Melting and Crystallization of Phase Change Materials (PCM) by Microcalorimetry

INTRODUCTION

A phase change material (PCM) is a substance with a high heat of fusion. Through melting and solidifying at a certain temperature, it is capable of storing and releasing large amounts of energy (see fig 1). Among other energy savings related applications, PCMs are used to design more efficient insulation systems. Indeed, these substances can be encapsulated in building materials such as bricks, or other wall, flooring, roofing, and ceiling materials. By successive melting and crystallization, they can dampen the effects of the day / night outside temperature variations and help maintain acceptable indoor temperatures.

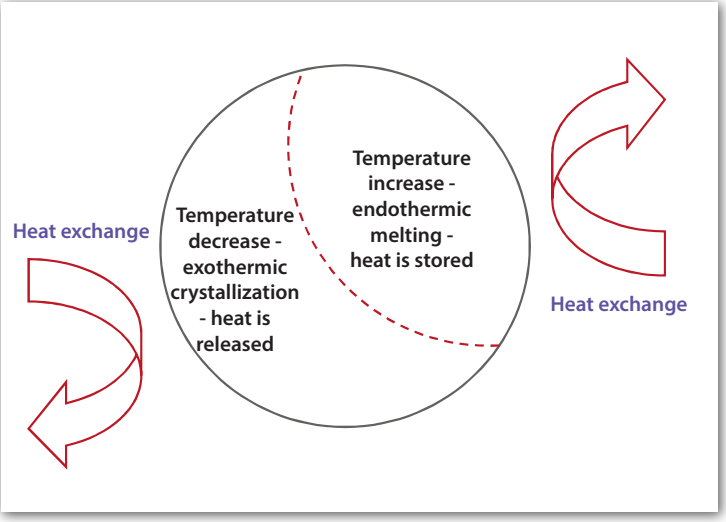


Figure 1 – Principle of a phase change material

Fatty acids, paraffins, organic substances, or inorganic salts can be used if they have high enough latent heat of melting and the temperatures of phase changes are adapted to the application. Moreover, the sensible heat, or the heat capacity change over the temperature range of the phase change being considered can play an important role. Of course, those relevant thermal properties have to be precisely known and with high accuracy. Using the MICROCALVET with the capability of applying very low scanning rates for a better simulation of the process is very well suited for such studies.

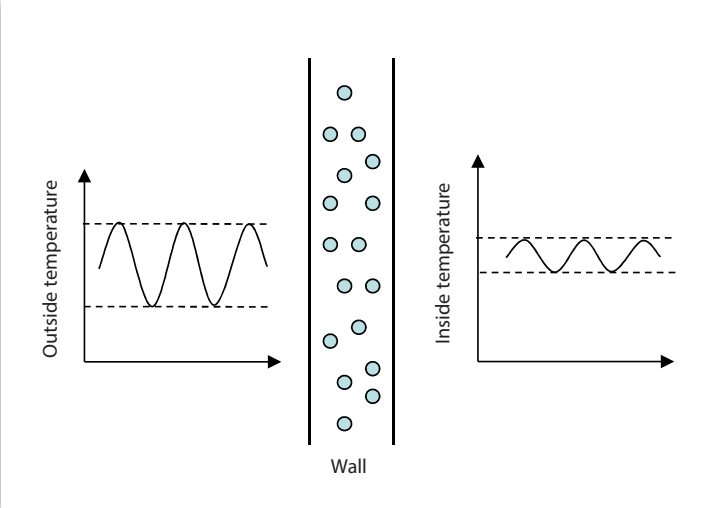


Figure 2 – Application of PCM in the building industry

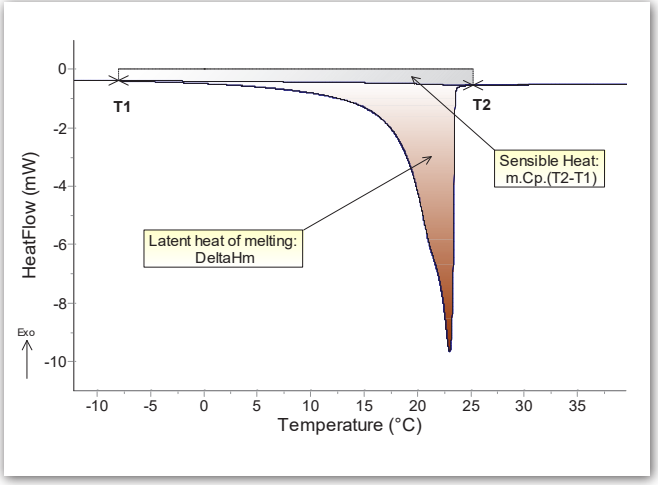


Figure 3 – DSC melting profile of a PCM

Property	Organic Paraffin	Organic Non-Paraffin	Inorganic Salt Hydrate	Inorganic Metal Eutectic
h_f (kJ/kg)	230 - 290	120 - 240	170 - 340	30 - 90
h_v ([J/m^3] x 10^6)	190 - 240	140 - 430	250 - 660	300 - 800
ρ (kg/m^3)	~ 810	900 - 1800	900 - 2200	~ 8000
k(W/m°C)	~ 0.25	~ 0.2	0.6 - 1.2	~ 20
Thermal Expansion	High	Moderate	Low	Low
Congruent Melt	Yes	Some Do	Most Do Not	Yes
Supercool	No	No	Most Do	No
Corrosion	Low	Some Are	Highly	Some Are
Toxicity	No	Some Are	Highly	Some Are

Table 1 – Thermal and other properties of common groups of PCM (adapted from [1])

EXPERIMENT

The sample is a homogeneous blend of polyolefins with the shape of soft beads. It weights 90.39mg which corresponds to about 10 beads. It is heated from -20°C to 50°C and then cooled from 50°C to -20°C at 1K/min and 0.04 K/min.

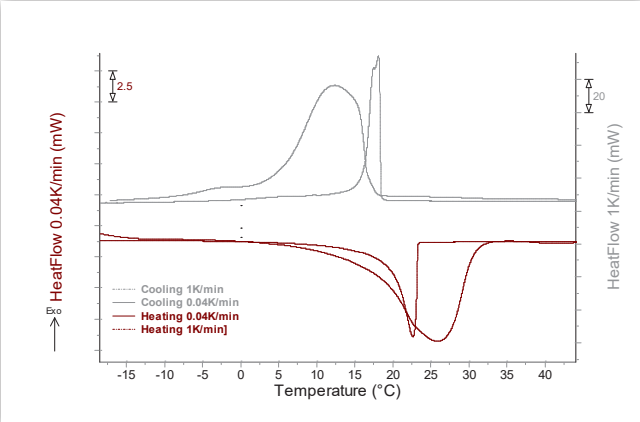


Figure 4 – Experimental DSC melting and crystallization profiles of the tested PCMs at 0.04K/min and 1K/min

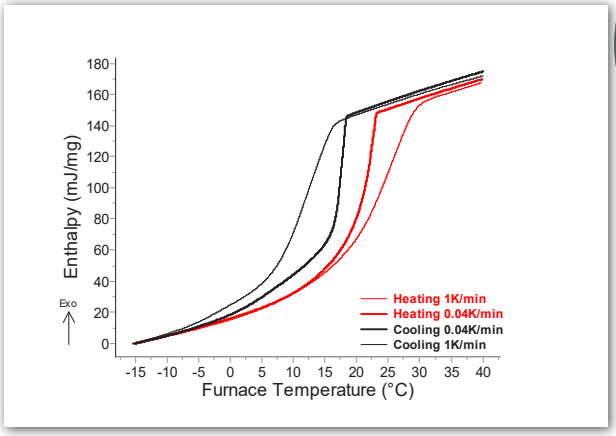


Figure 5 – Enthalpy variations of the tested PCMs during heating and cooling at 0.04K/min and 1K/min

RESULTS AND CONCLUSION

HeatFlow = f(Temperature) and derived enthalpy variation curves are showed in Figures 4 and 5. Latent heat, onset, offset and peak maximum temperatures are indicated in Table 2. Temperatures at which 90% of the product is crystallized (T90) during cooling operations is also indicated in Table 2.

The µDSC 3D sensor can handle large samples and odd shapes. It can scan temperatures at extremely low rates, which is representative of real conditions PCMs are used in, and approach thermodynamically stable conditions necessary for the determination of the heat stored at any temperature [2].

Conditions	T onset (°C)	Tmax (°C)	T offset (°C)	Latent Heat (J/g)	T_{90} (°C)
Heating at 0.04K/min	19.76	22.63	23.27	108.92	N/A
Cooling at 0.04K/min	18.43	18.11	16.21	-108.79	5.61
Heating at 1K/min	16.95	25.85	30.64	105.35	N/A
Cooling at 1K/min	16.87	12.34	4.55	-111.87	1.91

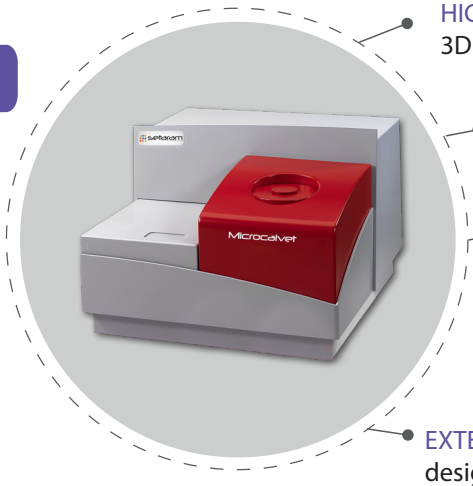
Table 2 – Results

[1] O'Conner, J., and Weber, R., "Thermal Management of Electronic Packages Using Solid-to-Liquid Phase Change Materials", ISPS. Proceedings, pp. 72 – 80, 1997.
[2] H. Mehling, H.-P. Ebert, P. Schossig, 7th IIR Conference on Phase Change Materials and Slurries for Refrigeration and Air Conditioning

INSTRUMENT

MICROCALVET

-45°C to 120°C



HIGHEST HEAT MEASUREMENT ACCURACY
3D sensor based on Peltier elements with Joule effect calibration.

MODIFIABLE TEMPERATURE CONDITIONS
for increased flexibility and replication of real life conditions.

CONVENIENT INTERCHANGEABLE CRUCIBLES AND CELLS
to perform even the most demanding experiments using one instrument :
- high pressure (1000bar) and high vacuum
- pressure measurement and control
- mixing experiment

EXTERNAL COUPLING CAPABILITY
designed to increase your research options including manometry, BET instrumentation, gas analyzers, humidity controllers and gas panels