

# 2008 DOE Hydrogen Program Review

## Electron-Charged Carbon-Based Hydrogen Storage Material

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Project ID  
STP28

# Overview

## Timeline

- Project start: July 1, 2005
- Project end: June 30, 2009
- Percent complete: 15%

## Budget

- Total project funding
  - DOE share: \$1,562K
  - Contractor share: \$390K
- Funding received in FY07: \$294,044
- Funding for FY08: \$255,011

## Barriers

- Cost: use inexpensive graphite
- Weight and volume: use high density graphite, maximizing capacity
- Efficiency: add electron charge to increase storage rate
- Durability: use electron charge to control cycles
- Refueling Time: use electron charge to increase fueling rate
- Codes and Standards
- System Life-Cycle Assessments

## Partners

**Superior Graphite Co.**  
**Chicago, Illinois**

# Objectives

Overall	Development of a hydrogen storage material and device for hydrogen quick charge and discharge, high wt% and vol% storage capacities, good durability over many cycles, and safe handling and transport.
2007	<ul style="list-style-type: none"><li>• Select and synthesize carbon-based materials</li><li>• Test and evaluate cycles for hydrogen storage</li><li>• Test external electron charge effect on hydrogen storage capacities</li></ul>
2008	<ul style="list-style-type: none"><li>• Combine internal electron-charge (doping) and external charge to increase hydrogen storage capacities</li><li>• Investigate performance optimization and prototype container systems</li></ul>

# Milestone or Go/No-Go Decision

Month/Year	Milestone or Go/No-Go Decision
July 30, 2008	<p>1) A Go/No-go decision will be made by DOE between 7/31/08 and 9/30/08 to determine if this project will continue into Phase 2. The results relating to the first Go/No-Go decision will be in the Quarterly Report due on 7/30/08.</p> <p>2) The first Go/No-Go decision point in the Statement of Objectives is essentially based on the performance of the electron charge device. A 50-100% increase is expected in the hydrogen absorption of carbon based materials at room temperature as a direct result of the electron charge device. Thus, an increase from 0.5wt% to 0.75/1.0wt% is expected. If the base material performs better at say 2wt%, then the electron charge device is expected to increase the capacity to 3.0wt%. In addition, a large sample size should be used if possible (~1gram) to ensure that the increase in capacity is not in the noise of the measurement.</p>

# Milestone or Go/No-Go Decision

<b>Month/Year</b>	<b>Milestone or Go/No-Go Decision</b>
July 30, 2009	<p>3) The second Go/No-Go decision point is to achieve 6wt% using charge control agent (CCA) at 77K or room temperature, but the key is that it would be using a carbon-based material.</p> <p>4) Additional information regarding the electron device and the materials properties of the carbon based material (pore size, density, surface area, etc) that are best suited for this technique should be investigated and reported.</p>

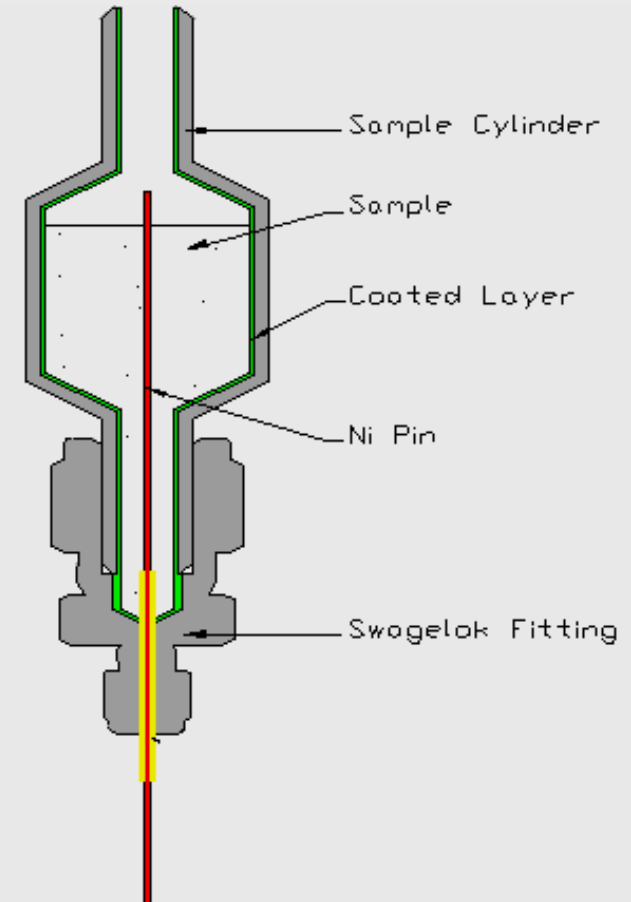
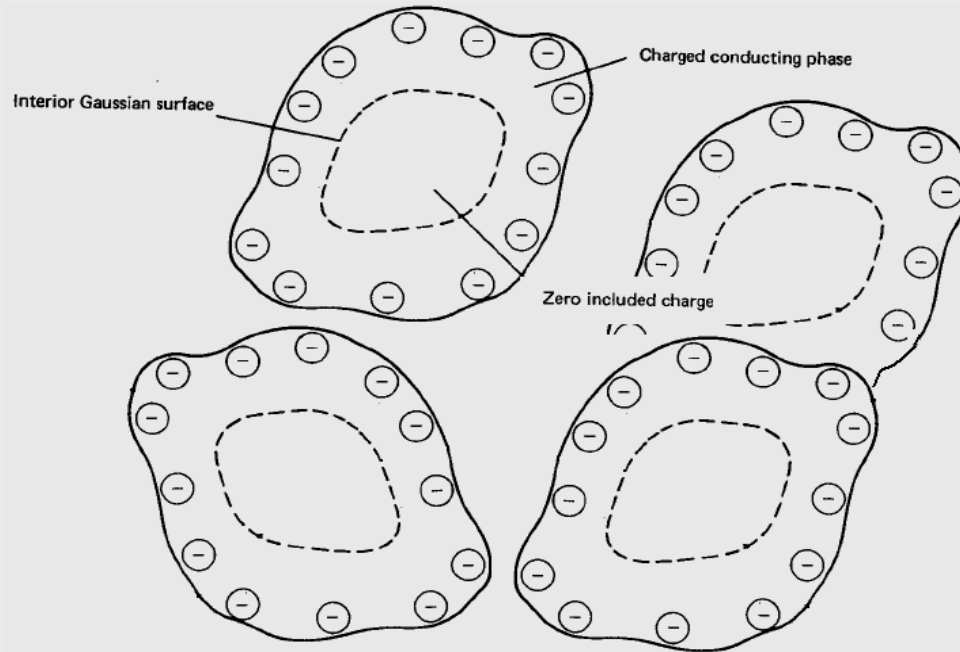
# Approach

- Hydrogen storage is based on hydrogen adsorption, which is involved in electron shift (physi-sorption) and electron transfer (chemi-sorption).
- Use external electron charge to increase hydrogen adsorption and alter hydrogen desorption kinetics.
- Use internal electron-rich or -poor material to change carbon-based material surface electron density, which affects hydrogen storage.

# Plan and Approach

- Select and synthesize carbon-based materials
- Test and evaluate cycles for hydrogen storage
- Test external electron charge effect on hydrogen storage
- Dynamic TGA and Sievert Tests

# Approaches





# Technical Accomplishments/ Progress/Results

- > Hydrogen storage improved about 31% at room temperature by using charge control agent (CCA)
- > Obtained 0.8 Wt% hydrogen Storage using carbon based material at ambient hydrogen pressure. The desorption temperature was approximately 70°C.
- > Cooperated with universities and industries to produce various high surface area carbons with different precursors.

# Technical Accomplishments/ Progress/Results

## Internal Electron Charge

>Electron charge polymer

Characteristic of the Charge Control Agent (CCA)\*

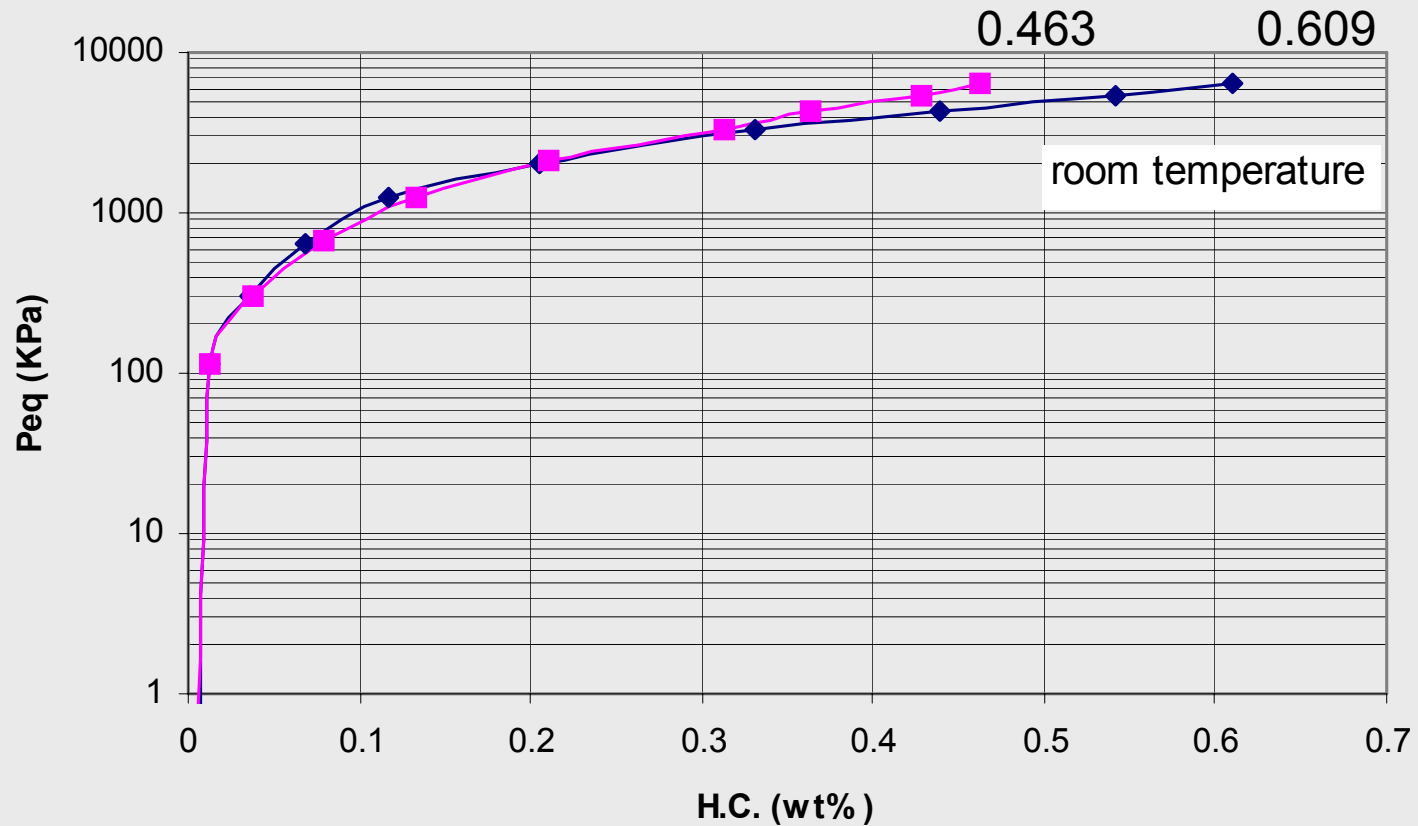
Item	Results
Appearance	Black powder
Charge measurement for 60 seconds (- $\mu\text{C}/\text{g}$ )	40.0
Average particle size ( $\mu\text{m}$ )	1.58
Apparent specific gravity ( $\text{g}/\text{cm}^3$ )	0.295

\*Obtained from a Japanese company

>Electron-rich or -poor materials

doping high surface area carbon with metals or other electron-rich/-poor elements

# Accomplishments/Progress/Results



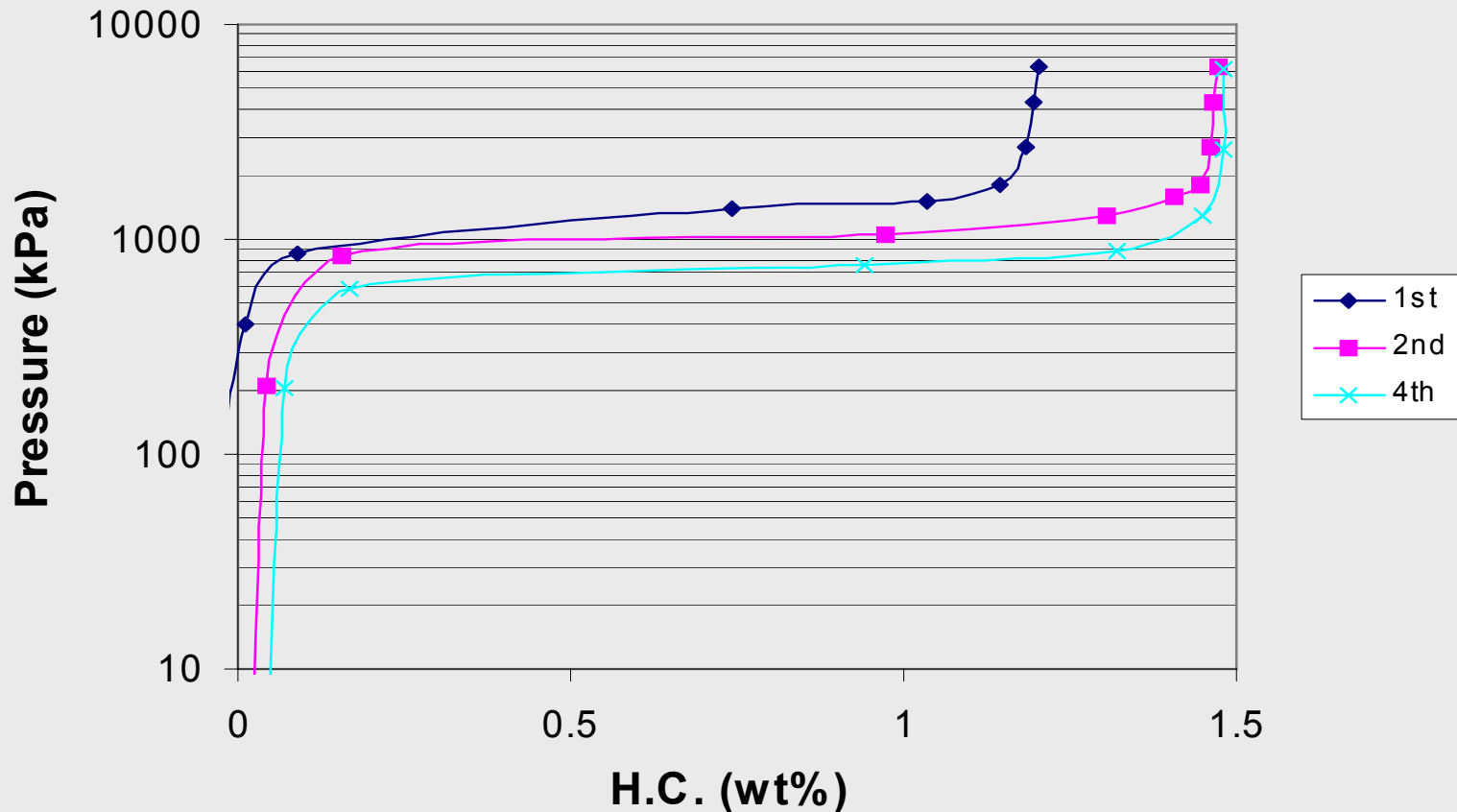
◆ APKI6S7-CCA Charge Agent      ■ APKI6S7 No Charge

CCA on carbon: the hydrogen storage rate increased from 0.463 wt.% to 0.609 wt.%, i.e. a 31% increase.



# Internal Electron Charge Effect

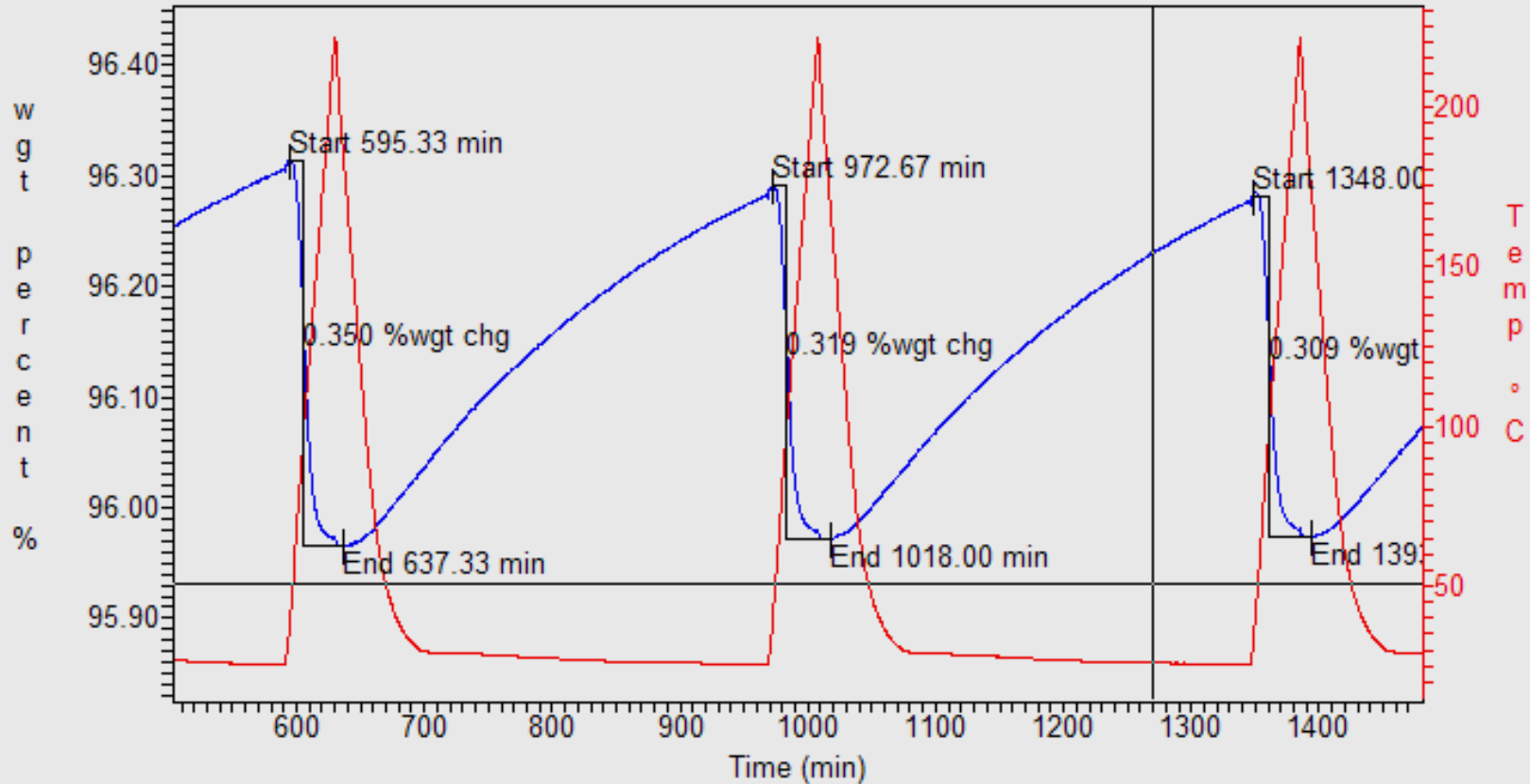
The hydrogen storage improvement under cycles at room temperature



CCA on metal based material: the hydrogen storage capacities improved from 1.185 wt.% to 1.476 wt.% (approximately 25% increase)

# GTI Synthesized Carbon-based Material

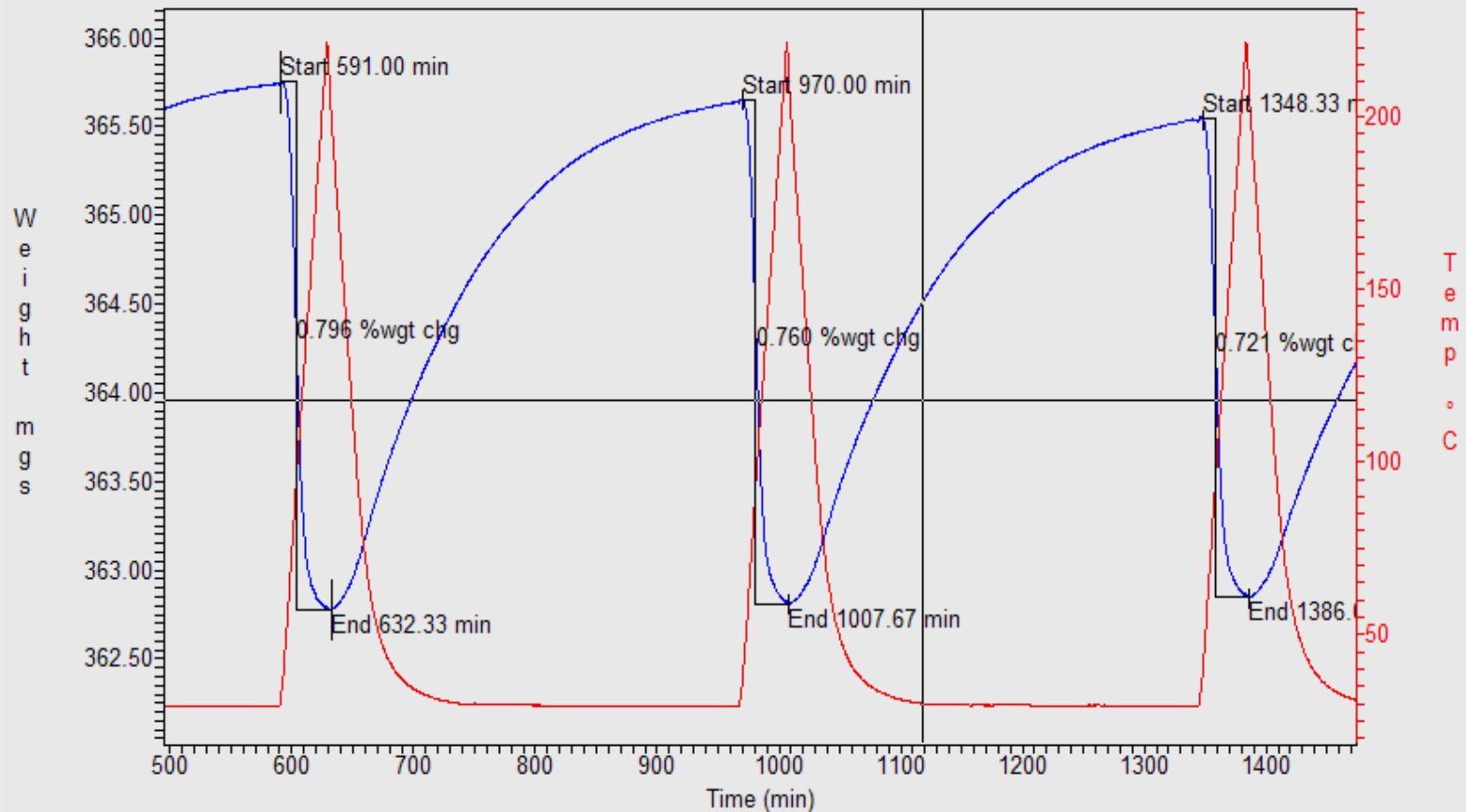
without Electron-rich Material Doping



**Dynamic TGA of hydrogen adsorption/desorption on GTI carbon-based material at ambient pressure**

# GTI Synthesized Carbon-based Material

with non-Metal Electron-rich Material Doping



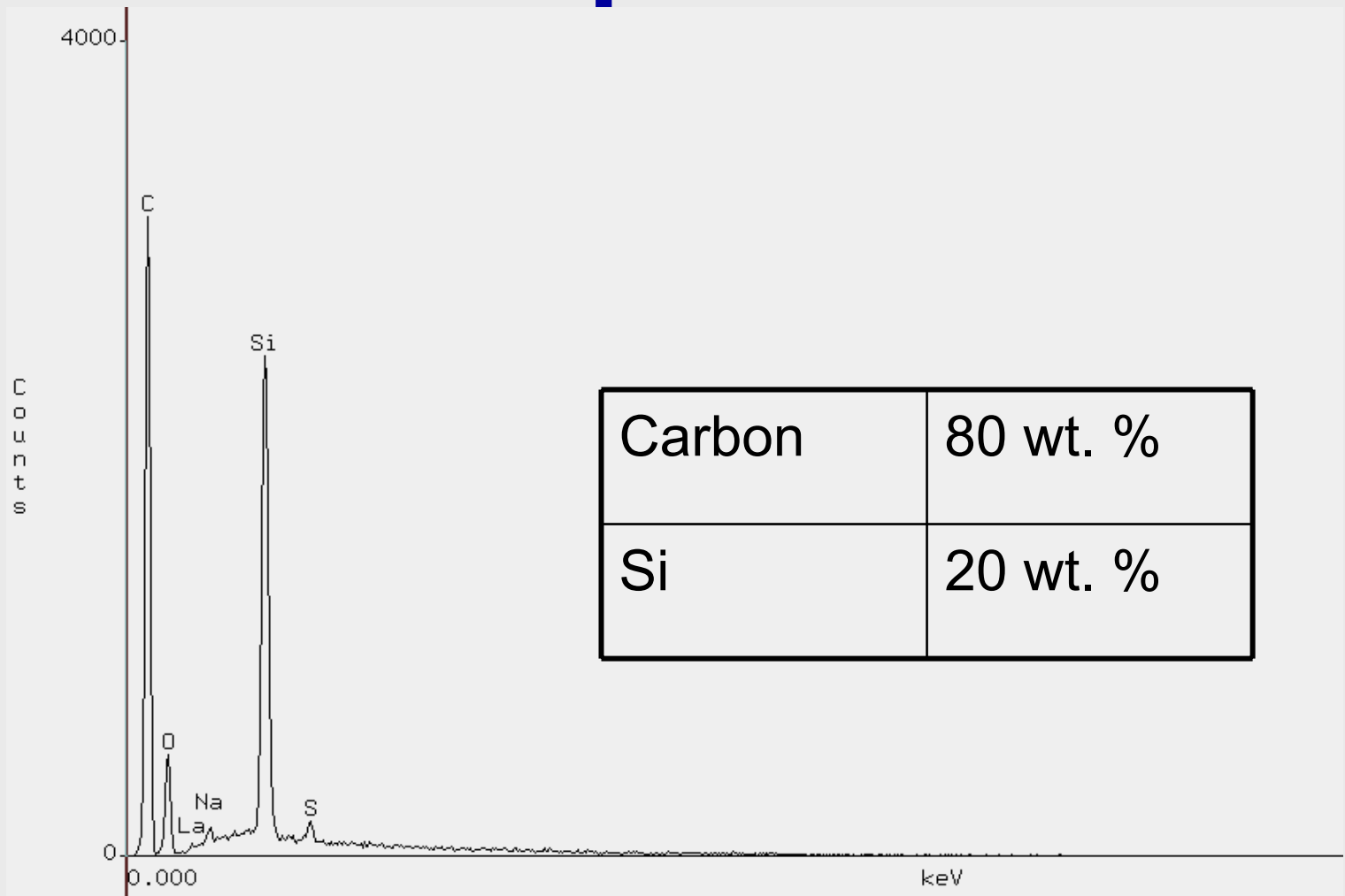
***Non-metal electron-rich doping doubled the hydrogen storage capacity from 0.35 wt% to 0.796 wt%***

# Pyrolysis Temperature Effect on Hydrogen Storage

Pyrolysis temperature (°C)	Hydrogen storage at ambient pressure (wt%)
350	0.350
400	0.328
600	0.223

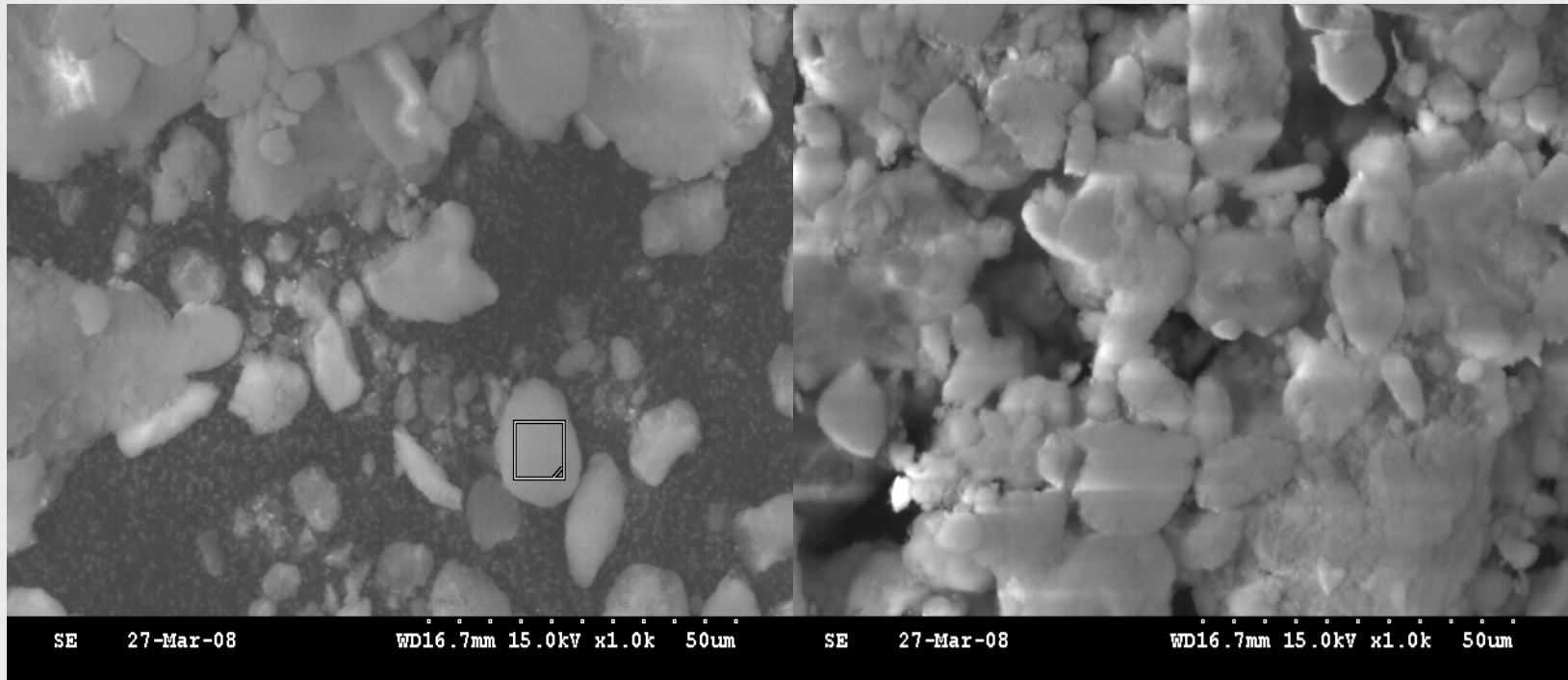
**Pyrolysis Temperature affects the powder surface area and porosity**

# EDX Analysis: Carbon Based Material Composition





# SEM Analysis Reveals High Surface Uniformity

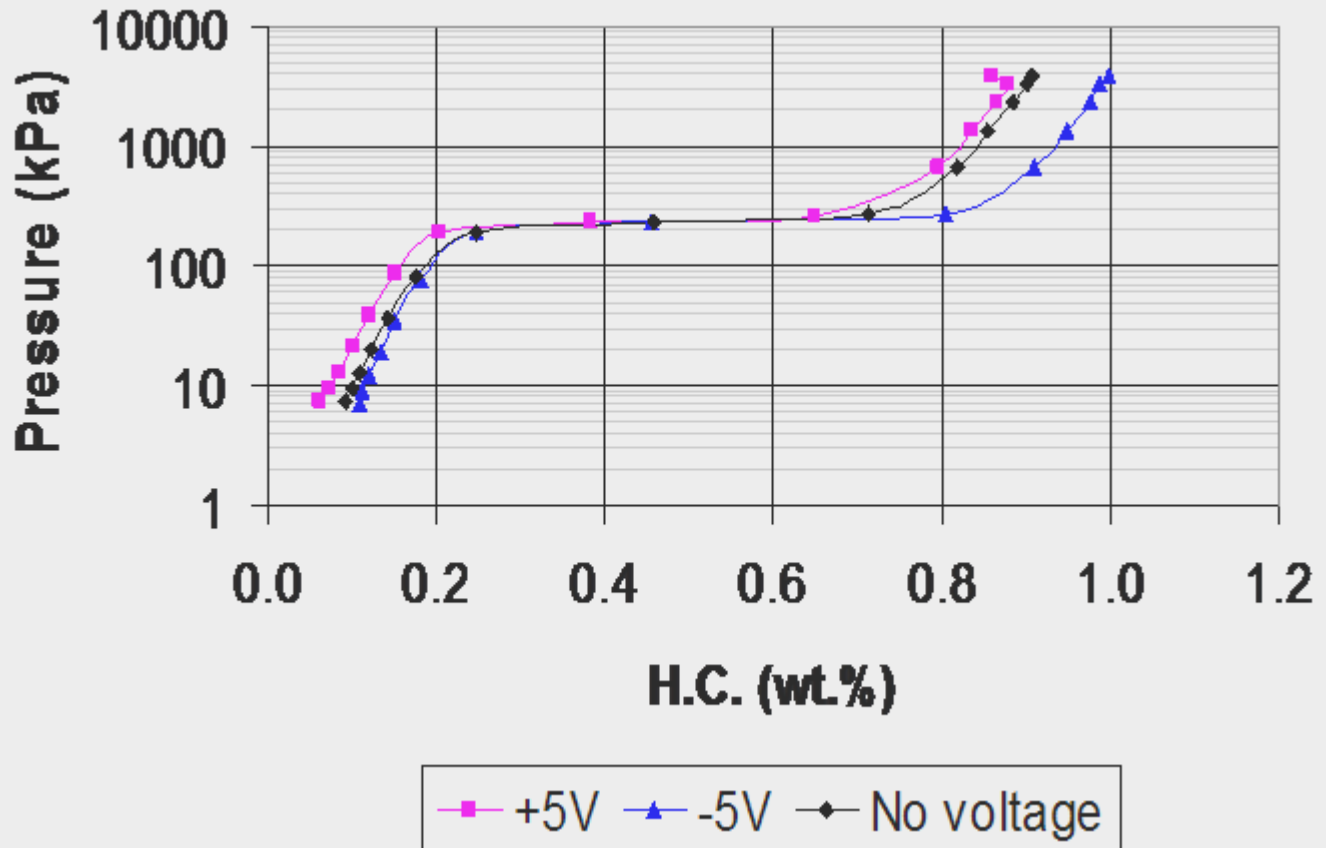


Before hydrogen storage test

After hydrogen storage test

# External Charge Effect

Room temperature



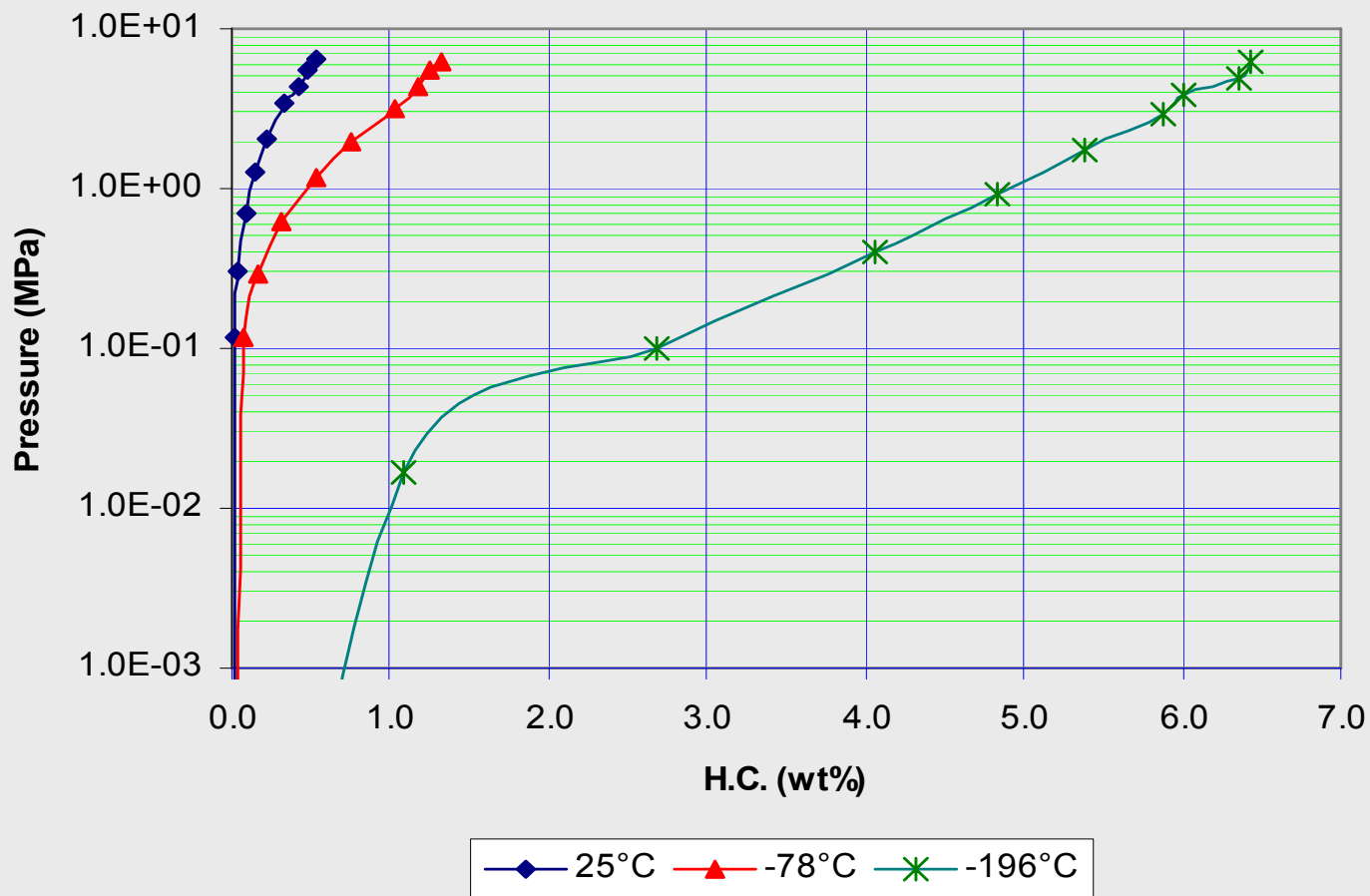
Could not perform 77 K experiment because the insulation on the electrode could not tolerate the low temperature. An electrical short was found in the system.

# Cooperation

- State University of New York at Syracuse:  
Prof. Cabasso group
- Japanese CCA Manufacturer
- ATMI
- University of Houston

# High Surface Area Carbon from SUNY at Syracuse

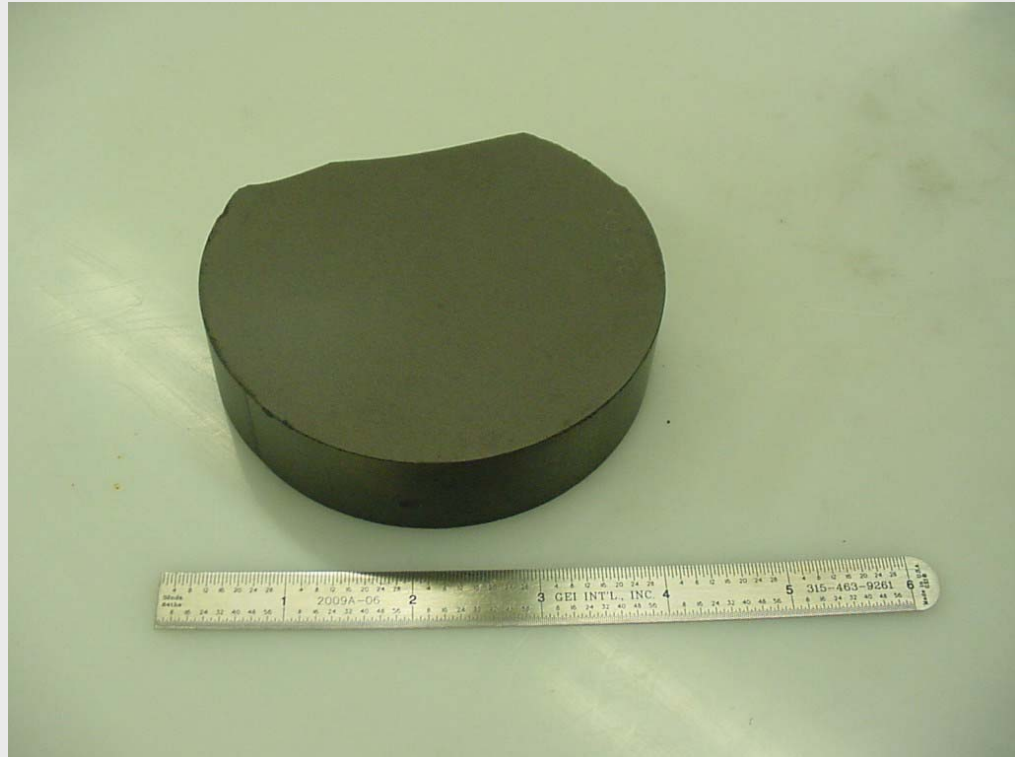
APO-R10



# Summary of Four SUNY Samples' Hydrogen Storage Capabilities at 60 Bars

	APKI6S7	APKI6S11	MK725	APO-R10
25°C	0.5 wt%	0.4 wt%	0.8 wt%	0.5 wt%
-78°C	1.3 wt%	1 wt%	1.65 wt%	1.3 wt%
-196°C	6.3 wt%	5.2 wt%	5.8 wt%	6.4 wt%

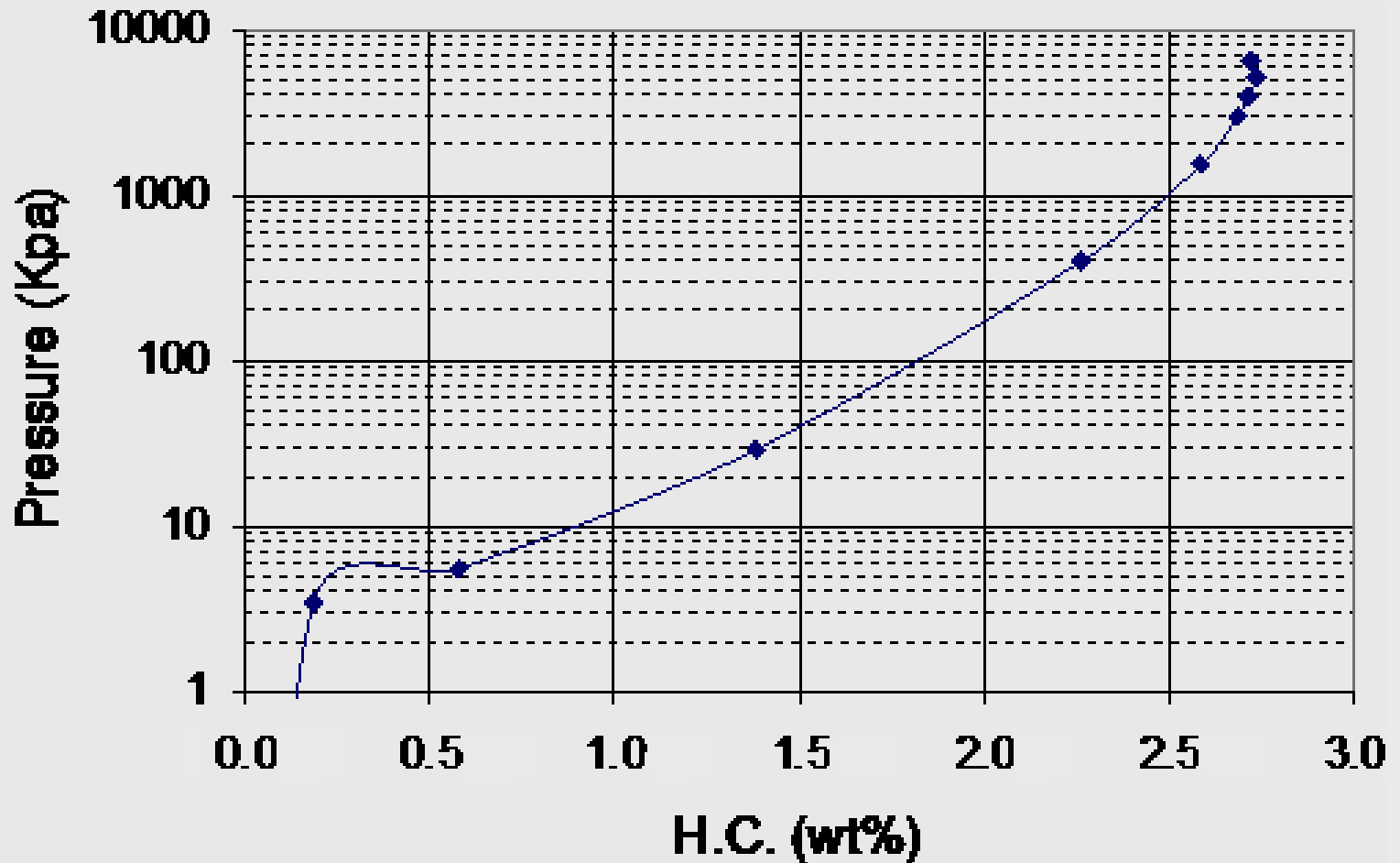
# High Surface Area Carbon Material from a Partner



Density 1.12g/cc. Pore size 0.5 to 0.8 nm. Surface area 1500 m<sup>2</sup>/cc.

# PCI Curve of H<sub>2</sub> Storage on a High Surface Area Carbon from a Partner

at 77K



# Future Work

- Test and evaluate cycles for hydrogen storage
  1. Modify testing apparatus
  2. Test the expanded and intercalated carbon based materials
  3. Investigate the electron charge effect on different hydrogen storage substrates.
- Investigate performance optimization and prototype container system
- Prepare samples for independent evaluation



# Summary

- Internal Electron Charge increases the hydrogen storage capacity by 31%
- Obtained 0.8 wt% hydrogen storage using a carbon based material at ambient hydrogen pressure
- Demonstrated that the external electron charge affects hydrogen storage. The PCT curves of the graphite-based materials show that the positive charge reduces the hydrogen storage, whereas negative charge increases the hydrogen storage
- The PCT shift is related to the electronic structure of the substrate. Electron-rich material needs positive charges and electron-poor material needs negative charges
- Synthesized high surface carbon-based material and found that non-metal electron-rich material doping doubled the hydrogen storage capacity