APPLICATION NOTE

Thermal Analysis

Tg of Glycerine using a Stainless Steel Gauze Scaffold

Summary

Determination of the glass transition temperature of viscous liquids is a challenge by mechanical methods. Traditionally, it is performed using Shear geometry, but this can be problematical with the material liable to flow at room temperature. An alternative is demonstrated in this application note by the use of a gauze scaffold to support glycerine. The glass transition is easily identified and correlates to data from a DSC experiment.

Introduction

Dynamic Mechanical Analysis (DMA) is one of the most appropriate methods to investigate relaxation events. When the sample is a viscous liquid at room temperature, often the only choice is to use a Shear geometry. This can be difficult due to the flow of the material and also the large stiffness range when cooling through the glass transition. By using a stiff scaffold to hold the viscous liquid, it is possible to investigate the material in bending mode. This is a similar principle to the Material Pocket concept. As long as the scaffold itself has no transitions in the temperature range of interest, any relaxation events from the PerkinElmer® DMA 8000 can be attributed to the viscous liquid.

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.

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Glycerine was chosen to demonstrate this technique as it is freely available and shows a clear glass transition. The support chosen was a piece of stainless steel (SS) gauze or mesh with a relatively large mesh size (about 1 mm). Surface tension held the glycerine in place sufficiently to perform the measurement. A DSC scan was also run on the same material to compare the glass transition data between the two techniques.

Experimental

1. Temperature scan of glycerine by DMA.

A sample of glycerine was added to the SS gauze scaffold and allowed to penetrate fully. The gauze was then mounted in the DMA 8000 in Single Cantilever Bending geometry and cooled to -150 °C before starting the experiment.

2. Temperature scan of glycerine by DSC.

A sample of glycerine was placed in a crimp seal pan and run in the DSC at 10 °C min^{-1}.

Equipment	Experimental Conditions	
DMA 8000 1L Dewar	Sample:	Glycerine
	Geometry:	Single Cantilever Bending
	Dimensions:	5.0 (l) x 3.0 (w) x 1.0 (t) mm
Shimadzu® DSC60A	Temperature:	-150 °C to 100 °C at 4° C/min^{-1}
LN2 cooling	Frequency:	1.0 and 10 Hz

Results and conclusion

Figure 1 shows loss modulus plotted against temperature. As glycerine goes through the glass transition, a peak is observed and it is shown that this is frequency dependant. The actual value of the loss modulus is from the glycerine and stainless steel gauze but the peak can be attributed solely to the glycerine. Stainless steel shows no transitions in this range. The glass transition temperature taken from the peak in the loss modulus is between -60 °C and -70 °C.

A heat flow against temperature plot from a heat-flux DSC is shown in Figure 2. A clear glass transition is observed through the baseline shift. The transition temperature under the scan conditions is around -70 °C which corresponds to the loss modulus peak values shown.

Although it is not expected to get exactly the same glass transition temperature from both techniques (the Tg is very dependant on how it is measured and in the DMA 8000 greatly depends on the frequency), the loss modulus peaks from the DMA and the slope change onset from the DSC are very similar. DMA Tg values are normally higher than those of DSC, as shown.

It has been demonstrated that DMA can be used to determine glass transition temperatures of room temperature viscous liquids by using a supporting gauze scaffold. The experimental technique is much easier than a Shear experiment and very accurate data can be achieved.

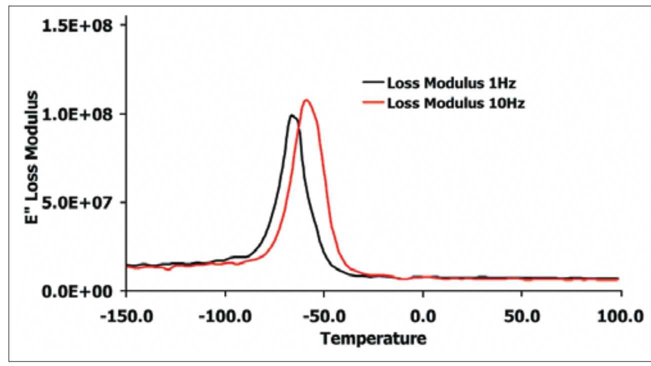

Figure 1. Loss modulus vs. temperature.

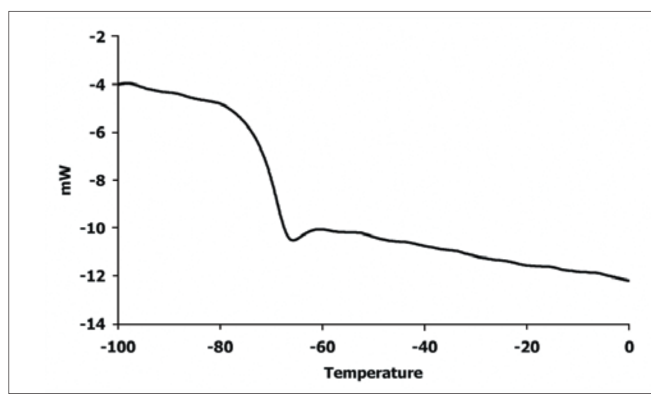

Figure 2. Heat flow vs. temperature.

PerkinElmer, Inc.
940 Winter Street
Waltham, MA 02451 USA
P: (800) 762-4000 or
(+1) 203-925-4602
www.perkinelmer.com

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