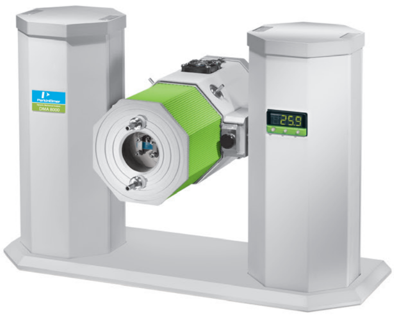
APPLICATION NOTE

Thermal Analysis

Dynamic Mechanical Analysis is of great importance to characterize and ensure optimal compound properties.

Material Characterization and Performance Assessment of Polybutadiene Rubber using Multifrequency Analysis by DMA



Introduction

Polybutadiene (butadiene rubber BR) is a synthetic rubber and one of the cheapest and largest-volume synthetic general-purpose elastomers which is sometimes used as a substitute for natural rubber. Polybutadiene has excellent abrasion resistance (good tread wear), low hysteresis loss, high elasticity, and low rolling resistance due to its low glass transition temperature (Tg). To optimize performance such as traction, rolling and abrasion resistance, it is typically compounded with other elastomers such as natural rubber and SBR.

Polybutadiene rubbers are used for the fabrication of car tires, wiper blades, gaskets, and other products where the ability to deform and return to the original shape are important. The glass transition temperature is of vital importance to both characterize the material and assess the performance of the final product. Dynamic Mechanical Analysis (DMA) is of great importance to characterize and ensure optimal compound properties.

The characterization of polybutadiene will be demonstrated in this application note. The glass transition temperature is very important when considering the material for various applications. Being a rubber, the modulus and the damping properties change dramatically on passing from the glassy to the rubbery state. Dynamic Mechanical Analysis will show the extent of the change of both the modulus and tan δ as the material goes through the glass transition. It will also characterize the glass transition temperature very precisely.

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Experimental

DMA is one of the most appropriate methods to investigate relaxation events. When the sample to be measured is rubbery at room temperature, this presents challenges to the experimenter in terms of clamping and to the instrument in terms of the stiffness range of the sample going from a glassy to a rubbery state. Rubbery samples are normally examined in either a shear or a bending geometry. This note examines single cantilever bending and how the unique design of the PerkinElmer DMA 8000 allows one to overcome experimental difficulties.

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.

Multifrequency temperature scan of polybutadiene. The sample was lightly mounted in the single cantilever bending clamps and cooled to approximately -150 °C (to ensure the sample was glassy). The clamps were tightened and the oven replaced to continue the cooling down to -170 °C.

Table 1: Need info.

Equipment	Experimental Conditions	
DMA 8000 1L Dewar	Sample:	Polybutadiene
	Geometry:	Single Cantilever Bending
	Dimensions:	7.5 (l) x 10 (w) x 2 (t) mm
	Temperature:	-170 °C to 50 ˚C at 3 ˚C/min^{-1}
	Frequency:	0.1, 1.0 and 10.0 Hz

Results and Conclusion

Figure 1 shows the glass transition of this material as a peak in the tan δ and a drop in storage modulus. As stated in the experimental section, the sample was clamped only after it had reached a glassy state. This avoids spread in the clamps and makes the measurement more accurate. The DMA 8000 is designed so that the oven can be removed from the instrument very quickly to allow reclamping. This feature prevents significant condensation of water on the surface of the sample and also avoids the sample returning to a rubbery state before it was possible to tighten the clamps. Also, it should be noted that this experiment was started at -170 °C (the instrument can easily cool to below -190 °C) demonstrating that this is an ideal instrument for investigating very low temperature relaxation events (low Tgs or δ relaxations for example).

The data also shows a drop in storage modulus of two orders of magnitude. This is well within the instrument parameters and means that experiments where the stiffness of a material changes dramatically can be examined in a DMA 8000 in one experiment over the entire temperature range of interest.

The glass transition is shown to be very sharp and frequency dependant as expected. Also, the tan response is well above zero even after the glass transition temperature has been passed. This demonstrates that this material has significant damping properties when in the rubbery state.

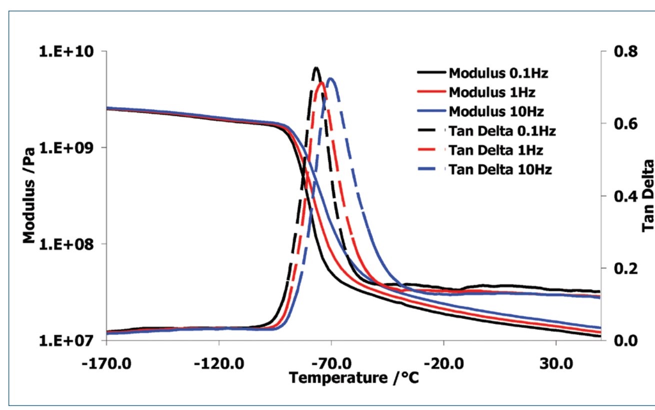

Figure 1: Glass transition of polybutadiene rubber.

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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