Combinatorial Synthesis and High Throughput Screening of Effective Catalysts for Chemical Hydrides

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– A participant in the DOE Center of Excellence for Chemical Hydrogen Storage –

May 23, 2005

DOE 2005 Hydrogen Program Annual Review
Washington, D.C., May 23-26, 2005

This presentation does not contain any proprietary or confidential information

Project ID #
STP12

Overview

Timeline

- Project start date: FY2005
- Project end date: FY2009
- Percent complete: New Start

Budget

- Expected total project funding
  - DOE: $1,100K
  - Intematix: $277K
- Funding for FY2005: $200K

Overview

Barriers

- Cost
- Weight and volume
- Energy efficiency
- System life-cycle assessment
- Spent material removal
- Regeneration processes

Partners

- Collaboration with LANL, PNNL, and UCLA on novel polyhedral boranes
- Future collaboration with Penn, NAU, LANL, PNNL on amine-boranes
- Other collaborations with Center partners based on future discoveries

Objectives

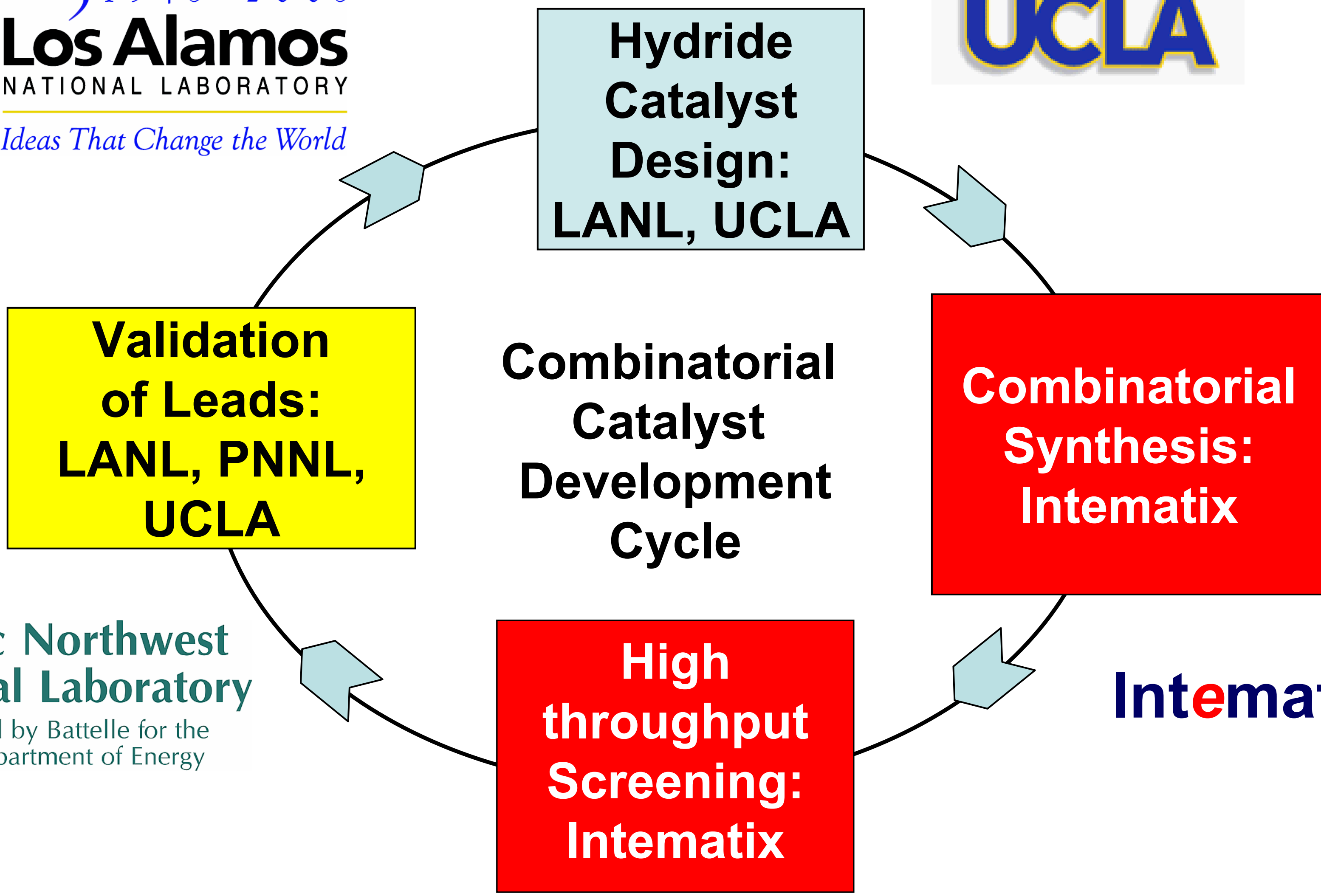
To assist DOE in achieving the DOE/ FreedomCAR target of a hydrogen storage system of 6.0 wt.% by 2010

- Develop and validate high-throughput synthesis and screening methods for new low-cost and effective compound catalysts for chemical hydrogen storage (current FY)
- Explore catalysts which could improve the kinetics of hydrogen release from candidate hydrogen storage materials (next FY and beyond)
- Explore new catalysts and catalytic processes which could significantly enhance regeneration processes (next FY and beyond)

Approach

1 9 4 3 - 2 0 0 3
Los Alamos
NATIONAL LABORATORY
Ideas That Change the World

UCLA



Pacific Northwest National Laboratory
Operated by Battelle for the U.S. Department of Energy

Intematix

Concept of Approach

- **Combinatorial Synthesis**
  - Intematix proprietary combinatorial synthesis technology can generate hundreds of different hydrides/catalysts combinations (thin film or nano-particles) in one experiment under oxygen free environment

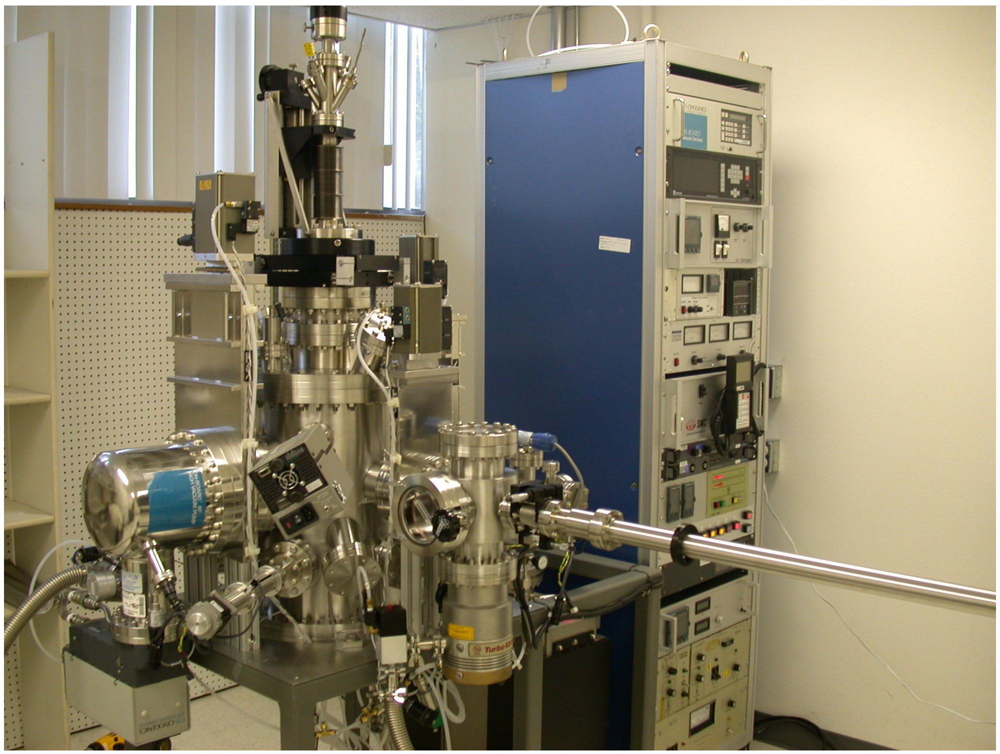
- **High-throughput Screening**
  - Intematix proprietary combinatorial high-throughput screening technology can test promising catalysts under realistic reaction conditions (high pressure/temperature, oxygen free)

Challenging Issues

- Reactant stability and catalyst surface contamination – in situ transfer between synthesis and screening
- High-throughput screening of arrayed catalysts (thin film or nano-particles)
- Defining initial screening parameters (hydrogen concentration, end products, temperature, reaction time, reaction rate, etc.) for the arrayed catalysts

Technical Accomplishments

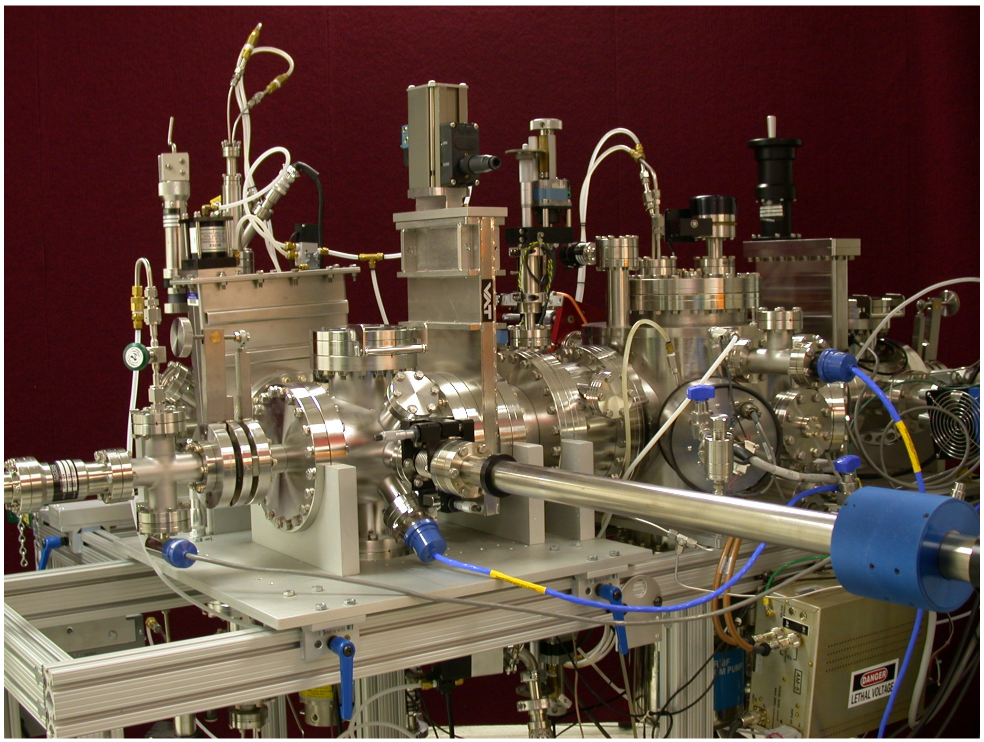
Validated a combinatorial molecular beam epitaxy (MBE) system for thin film deposition of compound catalysts



A high temperature effusion cell (up to 1900ºC) has been installed for catalyst elements (such as Ti) incorporation

Technical Accomplishments

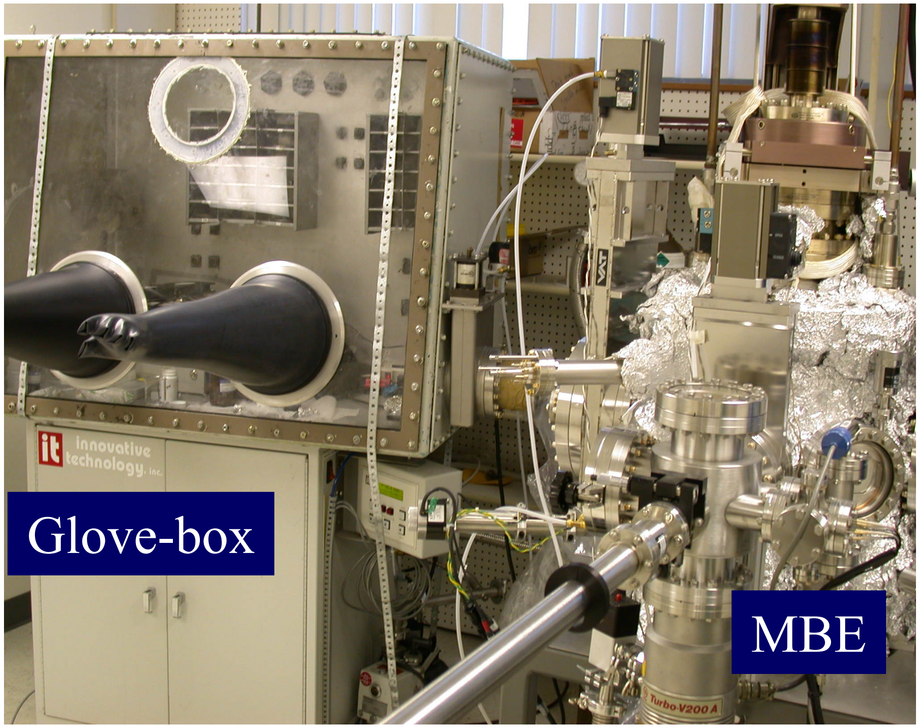
Validated combinatorial ion-beam sputtering (IBS) system for synthesizing catalysts and materials including air-sensitive compounds



A getter pump on the combi-IBS system has been installed to reduce the residual O_2

Technical Accomplishments

Set up an air-tight oxygen-free glove box for *in situ* sample transfer and characterization



The glove box and the MBE growth chamber are directly connected to allow sample transfer in oxygen-free environment.

Technical Accomplishments

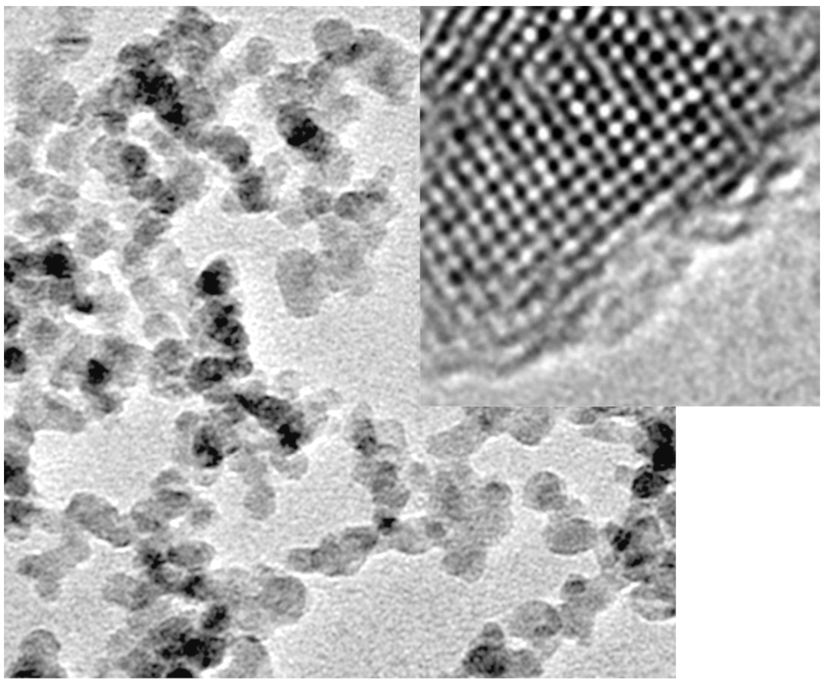
Validating combinatorial nano-particle (CNP) synthesis system – the third proprietary combinatorial materials synthesis technique Intematix has recently developed

The advantages:

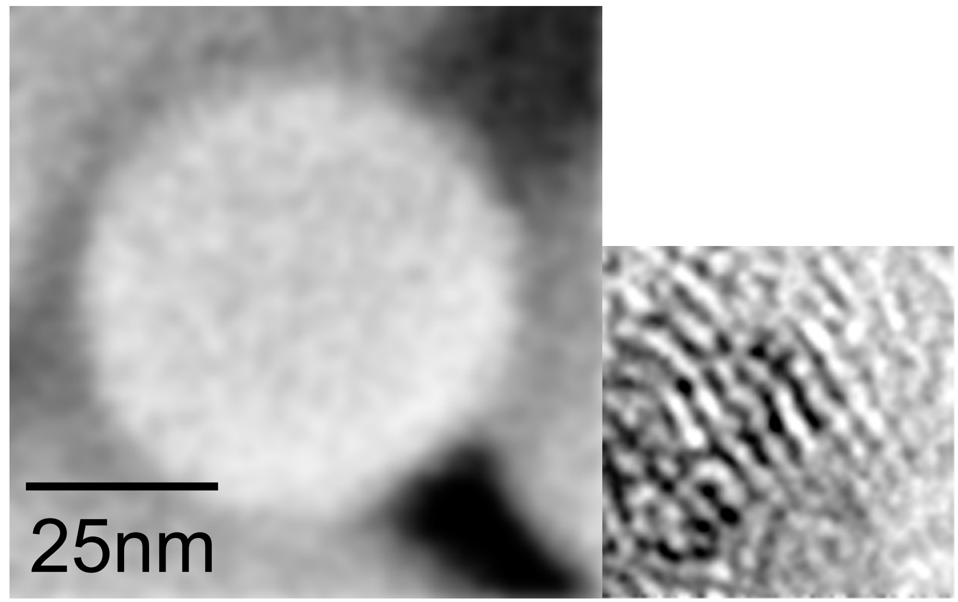
- Small particle size~10-50nm
- Narrow particle-size distribution
- Accessible to most elements
- Can optimize size and composition rapidly

Technical Results

The CNP system is very unique and powerful in high-throughput synthesis of nano-particles



TEM images of TiO_2 nano-particles prepared by CNP under optimized conditions.



YIG nano-particle prepared by CNP under preliminary condition. Particle has well-defined circular shape. The left image shows the crystal structure is not well-ordered.

Technical Progress

Designed and constructed micro-reactor arrays to screen combinatorial catalyst libraries for hydrogen release

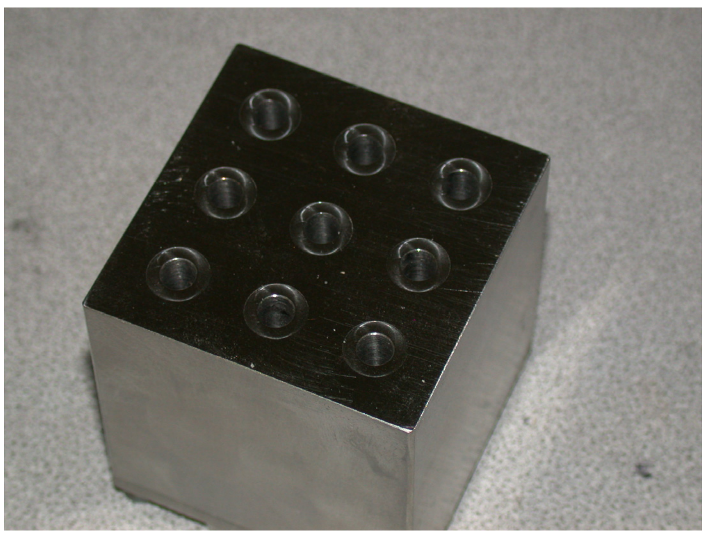
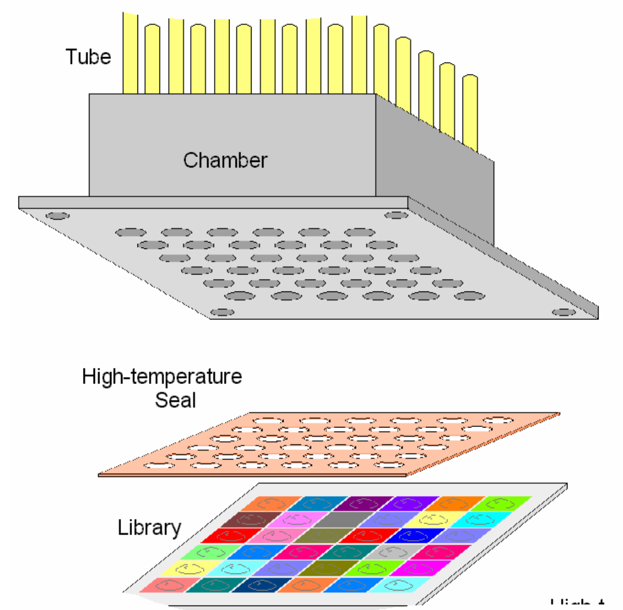


Image of a 3x3 micro-reactor arrays



Addressing Barriers

- Cost – *new non-precious metal alloy or compound catalysts will reduce the total system cost*
- Weight and volume – *new catalysts for high weight efficiency reactions will reduce the system weight and volume*
- Energy efficiency – *new catalysts enable high efficiency chemical hydrogen storage*
- Regeneration processes – *new catalysts may enable low cost regeneration process*

Future Plans

- **Remainder of FY2005**
  - *High-throughput in-situ catalysts screening*
    - Identify key parameters for high throughput *in situ* screening
    - Demonstrate effectiveness of catalyst screening methodology for model reaction:

$$NaBH_4 + 2H_2O \xrightarrow{\text{catalyst}} 4H_2 + NaBO_2$$

  - *Validate the capability of CNP for synthesizing catalyst nanoparticles*

Future Plans

- **FY2006**
  - *Synthesis and screening of catalyst libraries for hydrogen-release in polyhedral boranes, e.g.*

$$Na_2B_{10}H_{10}\bullet 16H_2O \xrightarrow{\text{catalysts}} 2\,NaBO_2 + 4\,B_2O_3 + 21H_2$$

  - *Characterization of material properties of lead catalysts*
    - Crystal structure, grain size, and alloy composition of catalyst materials

Summary of Future Plans