KEP Technologies

# MATERIAL CHARACTERIZATION **FOR HYDROGEN**

## THERMAL ANALYSIS , CALORIMETRY & GAS SORPTION SOLUTIONS

• HYDROGEN PRODUCTION •
• HYDROGEN STORAGE •
• HYDROGEN DELIVERY •
• HYDROGEN CONVERSION •

setaram
KEP TECHNOLOGIES

## YOUR CHALLENGES

For several years, hydrogen has been a highly coveted sector, both ecologically and economically. European countries are investing more and more in this energy vector, especially for its impact on reducing greenhouse gas emissions.

The Hydrogen cycle has become a real challenge in the development of our energies, and can be broken down into 4 steps : **production, storage, distribution and conversion.**

## COMMON HYDROGEN - STUDIES & SOLUTIONS

This brochure presents some of our solutions in this field and we encourage you to contact us for more information.

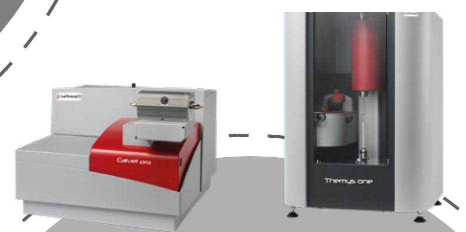
When hydrogen is transported in gaseous or liquid form, it has a significant maintenance cost due to the short life time of materials at high pressure and/or low temperature.
You may therefore have to characterize the stability of potential materials under these conditions.

**Hydrogen Distribution**

The cost and strength of materials used in hydrogen storage components and systems can be improved by characterizing their resistance to aging.
Storage in liquid or solid sorbents is a promising alternative. However, to gain efficiency and extend their application range, materials need to store more hydrogen and be faster to charge and discharge the stored hydrogen.
Setaram helps you characterize the capacity and speed of your materials.

**Hydrogen Storage**

**All Hydrogen steps**



This last stage of the hydrogen cycle can be achieved by several processes.
By combustion: turbines operating with hydrogen are subjected to higher temperatures and higher steam levels. Setaram solutions support you with the characterization of new, more resistant alloys.
By conversion in a fuel cell: the characterization of the materials' thermal stability (such as electrolytes and membranes) allows the development of less expensive materials, which can resist higher temperatures.

**Hydrogen Conversion**

The production of hydrogen from water or biomass requires a catalyst. The high cost of current catalysts requires optimization of these materials. Thermal analysis and calorimetry can be used to characterize the efficiency of new potential catalysts and thus achieve savings.

**Hydrogen Production**



## CUSTOMER TESTIMONIAL

*"I published more than 30 scientific papers on high IF journals in collaboration with leading groups in the frame of hydrogen storage research performing the analyses by the GASPRO equipment and the GASPRO connected to the CALVET PRO calorimeter.*
*I am fully satisfied by the instrument capabilities and performance and by the results we can obtain with it in my research field."*

***Chiara Milanese, Associate Professor, Scientific responsible***
*Pavia Hydrogen Lab - Chemistry Department – University of Pavia – ITALY*

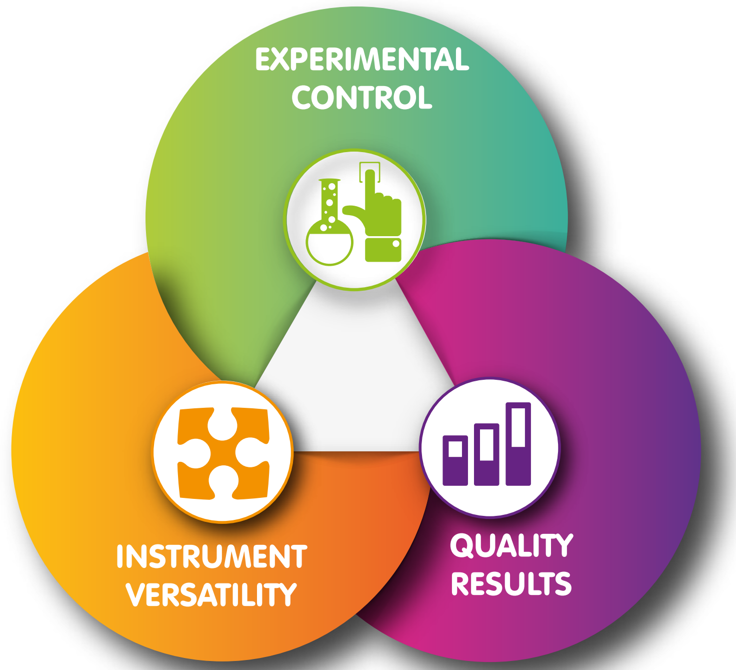
## THE KEP TECHNOLOGIES ADVANTAGE

KEP Technologies is addressing it's offerings to the battery market by making available the widest and most versatile choice of solutions. Now you can consult with one company, KEP Technologies, to address your challenges across the broadest number of battery studies on the market.

Each solution embodies our "Reimagine Material Characterization" value proposition by delivering the three core customer benefits of **Experimental Control, Instrument Versatility and Quality Results.**

We believe solutions that provide these benefits will deliver the highest value to our customers.

In addition to our core customer benefits, we are able to provide **customized solutions** by harnessing the engineering and project management of our highly skilled organization.




**CUSTOMIZED SOLUTIONS**

**Modular design allows for upgraded and tailored functionality**
**Access to all previous non-proprietary custom requests**
**Open access to our engineering development team**

## INSTRUMENT

### THEMYS

- **ACCURATE AND SENSITIVE ULTRA-HIGH TEMPERATURE**
  heat flow measurement with Tri- Couple DTA technology
- **ULTRA-HIGH TEMPERATURE CAPABILITY**
  to 2400°C with a single furnace
- **MODULAR ADAPTIONS ALLOWING**
  TGA only, DTA only, TG-DTA, and TMA up to 2400°C, DSC only and TG-DSC up to 1750°C all in one instrument
- **HIGH ACCURACY & VERSATILITY**
  hang-down symmetrical beam balance, specifically designed for TGA applications
- **EXTERNAL COUPLING CAPABILITY**
  designed for evolved gas analyzers (FTIR, MS, GCMS, MS-FTIR,or FTIR-GCMS)

## SPECIFICATIONS

| | |
|---|---|
| **Temperature range (°C)** | room temperature to 1750 or to 2400 |
| **Isothermal and temperature scanning (°C/min)** | 0.01 to 100 |
| **Sample volume (µl)** | up to 2500 in TGA |
| **Optional protected DTA rods for enhanced corrosion resistance, tricouple DTA rods for enhanced sensitivity, protected tricouple for combined advantages** | |

**For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com**



## APPLICATION

### Characterization of a catalyst used for the hydrogen production

### INTRODUCTION

To reduce our dependence on fossil fuels, we must diversify our energy solutions. Biomass is an abundant and renewable raw material. The transformation of biomass into hydrogen-rich gas requires catalysts that optimize this reaction. These catalysts accumulate carbon during the reaction, which affects their efficiency. The amount and reactivity of carbon deposited on the catalyst during biomass conversion was studied using THEMYS STA.

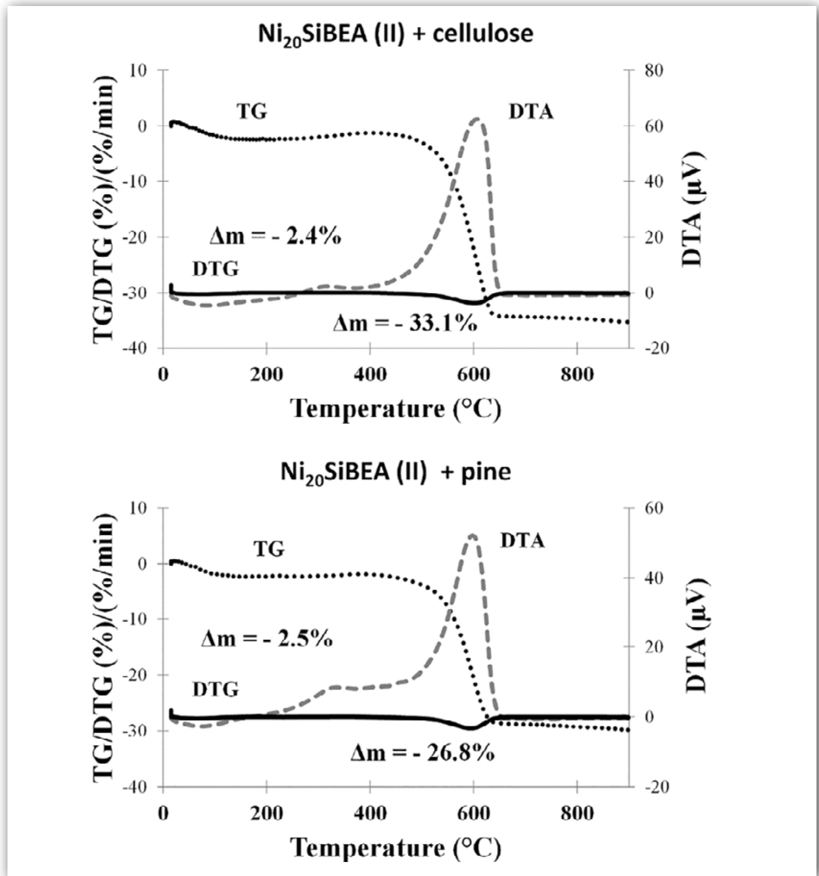
### EXPERIMENT

THEMYS with its TG-DTA configuration was used for the characterization of a catalyst used for the conversion of cellulose and pine into hydrogen rich gas.
The following profile was applied on the catalyst:
- Heating from 30°C to 900°C at 10°C/min
- Atmosphere: Air at 40ml/min

### RESULTS AND CONCLUSION

For both tests, mass losses and exothermic effects were observed. They are related to the combustion of carbon accumulated on the catalyst. The results obtained make it possible to determine the carbon content, with values of the order of 30% by weight. The combustion temperature gives information on the structure of the coke formed during the conversion of the biomass.

*Hydrogen-Rich Gas Production by Upgrading of Biomass Pyrolysis Vapors over NiBEA Catalyst: Impact of Dealumination and Preparation Method-Jacek Grams, Robert Ryczkowski, Renata Sadek, Karolina Chałupka, Kamila Przybysz, Sandra Casale, and Stanislaw Dzwigaj Energy & Fuels 2020 34 (12), 16936-16947*



## INSTRUMENT

### CALVET

**ISOTHERMAL OR TEMPERATURE SCANNING MODES**
for increased flexibility

**HEAT MEASUREMENT ACCURACY**
with Calvet 3D sensor capturing 93-95% of all heat forms. The highest level on the market

**CONVENIENT, INTERCHANGEABLE CELLS**
to perform even the most demanding experiments using one instrument

**WIDE TEMPERATURE RANGE**
with low temperature version CALVET CRYO and high temperature version CALVET HT

## SPECIFICATIONS

| | CALVET | CALVET CRYO | CALVET HT |
|---|---|---|---|
| **Temperature range (°C)** | Ambient to 300 | -196 to 200 | Ambient to 600 |
| **Temperature accuracy (°C)** | +/-0.3 * | +/-0.5 ** | +/-1* |
| **Temperature precision (°C)** | +/-0.15* | +/-0.25** | +/-0.5* |
| **Programmable temperature scanning rate** | 0.001 to 2°C/min | 0.01 to 1°C/min | 0.01 to 2°C/min |
| **Enthalpy accuracy** | +/-0.4 * | +/-0.2 ** | +/-1* |
| **Calorimetric precision (%)** | +/-0.4* | +/-0.5** | +/-1.5* |
| **Cells (ml)** | Up to 12.5 (standard cell) | Up to 12.5 (standard cell) | Up to 7 |
| **Pressure measured and controlled (bar [psi])** | 350 [5,075]; 600 [8,700]; 1000 [14,600] | 100 [1,450]; 600 [8,700]; 1000 [14,600] | 100 [1,450]; 300 [4,350]; 400 [5,800] |

** Based on indium melting tests ** Based on naphthalene melting tests*

**For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com**



## APPLICATION

### COMBINED CALORIMETRY AND HYDROGEN SORPTION MEASUREMENTS ON LANI5

### INTRODUCTION

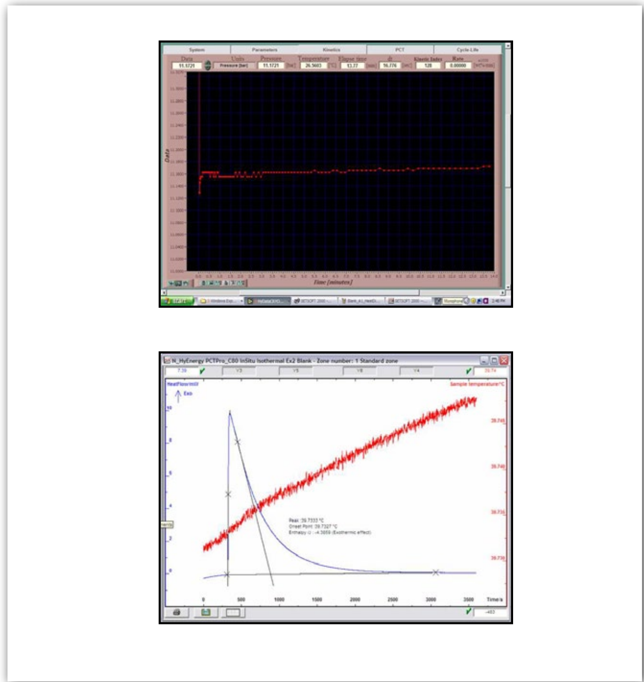
The thermodynamic stability of hydrides is a key parameter for practical applications in hydrogen storage. The measurement of the enthalpy of reaction in-situ during the gas sorption is particularly useful for unstable chemical hydrides where the H2 sorption is not reversible under normal conditions . By coupling a CALVET and a GASPRO, we can simultaneously quantify the heat of formation of the hydride and its hydrogen sorption capacity.

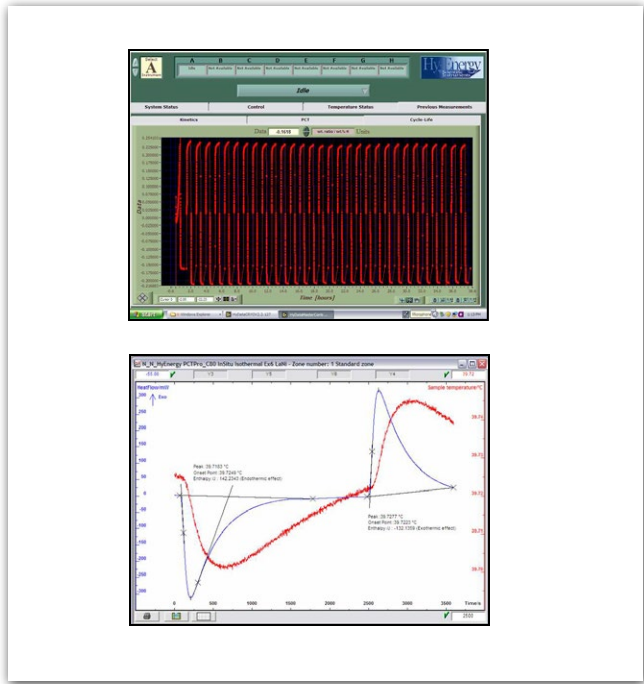
### EXPERIMENT

The adsorption and desorption of H2 by a LaNi5 alloy was studied using a GASPRO coupled with a CALVET.
Initially, a background was recorded without sample.
Then the powder (5 g) was analyzed using the following procedure:
• Activation of the powder by H2 absorption/desorption cycles at 40 °C.
• Measurement of the heat of reaction on the 20th cycle at 40 °C.
The CALVET signal obtained without sample not only showed that the influence of the heating on the gas is but, in addition, the measurement provided an accurate measurement of the heat capacity Cp of hydrogen gas.
Measured effect of gas heating 6 ml at 11.2 bar:
• → 29.8 J/K mol H2 ~ Cp,
• Baseline heating of gas ~ 0.3 kJ/mol H2 equivalent to a 1% effect on the measurement of the heat of hydride formation.

### RESULTS AND CONCLUSION

The analysis of the results demonstrates that:
1) Hydriding was steady but incomplete after more than 30 cycles. The material only reached 1/6 the full theoretical capacity.
2) This behavior is verified by the morphology of the powder following the sorption tests. Only 1/6 of the sample powder (fine grains) were hydrided.
3) Using the hydrogen sorption capacity measured by the GASPRO, the heat of reaction measured using the CALVET was in accordance with the theoretical heat of formation of the hydride.
•4.377 gm LaNi5 ↔ 1/6 LaNi5H6.
• Measured: 0.004875 moles H2.
• H: 142.2 J = 29.2 kJ/mol H2.





## INSTRUMENT


### CALVET PRO DSC

**HIGHEST HEAT MEASUREMENT ACCURACY**
Calvet 3D sensor based on thermocouples with Joule effect calibration.

**EXTERNAL COUPLING CAPABILITY**
Designed to increase your research options, including manometry, BET, gas analyzers, humidity controllers and gas panels.

**CONVENIENT INTERCHANGEABLE CRUCIBLES AND CELLS** to perform even the most demanding experiments with one instrument : high pressure (500bar) and high vacuum (10-4 mbar) studies, pressure measurement and control, packed bed reactor experiments.

## SPECIFICATIONS

| | |
|---|---|
| **Temperature range (°C)** | Ambient to 830°C<br>-120 to 200 °C (with cooling accessory) |
| **Temperature accuracy (°C)** | +/- 0.05* |
| **Temperature precision (°C)** | +/- 0.15* |
| **Programmable temperature scaning rate (°C/min)** | 0.01 to 30 |
| **Enthalpy accuracy (%)** | +/- 0.8* |
| **Calorimetric precision (%)** | +/- 0.4* |
| **Crucible or cells volume (ml)** | Up to 0.32 depending on the chosen design and material (alu-minium,<br>incoloy, graphite, alumina, platinum, etc) |
| **Pressure (bar [psi])** | 400 [5,800] (measured and controlled); 500 [7,250] (resistant) |

** Based on indium melting tests*

**For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com**



## APPLICATION

**Combined calorimetric and sorption measurements on Mg-based hydride with small sample mass**

### INTRODUCTION

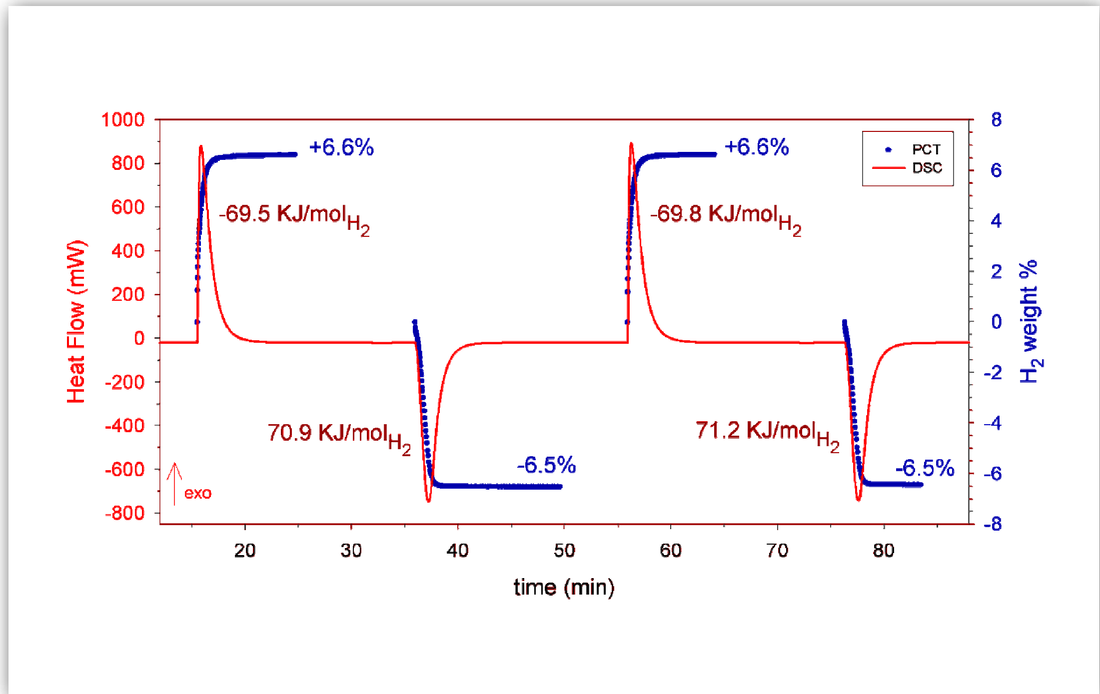
The thermodynamic stability of hydrides is key to practical applications in hydrogen storage. Both the heat of formation of the hydride and its hydrogen sorption capacity can be quantified. In this example, we demonstrate the capability to make combined calorimetry and sorption measurements on a very small (34mg) sample using the HP cell of a CALVET PRO DSC coupled with a GASPRO.

### EXPERIMENT

The reaction of hydrogen with 34 mg of a Mg-C-Nb2O5 composite was characterized by:
• PCT measurements to determine the hydrogen uptake,
• calorimetric measurements to study heats of reaction.
The sample was subjected to a series of absorption/desorptioncycles. The hydrogen absorption was started at 12 bar , and the desorption was carried out at 1 bar.

### RESULTS AND CONCLUSION

We have shown that the absorption reaction is fully reversible and that both the enthalpy and the hydrogen uptake are reproducible throughout the cycles.
This example demonstrates that sorption studies on small quantities of samples can be investigated by this unique coupling of the CALVET PRO DSC and GASPRO.




REIMAGINE MATERIAL CHARACTERIZATION

## INSTRUMENT

### GASPRO

- **VARIETY OF MODES OF OPERATION**
  ability to combine PCT, kinetics and cycle life modes to 200bar to determine the quantity and rate of sample gas interaction and its aging characteristics all in one instrument and operation
- **PRECISION MEASUREMENT OF SMALL SAMPLES**
  using the patented microdoser option to inject small doses of gas on the sample humidity controllers and gas panels.
- **HIGH ACCURACY VERSION**
  to reduce cumulative error across multiple measurements points
- **EXTERNAL CALORIMETER COUPLING CAPABILITY**
  to increase your research options

## SPECIFICATIONS

| | |
|---|---|
| **Temperature range (°C)** | -260 °C to 500 °C<br>with different sample holder options<br>Higher temperatures on request |
| **Calibrated reservoirs** | from ~5 ml to ~1.2 l |
| **Sorption gas (Test gas)** | Carbon Dioxide, Methane, Nitrogen, Argon, Hydrogen, Deuterium, Helium, Neon, Ammonia, n-alcanes from C2 to C6, more on request. |
| **Operating pressure range** | From vacuum to 200 bar |
| **Sample pressure measurement** | 1 transducer for vacuum to 200 bar<br>1 transducer for vacuum to 5 bar<br>Accuracy: 1% of the reading |
| **Maximum sensitivity** | 3 µmole of gas<br>(with the MicroDoser attachment) |

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## APPLICATION

### Measurement of PCT isotherms on sodium alanate with differentlevel of Ti doping

## INTRODUCTION

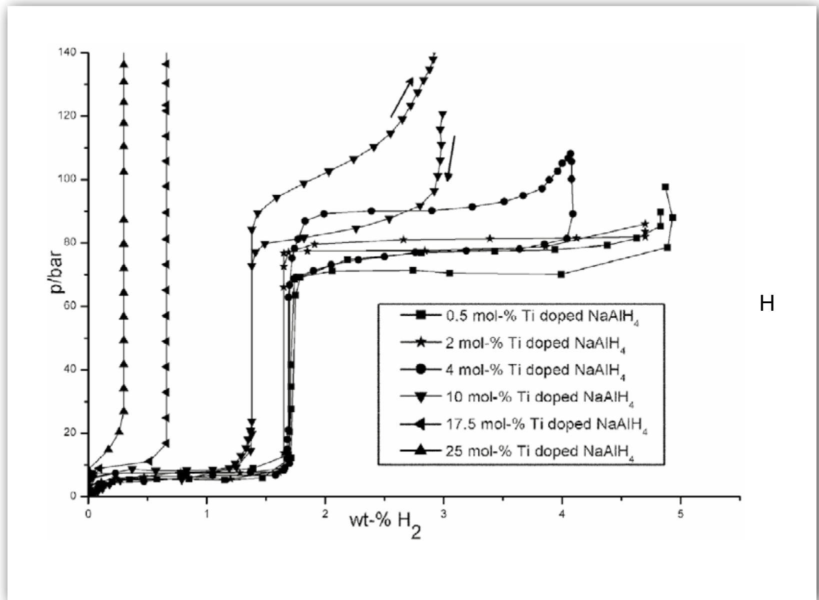
Bogdanović et al. demonstrated that Ti-doping of sodium alanate made the hydrogen sorption reaction reversible, despite the multi-steps reactions involved in the process. The precise role of the Ti in the system is unclear. The GASPRO was used by the same team to study the effect of various level of Ti-doping in the system.

## EXPERIMENT

Each sample (~2 g) of the Ti-doped sodium alanate was loaded into the sample holder in the glove box. Before recording the PCIs, samples were subjected to 3 hydrogen discharging/recharging cycles (dehydrogenation: 1 bar, hydrogenation:130 bar H2 pressure) which ensures stable performance. Then PCT isotherms were recorded at 160°C for each sample. PCT measurements for each individual doping level were repeated twice.
The equilibration time varied between 3.5 h (25mol.% Ti) and 20 h (0.5mol.% Ti) for each datapoint.

## RESULTS AND CONCLUSION

The PCT isotherms are strongly modified when the Ti-doping level is changed (fig. 1).
This is not consistent with the theory that the titanium is simply a catalyst. These observations clearly demonstrate that the titanium doping of the NaAlH4 does not just change the kinetics of the system, as expected for a catalyst, but also influences the thermodynamics quite drastically.
It has to be underlined that the conditions of the measurements were complicated with a system with extremely slow kinetics where high pressure is needed to reach the plateau.
The GASPRO permits work with the most complex hydride systems, like sodium alanate.



## INSTRUMENT

### THEMYS H2

- **ULTRA-HIGH TEMPERATURE CAPABILITY**
  to 1750°C with a single furnace
- **MULTIPLE BUILT-IN SAFETY SYSTEMS**
  for instrument and user's secured operations
  • up to 100% Hydrogen capacity
- **HIGH ACCURACY & VERSATILE**
  hang-down symmetrical beam balance
  specifically designed for TGA applications
- **EXTERNAL COUPLING CAPABILITY**
  to 1000 °C with Mass Spectrometers
- **ACCURATE AND SENSITIVE**
  Tri- Couple DTA and DSC technologies up to 1000°C
- **VERY HIGH TEMPERATURE**
  DTA technology up to 1750°C

## SPECIFICATIONS

| | |
|---|---|
| **Temperature range (°C)** | Ambient to 1 750 |
| **Programmable heating rate (°C/min)** | 0.01 to 100 |
| **Vacuum** | < 5.10-2 mbar |
| **Measuring range** | +/- 20 mg<br>+/- 200 mg |
| **Resolution** | 0.002 µg<br>0.02µg |

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## APPLICATION

### Thermogravimetric study of the hydrogen resistance of a titanium-based alloy

### INTRODUCTION

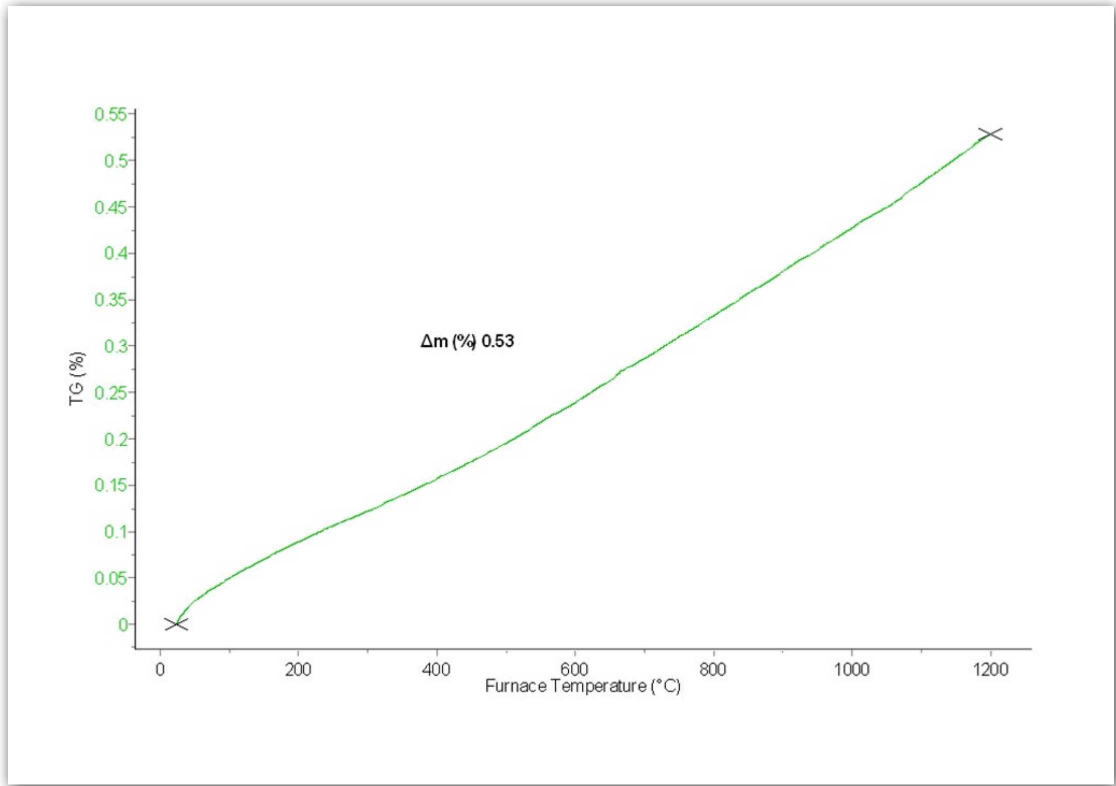
When hydrogen is transported in gaseous form, it represents a significant maintenance cost due to the short lifetime of materials (steel, composites, etc.). Thermogravimetric experiments at high temperature can help to understand degradation mechanisms of materials under hydrogen.

### EXPERIMENT

The resistance of a titanium-based alloy against hydrogen was study using THEMYS H2 TGA instrument.
The sample was heated up to 1200°C at 10°C/min under hydrogen.

### RESULTS AND CONCLUSION

The increase in sample weight indicates hydrogen adsorption in the titanium-based alloy. The hydrogenation of the alloy increases almost linearly with temperature up to the final temperature.



## INSTRUMENT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## SPECIFICATIONS

| | |
|---|---|
| **Temperature range (°C)** | Ambient to 830°C<br>-120 to 200 °C (with cooling accessory) |
| **Temperature accuracy (°C)** | +/- 0.05* |
| **Temperature precision (°C)** | +/- 0.15* |
| **Programmable temperature scaning rate (°C/min)** | 0.01 to 30 |
| **Enthalpy accuracy (%)** | +/- 0.8* |
| **Calorimetric precision (%)** | +/- 0.4* |
| **Crucible or cells volume (ml)** | Up to 0.32 depending on the chosen design and material (alu-minium,<br>incoloy, graphite, alumina, platinum, etc) |
| **Pressure (bar [psi])** | 400 [5,800] (measured and controlled); 500 [7,250] (resistant) |

** Based on indium melting tests*

**For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com**



## APPLICATION

### Heat capacity determination of elastomers used for hydrogen fuel cells

## INTRODUCTION

During the development of a hydrogen fuel cell, every part of the fuel cell requires careful consideration. Incorrect fuel gaskets can lead to gas leaks and reduction of the fuel cell performance.
Elastomers show an important variation of their heat capacity near the glass transition. The knowledge of the temperature of glass transition and the heat capacity before and after the glass transition is of a great utility for their uses inside hydrogen fuel cell.

## EXPERIMENT

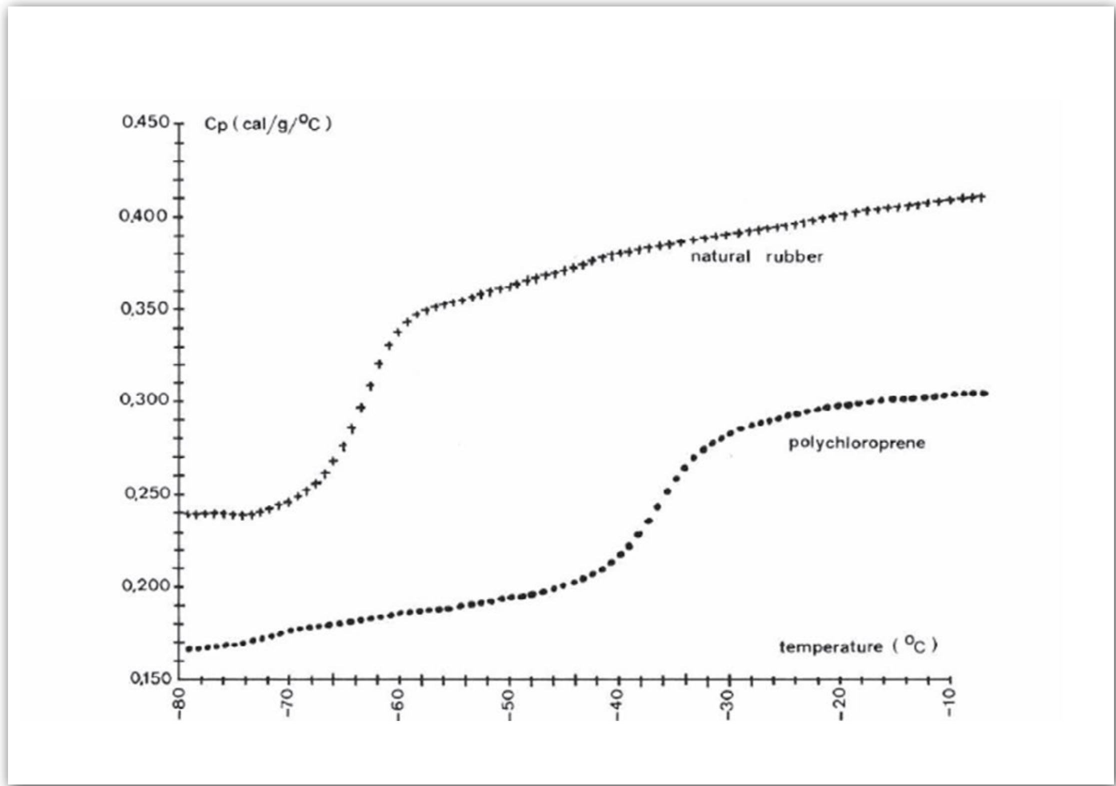
- Samples: natural rubber (156.7 mg) polychloroprene (230.8 mg)
- Crucible: Aluminum
- Heating mode: Scanning 5°C/min
- Use of the sub ambient cooling accessory

## RESULTS AND CONCLUSION

The mean values of specific heat before and after the glass transition are the following (in cal.g-1.°C-1) :

| | Before Tg | After Tg |
|---|---|---|
| Natural rubber | 0.240 | 0.360 |
| Polychloroprene | 0.190 | 0.300 |

The variation of heat capacity due to glass transition is 0.12 cal.g-1.°C-1 for natural rubber and 0.11 cal.g-1.°C-1 for polychloroprene




REIMAGINE MATERIAL CHARACTERIZATION

## INSTRUMENT


### THEMYS ONE

- **HIGH SENSITIVITY BALANCE FOR THE DETECTION OF SMALL MASS VARIATIONS** specifically designed for TGA analysis.
- **CONVENIENCE OF ONE FURNACE** to reach temperatures as high as 1600°C.
- **PLUG AND PLAY INTERCHANGEABLE RODS** to perform TGA only, TG-DSC, TG-DTA, and 3D high sensitivity/Cp measurements.
- **EXTERNAL COUPLING CAPABILITY** including evolved gas analyzers

## SPECIFICATIONS

| | |
|---|---|
| **Temperature range (°C)** | room temperature to 1600 |
| **Isothermal and temperature scanning (°C/min)** | 0.01 to 100 |
| **Sample volume (ml)** | up to 1 in TGA |
| **Evolved gas analyzers (FTIR, MS, GCMS, MS-FTIR, or FTIR-GCMS) for performing qualitative and quantitative gas characterization** | |

**For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com**



## APPLICATION

### Thermal stability study of polymers for electrolytes membrane fuel cells

### INTRODUCTION

High temperature fuel cell technology has a high efficiency for energy conversion. Since it operates at high temperatures, the thermal stability of the various components is crucial to ensure the performance of the fuel cell throughout its lifetime.
Thermogravimetric analysis is used to study the thermal stability of these components such as electrolyte membranes.

### EXPERIMENT

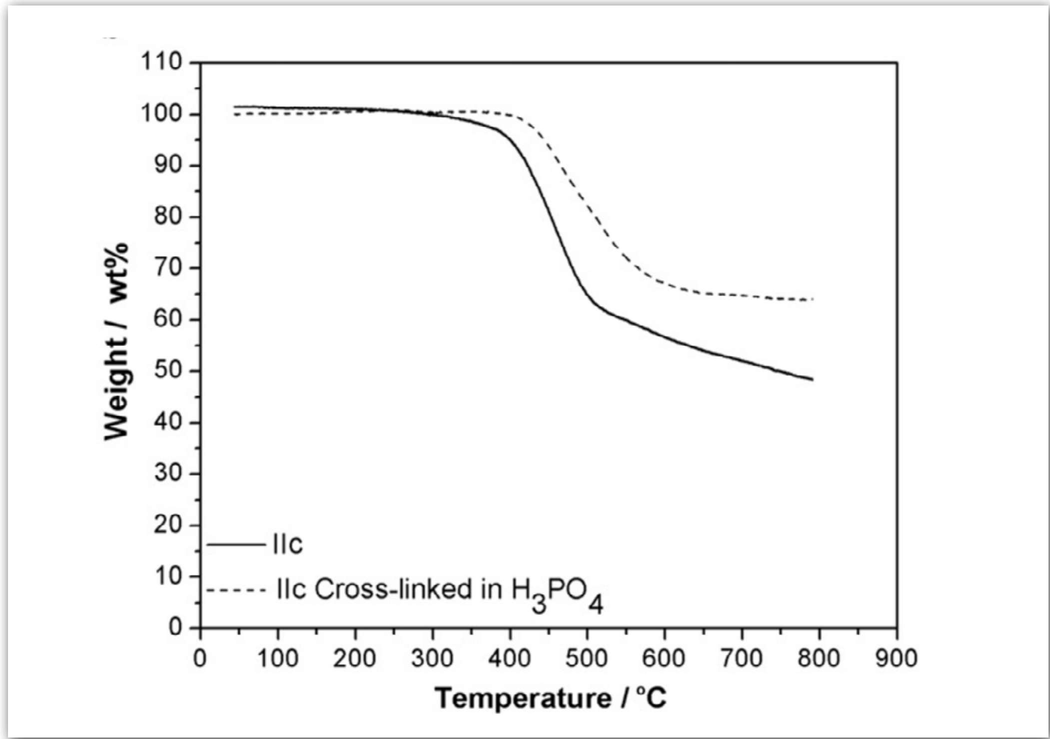
THEMYS ONE TGA was used with cross-linked polymer electrolyte membranes for high temperature fuel cells. The effect of crosslinking on the membrane's thermal stability was studied.

The virgin and cross-linked sample polymer was analyzed with the following thermal profile:
- Heating from 30°C to 800°C at 10°C/min
- Atmosphere: nitrogen

### RESULTS AND CONCLUSION

The virgin polymer is stable up to 280°C. At higher temperature, decomposition of the material can be observed on the TGA analysis result.
The TGA analysis of the crosslinked polymer shows an increase of the thermal stability: the decomposition temperature is 405°C, which is an improvement of 125°C.
These results highlighted that the crosslinking significantly improved the thermal properties of the membrane and extended its operational stability in a high temperature fuel cell system.

*Papadimitriou, Konstantinia & Geormezi, Maria & Neophytides, Stylianos & Kallitsis, Joannis. (2013). Covalent cross-linking in phosphoric acid of pyridine based aromatic polyethers bearing side double bonds for use in high temperature polymer electrolyte membrane fuelcells. Journal of Membrane Science. 433. 1–9. 10.1016/j.memsci.2012.12.051.*



## INSTRUMENT

### FLEXI HYCO

**COMPACT HP HYDROGEN DELIVERY SYSTEM**

• Compresses hydrogen from a low-pressure line or an optional, integrated electrolyzer
• Delivers ultrapure $H_2$ at a set pressure up to 200 bar (2900 psi)

**ROBUST AND PATENTED DESIGN**

• Stores and releases hydrogen from a metal hydride bed
• Operates without moving parts: silently and without maintenance

**PLUG & PLAY, EASY AND SAFE**

• Easy manual operation, no time-consuming setup is required
• Avoids the use of high-pressure hydrogen cylinders in your lab
• Safe by design with CE marking

## SPECIFICATIONS

| | |
|---|---|
| **Outlet pressure range** | From 10 to 200 bar (145 to 2900 psi) |
| **Hydrogen Storage Capacity** | Up to 90 Nl or up to 180* Nl |
| **Maximum Outlet Flowrate** | Up to 0.8 Nl/min or up to 1.6* Nl/min |
| **Inlet pressure** | 10 bar |
| **Hydrogen inlet options** | Electrolysers, low pressure gas line, high pressure cylinders stored outside |

**For more information on specifications please consult the product information and brochures available on our website : www.setaramsolutions.com**



## APPLICATION

FLEXI HyCo is the perfect accessory for laboratories that require **experimentations under hydrogen.**

FLEXI HyCo can simply be connected to small, pilot-scale small scale hydrogen based power generation systems, like fuel cell stacks testing units, fuel cell cells test benches.

It can also **be connected to any device** or to any reactor requiring operations under hydrogen pressures below 200 bar.

The purpose of the experimental setup may be the characterization of materials aimed at being used under hydrogen, or the study of reactions to be run under hydrogen.

**Hydrogen storage materials:** storage capacity, kinetics, heat of sorption, etc

**Fuel cells and electrolysers materials development** like membranes

**Catalysts, sorbents**: selectivity, reactivity, conversion, etc

**Metals and alloys:** resistance to corrosion and/or embrittlement by hydrogen, etc

**Chemical engineering**: safe scale-up of hydrogenation reactions, etc

**Food** (lipids): studies of hydrogenation reactions of vegetable oils, etc

REIMAGINE MATERIAL CHARACTERIZATION

Switzerland – France – China – United States – India – Hong Kong
For contact details: **www.setaramsolutions.com** or **setaram@kep-technologies.com**