DEVELOPMENT OF DOPED NANOPOROUS CARBONS FOR HYDROGEN STORAGE

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OBJECTIVES

The objective of the project is to understand the active adsorption sites in carbon materials that have been activated with nanocatalysts, and use this knowledge to enhance synergistic effects that create new adsorption sites and activate the carbon nanomaterials for adsorption in the DOE target temperature and pressure range.

To understand, identify, and optimize specific adsorption sites, *in situ* high-pressure analytical techniques will be used to fully characterize these sites at the pressures of interest. We will combine multi-wavelength resonance Raman, infrared spectroscopy (IR), X-ray diffraction, and temperature programmed desorption (TPD) techniques with 100 bar measurements of overall adsorption uptake and energetics. Delineation of surface sites by factors such as their hybridization state, potential to (reversibly) rehybridize upon application of pressure, attached chemical functional groups, local bonding environment, and the nature of their binding to hydrogen, combined with adsorption measurements will lead to site specific structure composition relationships and optimization of material design based on this site specific knowledge.

ACCOMPLISHMENTS TO DATE

To understand, identify, and optimize specific adsorption sites, *in situ* high-pressure analytical techniques are being used in parallel with hydrogen adsorption measurements. We have developed high-pressure *in situ* Raman techniques, including pretreatment and transfer in a controlled atmosphere and a differential volumetric high-pressure adsorption measurements. Currently, we are using single-wall carbon nanotubes doped with 1% platinum (1%Pt/SWCNT) as well as multi-wall carbon nanotubes with residual NiMgO (NiMgO/MWNT) catalyst as probe doped metal-carbon materials in *in situ* Raman and volumetric studies. During the synthesis stage of the process, we have synthesized exfoliated graphite nanofibers and explored metal intercalation directly into the graphite lattice.

The ultimate goal is to extend these measurements to 100 bar to delineate surface sites by factors such as their hybridization state, potential to (reversibly) rehybridize upon application of pressure, attached chemical functional groups, local bonding environment, and the nature of their binding to hydrogen, combined with adsorption measurements will lead to site specific structure composition relationships and optimization of material design based on this site specific knowledge.

FUTURE WORK

Now that the high-pressure volumetric system is complete, we are continuing calibration as the measurement temperature is varied. Once complete, we can move on fully to characterizing all the materials synthesized to date on this equipment. This includes our exfoliated graphite nanofibers and metal-interclated nanofibers, as well as 1% Pt/SWNT and NiMgO/MWNT. We are starting the high-pressure *in situ* Raman with SWCNT, as we feel the somewhat metastable and flexible structure shows the most potential to rehybridize upon hydrogen exposure. We plan to extend previous work that has shown re-hybridization of SWNT after exposure to 500 C hydrogen. We plan to repeat this work *in situ* at lower temperatures, and in the presence of a catalyst. We are able to utilize ultraviolet excitation in order to avoid photoluminescence effects of the previous report. We also plan to repeat previous work which has shown unique carbon-hydrogen interactions after MWNT, synthesized with a NiMgO catalyst, was exposed to hydrogen.[1] Our newly developed methods will allow us to repeat these experiments *in situ*, conducting both high-pressure Raman and high-pressure hydrogen adsorption experiments.

Ultimately, the research plan will incorporate a feedback look to optimize the hydrogen uptake of the catalyzed carbon material by incorporating fundamental knowledge gained by the *in situ* high pressure measurements, and knowledge about site-specific structure composition relationships into synthesis of new materials.

LIST OF PAPER PUBLISHED, U.S. PATENT/PATENT APPLICATION(S), CONFERENCE PRESENTATIONS, AWARDS RECEIVED AS A RESULT OF SUPPORTED RESEARCH

Jain, P.; Fonseca, D.A. Schaible, E.; **Lueking, A.D.** "Hydrogen Uptake of Platinum Doped Graphite Nanofibers and Stochastic Analysis of Hydrogen Spillover," *J. Phys. Chem. C* **111,** 1788-1800, (2007).

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Fonseca, D.A.; Gutierrez, H.R.; **Lueking, A.D.** "Characterization of Induced Lattice Defects in Exfoliated Graphite Nanofibers," *In preparation* 2007, 16 pages (current).

Fonseca, D.A.; Gutierrez, H.R.; **Lueking, A.D.** "Graphite nanofibers doped with Magnesium and Boron," *In Preparation* 2007, 14 pages (current).

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