

## Temperature cycling stability of Phase Change Materials

### Introduction

Some materials have the capacity to store and release large amounts of energy by changing their physical state: they are called Phase Change Materials (PCM). They can be used for instance to minimize the need of air conditioning and heating: incorporated into walls of buildings, they absorb heat when outside temperature is high, and release it when temperature decreases. This keeps the house into a "comfort temperature zone". Another utilization of the PCM is the use of residual heat from exothermic reaction in industry as a source of energy. An application fitting phase change temperature and a large melting enthalpy are two obvious requirements for these materials. However, there is another important parameter: the cycling stability, in order to use the PCM as many times for storage and release of heat as required by an application [1].

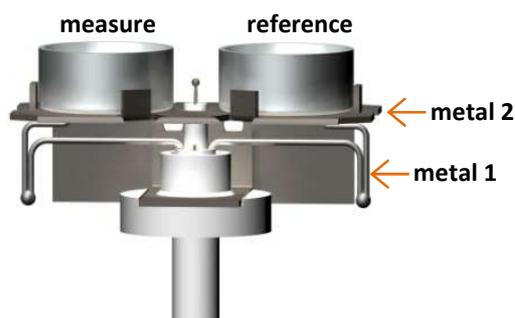


Figure 1 – Plate of the DSC 131evo

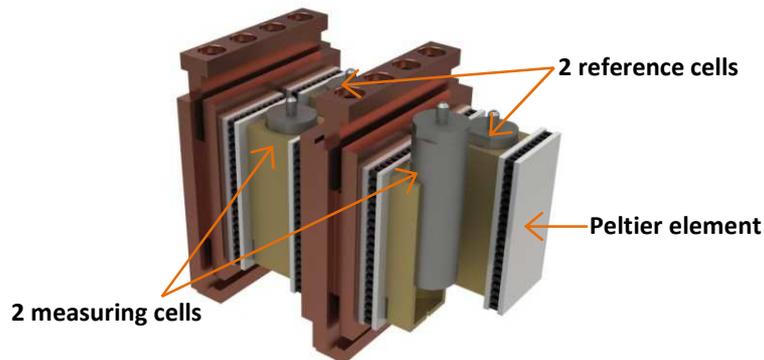


Figure 2 – The four wells of the  $\mu$ SC

### Experimental

Two PCM samples have been cycled in order to check their thermal stability after several phase changing: a mixture of paraffins and a dicarboxylic acid (sebacic acid) [2].

**The DSC 131evo (Figure 1)** is a flexible and powerful Differential Scanning Calorimeter, with fast heating and cooling performances. It allows to perform up to 100 melting/crystallizations of samples in 24 hours.

**The  $\mu$ SC (Figure 2)** is a state of the art multi-cells micro-calorimeter, with a wide operating temperature range (from  $-40$  to  $200^\circ\text{C}$ ). The very high sensitivity of the  $\mu$ SC enables transition studies to be performed on small but still representative quantities of products and at very low programming speeds.

The dicarboxylic acid is heated and cooled between  $100$  and  $150^\circ\text{C}$  at  $10^\circ\text{C}\cdot\text{min}^{-1}$  by DSC131evo. On **Figure 3** are presented the 1<sup>st</sup>, 20<sup>th</sup>, 60<sup>th</sup>, 100<sup>th</sup>, 140<sup>th</sup> and 200<sup>th</sup> heating thermograms.

The mixture of paraffin is heated and cooled between  $-20^\circ\text{C}$  and  $50^\circ\text{C}$  at  $5^\circ\text{C}\cdot\text{min}^{-1}$  a hundred times using the DSC131evo. Then, using the 2 measuring wells of the  $\mu$ SC, a simultaneous analysis of a fresh sample and an aged one is done at  $0.05^\circ\text{C}\cdot\text{min}^{-1}$  (similar to the real condition of use in building's walls).



$\mu$ SC  
 $-40^\circ\text{C}$  to  $200^\circ\text{C}$

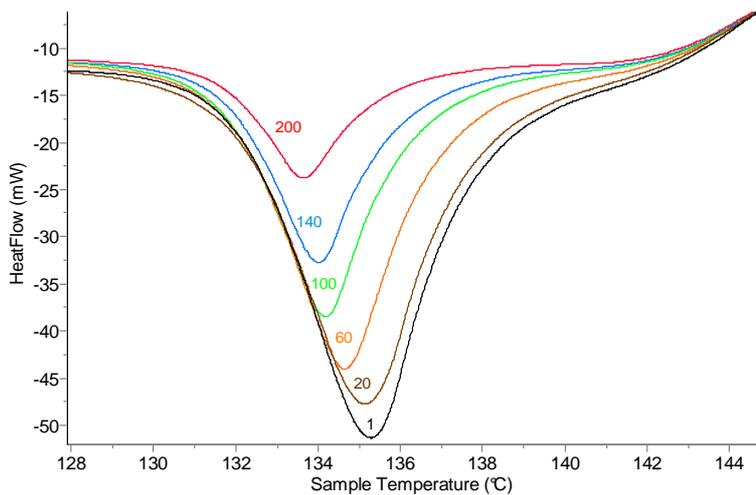
DSC131 Evo  
 $-170^\circ\text{C}$  to  $700^\circ\text{C}$



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## Results



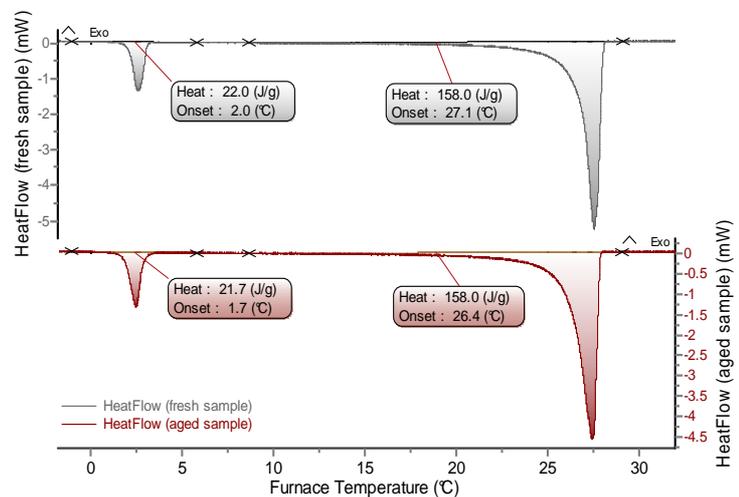
**Figure 3 – Superimposition of peaks linked to the melting of sebamic acid (numbers of cycling are indicated)**

The slow scan rate, and the two measuring wells of the microcalorimeter  $\mu$ SC allow to precisely obtain the latent heats of melting and crystallization of a fresh and an aged sample of paraffin .

A hundred successive phase changes did not affect the capacity of storage of this material, the latent heat of melting is still  $158 \text{ J.g}^{-1}$  (**Figure 4**). However, the difference in melting temperature observed is significant ( $0.7^\circ\text{C}$ ). It will be necessary to test on longer term (at least 1000 cycles) to ensure the effectiveness of this type of material over a very long period of time.

Experiments run with the DSC131evo on the sebamic acid show that there is a constant decrease of the latent heat of melting linked to the number of cycling done on this compound (**Figure 3**). Latent heat of crystallization presents a similar decrease (curves not showed here). This lost of energy is certainly due to a degradation of the sample at high temperature.

Due to its thermal instability, sebamic acid can not be considered as an interesting PCM.



**Figure 4 – Simultaneous analysis of a fresh and an aged sample of paraffins**

## Conclusions

Characterization of PCM could be done at low temperature rate using the new micro-calorimeter  $\mu$ SC; they can undergo an accelerated ageing using the DSC131evo in order to determine their cycling stability.

- [1] H. Mehling, L.F. Cabeza, *Heat and Cold storage with PCM – An up to date introduction into basics and applications*, Springer, 2008
- [2] D. Hailot, T. Bauer, U. Kröner, R. Tamme, *Thermal analysis of phase change materials in the temperature range 120-150°C*, *Thermochimica Acta* 513,p.49-59, 2011, Elsevier



$\mu$ SC  
-40°C to 200°C

DSC131 Evo  
-170°C to 700°C



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[sales@setaram.com](mailto:sales@setaram.com)

