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 $\delta$ 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 $\delta$ can be calculated. Tan $\delta$ 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 $\delta$ 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.
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 $\delta$ relaxation associated with the crystalline phase in this temperature range. The data here confirms this.

Figure 2 shows a more comprehensive frequency sweep of LDPE run at 22.5 °C. There is a general trend of decreasing $\tan \delta$ 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 $\tan \delta$ and modulus which is to be expected if no relaxation event is present. At 100 °C this relationship changes and $\tan \delta$ 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.

### Results and conclusion

Figure 1 shows the temperature scan data at three frequencies for LDPE. The large increase in $\tan \delta$ 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