Detection of Adhesive Layer Between Polyester Films Using DMA

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ABSTRACT

Dynamic mechanical measurements are performed on bi-layer films containing an adhesive between the two layers. A comparison of the dynamic mechanical analysis (DMA) results for the pure film with those of bi-layer samples with different adhesives, shows a difference in the thermal curve attributable to the glass transition of the adhesive.

INTRODUCTION

A linearly increasing temperature is one of the most commonly performed experiments using the DMA instrument. Such experiments involve sinusoidally deforming the sample at a constant frequency and small amplitude while linearly heating the sample. The viscoelastic properties of the sample are continuously evaluated during the temperature program. Small amplitude oscillatory experiments are non-destructive and enable the separation of the elastic and viscous components of the overall sample response and properties such as modulus, compliance and viscosity (*1, 2*). For solid materials, the modulus has an elastic or storage component (related to the ability of the material to store energy) and a loss component (related to the energy dissipative characteristics and is responsible for mechanical damping and internal friction) (*2*). Just like the overall modulus, the storage modulus (E') and the loss modulus (E") change with temperature.

For amorphous or semi-crystalline polymers, at very low temperatures, large-scale motions of an amorphous phase chain backbone are completely restricted and the bulk polymer is rigid and glassy (*2, 3*). As the temperature is increased, the thermal energy exceeds the potential barrier for chain motion. This marks the beginning of the glass transition region. Thus, the glass transition is associated with the cooperative segmental mobility within the amorphous phase of the polymer (*4*). Associated with this phenomenon are a large drop in the storage modulus and a large peak in tan δ, the damping characteristic. (Tan δ is the ratio of loss to storage modulus.)

Below the glass transition temperature, some polymers exhibit peaks in tan δ. These secondary transitions are attributed to local mode relaxations, crankshaft and kink motions, rotations of terminal groups and other side chain motions (*1*). Polyethylene terephthalate (PET) is known to have a broad secondary peak around –50 °C (*2*) believed to be due to small amplitude torsional oscillations and other vibrational motions of chain segments (*1*).

Adhesives usually have lower molecular weight than structural polymers. Hence, their glass transition is lower than the PET films that sandwich the different adhesives.

EXPERIMENTAL

The samples consist of two films of PET with a layer of adhesive between them. There are two such samples, each containing the same grade of PET, but different adhesives. Two multilayer films each with a different adhesive are compared with the "pure" PET film. Thus, there were 3 samples with the following characteristics:

1. Control : single PET film, no adhesives
2. Sample A : PET/Adhesive 1/PET film
3. Sample B : PET/Adhesive 2/PET film

Tension-film clamps were used with the Q800 DMA. The samples are loaded with care to ensure that there are no twists or folds. The load was added to the samples so that no pulsation of the sample was observed when viewed from the side. The instrument determines the exact length of the sample when the experiment is initiated. The sample is heated from –150 to 200 °C at 3 °C/min with a frequency of 1 Hz an amplitude of 20 μm.

RESULTS AND DISCUSSION

Figure 1 shows the storage modulus (in the solid line), the loss modulus (in the dashed line), and tan δ (in the dot-dashed line) of the control sample changing as a function of temperature. Two runs were performed to estimate the repeatability of the measurement.

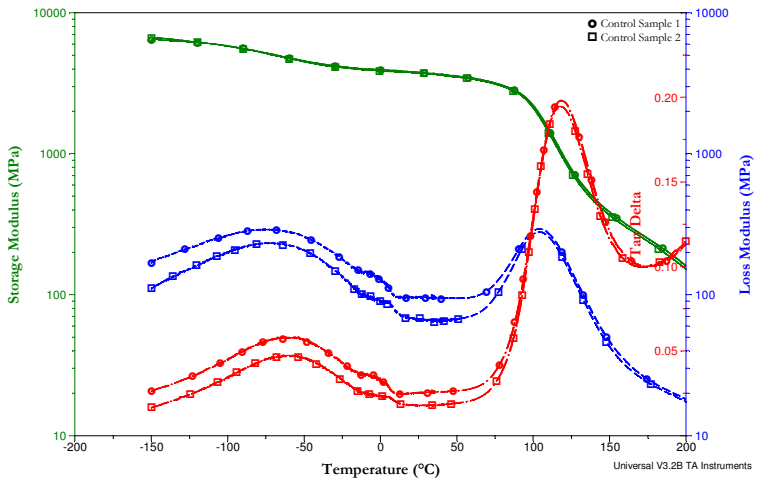

Figure 1 – DMA Curve of the Control PET Sample (no adhesive)

There seem to be two transition regions in Figure 1. The first of these is the glass transition near 120 °C and the second is the broad γ transition near – 70 °C characteristic of PET (*4,5*).

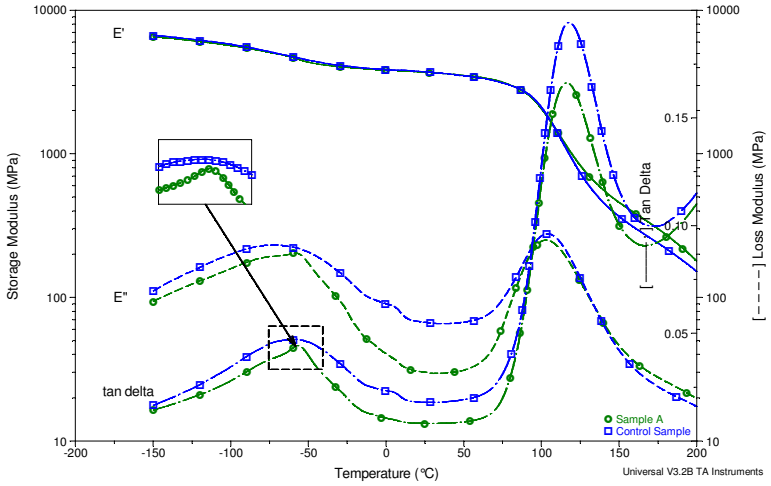

Figure 2 - Comparison of the PET Control and Sample A

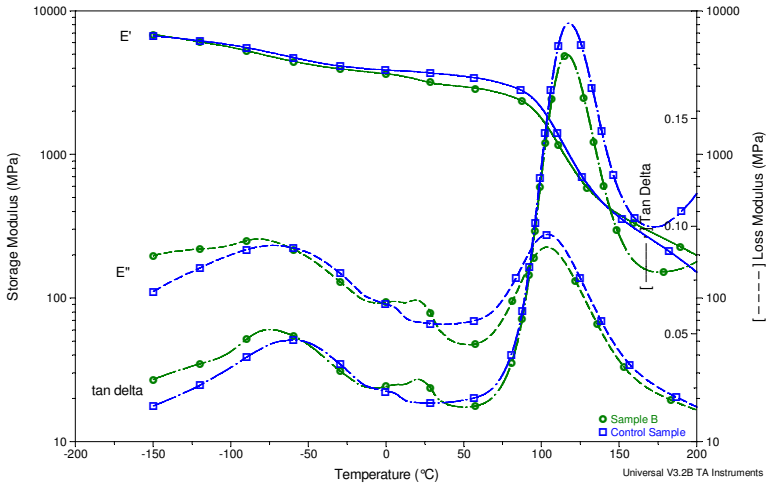

Figure 3 - Comparison of PET Control and Sample B

Figure 2 and Figure 3 show the temperature profiles of A and B samples respectively. A comparison with the control sample results in Figure 1, shows that there is a subtle tan δ peak in Sample A at –57 °C and one for Sample B at 19 °C. These peaks are also observed in the E" signal. These additional peaks are thought to be due to the adhesive layers sandwiched between the two PET films.

The storage modulus (E') extrapolated onset temperature, the loss modulus (E") and tan δ peak temperatures all used to indicate the glass transition temperature (T_g) at times. ASTM International Standard E1640 identifies the storage modulus onset temperature as the preferred indicator of glass transition (*6*). The following table shows the T_g of the three samples using different methods:

Table 1 - The Glass Transition Temperature of the Control, Sample A and Sample B, Using Different Signals

Sample	**E' Onset** (°C)	**E" peak** (°C)	**Tan(δ) peak** (°C)
Control	93.2	103.0	117.7
A	91.0	102.7	116.0
B	91.9	103.4	116.2

Since the adhesive does not interact with the PET chemically, the $T_{g,}$ does not change in samples A and B.

SUMMARY

The DMA is able to identify the glass transitions of a thin adhesive layers in the presence of a large excess of the adhered PET films. The glass transition temperatures are nearly 70 °C different for the two samples indicating that the laminated films are intended for different service.

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KEYWORDS

adhesives, dynamic mechanical analysis, films/fibers, glass transition, modulus, molecular spectrum, thermoplastic polymers

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