APPLICATION NOTE

THERMAL ANALYSIS

Glass Transition Measurement of an Adhesive Film in a Material Pocket

Summary

This application note demonstrates the ability of the PerkinElmer® DMA 8000 to investigate the mechanical properties of an adhesive, without the need to fabricate a self supporting film. The Material Pocket is used for this experiment and shows that this method of support can be used for film materials as well as powders.

Introduction

The glass transition (Tg) is a key process in any material, and can be observed with ease by DMA. This technique provides very revealing information about relaxations through the tan δ vs temperature data. The problem with measuring the glass transition of an adhesive by DMA is that traditionally, the material is either placed in a shear geometry or a self supporting film is required for running in tension. The problem of the shear experiment is that adhesives can be too stiff for this type of geometry. The problem with the self supporting film is that this can be very difficult to make, prepare for DMA measurement or obtain as a reproducible thickness. The alternative, proposed here, is to use the PerkinElmer Material Pocket to hold the adhesive in the instrument.

Experimental

DMA temperature scan of adhesive.
A small quantity of Super Glue was placed into the Material Pocket, folded and allowed to cure for a number of hours. It was then mounted into the DMA in Single Cantilever Bending geometry. 1 Hz and 10 Hz data were collected over the full temperature range.

Equipment	Experimental Conditions	
DMA 8000 Material Pocket	Sample:	Commercial branded Super Glue
	Geometry:	Single Cantilever Bending
	Support:	Material Pocket
	Frequency:	1 and 10 Hz
	Temperature:	Ambient to 200 °C at 5 °C min^{-1}

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. The Material Pocket is simply a piece of stainless steel that the adhesive is applied to and then the steel is folded to make a pocket. After curing, the pocket with adhesive inside can be run in the DMA. The pocket will give a background stiffness in the experiment so the modulus values are not useful, but there are no phase transitions in the temperature of interest so the tan δ response will be from the adhesive alone.

Results and conclusion

Figure 1. shows the tan delta response from the Super Glue sample. The glass transition is observed as a peak and is seen to be frequency dependant indicating a relaxation process. As this result was obtained in the Material Pocket, the observed modulus is not plotted as the main contribution was from the pocket and not the adhesive.

The sample preparation for this experiment was very simple and did not require the production of a self supporting film. In addition, the experiment was performed in the simplest geometry available and negated the need to try and obtain a shear result which is very difficult for these stiff adhesives.

The data demonstrate that the PerkinElmer Material Pockets can be used for adhesive film materials as well as powders. This was a very simple experiment and did not require expert knowledge to get the glass transition temperature. The glass transition temperature is a very important parameter for adhesives as the mechanical properties alter significantly if the adhesive enters its rubbery region rather than remaining as a glass. Often this results in failure above the Tg.

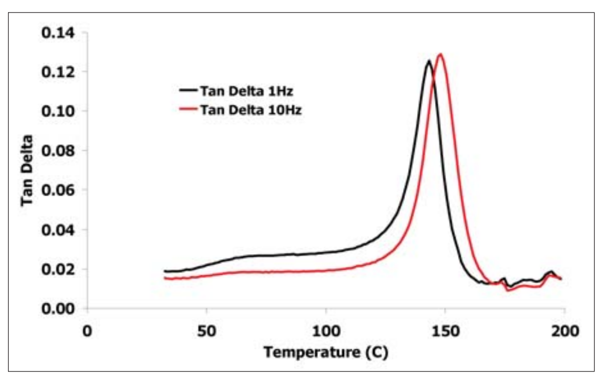

Figure 1. Tan δ response from Super Glue.

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