IV.F.11 Developing Improved Materials to Support the Hydrogen Economy*

Michael Martin
EMTEC (Edison Materials Technology Center)
3155 Research Blvd
Dayton, OH 45420
Phone: (937) 259-1365; Fax: (937) 259-1303; E-mail: hydrogen@emtec.org

DOE Technology Development Manager: Roxanne Garland
Phone: (202) 586-7260; Fax: (202) 586-9811; E-mail: Roxanne.Garland@ee.doe.gov

DOE Project Officer: Jill Gruber
Phone: (303) 275-4961; Fax: (303) 275-4753; E-mail: Jill.Gruber@go.doe.gov

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Start Date: March 1, 2004
Projected End Date: February 28, 2007

**Congressionally directed project*

Objectives

- DOE Application Approval
- Release Round 1 RFP
- Receive, review and approve Round 1 projects
- Release Round 2 RFP for white papers
- Receive, review and approve Round 2 projects
- Collect and review monthly project reports for go/no-go results
- Prepare overall Phase I project report from project reports
- Initiate Phase II projects as appropriate.

Technical Barriers and Technical Targets

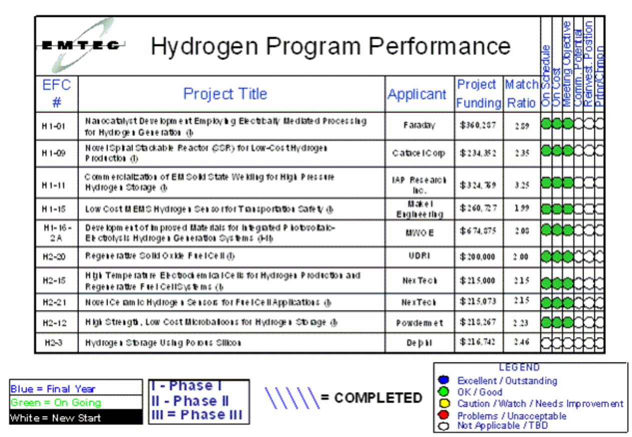
The Edison Materials Technology Center (EMTEC) will solicit and fund hydrogen infrastructure related projects that have a near term potential for commercialization. The subject technology must be related to the DOE hydrogen economy goals as outlined in the Hydrogen, Fuel Cells and Infrastructure Technologies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

Approach

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

Accomplishments

- DOE Application Submitted: April 29, 2004
- DOE Application Approved: September 27, 2004
- EMTEC prepared and released RFP1: June 11, 2004
- Round 1 proposal due date: July 2, 2004
- Proposal review completed (Advisory team): August 30, 2004
- EMTEC released RFP2 (white paper): October 22, 2004
- White paper due date: November 24, 2004
- Proposal review completed: December 7, 2004
- EMTEC released RFP2 (proposals): December 7, 2004
- EMTEC awarded two Round 1 projects: December 2004
- Round 2 proposal due date set for: January 12, 2005
- EMTEC awarded three more Round 1 projects: April 10, 2005
- EMTEC awarded four Round 2 projects: May 16, 2005
- EMTEC released RFP3 (white paper): July 8, 2005
- Round 3 White Paper due date August 12, 2005
- Invitation for full Round 3 proposals: August 29, 2005
- Full Round 3 proposal due date: September 30, 2005
- EMTEC attended and presented at the DOE Hydrogen Program review in Crystal City, May 23 – 26, 2005. EMTEC presented and exhibited at the annual Ohio Fuel Cell Coalition (OFCC) symposium held May 10-11, 2005 in Cleveland, OH.
- EMTEC hosted a highly successful one-day Fuel Cell Fundamentals Short Course on June 6, 2005 at the Engineers Club of Dayton, OH. This course was presented by fuel cell expert, Dr. Jack Brouwer of the National Fuel Cell Research Center, University of California, Irvine.
- EMTEC continued the negotiations for Round 1 and 2 proposals, culminating in a total award of nine active projects as of this period. Kick-off meetings were held and reporting and invoicing procedures were set and announced to each new active project team. EMTEC began the preparations for release of Round 3 RFP.
- Through June 30, 2005, the EMTEC advisory team has reviewed over 50 project proposals and accomplished over 30 site visits.
- As of June 30, 2005, EMTEC packaged a total of 12 project awards with 3 negotiations in progress. Of the 12 packaged projects, 9 have been awarded and



Hydrogen Program Performance

EFC #	Project Title	Applicant	Project Funding	Match Ratio	On Schedule	On Cost	Meeting Objective	Comm. Potential	Reinvest. Position	Prmr/Chmpn
H1-01	Nanocatalyst Development Employing Electrically Mediated Processing for Hydrogen Generation (I)	Faraday	$360,287	2.89	●	●	●	○	○	○
H1-09	Novel Spiral Stackable Reactor (SSR) for Low-Cost Hydrogen Production (I)	Catacel Corp	$234,352	2.35	●	●	●	○	○	○
H1-11	Commercialization of EM Solid State Welding for High Pressure Hydrogen Storage (I)	IAP Research Inc.	$324,769	3.25	●	●	●	○	○	○
H1-15	Low Cost MEMS Hydrogen Sensor for Transportation Safety (I)	Makel Engineering	$260,727	1.99	●	●	●	○	○	○
H1-16-2A	Development of Improved Materials for Integrated Photovoltaic-Electrolysis Hydrogen Generation Systems (I-II)	MWOE	$674,875	2.08	●	●	●	○	○	○
H2-20	Regenerative Solid Oxide Fuel Cell (I)	UDRI	$200,000	2.00	●	●	●	○	○	○
H2-15	High Temperature Electrochemical Cells for Hydrogen Production and Regenerative Fuel Cell Systems (I)	NexTech	$215,000	2.15	●	●	●	○	○	○
H2-21	Novel Ceramic Hydrogen Sensors for Fuel Cell Applications (I)	NexTech	$215,073	2.15	●	●	●	○	○	○
H2-12	High Strength, Low Cost Microballoons for Hydrogen Storage (I)	Powdermet	$218,267	2.23	●	●	●	○	○	○
H2-3	Hydrogen Storage Using Porous Silicon	Delphi	$216,742	2.46	○	○	○	○	○	○

Figure 1. EMTEC Hydrogen Project Status

funded (see Figure 1). Individual quarterly progress reports for the projects funded through this quarter are included in this report.

Future Directions

- Release Round 3 RFP
- Initiate Phase II projects as appropriate
- Collect and review monthly project reports for go/no-go results

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Results

Figure 1 provides an overview of the status of the projects funded by this project to date. A brief description of each project follows:

Faraday Technology, Inc.

Project Title. Nanocatalyst Development Employing Electrically Mediated Processing for Hydrogen Generation

Project Objectives. The overall objective of the project is to develop a low-cost, mass fabrication technology for catalyzation of membrane electrode assemblies (MEAs) for polymer electrolye membrane (PEM) electrolyzers and regenerative fuel cells. This project meets the mission of EMTEC, 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 project is to develop nanomaterials to support 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. A PEM regenerative fuel cell is a technology that 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 which directly affect electrolyzer performance, e.g., loading, dispersion and utilization of catalyst, must be improved using methods amenable to low-cost manufacturing.

In this project, Faraday will develop a sophisticated electrochemical process for catalyzation of MEAs 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 proposed technology is protected by a U.S. Patent. (Taylor, E.J. and M.E. Inman [2000], Electrodeposition of Catalytic Metals Using Pulsed Electric Fields, U.S. Patent No. 6,080,504.)

Status. To meet the economic and performance requirements of the project, we are developing a novel reel-to-reel electrodeposition approach. 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, but 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.

Catacel Corp.

Project Title. Novel Spiral Stackable Reactor (SSR) for Low-Cost Hydrogen Production

Project Objective. The objective of this Phase I project is to demonstrate the technical and commercial feasibility of a novel Spiral Stackable Reactor (SSR) for low-cost stationary hydrogen production. Specifically, we expect to demonstrate that when conventional steam reforming hydrogen plants adopt SSR technology, their life-cycle production costs will be significantly reduced by improvement in catalyst performance, and reduction or elimination of catalyst replacement and disposal costs.

Background. This Phase I project is designed to: I) Determine SSR cost structure by determining if life cycle cost appears to be less than life cycle cost for currently used ceramic media technology, and by determining if initial product manufactured cost is such that the SSR can be sold for a price comparable to ceramic media; II) give initial input regarding the durability of the SSR; and III) develop a commercialization plan.

In FY 2005, a safety review was conducted. Discussions were held with potential catalyst suppliers, resulting in a number of test strips being made. Several samples tested to date show very promising results. A market study was initiated and preliminary results reported. A computer and FEMLAB software were purchased, and computational fluid dynamics (CFD) modeling efforts were initiated. Model results led to the realization that the original product design has some performance issues. A modified design was conceived that shows promise. Equipment needs were reviewed; a used powder blender was purchased. Modifications to the flow bench were specified and components placed on order. Discussions were held with potential customers about performing a (free to them) field demo in place of a marginally useful lab demo.

Status. Rapid progress has been made on the project, particularly in late May, as resources from other projects have become available. A significant breakthrough on the design was realized as a joint effort between CFD modeling and bench prototyping efforts (Tasks I-B and I-C). Original attempts to make prototypes that had good performance predicted by CFD models proved difficult. Intense collaboration between prototyping and CFD personnel revealed two (closely-related) designs that are easily produced and are expected to have good performance. One variation was prototyped and tested on the flow bench (Task I-D), and showed superior heat transfer performance when compared to ceramic materials. We expect to have the other variation prototyped and evaluated by mid-June. Depending on the variation selected, some final tooling issues will need to be worked out.

Flow bench (Task I-D) is working in manual mode. Automatic controls should be on-line in mid-June. Testing of catalyst samples (Task I-A) is proceeding at the University of Toledo (Task I-E). A furnace and some auxiliary parts were purchased for the Catacel lab reactor (Task II-A), but overall system design is proceeding slowly. Enspec Engineering (Tom Tomsik) was engaged to do some system design to speed this along.

Iap Research, Inc.

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

Project Objective. In Phase I, 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 Research, Development, and Demonstration Plan for compressed hydrogen storage while at the same time have excellent near term commercialization potential in HVAC, chemical processing and power industries.

Background. The Hydrogen, Fuel Cells & Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan identifies that on-board hydrogen will require storage pressures as high as 10,000 psi. Due to safety issues with hydrogen, all joints and fittings associated with storage at this pressure must have extremely low leak rates.

These new requirements will severely challenge commonly used joining and assembly technologies. It is unlikely that commonly used threaded fittings and compression joints will be able to meet the proposed pressure ratings and leakage requirements.

In industries such as the nuclear industry, where safety is critical, tubular pressure joints are often made by conventional fusion welding processes. In order to meet safety requirements, these welding operations are often 100 percent inspected via expensive quality assurance testing. In addition, highly certified welders using no automation normally perform these welding operations. It is unlikely that the cost structure associated with these types of operations will be transferable to the on-board storage of compressed hydrogen.

The storage of hydrogen creates additional problems with respect to conventional fusion welding processes. Fusion-welding processes often significantly reduce the strength of the joint in the heat-affected zone. Problems with hydrogen embrittlement are well documented for materials commonly used in pressures vessels. Materials exposed to hydrogen gas can become even more susceptible after they have been welded via standard fusion processes. Pressure joints using alternative materials to reduce hydrogen embrittlement effects will be required. This may generate the requirement for creating high-pressure joints between dissimilar materials, which will prevent the use of almost all fusion welding processes.

Future requirements for hydrogen storage will require alternatives to conventional welding processes. The need for high strength joints between dissimilar will be required to make on-board hydrogen storage a reality. In addition these joints must be robust, reliable and cost effective to produce.

Status. Under this project we have undertaken in a Phase I effort to evaluate the commercial potential of using electromagnetic solid state welding for the production of high pressure, low leakage joints in tubular structures. During the past month we have initiated Task 1 – Scope of Application and Task 2 – Solid State Welding Parameter Study. Under these tasks we are developing the initial pressure sealing applications and identifying the associated welding parameters that will be assessed under the Phase I project.

Makel Engineering, Inc.

Project Title. Low Cost Micro Electromechanical Systems (MEMS) Hydrogen Sensor for Transportation Safety

Project Objective. The objective of this research is the development of advanced hydrogen sensor systems 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 MEMS and National Energy Modeling System (NEMS) based sensor technology provides the means for low-cost, compact, low power consumption, and miniaturized systems, suitable for mass production. Makel Engineering (MEI) will demonstrate hydrogen sensor technology suitable for hydrogen fuel transportation control and safety applications, which will lead to a low-cost, robust, and commercially viable automotive hydrogen sensor.

Background. This project addresses the use of hydrogen sensors for hydrogen fuel safety. The sensors will monitor hydrogen levels in the ambient air in the vehicle, and will be applicable to safety monitoring for hydrogen delivery infrastructure. MEI will adapt its current state-of-the-art wide range hydrogen sensor, originally developed for aerospace applications, to the automotive market. Hydrogen leaks must be promptly and accurately detected so corrective action (e.g., shut down of hydrogen supply, ventilation of passenger cabin) can be performed well before its concentration in air reaches the lower explosive limit.

Our sensor combines in a single chip a hydrogen sensitive Schottky diode made of a palladium alloy on a silicon substrate for measurements in the low concentration range (50 ppm to a few percent) and a hydrogen sensitive Pd alloy resistor for measurements in the high concentration range (1-100 percent).

Progress Status. During the FY 2005, the following was accomplished for this project:

- MEMS sensor fabrication (Case Western Reserve University [CWRU]) - ongoing
- NEMS test sensor fabrication (Argonne National Laboratory) – delayed, ongoing
- Continue lab testing of NEMS and MEMS hydrogen test sensors
- Initiation of commercial hydrogen sensor system design

- Project update meeting with Elliot Salmon on 4-14-2005
- Generation of product development plan and common commercial test plan

Ongoing tasks encompass the design and initial fabrication of the commercial prototype. In addition, sensor testing and characterization is ongoing and CWRU is continuing MEMS sensor fabrication.

Midwest Optoelectronics, LLC (MWOE)

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

Project Objectives and 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 integrated photovoltaic electrolysis (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. In Phase 1, there are three tasks:

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

Status. Task 1. The University of Toledo (UT) fabricates 100cm^2 triple-junction a-Si based solar cells using its small-area cluster tool, on a routine basis. Efforts are made to fabricate these solar cells having reversed structures, desirable for PEC applications. As was described in the original proposal, this effort is supported by complementary development projects at UT, with funding from the Air Force Research Laboratory (AFRL/WPAFB/UTC) and the National Renewable Energy Laboratory. The effort under the EMTEC project is on the transfer of technologies developed under these on-going projects for MWOE's use in the fabrication of large-area PEC panels. Under the AFRL supported project, UT has made several deposition runs for the reversed-structure solar cells (p-i-n type instead of the usual n-i-p type) on glass superstrate for PEC applications. The research effort included the optimization of p-type material deposited at higher substrate temperature and the insertion of a ZnO layer between the SnO_2 transparent conducting layer and the n-type doped amorphous silicon layer. Solar cells having p-i-n structure have been successfully deposited on glass superstrates for PEC applications. Task 1 effort represents a marginal portion of the Phase 1 effort, while Task 2 effort represents the focus of the Phase 1 effort.

Task 2. The photovoltaic panels used in this task are produced by Energy Photovoltaics. These a-Si cells are double-junction a-Si/a-Si solar cells. The layered structure of the cells is: Glass/SnO/a-Si-p-layer/a-Si-intrinsic/a-Si-n-layer/a-Si-p-layer/a-Si-intrinsic/a-Si-n-layer/aluminum. Scribe lines are made so that the cells are interconnected for conventional photovoltaic series interconnection. These unencapsulated solar cells deposited on 2 ft x 4 ft glass substrates are shipped to MWOE for the assembly of PEC panels. At MWOE, additional interconnection and isolation steps are taken to convert the panels for integration with electrolysis components.

Task 3. Under this task, MWOE is currently focusing on two areas: 1) a market study to collect and analyze available public reports that focus on the forecast of future market size by the segments and applications and the growth potential for each of these segments and 2) a financial planning for future production and expansion plan. Preliminary studies in these two areas have been completed. Detailed studies in these two areas are underway so that a comprehensive commercialization plan can be developed.

University of Dayton Research Institute

Project Title. Regenerative Solid Oxide Fuel Cell

Project Objective. The successful demonstration of a commercially viable, regenerative solid oxide fuel cell (RSOFC) and solid oxide electrolyzer cell (SOEC) based systems depend upon the development of cost-effective and reliable cell material exhibiting a high operating power density and high hydrogen production efficiency, while minimizing system size. The overall objective of the Phase I effort is to select anode, cathode, and electrolyte materials and test these materials using SOEC cells for intermediate temperature ranges from 650 to 850C.

Background. Water electrolysis will be a viable approach to achieve an environmentally benign source for hydrogen production. A high temperature steam electrolyzer SOEC or RSOFC does not require precious metals for catalytic activities, but has the advantages of high efficiency for hydrogen production compared to a low temperature water electrolyzer.

Status. Work continued on the fabrication and evaluation of the laboratory scale RSOFC/SOEC cells. A cell supported on Ni/YSZ anode was fabricated using the powder (solid state sintering) processing technique. The electrolyte and cathode for the cell were YSZ and Pt-paste, respectively. The thicknesses of anode, electrolyte and cathode were approximately 2 mm,
15 mm and 10 mm, respectively. The cell was inserted into the RSOFC/SOEC test apparatus and evaluated using H_2:H_2O as the fuel and air (O_2:N_2) as the oxidant. The unit allowed the determination of I-V plots of a cell structure.

H_2:H_2O, Ni-YSZ/YSZ/Pt, O_2:N_2

The lower open circuit voltage (OCV) as compared to theoretical OCV suggests leakage of hydrogen or migration of platinum across the electrolyte.

It is believed that either the electrolyte is too thin or it is not fully dense. An existing effort is being focused to produce a dense, thick electrolyte to obtain a higher OCV so as to increase the power output and, hence, enhance hydrogen generation efficiency.

Nextech Materials, LTD.

Project Title. Novel Ceramic Hydrogen Sensors for Fuel Cell Applications

Project Objectives and Background. The objective of this project is to develop a novel ceramic based sensor for hydrogen detection for fuel cell safety systems. The ceramic sensor materials being used will result in a low-cost sensor technology with improved gas sensitivity, selectivity, and response time which are critical for the fuel cell safety application. NexTech will improve sensor performance by the following methods:

- Improved compositional control by NexTech's proprietary chemical synthesis process.
- Addition of dopants to enhance sensitivity and limit cross-sensitivity to other combustible species (i.e. CH4, CO, etc.)
- Addition of catalytic promoters to improve sensing performance.
- Optimumization of the operating temperature for hydrogen sensing with limited cross-sensitivity to other contaminant gases
- Use of a novel component design that improves improves sensitivity and minimizes power requirements.

Throughout this project, NexTech will conduct materials synthesis, characterization and testing work. In Phase I of this project, NexTech will evaluate the feasibility of novel materials strategies. At the end of the Phase I, NexTech will fabricate and test a breadboard prototype for delivery to EMTEC and for evaluation by fuel cell system developers.

Status. All raw materials have been ordered, and initial characterizations of the active sensing materials have been completed.

Inks were prepared and coatings have been deposited on alumina substrates with inter-digited electrodes for testing. Temperature screening and sensitivity studies have begun on ceria sensors. Temperature screening and sensitivity measurements have been initiated. Tempeature versus sentivity measurements are being conducted at 300, 350, 400, 450 and 500°C. Preliminary test on one of the baseline materials showed very good sensitivity to hydrogen at the 500 ppm level, which is well below the 25% lower explosive limit (LEL) of 1%H_2 in air. This sensor had an average sensitivity of 80% at the 25% LEL.

Powdermet Inc.

Project Title. High Strength, Low Cost Microballoons for Hydrogen Storage

Project Objectives and Background. The Phase I technical approach will be to apply high strength glass, ceramic and metallic nanocomposite coatings to carbon microballoons using chemical vapor deposition in a fluidized bed as well as various other coating methods. By applying a defect-free high strength coating using chemical vapor deposition in a fluidized bed, very thin, high strength coatings (1 micron or less) can be applied to the ultra lightweight (0.05g/cc) carbon microballoon *seeds* in a very cost-effective manner. Furthermore, this coating is applied in vacuum, leaving no residual gas inside the spheres to reduce capacity. Coatings on microballoons with a thickness of 0.8-1.1 microns will be deposited. This will result in a microballoon with burst strength exceeding 15,000 psig and a storage capacity of >10 wt% as well as exceptional crush strength for structural applications. High strength microballoons will be used for the hydrogen economy. The high strength microballoons will also be used in the creation of light weight structural solids that can be used in various applications including transportation and aerospace applications.

Status. During this reporting period initial models for microballoons were created in Ansys and evaluated under burst and crush pressures as well as with selected voids under static conditions to give a modeling base line for how perfect microballoons would behave. The models show that for both internal and external pressures the maximum stresses occur on the interior of the balloon. The initial single walled balloon modeled was and elastic material simulation of a glass balloon 100 um in diameter with a wall thickness of 10 um (typical for a glass microballoon). A small defect was also modeled on the surface of the balloon to show how a defect can magnify the stresses in a particular area. The stresses on the perfect balloon were greater for a 10,000 psig load in burst than in compression. The stresses in burst are almost double compared to the stress in compression. The maximum stress occurs on the interior wall of the balloon but in burst the interior is in tension and in crush it is in compression. When the defect is created the stresses are maximized at the center of the defect and depending on the material and size and shape of the defect they approach or exceed the tensile strength of the glass. More analysis is being conducted on how a coating affects the stresses on the balloon and if a coating can reduce the stress maximization at defects by filling in the defects.

Conclusions

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

FY 2005 Publications/Presentations

1. EMTEC presented an overview at the DOE Program Review in Washington, DC May 23-26, 2005