Thermal Cycling 101

A thermal cycling study is designed to determine the cycling stability of a phase change material. During the study, a PCM goes through 10,000 or more cycles as quickly as possible to see if any thermal or chemical degradation is observed. The results help determine the useful lifetime of a PCM and its suitability for various applications.

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The purpose of a phase change material (PCM) is to store thermal energy for later use**.** These materials store energy by absorbing heat, which causes a phase change from solid to liquid to occur. When the material undergoes a phase change from liquid to solid, the energy that was previously stored is released. The ability of a PCM to perform consistently and reliably over numerous thermal cycles determines its viability in various applications. In this paper the importance of thermal cycling and how a study is conducted will be addressed.

What is a thermal cycle?

A thermal cycle is observed when a PCM undergoes a melt (solid to liquid) followed by a freeze (liquid to solid). During the melt, the PCM absorbs thermal energy from its surroundings. When it freezes, this energy is then released to its surroundings.

What is a thermal cycling study?

A thermal cycling study is an experiment designed to determine the cycling stability of a PCM. During a thermal cycling study, a PCM goes through numerous (≥10,000) cycles as quickly as possible to see if any thermal or chemical degradation is observed.

What type of information does a thermal cycling study provide?

The results of a thermal cycling study help determine the useful lifetime of a PCM and its viability in various applications.

What quality criteria are assessed during a thermal cycling study?

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During a thermal cycling study, the phase transition temperature, latent heat and purity must be analyzed at each testing interval within the study. The thermal conductivity and change in mass may also be monitored.

How frequently are the quality criteria assessed during a study?

According to RAL[1], the frequency of testing is dependent on the total number of cycles targeted by the entire study. The RAL design allows for 4 to 10 testing intervals per study. For example, if the target was for 1,000 cycles to be reached, the PCM quality criteria would be tested every 250 cycles. The values obtained from each analysis are compared to the data obtained prior to the initiation of the thermal cycling study. Thermal cycling studies performed by Entropy on its PureTemp PCM line allow for the mandatory quality criteria to be assessed 5 times en route to 10,000 cycles. The PCMs were tested after 131, 500, 1,000, 5,000 and 10,000 cycles.

How is a pass or fail designated for the quality criteria being assessed?

Deviations within the limitations of the DSC being used to measure the latent heat and phase transition temperature are tolerated for this study. Maintenance of sample purity is the primary method used by Entropy to determine whether or not a PCM has been damaged during the cycling study. The tolerable degree of deviation for purity is ~2% from the initial measurement.

What apparatus is needed to conduct a thermal cycling study?

Typically, thermal cycling studies are conducted using equipment that has been designed to go through as many cycles as possible in the shortest period of time. Entropy uses a TestEquity Model 107 temperature chamber, shown above, to conduct thermal cycling studies.

How are temperature parameters chosen for a thermal cycling study and how long does it take?

The time required for a thermal cycling study is dependent on the temperature chamber. The duration of a cycle is determined by the time required for the soak/hold at the high and low temperatures to ensure that a complete phase

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transition is observed, and the time needed to transition between those temperatures.

The temperature parameters used for a thermal cycling study are similar to those used during DSC. The upper limit is set 25° C above the transition temperature of the PCM to ensure that the PCM melts completely. The lower limit is set 25° C below the transition temperature so that a complete freeze is achieved. In a typical temperature chamber used for Entropy's studies the PCM is allowed to soak/hold for up to 30 minutes at both the high and low temperatures. Depending on the ramp rate between these temperatures, a cycle will take a minimum of 72 minutes. This would require 500 days for a study over 10,000 cycles to be completed.

When and how are thermal cycling study results reported?

Thermal cycling study results are reported once the study is completed. The results are reported as a comparison of the interval quality criteria assessment results to the initial quality criteria results. If the results of the final interval testing are within the acceptable deviation limit for each quality criterion, the PCM is considered to have passed the thermal cycling study and is approved for the total number of cycles targeted by the study. If the results of the final interval testing are not within the acceptable deviation, then the PCM is considered to have failed the thermal cycling study for the targeted number of cycles and is approved for the previous number of cycles for which it yielded passing results.

References

[1] RAL (www.ral-guetezeichen.de) is the governing association responsible for issuing quality marks that regulate the quality and performance of various products produced and sold in Germany and throughout the European Union.

About the author

Dr. Aymara Albury joined Entropy Solutions in 2013 after earning her Ph.D. in synthetic organic chemistry from the University of Alabama. She earned a Bachelor of Science degree in chemistry at Alabama in 2007.

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