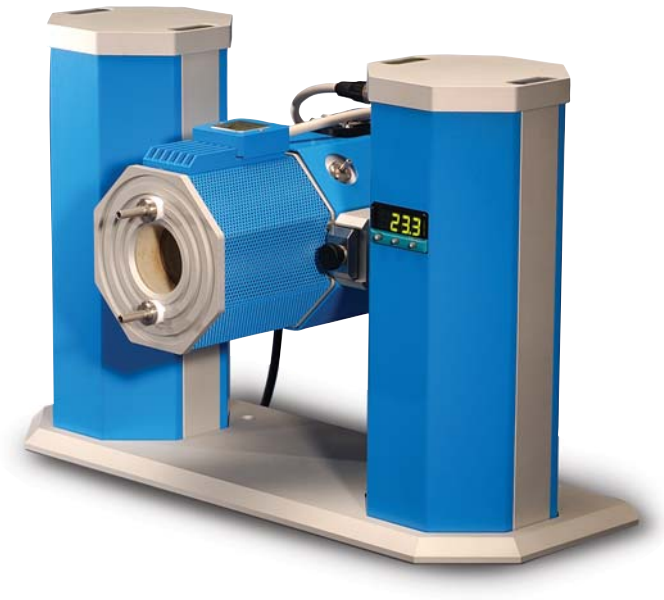
Tg Comparison of Material Pocket and Bar Sample of Polystyrene



Summary

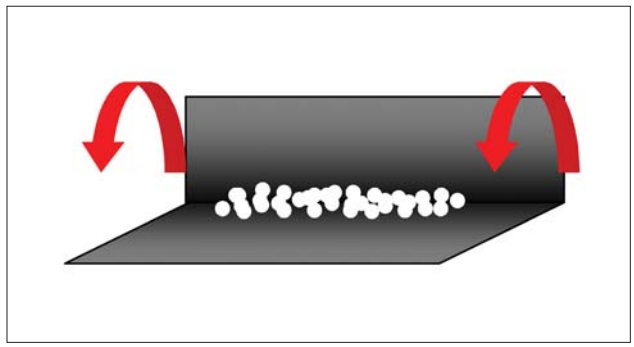
The PerkinElmer® Material Pocket has been proposed as a mechanism to support non-rigid materials within the PerkinElmer DMA 8000. It enables powders, flakes, films, liquids and semi-solids to be investigated. In order to have confidence in this as a sample support mechanism, a bar sample of polystyrene and a grated powder in a Material Pocket were analyzed and compared.

Introduction

Dynamic Mechanical Analysis (DMA) is one of the most appropriate methods to investigate relaxation events. The Material Pocket is a device to support non rigid materials in the DMA 8000. It is important to note that it can be proved that the Tg observed from a material in a Pocket is comparable to that from the same material without the Pocket present. To that end, polystyrene was chosen as a test material as it has a clear glass transition and can be formed into both a bar and can be powdered to go into a Material Pocket.

DMA works by applying an oscillating force to the material and the resultant displacement of the sample is measured. From this, the stiffness can be determined and the modulus and tan δ can be calculated. Tan δ is the ratio of the loss modulus to the storage modulus. By measuring the phase lag in the displacement compared to the applied force it is possible to determine the damping properties of the material. Tan δ is plotted against temperature and glass transition is normally observed as a peak since the material will absorb energy as it passes through the glass transition.

When analyzing data from a Material Pocket, it is not useful to look at the modulus value. The Material Pocket does not display any transitions in the temperature of interest as it is made from stainless steel. It does, however, include the modulus of the Pocket as a background signal. Although it is sometimes possible to subtract this out of the data, for most routine experiments, it is preferable to look at just the tan δ signal or changes in the modulus signal only.



PerkinElmer®
precisely.

Experimental

Temperature scan Polystyrene.
The sample was mounted in the Single Cantilever Bending clamps and run from ambient to 180 °C. The same was repeated for the Material Pocket sample.

Equipment	Experimental Conditions	
DMA 8000 1L Dewar	Sample:	Polystyrene (grated and bar)
	Geometry:	Single Cantilever Bending
	Dimensions:	(bar) 10.4 (l) x 4.9 (w) x 3.8 (t) mm
	Temperature:	30 °C to 180 °C at 5 °C min^{-1}
	Frequency:	1.0 Hz

Results and conclusion

Figure 1 shows the tan δ response for polystyrene both as a bar sample and as a powdered sample in the Material Pocket. The Tg is shown to be in agreement for both samples. This proves the use of the Material Pocket for investigating Tg effects in samples. It should be noted that the magnitude of signal is different between the two samples. This is to be expected due to the effective dilution of the signal by the stiffness of the Material Pocket. The pocket contained about 20 mg of polystyrene, whereas the bar was significantly larger. Despite the reduction in magnitude, the Tg values overlap exactly.

The storage (E') and loss (E'') modulus for the two samples is shown in Figure 2. As discussed in the introduction, it is not possible to use these data to quantitate the glass transition temperature due to the influence of the Material Pocket to the data. It is possible to see similarities in the data however and conclude that the material studied was the same for both experiments.

This application note has demonstrated the ability of the Material Pocket to be able to precisely determine the glass transition temperature of a material. Excellent agreement is seen between a grated sample and a bar sample of polystyrene.

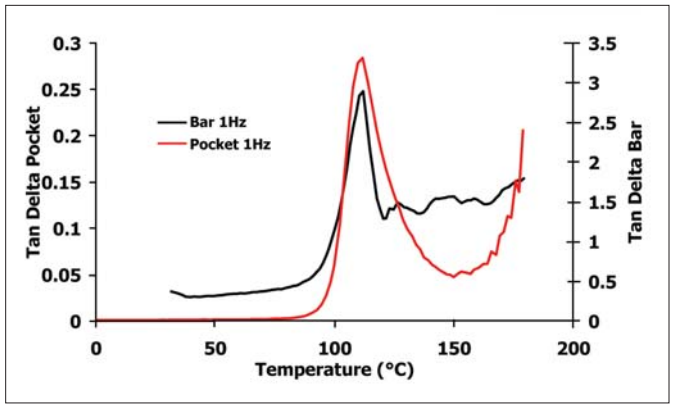


Figure 1. Tan δ response for polystyrene in both bar and powdered samples.

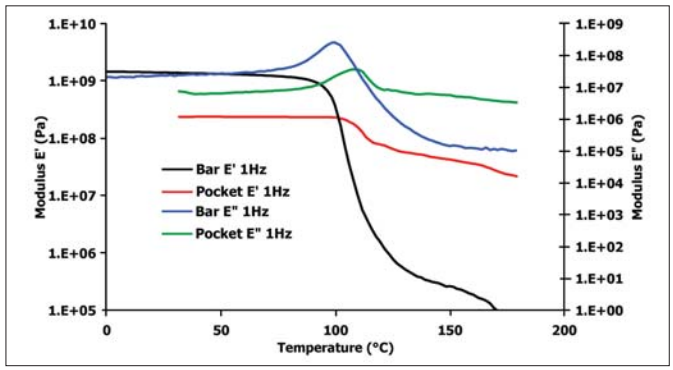


Figure 2. Storage and loss modulus for polystyrene in both bar and powdered samples.

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