Title: **A Radically New Method of Hydrogen Storage in Hollow Glass Microspheres**

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OBJECTIVES

This project will demonstrate that hydrogen can be stored and delivered using hollow glass microspheres (HGMS) with efficiencies superior to that of other methods by application of the newly discovered phenomenon of photo-enhanced hydrogen diffusion in glasses. Studies will provide an understanding of the mechanism(s) behind this effect, allowing use of that knowledge to optimize application of this phenomenon to a hydrogen generator, to develop the technology necessary to produce HGMS of the desired composition in sufficient quantities for proof-of-concept testing, and to determine the parameters necessary for filling and the rate of loss of hydrogen from HGMS under ambient conditions.

ACCOMPLISHMENTS TO DATE

Results to date demonstrate the increased release rate of stored gases during exposure to intense light as compared to traditional heating. The initial release of gas occurs within 1 second (our minimum detection time) of exposure to light. This release rate is much faster than that obtained by heating the sample using a preheated furnace.

Hollow glass microspheres containing different concentrations of iron oxide have been produced by MoSci, Corp. under a subcontract on this project. A pressure-volume-temperature (PVT) apparatus for photo-enhanced diffusion measurements has been designed, constructed, and calibrated. A residual gas analyzer (RGA) system for study of the kinetics of outgassing has also been constructed and used for preliminary studies.

The effect of photo and thermally induced gas diffusion in HGMS has been studied as a function of gas identity and pressure, temperature, and dopant content in the HGMS. Linearity between applied gas pressure and amount of gas contained within the HGMS has been demonstrated. The effect of temperature on fill rate has been established for this glass. The photo-induced response increases, i.e. the response time decreases, with increasing dopant concentration.

The base glass used by MoSci Corp to produce these HGMS consists of approximately 85% recycled container glass. Various concentrations of iron oxide and other dopant oxides are added to cullet of this glass, along with small amounts of other component to produce a melt containing

the dopant. This glass is then crushed and flame processed to produce HGMS. MoSci Corp can now deliver approximately 100 gram samples as needed for future study.

Other properties of the glasses have also been determined, including the optical absorption spectra of the microspheres in the uv-vis-nir and infrared regions of the spectrum and the thermal expansion coefficients and glass transformation temperatures of the bulk glass. Studies are underway using electron paramagentic resonance, magnetic susceptibility, and other methods to determine the degree of reaction of the glass with hydrogen during filling, storage, and outgassing. Characterization of the microspheres for size distribution, wall thickness, and compositional variation is underway.

FUTURE WORK

Testing will continue using both photo-enhanced and furnace induced diffusion for comparison of kinetics of the process. These tests will include both PVT and RGA methods (these methods cover different aspects of the project). Mathematical models for the diffusion rate for loading and outgassing the spheres will be developed. Studies of the effect of pretreatment of the HGMS to alter the oxidation state of the iron will be carried out. The effect of microsphere size on outgassing kinetics will also be studied.

Additional dopant concentrations will be studied in order to optimize the material. It has been found that too much dopant limits the penetration depth of the light in the samples without contributing a significant improvement in kinetic response. Studies of the loss of hydrogen under ambient conditions (0 to 50°C) will continue to establish the retention time for the spheres.

Presentations:

Fabienne C. Raszewski, Mathew M. Hall and James E. Shelby, "Thermal and Photo-Induced Outgassing of Hollow Glass Microspheres," GOMD meeting ACerS, Greenville, SC, May, 2006.

James E. Shelby and Mathew M. Hall, "Separation, Purification, and Storage of Hydrogen in Hollow Glass Microspheres," GOMD meeting ACerS, Greenville, SC, May, 2006.

James E. Shelby and Mathew M. Hall, "Novel Applications of Hydrogen-Filled Hollow Glass Microspheres," University Conference on Glass, Penn. State U., June, 2005. (Invited)

Matthew M. Hall and James E. Shelby, “Hollow Glass Microspheres for Hydrogen Gas Storage,” NHA Annual Hydrogen Conference, Mar. 2005, Washington, DC.

Matthew M. Hall, Scott T. Misture, and James E. Shelby, “Glass as an Enabling Material for the Hydrogen Economy,” GOMD meeting of the Am. Ceramc. Soc., Nov. 2004, Cocoa Beach, FL.

Student supported:

Ms. Fabienne Chantal Raszewski, B.S. Ceramic Engr., 2003, M.S. Glass Science, 2004, Alfred University, currently a PhD student.