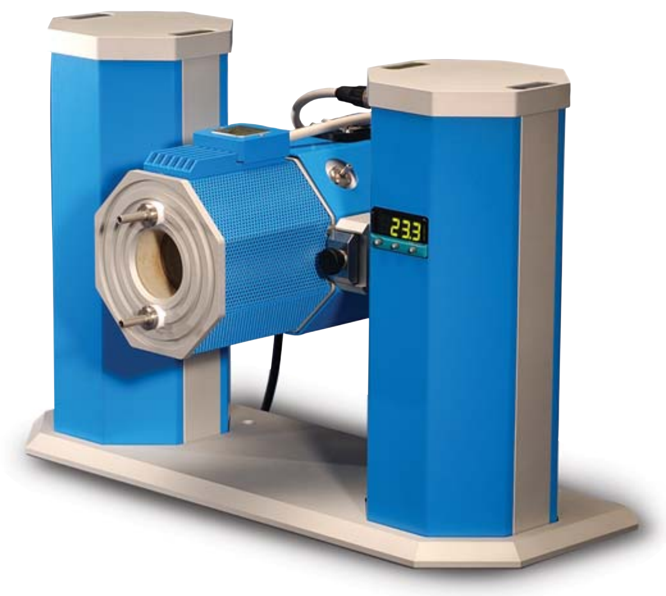
APPLICATION NOTE

THERMAL ANALYSIS

Characterization of LDPE Over a Large Frequency Range



Summary

LDPE (Low Density Poly Ethylene) is an important polymer used in the manufacture of plastic products. This application note details DMA experiments over a temperature and frequency range. The combination of frequency and temperature are important factors for the practical application of LDPE. Relaxations are frequency dependant, so they will occur at different temperatures depending on the distortion frequency the material is subjected to. A temperature scan of the material will be discussed, as will a frequency sweep at a series of discrete temperatures.

Introduction

Dynamic Mechanical Analysis (DMA) is one of the most appropriate methods to investigate relaxation events. The PerkinElmer® DMA 8000 is capable of measuring data over a very large frequency range (up to 300 Hz in all geometries). When investigating the frequency dependant behavior of materials, this high frequency range is very useful. The temperature dependence of the material is also important so a temperature scan will also be displayed with these data. LDPE is semi-crystalline and it has been reported that the δ relaxation event is associated with a crystalline phase relaxation.

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.

Results and conclusion

Figure 1 shows the temperature scan data at three frequencies for LDPE. The large increase in tan δ and decrease in modulus at around 100 °C is the onset of melting. Note, just prior to the melting event, there is a frequency dependence. As melting behavior is not frequency dependent, this event has to be a relaxation quite separate from melting. As mentioned in the introduction, it has been reported that there is an δ relaxation associated with the crystalline phase in this temperature range. The data here confirms this.

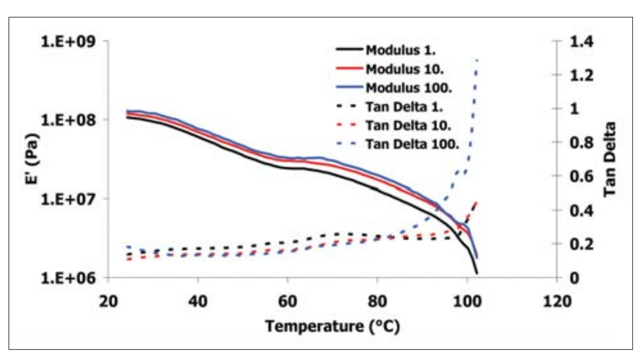


Figure 1. Temperature data at three frequencies.

Experimental

1. **Temperature scan of LDPE.**
   The LDPE sample was mounted in the Single Cantilever Bending clamps and run from ambient to melting temperature. Data was collected at three frequencies.

2. **Frequency scan of LDPE at five temperatures.**
   The LDPE sample was mounted in the Single Cantilever Bending clamps. The temperature was stepped from 0 °C to 100 °C in 25 °C increments. A series of discrete frequencies were investigated at each temperature.

3. **Isothermal frequency scan of LDPE (30 discrete frequencies).**
   The LDPE sample was mounted in the Single Cantilever Bending clamps and run at ambient temperature at multiple frequencies.

Equipment	Experimental Conditions	
DMA 8000 1L Dewar	Sample:	LDPE
	Geometry:	Single Cantilever Bending
	Dimensions:	5.0 (l) x 3.6 (w) x 5.0 (t) mm
	Temperature:	0 °C to 100 °C in step isothermal mode
	Frequency:	0.1 to 300 Hz

Figure 2 shows a more comprehensive frequency sweep of LDPE run at 22.5 °C. There is a general trend of decreasing tan δ as frequency is increased and the modulus remains approximately constant.

Figure 3 shows frequency data at a range of temperatures. Fairly linear relationships are observed for tan δ and modulus which is to be expected if no relaxation event is present. At 100 °C this relationship changes and tan δ increases with frequency. This result further supports the theory of a relaxation event at around 100 °C.

Note, the distortion in the data at 250 Hz corresponds to the resonance frequency of the sample. The instrument was able to continue with the frequency sweep through resonance to collect the full range of data.

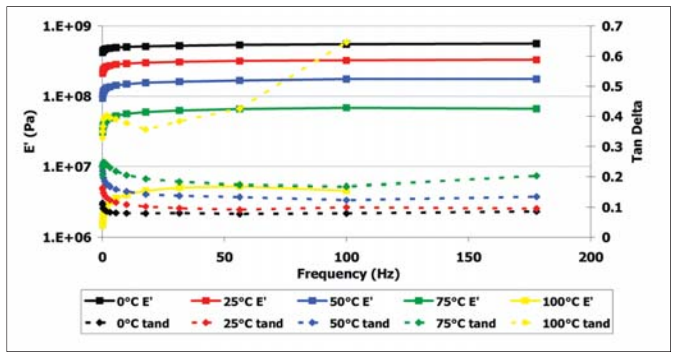


Figure 2. Frequency data at various temperatures.

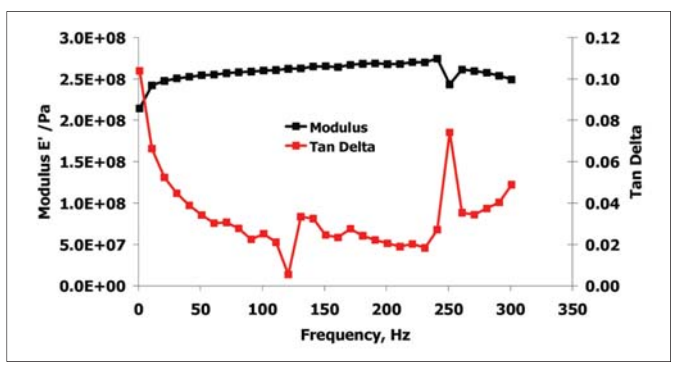


Figure 3. LDPE run at 22.5 °C.

PerkinElmer Life and Analytical Sciences
710 Bridgeport Avenue
Shelton, CT 06484-4794 USA
Phone: (800) 762-4000 or
(+1) 203-925-4602
www.perkinelmer.com

For a complete listing of our global offices, visit www.perkinelmer.com/lasoffices

007771_13 Printed in USA