

Introduction

PTFE is a material that is widely used for its many qualities. This polymer exhibits two phase transitions at temperatures close to ambient. These correspond to the transition from triclinic to hexagonal crystal (around 20°C), and from hexagonal to pseudo-hexagonal crystal (around 35-40°C). These two transitions are particularly close in temperature. Their thermal signal is therefore difficult to observe by calorimetry, as the two exothermic peaks are often merged into a single peak that encompasses both transitions.

One of the classic ways of differentiating between two close peaks in differential scanning calorimetry is to reduce the heating or cooling rate. However, this operation also reduces the amplitude of the thermal signal measured. The sensitivity of the calorimeter can then become limiting to obtain usable thermograms.

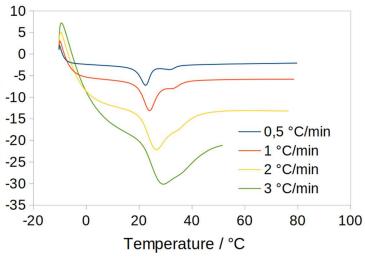
It is in this context that Calneos has developed the Ultimate Micro Calorimeter (UMC), which combines high heat flow sensitivity with a large sample volume of 750 microlitres. Samples are placed in removable and reusable stainless steel cells. One of the advantages of this new device on the market is the ability to create precisely controlled heating and cooling ramps at fairly high rate (up to 5~°C/min).

Experimental protocol

A 524 mg PTFE cylinder was placed in the measurement cell while the reference cell remained empty.

The Ultimate Micro Calorimeter was programmed to perform several heating ramps between -10°C and 80°C, at scanning rate of between 0.5 and 3°C/minute. The thermograms obtained are shown here after.

At all the rates, the two Teflon phase transitions were observed. The separation of the two transitions is better on the thermograms with the low scan rate.



Conclusion

The Ultimate Micro Calorimeter is an effective tool for studying phase transitions in materials. Its high sensitivity makes it easy to study very low-energy phenomena.

The Ultimate Micro Calorimeter has other types of vessel specifically adapted to measurements on liquids, mixtures, etc...

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