HGMS: Glasses and Nanocomposites for Hydrogen Storage

2011 DOE Hydrogen and Fuel Cells Program and Vehicle Technologies Program Annual Merit Review and Peer Evaluation Meeting

May 9-13, 2011, Arlington, Virginia

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3/11/2011

Project ID #ST085

Overview

Timeline

New Project:

- Start: Nov. 2009
- End: Oct. 2011
- ~30% complete

Barriers

- Comprehensive understanding of storage material properties
- Weight and cost of hydrogen storage system
- Durability/reversibility of hydrogen storage system

Budget

- Total project funding
  - DOE: $523,325
  - UNLV: $130,831

Partners

Independent Project:

- Assoc. Res. Prof. K. Lipinska (PI - UNLV)
- Res. Prof. O. Hemmers (co-PI - UNLV)
- Post-Doctoral Scholar – UNLV
- Team has established collaborations in materials science (LBNL, Coe College, ANL, Illinois Institute of Technology, University of Verona, Italy)

Relevance - Objectives

Global Objective

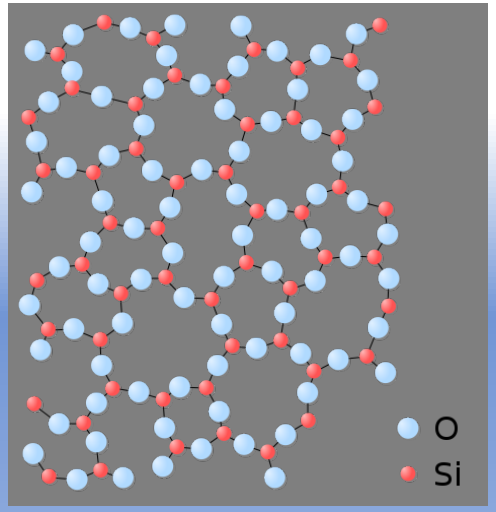
- endeavor to extend the concept of using glass-based material as hydrogen storage media
- demonstrate a pathway to the finding of a class of materials for hydrogen storage media that can hold hydrogen at ambient conditions through physisorption.

Objective for Current Project Year:

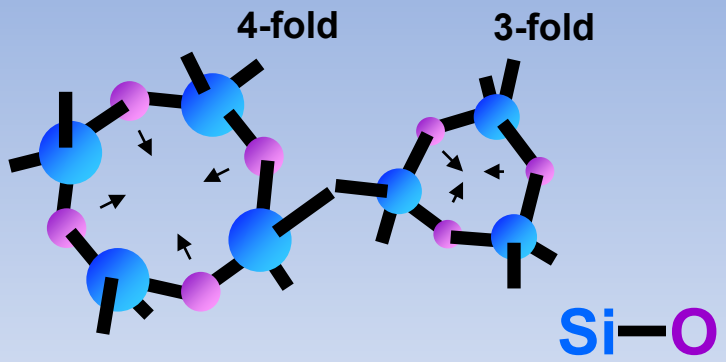
- develop glass-based materials with structural properties that would make them promising candidates for use in H-storage: either as material for glass microspheres or for sponge-type storage.

Approach – Uniqueness

- **The concept of Structural Free Volume:** the disordered structure of glass has ample free spaces that can be modified by composition of glass network and functional dopants etc.
- or by growing **nanosized crystals** within the glass architecture
- could open doors for **new material's functionalities** in respect to hydrogen storage.
- **glass itself could be a sponge for H-storage if endowed with "H-sponge" functionality**



Ex: 2D structure of glass showing structural Free Volumes



Ex: different ring structures found in SiO_2 glass

Approach – Program Barriers

Barrier: Comprehensive understanding of storage material properties

Barrier: Weight and cost of hydrogen storage system

Barrier: Durability/reversibility of hydrogen storage system

- Modulation of Structural Free Volume in Glass as well as nano-crystallization provide an avenue to reach a sponge-like material for H-storage, but requires a better fundamental understanding of the materials themselves
- A glass-based H-storage material could be made from low-cost and light-weight components
- Modulations of glass network which could be induced by external fields (temperature, E&M) promise reversibility of H-storage

Approach- Integration with H_2 Progs.

- Complements previous project on hydrogen storage using hollow glass microspheres

Approach – Milestones FY10 &FY11

Changes to baseline schedule

Planned: Jan 2010 – Dec 2011 (24 mo.)

Delayed by ~12 months: laboratory reconstruction works required before installation of state-of-the art instrumentation

New End Date: to be established; does not involve re-scoping

Milestone M.1

- equipment purchased, installed and tested →90% complete
- post-doctoral researcher is hired → 100% complete

Milestone M.2

- Fabrication of series of glasses →10% complete

Milestone M.3

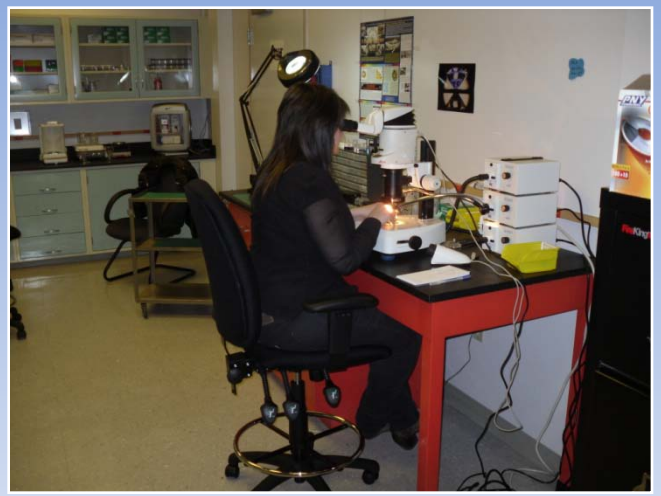
- comprehensive understanding of structure and packing density in the fabricated glasses (…). →not started

Milestone M.4.1

- Fabrication of glass-ceramic nanocomposites (…) → 10% complete

Technical Progress – Previous Accomplishments

- This is a brand new project and requires the establishment of 2 new laboratories
- The project is composed of 4 Tasks which are sequential
- The execution of the project is conditioned by laboratory reconstruction and equipment purchase, installation and testing
- The first laboratory space to be renovated was the **Materials Synthesis Lab.** combined with **Materials at Extreme Environments Lab.** → **completed**



Relates to accomplishment of Milestone M.1

Technical Progress – FY10 & FY11

Task I: Equipment Purchase, Laboratory Setup and Personnel Hiring

- **Subtask 1.1** Equipment purchase → **100% completed**
- **Subtask 1.2** Laboratory Reconstruction Works → **100% completed**
- **Subtask 1.3** Setup and Testing of Instrumentation →**90% completed**

Comment: large portion of the experimental instrumentation had to be selected, negotiated and purchased. This included:

- Raman spectrometer, combined with a confocal Raman microscope
- Mid-temperature research furnace
- Optical tables
- Multi-wavelength gas laser
- High-temperature research furnace
- other minor equipment and lab supplies

Technical Progress – FY10 & FY11

Lab space reconstruction included: electrical, HVAC, water, fireproofing to adapt the lab space to requirements of state-of-the art new instrumentation.

Subtask I.2: Laboratory Reconstruction Works → 100% completed

Relates to accomplishment of Milestone M.1

Technical Progress – FY10 & FY11

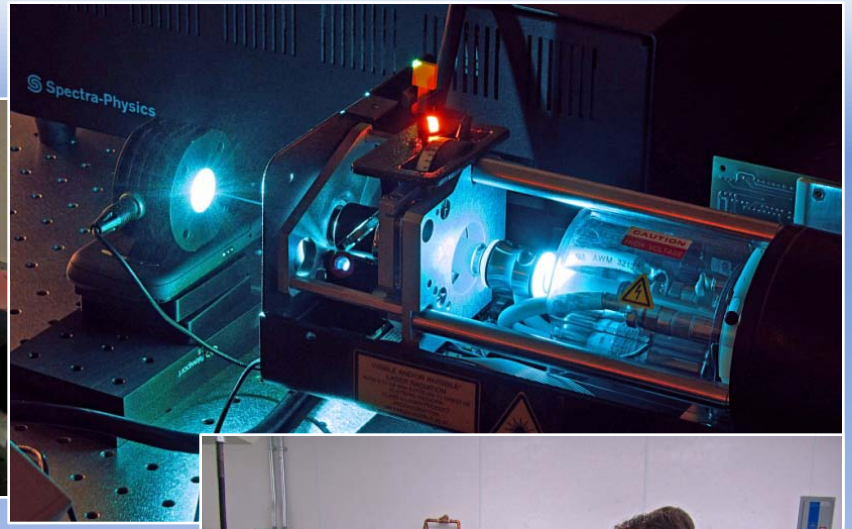
Purchase and installation of a double vibration control system for optical instrumentation: Custom Doubler Table, Air Compressor and 4 Vibration Isolators

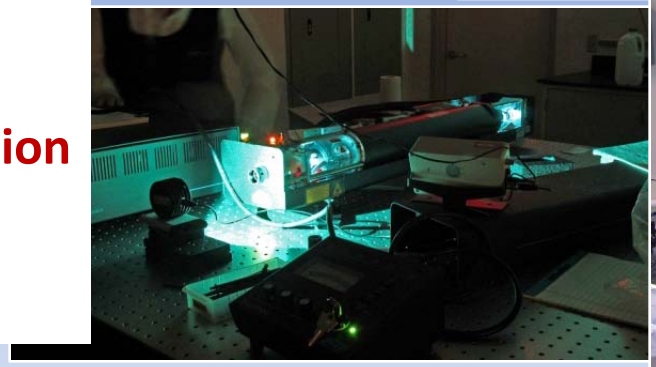
Subtask I.3: Setup and Testing of Instrumentation →90% completed

Relates to accomplishment of Milestone M.1

Technical Progress – FY10 & FY11

Purchase, installation and testing of multi-wavelength Ar-Kr class IV Gas Laser and Water Conditioning System





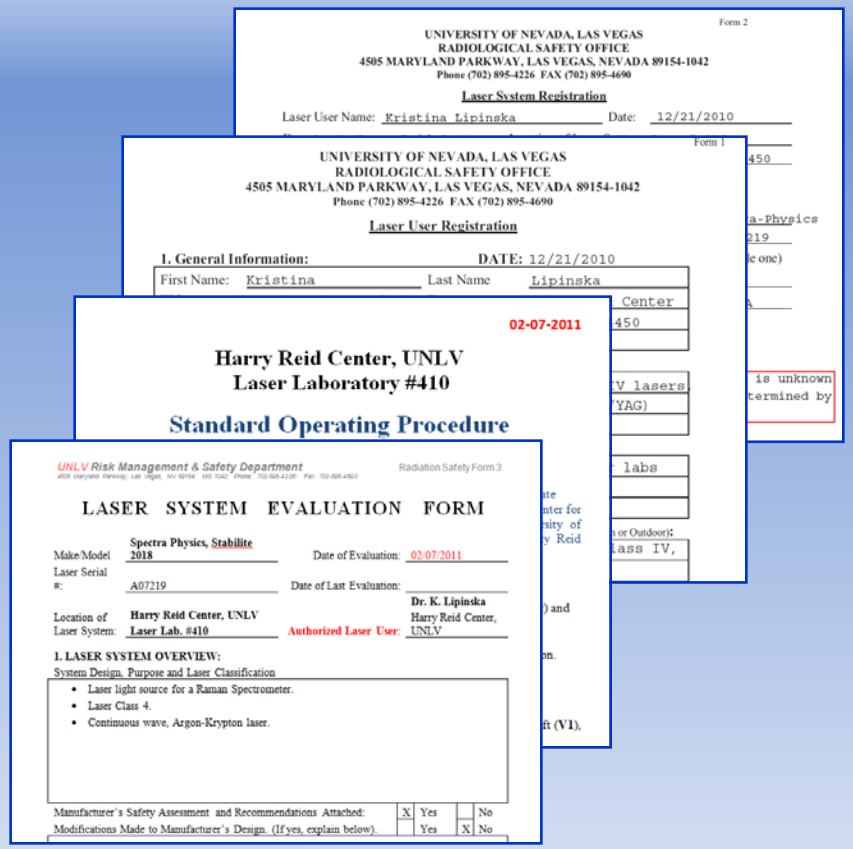
Subtask I.3: Setup and Testing of Instrumentation →90% completed

Relates to accomplishment of Milestone M.1

Technical Progress – FY10 & FY11

Subtask I.3: Setup and Testing of Instrumentation →90% completed

- Established safety rules, Dos and Don'ts of Laser Operation, Lab Access and Use Rules, Standard Operating Procedures according to University Regulations and National (ANSI) Standards
- Created an access controlled laboratory space: Prox Cards on doors, interlocks, warning lights
- Established physical and safety barriers for safe operation of laser and spectrometer.



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4505 MARYLAND PARKWAY, LAS VEGAS, NEVADA 89154-1042
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Laser System Registration

Laser User Name: Kristina Lipinska Date: 12/21/2010

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4505 MARYLAND PARKWAY, LAS VEGAS, NEVADA 89154-1042
Phone (702) 895-4226 FAX (702) 895-4690

Laser User Registration

I. General Information: DATE: 12/21/2010

First Name: Kristina Last Name Lipinska

02-07-2011

Harry Reid Center, UNLV
Laser Laboratory #410

Standard Operating Procedure

UNLV Risk Management & Safety Department

LASER SYSTEM EVALUATION FORM

Make/Model: Spectra Physics, Stabilite 2018 Date of Evaluation: 02/07/2011

Laser Serial #: A07219 Date of Last Evaluation:

Location of Laser System: Harry Reid Center, UNLV Laser Lab. #410 Authorized Laser User: Dr. K. Lipinska Harry Reid Center, UNLV

I. LASER SYSTEM OVERVIEW:

System Design, Purpose and Laser Classification

- Laser light source for a Raman Spectrometer.
- Laser Class 4.
- Continuous wave, Argon-Krypton laser.

Manufacturer's Safety Assessment and Recommendations Attached: X Yes No

Modifications Made to Manufacturer's Design. (If yes, explain below). Yes X No

Relates to accomplishment of Milestone M.1

Technical Progress – FY10 & FY11

Purchase, installation and testing of High-Resolution Raman Spectrometer System with Confocal Microscope and Motorized Mapping stage

Subtask I.3: Setup and Testing of Instrumentation →90% completed

Relates to accomplishment of Milestone M.1

Technical Progress – FY10 & FY11

Purchase, installation and testing of 2 High Temperature Furnaces for synthesis of glasses and of nano-composites

Subtask I.3: Setup and Testing of Instrumentation
→90% completed

Relates to accomplishment of Milestone M.1

Technical Progress – FY10 & FY11

Purchase, installation of lab benches, instrumentation carts and computer desks.

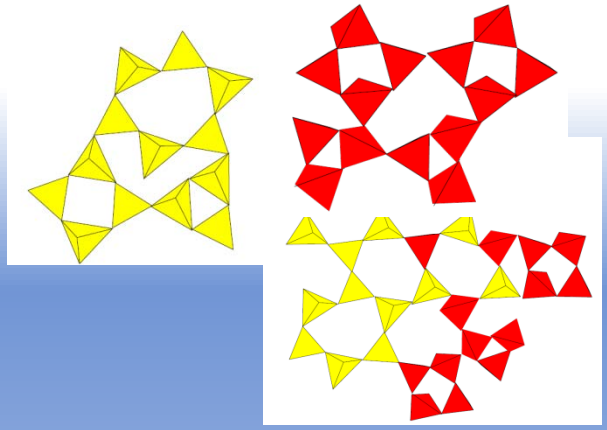
Subtask I.3: Setup and Testing of Instrumentation
→90% completed

Relates to accomplishment of Milestone M.1

Technical Progress – FY10 & FY11

Task 2: Synthesis and Processing of Glass Materials

→ 10% completed

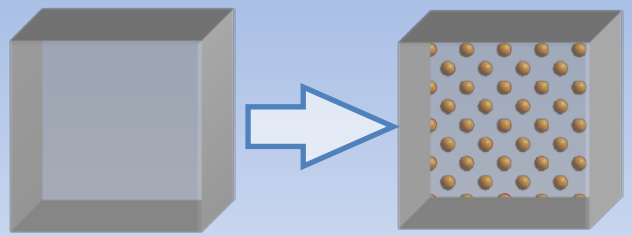


Task 3: Micro-structural and Nano-structural Studies

→ 5% completed

Task 4: Synthesis of Glass-Based Nanocrystalline Composites

→ 10% completed



Technical Progress – FY10 & FY11

Task 2: Synthesis and Processing of Glass Materials

Subtask 2.1: Fabrication of Glass Materials → 10% completed

Glass batch #1
doped with Ti

Glass batch #2
doped with Ta

Glass batch #3
doped with Ga

Successful fabrication of silica based glasses with titanium, tantalum and gallium acting as network formers and/or network modifiers

Relates to accomplishment of Milestone M.2

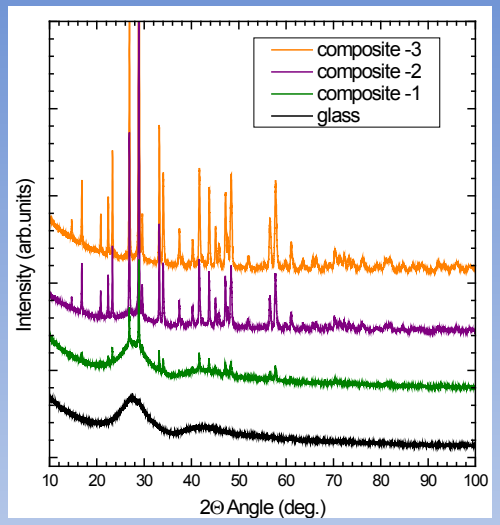
Technical Progress – FY10 & FY11

Task 4: Synthesis of Glass-Based Nanocrystalline Composites

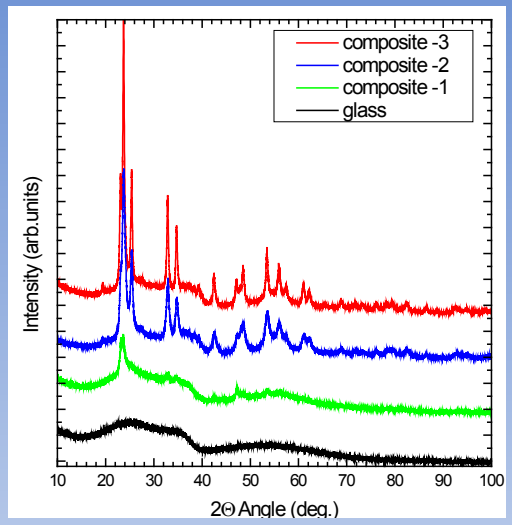
Subtask 4.1 and 4.3 → 10% completed

progress of nano-crystallization

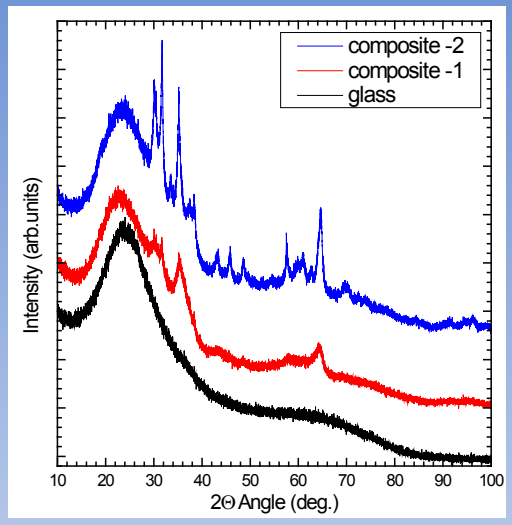
Composites based on glass batch #1 doped with Ti



Composites based on glass batch #2 doped with Ta



Composites based on Glass batch #3 doped with Ga



Successful fabrication of several nanocrystalline composites based on glass batches related to Subtask 2.1. As seen on XRD patterns wide bumps indicate glass and the sharp lines show presence of nanocrystals inside the host glass. This is an important accomplishments because there exist only very few glass compositions that allow for formation of nanocrystals in a glass matrix.

Relates to accomplishment of Milestones M.4.1 and M.4.2

Collaborations

- **Team:**
  - Assoc. Res. Prof. Kris Lipinska
  - Res. Prof. Oliver Hemmers
  - Post-Doctoral Researcher

- Project team has established collaborations on materials research with:
  - LBNL (synchrotron X-ray spectroscopy methods)
  - Coe College (glass synthesis methods)
  - ANL (XRD at elevated pressures using synchrotron X-rays)
  - Illinois Institute of Technology (synchrotron X-ray spectroscopy methods)
  - University of Verona, Italy (Raman spectroscopy methods)

Proposed Future Work

FY 2011 & FY 2012

- **Task 1.0:** Complete setup and testing of instrumentations
  - **Milestone M.1:** labs are ready, equipment is installed and tested;
- **Task 2.0:** Continue synthesis and processing of glass materials
  - **Milestone M.2:** materials are fabricated; characteristic glass temperatures are determined
- **Task 3.0:** Micro-structural and Nano-structural Studies using a multi-technique approach
  - **Milestone M.3:** comprehensive understanding of structure and packing density in the fabricated glasses
- **Task 4.0:** Synthesis of Glass-Based Nanocrystalline Composites
  - **Milestone M.4.1:** comprehensive understanding of structure and packing density in the fabricated glasses
  - **Milestone M.4.2:** Determination of microstructural changes produced in glass networks as a result of nucleation and growth of specific nanocrystalline phases.
  - **Milestone M.4.3:** Determination of the local structural environment (XAS) of selected atoms of dopants that contribute to the formation of nanocrystals.
- Project Management

Summary

- **Project Objective:** develop glass-based materials with structural properties that would make them promising candidates for use in H-storage: either as material for glass microspheres or for sponge-type storage.
- **Progress (i):** In the first 13 months of project (Jan 2010 – Feb 2011) focus has been on laboratory space remodel, equipment purchase, installation and testing, hiring of personnel and literature studies. Task 1.0
- **Progress (i):** Two Laboratories were established "from scratch": the **Materials Synthesis Lab.** combined with **Materials at Extreme Environments Lab.** as well as **the Laser Spectroscopy Lab.**
- **Progress (i):** Materials synthesis and characterization in Task 2.0, Task 3.0 and Task 4.0 has been started.