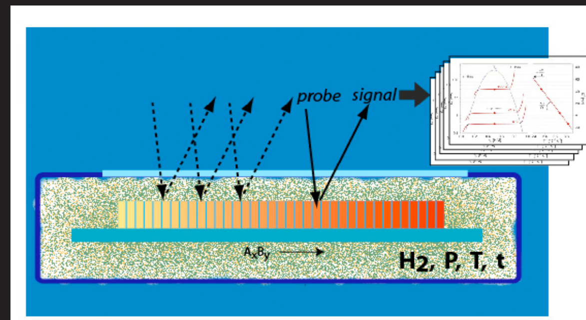
# Hydrogen Storage

## Objective

The goal of this project is to develop the metrology necessary for rapid, high-throughput measurement of the hydrogen content of novel materials proposed for hydrogen storage. A focus is bringing Prompt Gamma Activation Analysis, a direct method requiring a neutron source and thus not commonly available, to the stage where it can be used as a reference method for the calibration of indirect methods that are more readily available to the research community, such as infrared spectroscopy (IR).




METALS

## Impact and Customers

- Hydrogen is promoted as a petroleum replacement in the Hydrogen Economy, presenting an attractive alternative for fueling automobiles and trucks while maintaining a healthier global environment. A major roadblock associated with the use of hydrogen is the inability to store it efficiently.

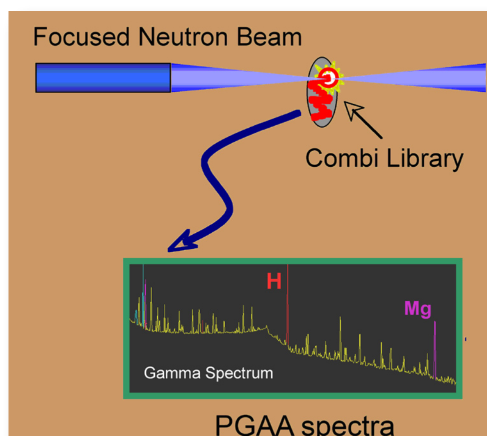
- Enable high throughput measurement tools for determining hydrogen absorption/desorption characteristics will accelerated discovery of new materials which can store hydrogen in a useful manner.


- A correlation of direct and indirect measurements of the hydrogenation process and methods to measure combinatorial samples are essential in developing the high throughput methodology.

- Industrial R&D, academia, and national labs are potential customers to use the developed methods as a fast screening tool in their research of novel materials for hydrogen storage.

## Approach

The evaluation of candidate storage materials is complicated by a lack of readily available methods for the direct measurement of hydrogen content. MSEL is working together with researchers from CSTL, NCNR, and PL to provide measurement tools to fill this gap. Prompt Gamma Activation Analysis (PGAA) is a direct method for measuring hydrogen, but is not commonly accessible since it requires a neutron source. Infrared (IR) and Raman imaging/spectroscopy methods, being developed at NIST, could be made widely available, but are not capable of directly measuring hydrogen content. Using measurements of hydrogen content with PGAA to calibrate IR and Raman imaging/ spectroscopy methods, NIST will provide an accurate screening methods widely usable by industry with the high spatial resolution needed for the development of storage materials. These methods are being tested on combinatorial thin films that the Metallurgy Division prepares using a multilayer-deposition technique in a customized e-beam deposition chamber. Currently the focus is on Mg-based films, which are of great interest to the hydrogen storage scientific community.



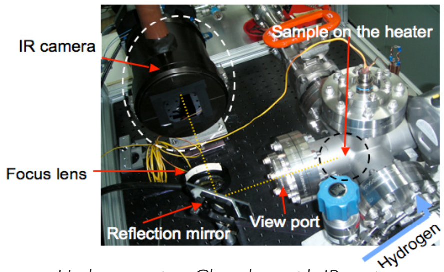
## Accomplishments

As the first step towards correlating IR results with those obtained using PGAA, the Metallurgy Division has successfully demonstrated that in-situ IR emissivity imaging method is able to capture the reaction between Mg/transition metal thin films and hydrogen gas. The method has been shown to be sensitive to variations in composition and microstructure.

Thin films of $Mg_xNi_{1-x}$ and $Mg_xTi_{1-x}$ with a composition gradient corresponding to 0.95>x>0.4 were capped with Pd to prevent oxidization of the films and to catalyze the hydrogenation reaction. The composition of the films was selected for two reasons: (1) high gravimetric density of hydrogen in $MgH_2$ and the need for better knowledge of the thermodynamics and kinetics of the process; (2) the desire for two-phase microstructures in the material, with one phase responsible for hydrogen storage and the other for fast hydrogen delivery.

Hydrogen absorption/desorption of the films was studied by acquiring IR images in-situ using the chamber pictured below.

The compositionally graded films are

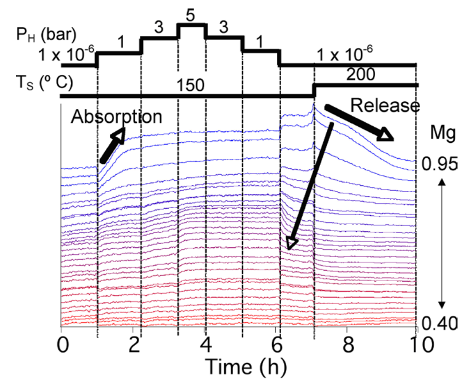



*Hydrogenation Chamber with IR optics*

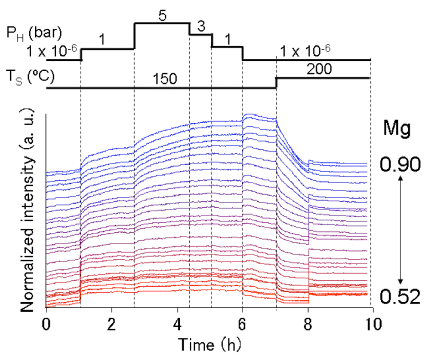
clamped to a heating stage inside the hydrogenation chamber and IR emission images are continuously collected through a sapphire window. The IR camera used permits "snap-shot" imaging (with 10 microsecond or longer integration times). The camera has its peak sensitivity at a wavelength of 5 micrometers, but it is able to detect over an integrated range of 1.0 to 5.5 micrometers.

The IR emission images are analyzed post acquisition by means of an image analysis software to extract the temporal intensity evolution for each pixel, corresponding to a given value of the composition gradient. Normalized IR intensities (with respect to a fixed region in the Si substrate) are then plotted as a function of measurement time (therefore, as function of hydrogenation conditions) along the composition gradient.

Evolution of the normalized IR intensities during hydrogenation experiments are shown to the right for $Mg_xNi_{1-x}$ and $Mg_xTi_{1-x}$ films. In these experiments the films were initially equilibrated at 150 °C prior to exposing the samples to hydrogen, thus the change in the film's IR emissivity during hydrogenation could be attributed only to the changes in the amount of hydrogen in a film. The curves were offset to show the evolution of IR intensity with time for the given compositions across the gradient. The conditions of the hydrogenation experiments: hydrogen pressure $P_H$ (bar) and film's temperature $T_S$ (°C) are depicted with each figure.




*Normalized IR intensity of $Mg_xNi_{1-x}$.*




*Normalized IR intensity of $Mg_xTi_{1-x}$.*

## Learn More

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## Publications

Oguchi H, Takeuchi I, Josell D, Heilweil EJ, and Bendersky LA, *Investigation of hydrogen storage using combinatorial thin films and IR imaging*, MRS Proceedings 2007