



# **Synthesis of Nanophase Materials for Thermodynamically Tuned Reversible Hydrogen Storage**

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with Metal Hydride Center of Excellence  
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This presentation does not contain any proprietary or confidential information

Project ID # STP21 AHN

# Overview

## Timeline

- Project start date:  
October 1, 2004
- Project end date:  
September 30, 2009

## Budget

- Total project funding
  - DOE share \$1.15M (5 yrs)
  - Contractor share \$287.5k (5 yrs)
- Funding for FY05
  - DOE share \$150k
  - Contractor share \$37.5k

## On board hydrogen storage Barriers and Targets

- (B) Weight and volume of on board hydrogen storage systems
- (M) Reversibility of high capacity solid state storage materials
- (N) Kinetics (fueling/refueling times) associated with current solid state storage materials

## Partners

### Interactions/ collaborations:

Jet Propulsion Laboratory

(Robert C. Bowman, Jr.)

HRL Laboratories

(John Vajo and Greg Olson)

University of Hawaii (Craig Jensen)

Stanford (Bruce Clemens)

Univ. Pittsburgh (J. Karl Johnson)

NIST (Terry Udovic)

CECM-CNRS, Vitry, France

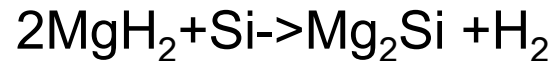
(Yannick Champion)

# Objectives

- Address the role of nanoscale dimensions in the kinetics (hydrogen fueling/refueling rates) of light metal hydride materials.
- To address the problems associated with large, light-metal-hydride enthalpies (hydrogen fueling/refueling temperatures) and develop strategies to address thermodynamic issues surrounding the use of these materials through hydride destabilization.

# Approach

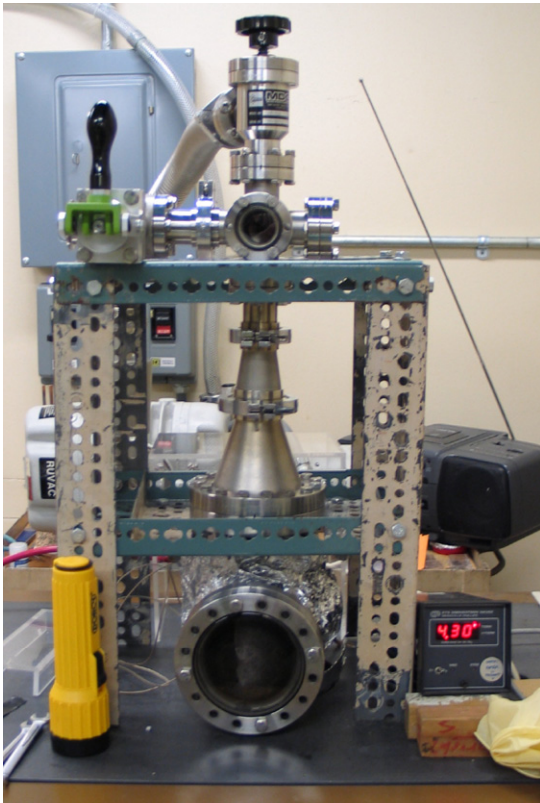
- Two approaches are being employed for nanophase hydride and hydride precursor synthesis
  - A) Gas condensation/consolidation B) cryo-melting (these approaches generate smaller size particles (10-100nm) than mechanical attrition (1 $\mu$  typical). Kinetics are expected to go as at least  $1/d^2$  of particle size so hydrogenation/dehydrogenation rates will be 100 to 10000 x faster.
- Hydrogenation/dehydrogenation analysis using volumetric Sieverts apparatus.
- Initial studies to concentrate on Mg<sub>2</sub>Si which can be dehydrogenated via the reaction



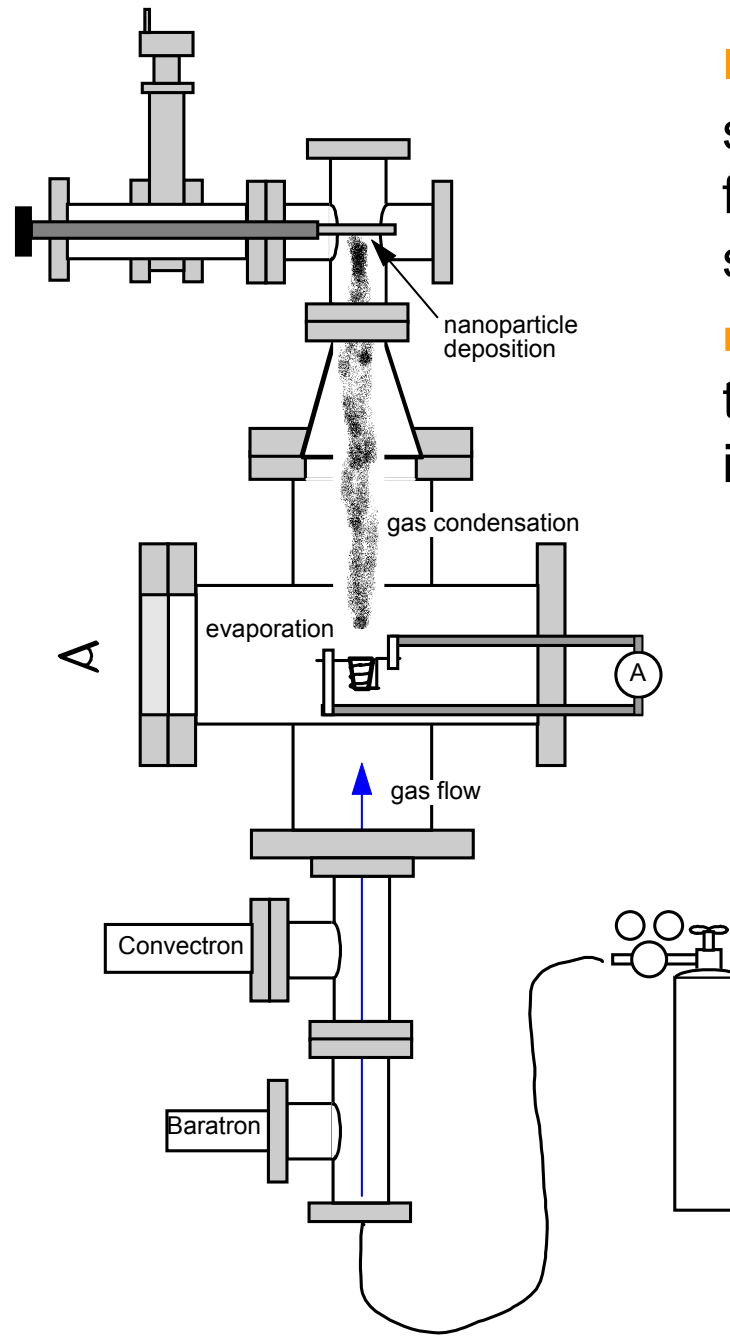
which should be reversible but does not appear to be in initial experimental efforts.

Issues related to solid state diffusion, gas solid interactions, grain growth, and the role of surface/interface energies will all be vital in order to understand the kinetics of hydrogenation/dehydrogenation reactions.

# Technical Approach (Gas Condensation) at Caltech

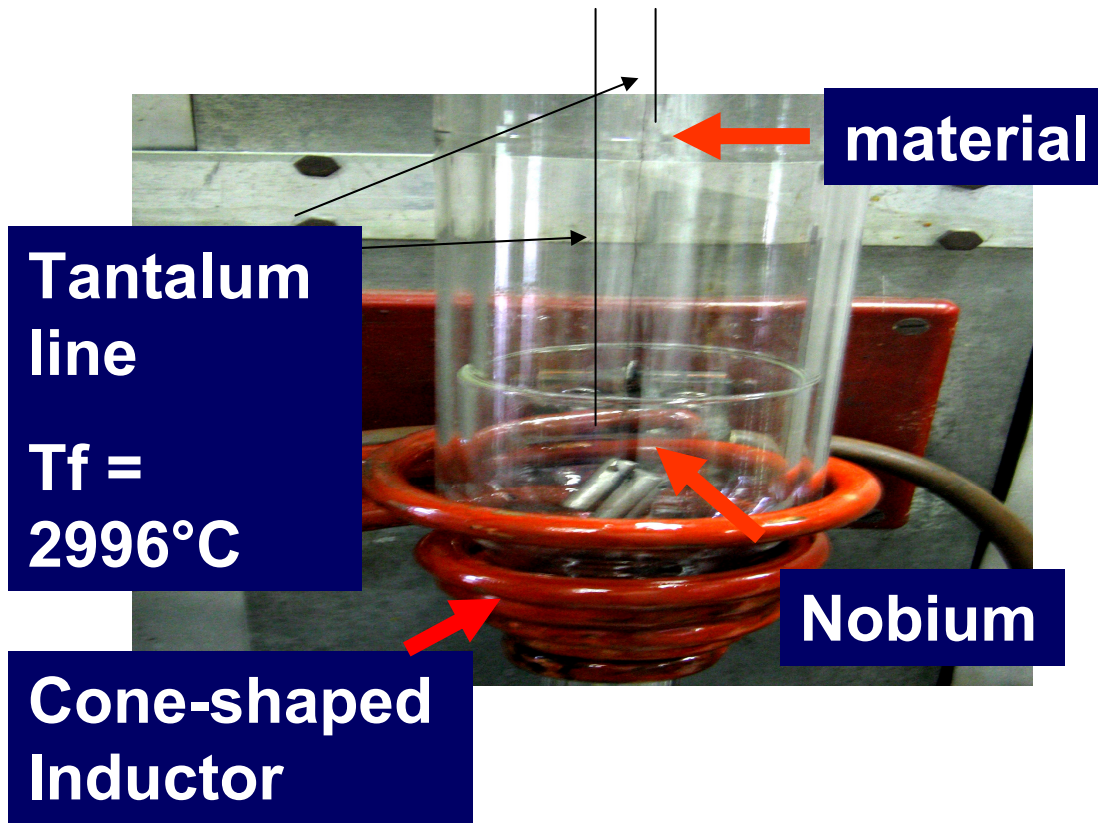


Shown above is our operational chamber for synthesis/deposition of nanoscale hydrides and hydride precursors based on gas condensation. Schematic of chamber operation is shown at right.



- Small pilot batch synthesis system for fundamental feasibility studies
- Yields presently on the order of ~15% of initial charge.

# Technical Approach (Cryo Melting) at CECM-CNRS, Vitry, France



Collection Filter

Mixture of Ar gas and nanoparticles

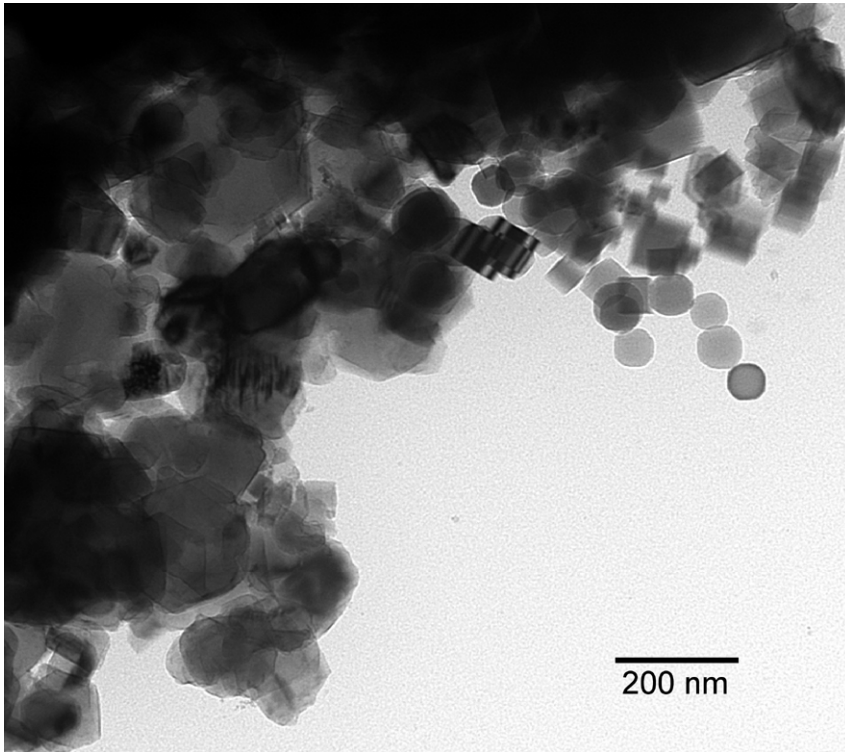
Induction heater

Nobium droplet 3000K

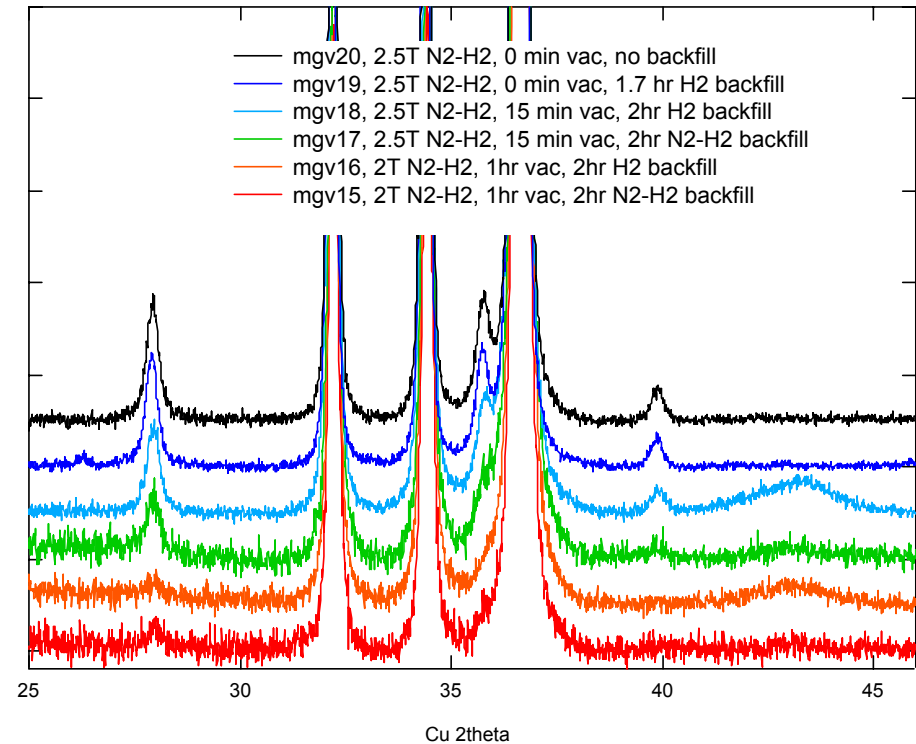
Argon 87K

New collaboration with Yannick Champion in France to employ semi-continuous process for nanophase material synthesis with gm/min yields. This approach will make large quantities of material available for testing.

# Progress/Initial Results from gas condensation



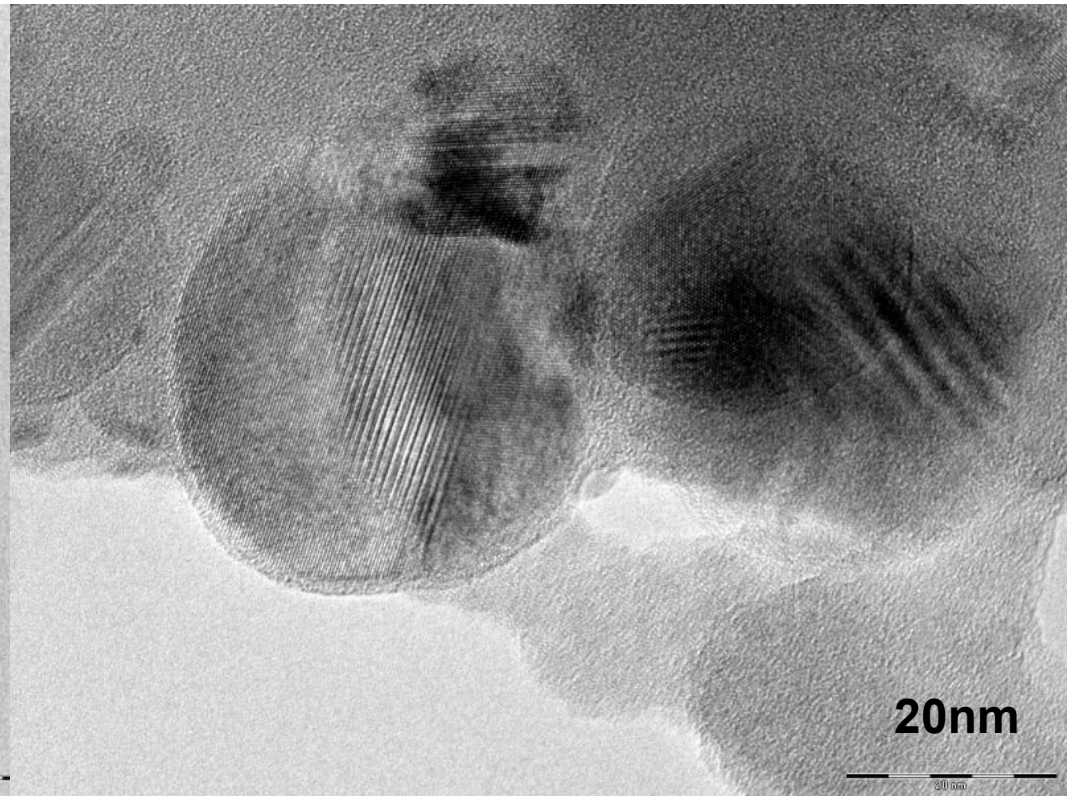
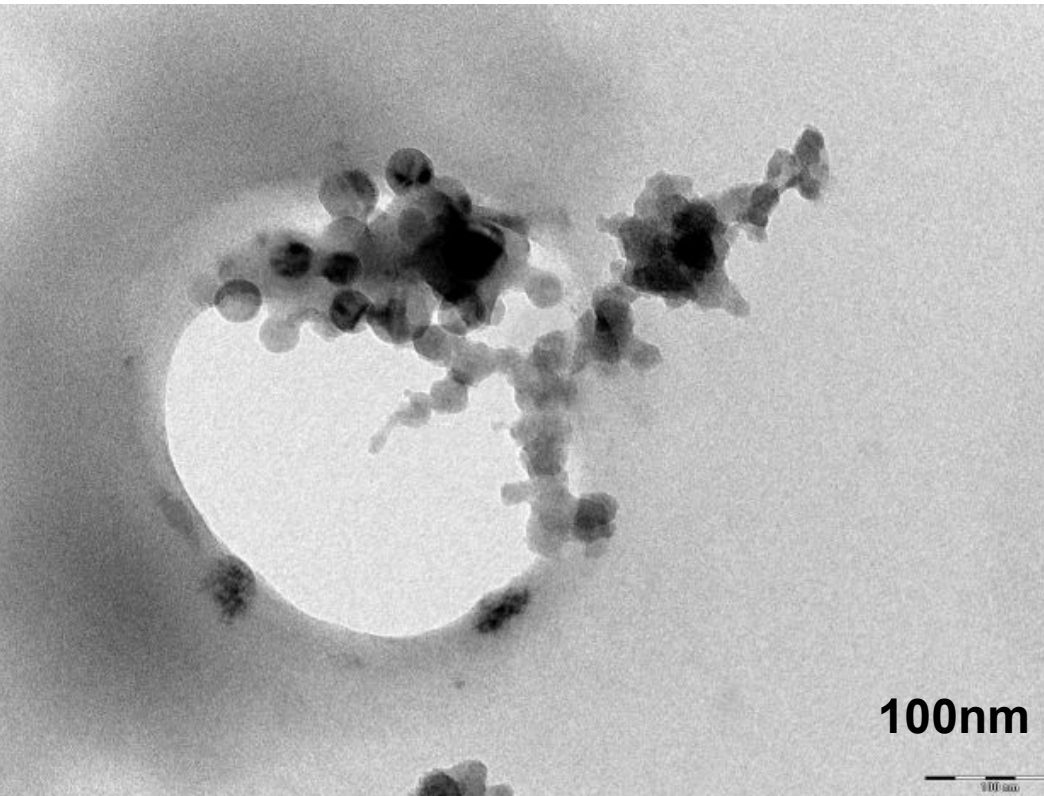
TEM micrograph of Mg metal using Ar-10% $H_2$



X-ray diffraction of initial results of Mg synthesis/deposition attempts showing formation of  $MgH_2$  during synthesis.

- Initial results from Mg tests show ~8%  $MgH_2$  formation during nanoparticle synthesis.
- Yields presently on the order of ~15% of initial charge.

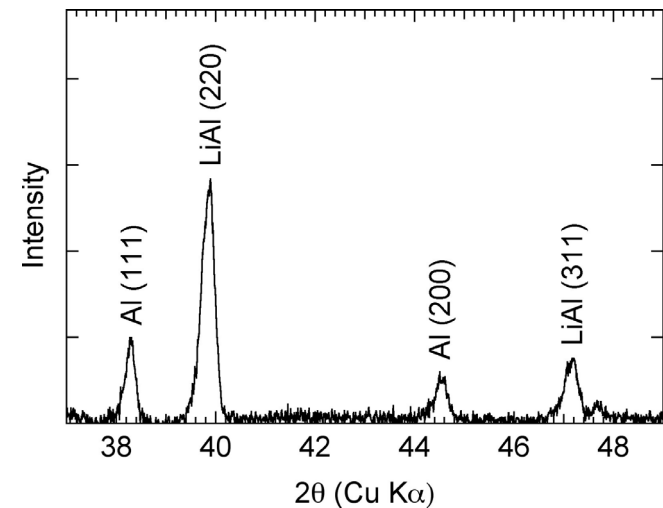
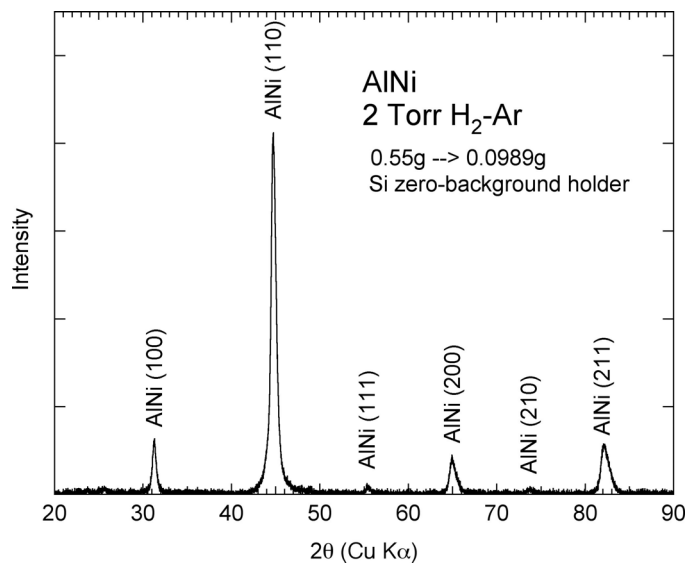
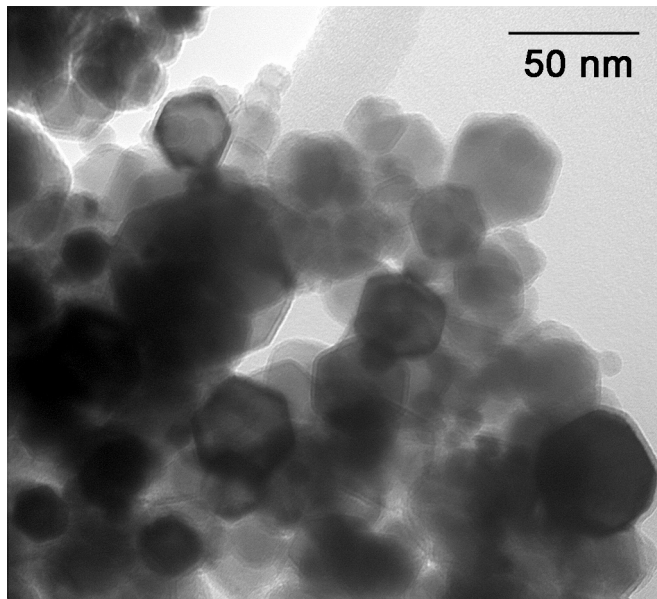
# Progress/ Initial Results from cryo melting



Results from Si show  $< 50\text{nm}$  particle size generation and revealing that the particles are typically crystalline.



# Previous results of gas condensation from congruently melting systems (cont'd)



Nanoparticle synthesis of more complex systems will be possible. We have previously synthesized alloys using gas condensation. Upper left shows a TEM micrograph of NiAl nanoparticles that show surface faceting, presumably from (110) planes in this material.

Left side shows x-ray diffraction pattern of gas condensed NiAl, that started with a NiAl charge. Only peaks from NiAl are present. Collection efficiency of material from this run is 18%.

Plot in upper right shows some disproportionation of LiAl during gas condensation.

# Hydrogenation/Dehydrogenation evaluation with volumetric Sieverts apparatus



- Over 10 years experience with volumetric hydrogen sorption measurements.
- Computer controlled unit is 3rd generation Sieverts, built at JPL from unit originally built at Caltech.

# Future Work for FY2005

- Follow-up on  $\text{MgH}_2$  phase formation
- Increase yields of elemental nano Mg and Si in order to study the reaction kinetics of intermetallic formation.
- Direct nano  $\text{Mg}_2\text{Si}$  alloy synthesis
- Kinetic evaluation of this system.

# Task and Milestone chart

