels and transparent ports parallel to each channel allowing introduction of the diagnostic laser*: Faraday, with input from PSI, designed and built a bipolar plate with sensor ports incorporated into the side of the plate, such that the TDLAS sensor was able to measure the moisture content of the channel.

2. *Redesign/adaptation of the TDLAS for sensing down the length of a channel*: PSI redesigned the TDLAS sensor for insertion into the ports incorporated into the bipolar plate. Design considerations included the placement and angle of insertion of the sensor.

3. *Demonstration of the TDLAS for moisture or oxygen sensing down the length of a channel in a bipolar plate*: Faraday and PSI successfully demonstrated the ability to sense moisture in the channel of a bipolar plate using the TDLAS, with moisture contents from 0 to 30,000 ppm.

4. *Conceptual design of a TDLAS capable of continuous, sequenced in-line sensing of a fuel cell or electrolyzer stack*: Faraday completed a conceptual design of a TDLAS application for continuous in-line sensing of multiple ports in a bipolar plate and stack, including monitoring of the interior channels of a bipolar plate.

5. *Estimation of the capital and operating cost of the TDLAS for in-line fuel cell or electrolyzer stack monitoring*: At this early stage of development, an estimate has been made of approximately $10,000 for the TDLAS control unit and $500-1000 for each measurement port. With future development, these cost estimates may decrease.

Technology Management, Inc. - SOFC

On-Farm Soybean-Powered TMI SOFC System Demonstration

EFC-H3-14-1A; EFC-H4-14

Project Objective:
Under the Department of Energy Hydrogen Infrastructure Program, the Edison Materials Technology Center selected Technology Management, Inc. (TMI) as a recipient of a phase III award to continue commercialization development of advanced energy generation project. Under this award, TMI utilized this award to augment work for the demonstration of a fuel cell system producing one kilowatt of electricity operating directly on soybean oil being conducted on behalf of the Ohio Soybean Council (OSC). The overall demonstration project continued the commercialization efforts of TMI by advancing prototype system testing at an end-user site in order to provide additional engineering data necessary for downstream product development. TMI will use the Phase III funding to improve the long-term performance of cell and stack components necessary for the demonstration and downstream products.

Building on knowledge from previous short-term demonstrations (at the 2006 OSC board meeting and the 2007 Farm Science Review), this project tested a TMI SOFC system operating on actual end-user loads providing useful AC power for a period of approximately 30 days. For a successful demonstration, improved stack longevity was demonstrated. Under this program, several advanced electrode materials developed by TMI were evaluate for long-term performance in tests of >1000 hours. These materials were selected for mechanical stability with chemical stability considerations but were verified to ensure satisfactory long-term performance. A preliminary characterization of the advanced electrode degradation under several operating conditions was conducted. A variation was used as a comparison to the base case. A large stack final evaluation was be conducted and compared to small stack testing. Testing primarily focused on single cells and small stacks but two full-kilowatt scale tests were conducted to benchmark performance as well. In addition, TMI evaluated fabrication methodologies relating to component quality and the impact on long-term degradation.

The system was modified to accommodate the particular site requirements in this case, a rural farm setting. Electrically, the system was designed to accommodate interface to the existing electrical system. Safety and security for the system, the host site and personnel, and the public utility were all considerations. Grid parallel operation (isolated loads that do not automatically interconnect with the utility grid) was the operating mode of choice. Grid parallel minimized the potential for unsafe interactions with the grid and included manual switching to change from fuel cell power to grid power, to minimize impact to farm operations. The location influenced the mounting and system protections (filters, circuit breakers, etc.) that must be included. Fuel availability and storage was determined and systems designed to accommodate operations. Soybean oil as a fuel is particularly friendly to site because of very low flammability and no toxicity concerns.

UltraCell Corporation – Hydrogen Generation

Manufacturing UltraCell's Reformed Methanol Micro Fuel Cells in the State of Ohio For Military and Commercial Markets

EFC-H3-34

Background/Objectives:
UltraCell has developed a revolutionary reformed methanol fuel cell. Micro-reforming is a novel way to generate hydrogen to support the hydrogen economy. In its pre-production prototype stage this technology has already demonstrated "leap ahead" improvements in energy density and weight savings relative to batteries. For extended missions it offers virtually continuous, quiet, fume-free, and reliable DC power by just hot swapping methanol cartridges. Key development customers to date include the military, first responders, and notebook computer manufacturers. The company intends to transition this technology into high volume manufacturing. In UtraCell's pilot line in Livermore, California, the company is developing and refining highly automated, scalable manufacturing processes for its fuel cell core engine. These processes would be transferred to the manufacturing plant in the State of Ohio by mid-2007.

UltraCell wants to keep its fuel cell core engine technology in the United States. And, the company needs s micro fuel cell "ecosystem" inside the US capable of supplying production quality components. By locating UltaCell's high volume fuel cell manufacturing plant in the Dayton, Ohio, region and by growing suppliers and skilled personnel, the State of Ohio and the US would gain a world class fuel cell technology hub. This hub would position the Dayton region for leadership in the global race for alternative energy technologies and the manufacturing.

Inorganic Specialists, Inc. – Hydrogen Generation

Nanofiber Paper for Efficient Hydrogen Generation

EFC-H3-6-1B

Project Objective: Demonstrate nanofiber paper for hydrogen production, and compare it to existing products. Develop continuous nanofiber papermaking for the hydrogen generation application. Compare different methods of catalyst deposition for its impact on hydrogen generation efficiency.

Background: Our studies have shown that nanofiber paper produces excellent catalyst utilization in fuel cells. Thus it makes sense to examine electrolyzers, which are essentially fuel cells operated in reverse, to see if the high surface area and catalytic enhancement of the nanofiber paper produces high efficiency, high output hydrogen generation. Furthermore, the practical application of this technology hinges on being able to make nanofiber paper commercially, and so the project includes the development of the first demonstration unit for continuous nanofiber papermaking.

Conclusion: Catalyzed nanofiber paper has been shown to enhance the performance of fuel cells through improved catalyst utilization. This project sought to translate those benefits to the reverse process of a fuel cell, electrolysis and hydrogen generation. It was ultimately found that the turbulent conditions and/or stressful voltages that accompany electrolysis were not compatible with the morphology of our catalysts on our nanofibers. Although we cannot rule out that the approach itself has merit, the procedures and materials we examined did not give the results that were sought.

Nonetheless, the design and creation of the first carbon nanofiber papermaking unit was a major achievement from this project. Nanofiber paper is a platform technology for a host of energy-related and composite applications, and the ability to manufacture it represents a true milestone in transitioning it from the lab to the commercial sphere.

Proton Energy Systems – Hydrogen Generation

Development of High Pressure Electrolyzers for Backup Power Systems

EFC-H3-3-1A

Project Objective: This project seeks to develop new materials for high pressure electrolyzers associated with backup power systems. Efforts are focused on finding lower cost membrane materials suitable for high pressure electrolysis that have increased ionic conductivity and/or decreased gas/water permeability as compared to typically used membrane materials.

Background: Currently, various types of batteries are used to deliver backup power in applications such as telecommunications towers. Commonly used battery types, such as lead-acid and Nickel-Cadmium are known to contribute to environmental degradation by introducing toxins into the environment throughout their life-cycle. An alternative to these technologies is the Regenerative Fuel Cell, which combines a proton exchange membrane (PEM) fuel cell with a PEM electrolyzer that produces hydrogen at elevated pressure from water and electricity. This technology holds great promise, but further advances in electrolyzer membrane materials are needed to facilitate wide-spread implementation. In order to achieve this objective, various candidate materials will be obtained and tested for use in high pressure electrolyzers. The project seeks to assess physical properties such as high pressure water and gas permeability and ionic conductivity to identify candidate materials for electrolysis operation. One or two of the tested membrane materials will be operated in an electrolyzer cell stack to generate hydrogen gas at high pressure.

Progress and Status: High pressure permeation testing was completed on Membrane Sample C (from Supplier A) along with bench-top acid capacity and water content testing. Contact with Supplier H (maker of Membrane Sample B) continued. As part of that contact, other samples were identified that might be of potential interest for this application and may be evaluated if schedule and resources allow. Budgetary quote for large volumes of Membrane B was received. One of three materials was received from Supplier L (Membrane D) and diffusion testing was begun. Other materials from Supplier L have been delayed and are expected to arrive in December 2006 and January 2007.

MetaMateria Partners LLC – Hydrogen Generation

Preparation of Nanoscale Tubular Membrane for Hydrogen Purification/Separation

EFC-H3-33-1C

Program Summary: The objective of this project was the development and demonstration of a low-cost, high-flux, nano-enabled tubular membrane technology for the purification/separation of hydrogen obtained by steam methane reforming or a similar method. This technology was developed through the combination of low-cost ceramic supports developed by MetaMateria Partners (MMP) and inorganic separation membrane technologies developed by Professor Henk Verweij of The Ohio State University (OSU). Through this program, a multilayer, tubular, ceramic-supported, ultrathin, dense palladium membrane was developed which exhibited hydrogen permeability of 1x 10-6 mol/(m2·s·Pa) [6x10-4 mol/(m2·s·Pa1/2)] at 320°C. For comparison, the best reported value available from the literature for any supported pure palladium membrane at this temperature is 3.7x10-4 mol/(m2.s.Pa1/2).1 The ultra-thin membrane, which is gas-tight and fully dense at a thickness of about 200 to 300 nm, is enabled through the quality of the graded porosity alumina support which contains two thin, pore-size-reducing intermediate layers and use of a novel nanocomposite catalytic seed layer for electroless deposition of the membrane. Because of the ultra-thin structure of the precious metal palladium membrane layer and the lower-cost manufacturing methods used in preparing the membrane support, this high-performance tubular membrane is anticipated to offer a lower-cost, high-volume solution for the growing hydrogen purification needs. The next steps for this project are to scale up the tubular membrane preparation and demonstrate through a cross-flow, or alternate construction, the viability of the system. This continuation will require a business relationship with a company interested in designing a hydrogen separation system utilizing the technology developed in this one-year program.

Background: The availability of inexpensive energy is perhaps the most important enabler of human technological advancement. Unfortunately, petroleum, which was the fuel of choice for most of the 20th century, has inherent problems which include its dwindling supply, rising cost and the production of greenhouse gases. Many countries have already passed legislation to limit the production of greenhouse gases in an attempt to curb the threat of global warming. Hydrogen has been identified as a fuel of the future in that it can be made without generating carbon dioxide and is a renewable form of energy.3-5 However, since hydrogen is not readily available in its pure, usable form on Earth, it must be manufactured and then separated from other production byproducts. In order to lower the end-user cost of hydrogen fuel, separation technology for hydrogen refinement must be improved. Inorganic membrane separation technology is of particular interest because of the simplicity of design, low energy-cost to use and ease of scalability for manufacturing-scale installations. While several membrane separation technologies are currently under development, a DOE report states:

"Currently no membrane separation technology can simultaneously meet all of the performance criteria for hydrogen fuel production, including high hydrogen flux at low pressure drops; tolerance to contaminants; low cost; operation at system temperatures of 250-600ºC; durability; and robust performance under harsh operating conditions."

Dense metallic palladium is an excellent permselective membrane material for hydrogen separation from other gases as it exhibits selectivity of >1000:1 for hydrogen versus other gases at modest temperatures.3,7 However, thick, self-supporting membranes of palladium are unattractive for use due to the high material cost of palladium metal, low hydrogen flux and susceptibility to chemical attack, especially from sulfur. By moving toward supported thin membranes, the hydrogen flux is increased as the transport length is decreased and the contribution of the material cost of palladium metal is reduced by a factor of between 10 and 1000, depending upon membrane thickness. Alloying elements such as silver and copper in these supported membranes have also been shown to increase hydrogen flux and improve stability against hydrogen embrittlement and chemical attack.3 However, further improvements in the actual performance of these supported membranes are still required. Based upon the current DOE goals, H2 permeance >>3x10-7 mol/(m2·s·Pa) and selectivity greater than 1000:1 at operational conditions are required. In moving to a supported membrane to improve flux by decreasing membrane thickness, the properties of the support become very important in determining the performance of the composite membrane module.9 Qualities of the support to control include the strength, the upper limit of the permeability and the lower limit of the membrane thickness. As implied by the name, the support provides, as its primary function, the mechanical integrity for the membrane during operation. Ceramics, such as aluminum oxide, are a good choice as support materials because they offer high strength and rigidity, are stable under the anticipated operational conditions of the membrane and are inert with respect to palladium. Since hydrogen does not permeate through dense alumina, the support must be porous. This porosity affects both the strength of the support as well as the surface finish onto which the membrane is deposited. In order to maintain the high selectivity inherent to palladium; the membrane must be free of pin holes and other defects. The minimum thickness of the membrane is thus determined by the surface roughness and pore size of the support surface. If larger defects are present in the surface due to poor processing control, the thickness of the membrane will increase. Small pores and a surface that is free of defects are required to minimize the thickness of the membrane, which is desired. On the other hand, small pores in the support will limit the permeability of hydrogen through the support. As with the membrane, the flux decreases as the thicknesses of these fine pore layers increase. If the entire support is constructed with fine pore alumina, the hydrogen permeability drops below the minimum DOE requirement even in the absence of the membrane layer. In order to maximize the permeability through support, the majority of the support structure must be comprised of large pores. This program was very successful at developing and demonstrating a novel, high performance approach toward preparation of an inorganic hydrogen membrane system. The desired outcomes were achieved for each of the tasks. A porous ceramic tubular support was developed using MMCP which was successfully coated with two intermediate layers with reducing pore sizes. Onto this support was deposited a novel, patent-pending nanoparticle seed layer which acted as the catalytic seed layer for the electroless deposition of an ultra-thin, dense palladium membrane layer. The palladium membrane layer is deposited in about 10 minutes by the electroless deposition process and is gas-tight at a thickness of about 200 to 300 nm. Most importantly, the resulting multilayer structure exhibits hydrogen permeability of 1x10-6 mol/(m2·s·Pa) [6x10-4 mol/(m2·s·Pa1/2)] at 320°C which is better than the best reported value found in the literature for any supported dense palladium membrane at that temperature.1 Further improvements are expected with the addition of silver or copper. This high permeability, coupled with the ultrathin membrane layer that requires significantly less palladium than other demonstrated technologies, presents a commercial path toward production of a low-cost membrane system for hydrogen separation. OSU is preparing a patent application for the novel composite seed layer developed in the program for electroless deposition of the palladium membrane layer.

Business Development: Conventional SMR (steam methane reforming), which produces over 90% of merchant hydrogen in the US, and coal-hydrogen both require, conceptually, a steam/oxygen reforming step, followed by two stages of water gas shift, hydrogen separations, and various cleanup/polishing steps. Hydrogen separation is now conducted by pressure swing absorption (first commercialized 40 years ago). PSA is highly capital intensive, and therefore only suited for large, central site production – with a consequential need for (expensive) transportation to smaller users. The newly demonstrated MMP/OSU membrane structure utilizes improved materials in the form of thin, supported membranes to replace the mechanical complexities of PSA. In addition to replacing PSA in central hydrogen production, the inorganic nano-featured membrane at the core of this proposal also represents a disruptive technology for the generation of hydrogen by enabling the manufacture of smaller, cost-effective on-site gas plants that are scaled to suit individual facility demand. With on-site manufacture, the expensive transportation costs can be eliminated. For example, a system using a 1 square meter of the proposed inorganic membrane and a balance of plant with a footprint less than 250 cubic feet, is anticipated to produce approximately 200,000 standard cubic feet of hydrogen per day. This could result in the cost of the hydrogen to be comparable to or lower than the cost of hydrogen delivered via pipeline or gas tube trailer. The ability of flexibly for on-site or distributed generation can dramatically reduce the risk of over-the-road distribution of hydrogen while making the gas convenient to make as the hydrogen economy develops. On-demand generation could also reduce the requirement for high pressure gas storage. In addition to the existing and robust growth in industrial demand for hydrogen, automobiles running on pure hydrogen may also create a significant demand for the gas. Estimates are that there will be at least 1,000,000 vehicles on the road by 2015 requiring pure hydrogen. Hydrogen to support the transportation industry requires the development of a highly integrated national network of manufacturing stations and distribution. The availability of cost-effective, small-scale, on-site, production of hydrogen at fuel stations, could dramatically reduce the investment required to enable the hydrogen-fueled fleet. Combining all current and pending demands for hydrogen, the US market for the commodity is expected to exceed 14,000 billion cubic feet per year by 2012 and new technology that can reduce the production cost of hydrogen is desirable. This project demonstrated the potential for thin, supported palladium membranes for hydrogen separation; however, more experimental development is needed for the system developed in this Phase I (12 month) program to be tested commercially. For this, MetaMateria will work to identify a commercialization partner with an interest in further development and demonstration of the technology. Originally, MetaMateria expected to work with Innovative Gas Systems or Air Products which both provided support letters for this program and had there been availability of funds for a Phase II program, follow on with them would likely have occurred. A Phase I STTR was submitted to DOE in November 2007. Additionally, MetaMateria, with its parent NanoDynamics Inc., which contributed some funds for this project, will actively seek alternative funding in order to continue development and demonstration of this technology. Several potential partners have been identified, summary presentations and marketing materials will be prepared and these will be used to determine interest by organizations interested in commercialization.

The Ohio State University - Hydrogen Generation

Point Source Hydrogen Electrolysis to Enable MOCVD Development of III-V Solar Concentrator Cells

EFC-H3-36-1A

Project Background: The changing environmental and political climates in the United States and in the world have resulted in new priorities for research, development, and commercialization of photovoltaic (PV) cells for the generation of clean electrical energy. For example, compared to many other solar technologies, concentrator photovoltaic (CPV) technology, in particular, may be uniquely capable of achieving the year 2015 targets for levelized cost of electricity as described by the National Solar Technology Roadmap for this technology [1]. However, the design and fabrication of new highly efficient, low-cost cells for CPV applications is absolutely essential to the success of this technology and, indeed, may be a key to future US alternative energy solutions.

Motivated in part by this goal and the promising future applications for all PV technology, in June 2007 the Ohio Department of Development funded a Wright Center of Innovation for Photovoltaic Innovation and Commercialization (PVIC) as a means of strengthening the research and manufacturing base for PV in Ohio. As part of this large PV development center led by The Ohio State University (OSU) and University of Toledo and including more than fifteen Ohio industry members, a primary thrust of the OSU team is dedicated to research and development of advanced solar cell materials, devices and technologies, particularly those related to III-V solar concentrators for terrestrial application.

As a vital enabling asset for this effort, PVIC is establishing a metal organic chemical vapor deposition (MOCVD) laboratory capable of providing epitaxial III-V films for PV research and development to all PVIC member organizations. Through PVIC's capital equipment support, this laboratory will be located at the OSU Nanotech West facility and, in addition to other supporting processing and characterization equipment will be facilitated with an MOCVD reactor to be operational in 2Q08. However, while the current infrastructure of the Nanotech West facility, including more than 6,000 square feet of cleanroom space, is almost perfectly suited to support the significant operational requirements of an MOCVD system and the associated research needs (device processing, metrology, etc.), one of the system installation requirements which requires special consideration is the supply hydrogen, the primary process gas. Hydrogen gas, the industry standard carrier gas utilized in MOCVD processes due its' thermal properties and the ability to purify the hydrogen to levels exceeding 99.99999%, is a key component to minimizing contamination levels in the epitaxial films and achieving acceptable film quality and device performance. This proposal addresses a solution to both the quality and quantity of hydrogen gas required to enable PVIC to operate the MOCVD facility in a manner which also addresses some primary safety concerns.

Statement of Work: In this project, we propose the purchase and implementation of a hydrogen electrolysis system for the supply of primary process gas to support the research and development of advanced solar cell materials, devices and technologies in PVIC's new MOCVD laboratory. For the research sized (capable of growing on 3 two inch wafers) MOCVD system to be installed at OSU to develop innovations in PV technology and related fields based on III-V semiconductor devices, as much as 35 standard liters per minute (slm) of hydrogen gas is required during the epitaxial process while approximately 5 slm is still continuously consumed when the system is in an idle state. This idle flow is critical for keeping the system chamber clean and moisture-free in order to enable good electronic quality of the epitaxially grown films. However, combined with the typical process flows required, not only is a significant amount of hydrogen gas consumed but a significant amount of hydrogen gas is required on-site (or in the laboratory) to maintain system operation and minimize manual handling which is required during high pressure cylinder changes.

In many labs, a solution requiring multiple 6-pack or 12-pack hydrogen cylinder banks is employed. Here, we propose to use a hydrogen electrolysis system instead of the conventional high pressure cylinder solution for two primary reasons. First, the cost of hydrogen high pressure cylinder gases over the decades of the MOCVD tool life is significant. In fact, due to the continuous quantity of hydrogen required to maintain the operation of the MOCVD system alone, limitations in space and safety concerns over cylinder quantities internal to the Nanotech West facility would dictate that a hydrogen gas "bunker" be constructed external to the facility in order to house such a high pressure cylinder supply. In addition to the facility modification costs, increasing tool usage over the duration of the program (and beyond) will result in continually increasing gas costs which could be avoided. In fact, based on modest usage projections the capital cost of a hydrogen generation system will be recouped in only two to four years of operation.

Second, and maybe most compelling for this installation, the hydrogen generator system is a much safer hydrogen supply solution. Safety concerns over the quantity of stored hydrogen and high pressure cylinder handling are always an issue of debate. In addition to the need to construct a separate outdoor hydrogen bunker for a cylinder gas solution, the laboratory's location relative to neighboring properties make alternative liquid hydrogen or gas tube-trailer solutions to meet the process needs an additional safety concern which has received resistance from local agencies for safety considerations. Fortunately, recent advancements in the capabilities of point source hydrogen electrolysis equipment make it a perfect solution for our application. Specifically, the HOGEN 2M unit (one possible hydrogen generator solution produced by Distributed Energy Systems being considered for implementation) is capable of supplying 0-36 slm of high purity hydrogen gas on demand with less than 1 liter of hydrogen gas "stored" when the unit is in an idle state.

Finally, minimal maintenance requirements of less than 4 hours per year are also advantageous and result in minimal impact on the tools research goals. In addition, the HOGEN 2M or similar systems can be expanded with the simple insertion of additional cells to meet greater hydrogen supply needs of the MOCVD or Nanotech West laboratories in the future. Therefore, from both a cost and more importantly the alleviation of many safety concerns present with any other hydrogen solution, the hydrogen generator proposed here is not only a critical enabling addition to the MOCVD lab for PV research and development but a perfect solution for this laboratory's application needs.

Protonex Technology Corp – Hydrogen Generation

METHANOL REFORMED HYDROGEN FOR PORTABLE PEM FUEL CELL SYSTEMS

EFC-H3-19-1B

Project Objective: The proposed program is primarily aimed at addressing issues relating to a methanol-reformed hydrogen based solution for PEM fuel cells in the portable power range. Successfully completed, the project will have significant impact on the development of portable hydrogen infrastructure leading to speedy commercial breakthrough of the emerging technology in applications that can support near-term systems costs. The specific objective of the program is to 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. The main thrust of the Phase I work addressed the technical and functional issues of reformer prototypes operating in conjunction with a fuel cell system. The Phase II effort will be dedicated to optimizing the design and integration of the reformer and fuel cell subsystems. Phase I focused on a 150W scale reformer as a Protonex 150W fuel cell system had already been developed.

Summary: Prior to the beginning of the project, work was done at Protonex on both a reformer prototype as well as a fuel cell stack built to operate on a reformate stream. The reformer prototype was built by Battelle prior to the program and was tested at Protonex primarily to help the Protonex team become familiar with its operation. The testing of the reformer provided valuable information that was incorporated into the designs for the full reformer system, specifically the balance of plant components. The fuel cell stack was constructed on a reliable Protonex stack platform with the exception that its membrane electrode assembly (MEA) was designed specifically for reformate streams with unknown concentrations of CO, CO2, CH4, H2, etc. The stack was operated for approximately 50 hours on a reformate stimulant mixture with CO concentrations ranging from 10 - 250ppm. The stack was able to produce 100-150 watts electrical power while utilizing 55-75% of the hydrogen in the reformate stream.