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HT Combinatorial Screening of Novel Materials for High Capacity Hydrogen Storage

Ali T-Raissi

Director, Hydrogen & Fuel Cell R&D Division

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This presentation does not contain any proprietary or confidential information

Objectives

- Develop (*i.e.* design, build, test and verify) a high throughput screening device based on FSEC-developed H_2 sensing materials applicable to a broad range of adsorbents, including MOFs, various doped & undoped hydrides so that methods for increasing H_2 adsorption energies beyond 5 kJ/mol can be identified.
- Develop a rapid screening method & demonstrate the ability to reproducibly screen **10/100** hydrogen sorbing materials per run.
- Measure the amount of H_2 released for up to **10/100** solid adsorbents, simultaneously, from SA/LN2 to 423 K (150^{o}C) and pressures from 10 torr to 50 bars.

FSEC's Chemochromic H_2 Sensing Materials & Applications

- Industries producing or consuming H_2 (*e.g.* refineries) & others (*e.g.* NASA).

- Hydrogen Economy
  - Transportation
  - Storage
  - Fuel Cells

Accomplishments

At FSEC, we have:

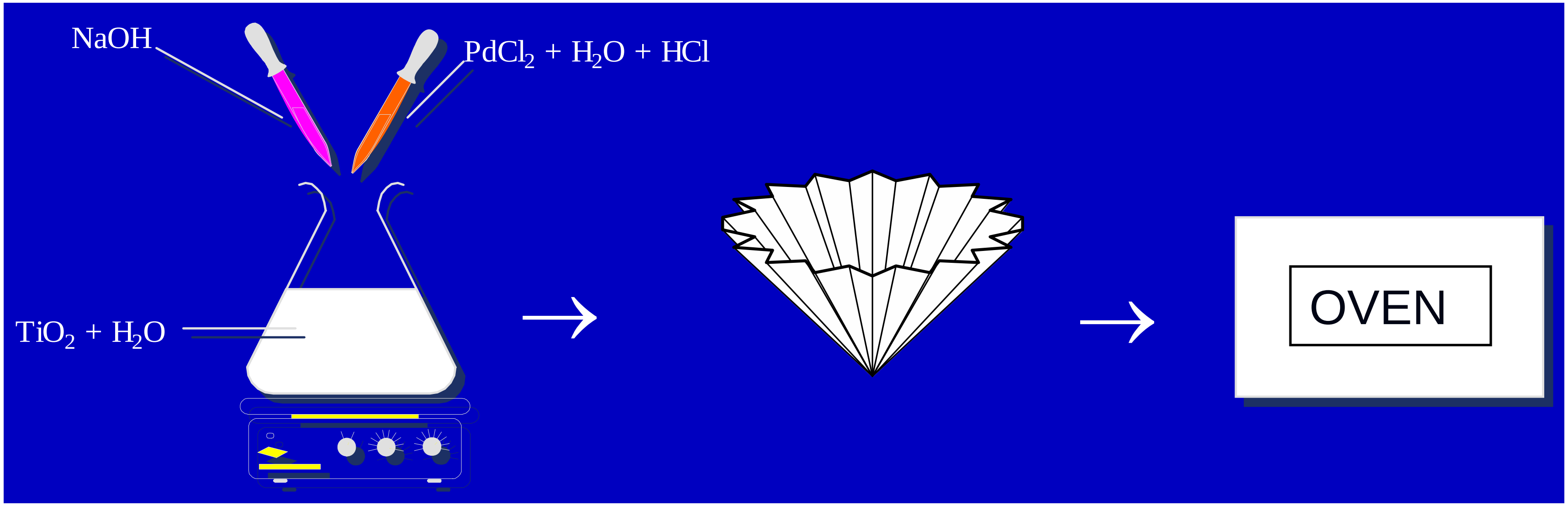
- Formulated, prepared & characterized many novel chemochromic materials for the visual detection of hydrogen gas.
- Developed a technique for the implementation of PdO-based & other “smart pigments”.

  *Bokerman, G., et al. “Gas Permeable Chemochromic Composition for Hydrogen Sensing,” U.S. Patent and Trademark Office Serial No. 11/414,900, May **2006**.*
- Synthesized novel chemochromic materials based on POM complexes and others.

PdO Based Pigments

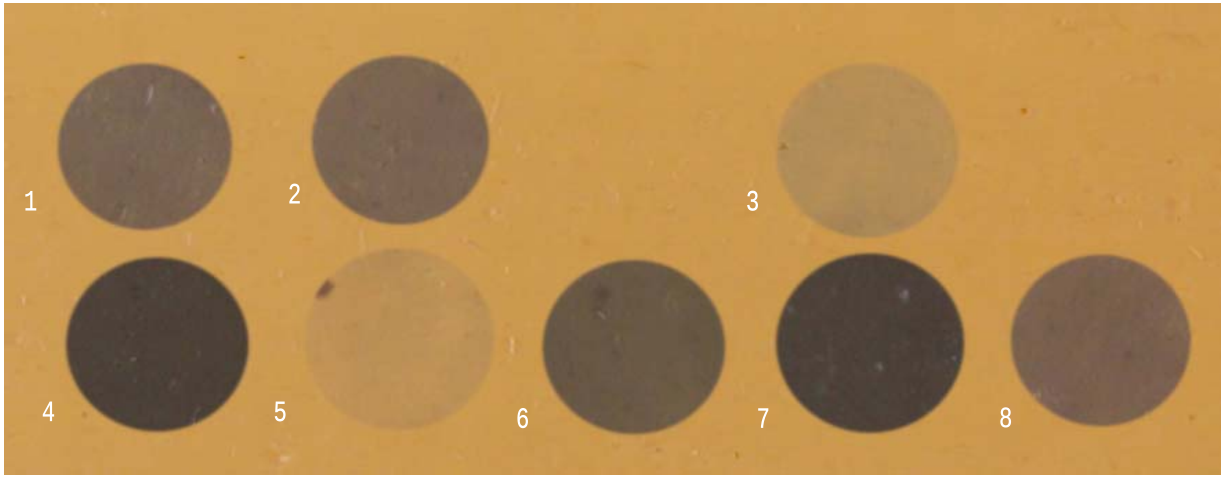
- Change color at temperatures as low as -40°C
- Employ gas permeable matrices for the pigment encapsulation that make them selective toward hydrogen detection
- Has been fielded by NASA.

Synthesis of TiO_2/PdO Based Pigments



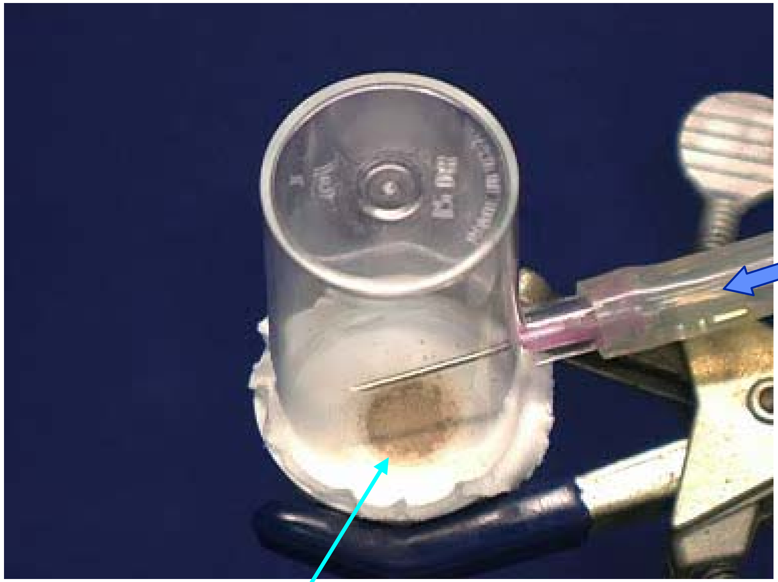
TiO_2 Sample	Degussa P-25	Aldrich	Fisher	DuPont R103
Particle size (μm)	0.02-0.04	0.5	0.1-0.5	0.05-0.2

Implementation of TiO_2/PdO Based Pigments



A 25-mil thick section of chemochromic film after exposure to various quantities of hydrogen gas.

PdO on Degussa P-25 TiO_2



H_2 FLOW

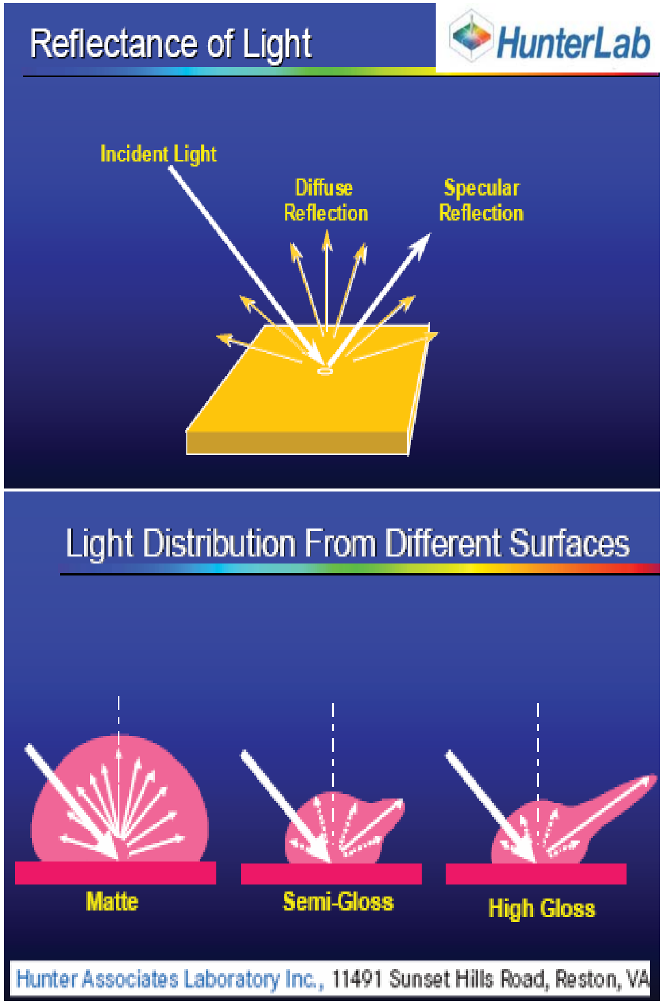
CHEMOCHROMIC MARKER

Quantifying Color Change

Parameters affecting the appearance of color are:

- *Basic pigment formulation*
- *Surface characteristics (gloss, texture, pattern, etc.)*

0/45- or 45/0-degree instruments are most often used as they avoid the specular component, like a person does when examining the appearance of color.



Quantifying Color Change (cont'd)

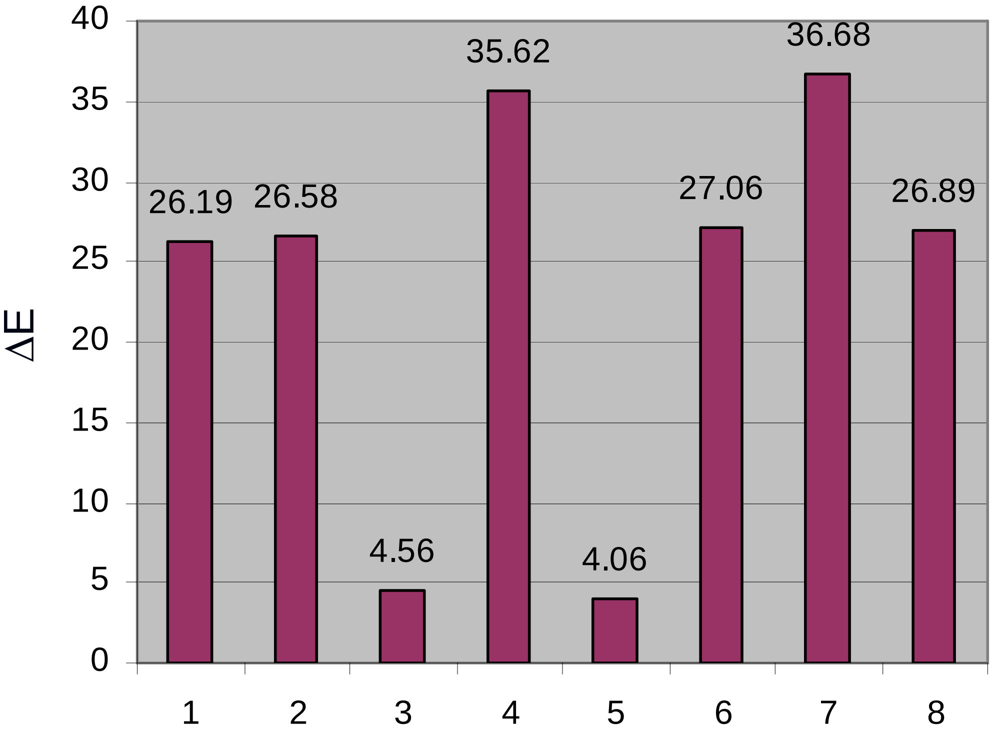
- Using ColorTec-PCM colorimeter

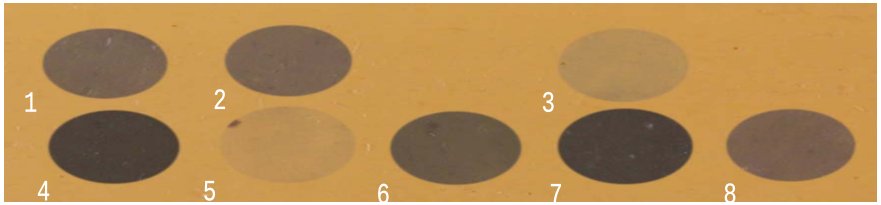
$$\Delta \boldsymbol{E}^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{½}$$

L*- Lightness Value
a*- position on red-green axis
b*- position on yellow-blue axis

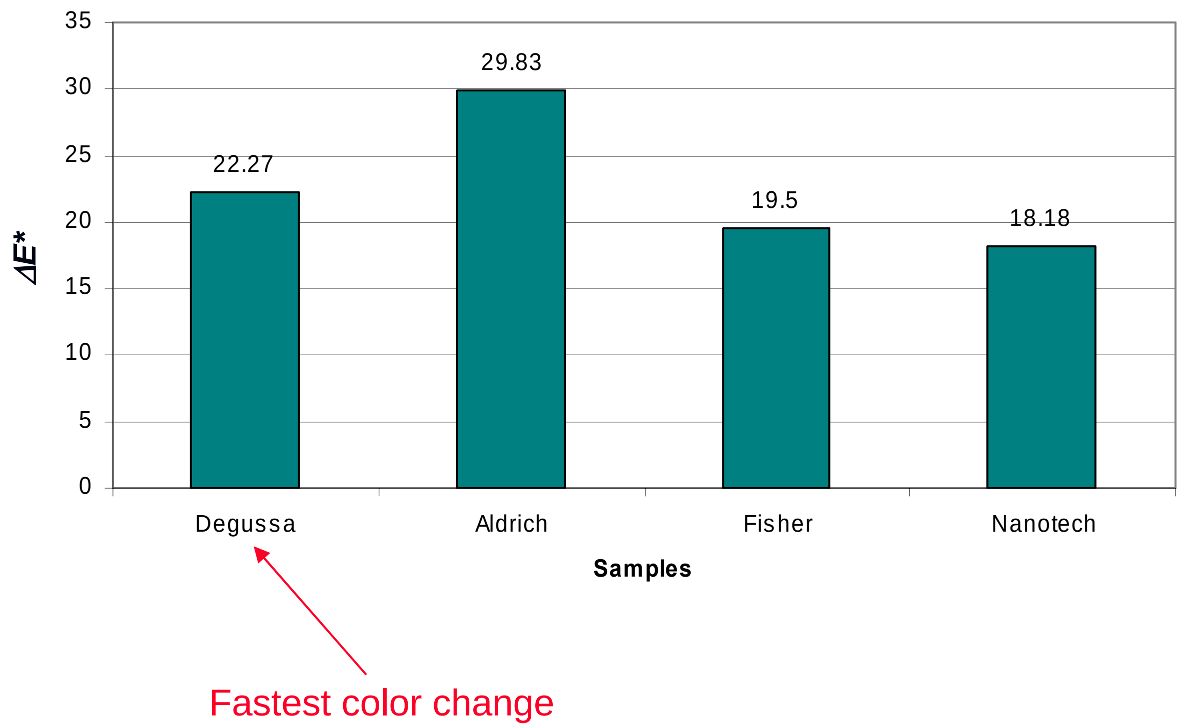
- Greater the $\Delta \boldsymbol{E}^*$ value, higher the color contrast

Quantifying Color Change (cont'd)



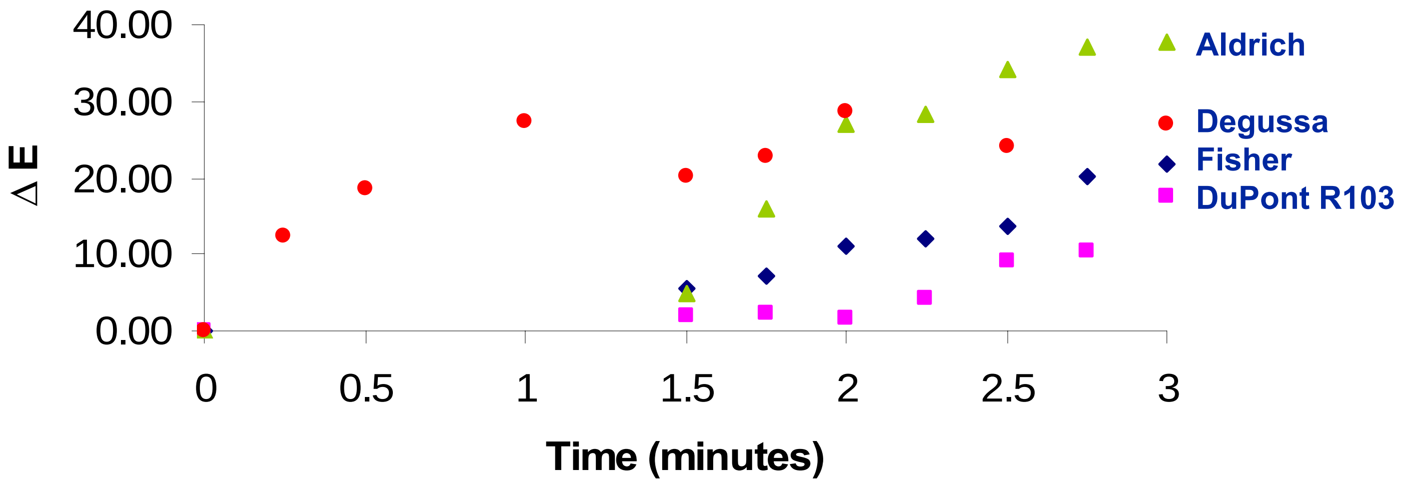


Quantifying Color Change (cont'd)



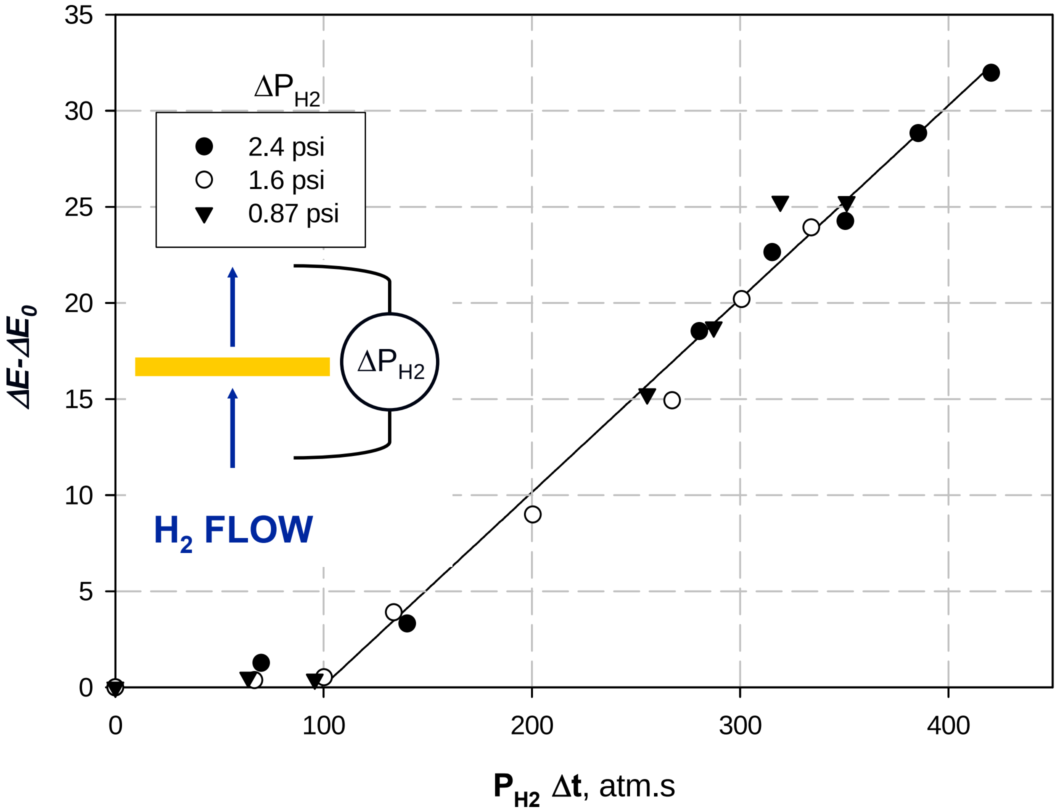
Captain, J.E., et al. "Chemochromic Hydrogen Detection," *Proceedings of SPIE Conference*, 17–21 April **2006**, Orlando, Florida.

ΔE vs. H_2 Exposure Time for Various TiO_2 Supports



Mohajeri, et al., “Effect of PdO on TiO_2 Loading on Chemochromic Detection of Hydrogen,” submitted for publication in the Int. J. Hydrogen Energy.

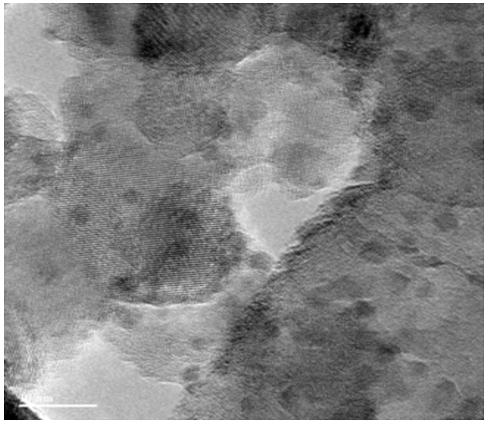
Color Contrast vs. Total H_2 Reacted within Membrane

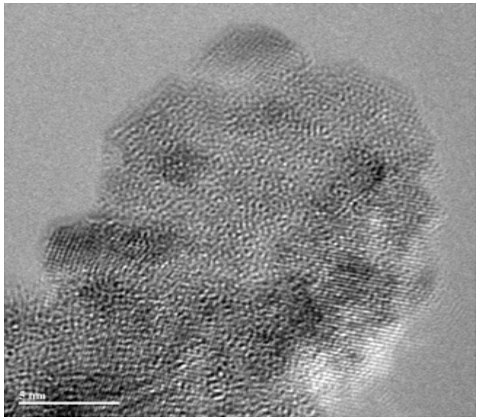


Data courtesy of NASA-KSC & Arctic Slope Research Corp.

TEM of PdO on Degussa P-25 TiO_2

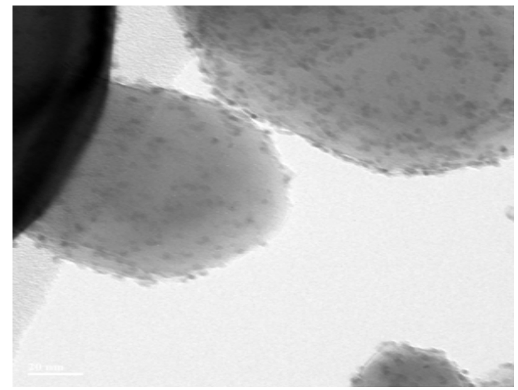
- Well dispersed PdO particles
- Strongly attached to TiO_2 support
- Homogenous
- Particle size ~2.5 nm

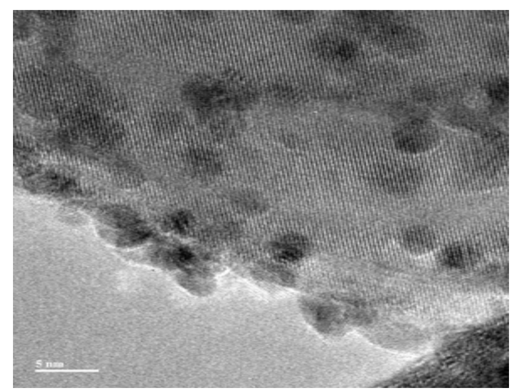




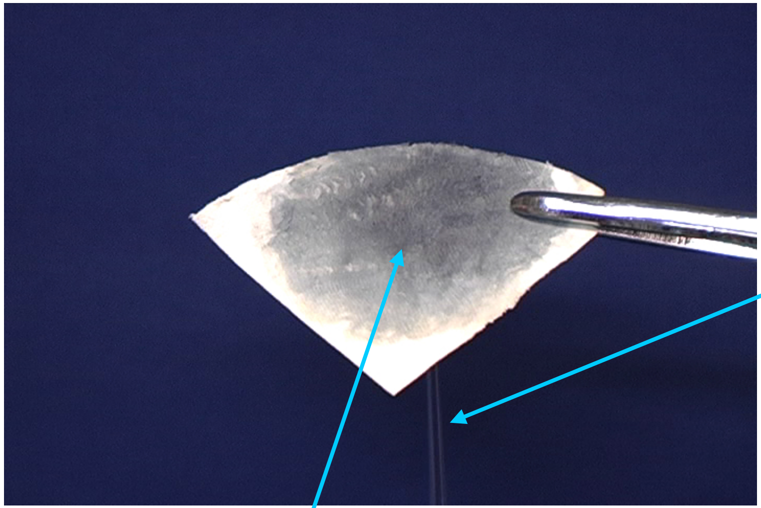
TEM of PdO on DuPont R103 TiO_2

- Well dispersed spherical PdO particles
- Loosely attached to TiO_2 support
- Non-homogenous
- Particle size > 5 nm





FSEC's New & Proprietary H_2 Sensing Pigment

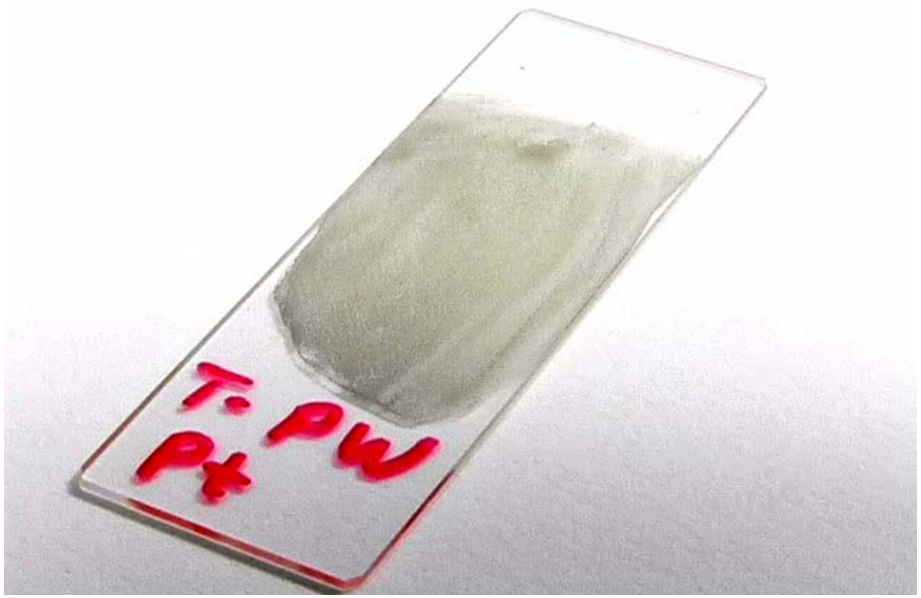


H_2 FLOW

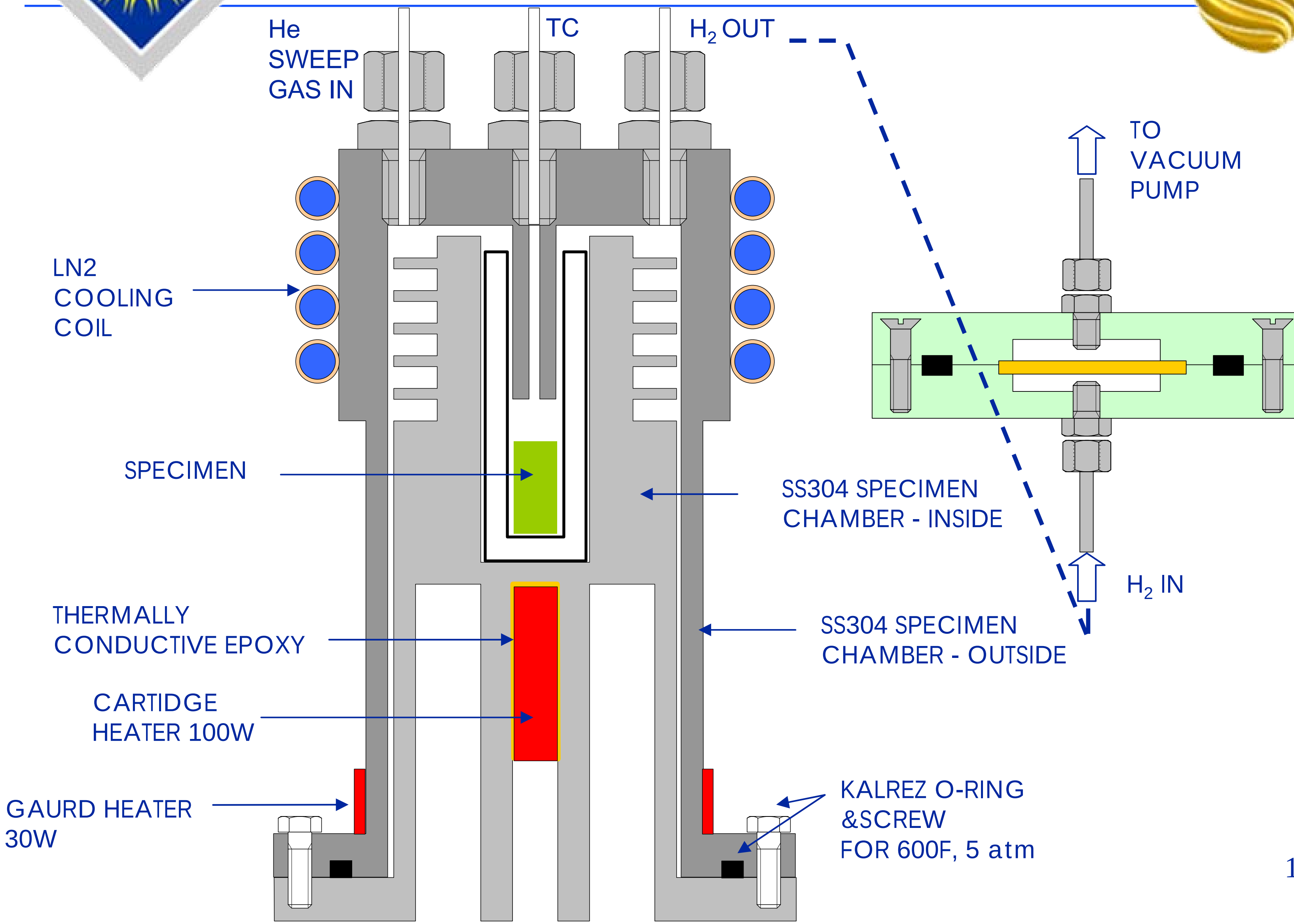
H_2 SENSING PIGMENT & SUPPORT

FSEC's Reversible Pigments

Several formulations developed at FSEC can be used in "repeated coloration/discoloration" applications including HT screening of hydrogen storage materials as the following video shows.



Single Cell Design of FSEC's HT Combinatorial Screening Apparatus



Summary

- A large number of hydrogen sensing pigments suitable for HT H_2 storage materials screening have been synthesized using PdO on TiO_2 supports and other compounds

- The extent of color change has been determined as a function of time and amount of H_2 within the membranes

- PdO on Degussa P-25 with smallest TiO_2 particles shows fastest discoloration and highest color contrast

- A HT combinatorial screening apparatus has been designed for testing up to 100 individual samples, in few minutes, at temperatures & pressures in the range of LN2-150^{o}C and 10-38 ktorrs, respectively .

Acknowledgment

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