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# Using the Linkam CCR1000 to follow the phase transition with Raman spectroscopy of LiBH<sub>4</sub>

**Tadworth, UK** –**2012:** Market leaders in temperature controlled microscopy, Linkam Scientific Instruments report on the use of their CCR1000 stage at the University of South Carolina to visualise and measure raman in-situ phase change of LiBH<sub>4</sub>.

WHO + WHERE

At the University of South Carolina Xiaojing Sun, a graduate student in Dr William's team, and her colleagues are using the Linkam CCR1000 stage to look at the phase change of LiBH4, which is the focus of study for scientists as a possible Hydrogen ( $H_2$ ) store. Scientists are exploring several ways of storing  $H_2$  including under high pressure, cryogenics and chemical compounds that absorb/bind  $H_2$  and reversibly release  $H_2$  on heating. The New York Times recently reported that the University of South Carolina was the leading national force in driving research and making the  $H_2$  economy a reality. Home to the nation's only National Science Foundation-funded Industry/University Cooperative Research Center for Fuel Cells, the University partners with more than 15 private companies.

One of the main barriers to  $H_2$  gas becoming a viable alternative fuel is the need to identify a suitable, safe, reliable method of storage with an acceptable energy density. LiBH<sub>4</sub>, a complex hydride is one mineral that can store  $H_2$ ; when it decomposes it breaks down into B,  $Li_2B_{12}H_{12}$ , LiH and  $H_2$ .

## WHAT? HOW?

 $LiBH_4$  is in an orthorhombic phase at room temperature and changes to a hexagonal phase at higher temperatures. At around 300°C it begins to melt and release  $H_2$ . There is a corresponding phase change; the white solid powder  $LiBH_4$  very quickly translates into a liquid phase. The scientists are attempting to follow the phase transition and identify the crystallographic structures with Raman spectroscopy to further understand the decomposition mechanisms. The products and intermediates are not well understood.

Raman spectroscopy has a dual advantage: it can be used to probe the bonding between the elements and establish intermediates and it can be used to examine the volume change that occurs when  $H_2$  is released.

The Linkam CCR1000 stage can be used to study hydride samples at high temperatures and at high pressures. Samples are mounted on a ceramic fabric filter placed inside the ceramic heating element and can be heated from ambient to 1000°C very quickly.

The scientists at the University of South Carolina are using a little aluminium foil 'boat'" on the top of sample filter. They load their sample into the boat to prevent

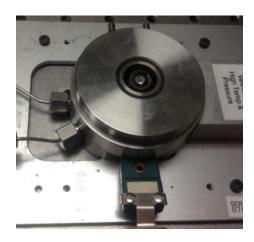


the sample melting and penetrating the sample filter and flowing into the cell with gas flow at temperatures over 300°C. The sample is heated to 600°C with 5°C/min ramp and Raman spectra taken every 4 mins.

Xiaojing Sun says: "The cell and temperature controller are very well designed and working well."

#### WHY?

She added: "The the goal for our research is to increase the  $H_2$  production and most importantly have a high efficiency in recycling the reactant." So far they have found that when the reactant decomposes to form  $H_2$  around 400°C, and is cooled fully, and heated again, the amount of  $H_2$  released for this second cycle is significantly decreased. The scientists intend to learn what chemical changes occur during the increasing temperature and then they may introduce some other metallic element to the reactant to achieve a better recycle.



Visit Linkam at <u>www.linkam.co.uk</u> and learn about the broad range of applications in the field of temperature controlled microscopy.

For high resolution copies of these and other images, please contact Jezz Leckenby at Talking Science Limited.

## **About Linkam Scientific Instruments**

Linkam develops and manufactures a broad range of heating and freezing stages for both OEM and end users to visualize and explore materials properties. Used in conjunction with light microscopes and other forms of spectroscopy, Linkam stages are found in thousands of laboratories worldwide with the most successful microscope heating stage, the THMS600, selling over 4,000 units alone. Linkam is the market leader in temperature controlled microscopy.

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