Amorphicity and Crystallization of PEEK

Summary

This application note describes a simple experiment on an amorphous polymer. PEEK (polyetheretherketone) is a common polymeric material used for a variety of applications. Normally it is supplied as an amorphous material. This material, when examined in a PerkinElmer® DMA 8000, shows classic relaxation behavior with a sharp and defined glass transition. The material also displays a recrystallization after passing through the Tg. On cooling, the size of the Tg is greatly decreased indicating that a large proportion of the material is now crystalline.

Introduction

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 tan δ can be calculated. Tan δ is the ratio of the loss component to the storage component. 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.

Monitoring recrystallization events in the DMA is relatively simple. On passing through a Tg, there is sufficient mobility in the molecules to allow recrystallization to occur. This produces dramatic effects in the DMA response. In addition, if the material is then cooled, the cooling Tg can be obtained. If this is smaller than in the increasing temperature ramp, then it is proof that a recrystallization event has occurred. The data in this note demonstrates this phenomenon.

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Experimental

Temperature scan on PEEK

The sample was supported in the tension clamps and the temperature allowed to equilibrate.

The sample was scanned to 220 °C and held isothermally at this temperature for 10 minutes.

The down scan was then initiated and data collected until the temperature reached 100 °C.

Equipment	Experimental Conditions	
DMA 8000	Sample:	PEEK
	Geometry:	Tension
	Dimensions:	9.5 (l) x 4.2 (w) x 0.3 (t) mm
	Temperature:	25 °C to 220 °C to 100 °C at 5 °C min^{-1}
	Frequency:	1 and 10 Hz

Results and conclusion

Figure 1 shows the frequency data for the up ramp (increasing temperature) of PEEK.

The glass transition is identified as the peak in the tan curve and the large drop in the modulus curve. Both data show a frequency dependence as expected. After the Tg, the modulus curve starts to increase which is as a result of the recrystallization process. Crystalline PEEK is more stiff than amorphous PEEK. The tan δ curve shows a broad curve at this point. Note that there is no frequency dependence during this event indicating that this is not a relaxation event.

Only the 1 Hz data for clarity is shown in Figure 2. Added to the up scan data is the down scan where the temperature was decreased after the initial thermal ramp. The part of the data assigned as the recrystallization event is not replicated on the down scan indicating an irreversible process. Also, the size of the glass transition is much smaller in the tan δ response. This reduction in the size of the Tg is indicative of a reduction in the amount of amorphous material in the sample on the down scan. This again supports the idea that there has been a recrystallization in the sample between the two scans. The fact that a Tg can be observed at all indicates that the recrystallization process was not complete and that some amorphous material was left in the sample.

This application notes has demonstrated the ability of DMA to investigate both relaxation events and recrystallization in a polymeric sample. By utilizing a downward temperature ramp after an initial heating it is possible to confirm the up scan data and evaluate the degree of crystallization in the sample that is below.
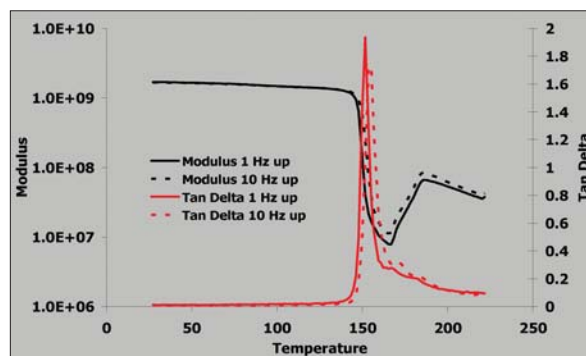

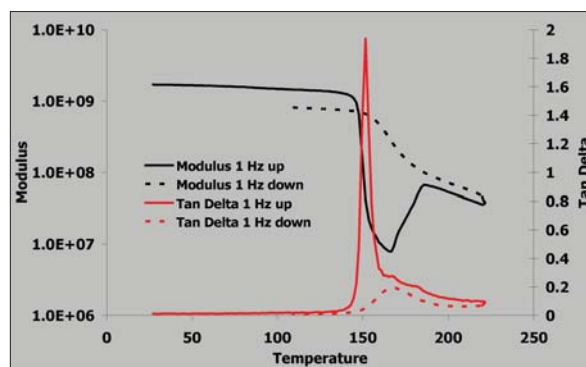
Figure 1. Modulus and tan δ of PEEK at two frequencies.

Figure 2. Modulus and tan δ in heating and cooling.

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