APPLICATION NOTE

Thermal Analysis

Better Characterization of Polymer Blends Using StepScan DSC

Introduction

Polymer blends are widely utilized for a variety of applications as the particular thermo-physical properties of the individual components comprising the blend result in better overall properties. This is particularly the case of polymer blends used in the automotive industry where impact resistance, toughness, stiffness and paintability all become important issues. The blending of polymers yields a better overall product with the desired end use properties and characteristics. Polymer blends are also used for container applications where there is a need for good impact resistance coupled with long term stability, barrier resistance and opacity.

With polymer blends, it can become difficult to characterize their physical properties, especially the glass transition event, as the components can interfere with one another or mask the other's presence. Additionally, the processing of the blends, via extrusion or injection molding, can lead to the occurrence of frozen-in stresses, which can make the interpretation of DSC results difficult. A large enthalpic relaxation peak or other interfering, irreversible thermal event may accompany the Tg's of the different polymer phases. All of these factors can make it difficult to observed the unique glass transition events of the polymer components in a blend.

One means of greatly improving data interpretation from DSC is using StepScan™ DSC from PerkinElmer.

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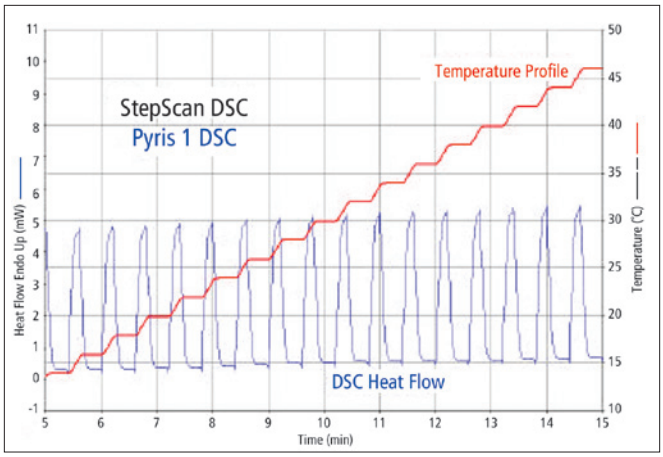

Figure 1. StepScan DSC approach using time-temperature profile.

StepScan DSC

StepScan DSC is software for the enhanced characterization of the thermal properties of materials. The technique permits the separation of DSC results into thermodynamic (reversible) and kinetic (irreversible) components for better interpretation. The method is straightforward and utilizes the traditional approach for measuring the heat capacity, Cp, for the highest possible reliability of results without interfering experimental problems. The StepScan DSC approach is only possible with the design of the power compensated Pyris™ 1 DSC, with its very low mass sample and reference furnaces and rapid response time.

Figure 1 shows the StepScan DSC approach with the application of a repetitive sequence of short heating – isothermal hold segments.

With the application of heating (10 °C/min) over small temperature increments (1.5 or 2 °C), and by holding for a short time interval (e.g., 30 seconds), the heat capacity that is yielded reflects the reversible aspects of the sample. Kinetic or irreversible effects (on the time scale of the experiment) are eliminated in the ***Thermodynamic Cp*** data set, which reflects 'fast' or reversible phenomenon, such as the sample's heat capacity (molecular vibrations) or Tg (molecular rotations). For example, if a sample has a Tg, with an overlapping enthalpic relaxation, moisture loss or crystallization event, the Thermodynamic Cp signal will show the classic, stepwise change in the heat capacity. This then makes it simple and straightforward to analyze and interpret. The StepScan DSC approach also provides the kinetic or ***IsoK Baseline*** data set, which is reflective of the irreversible or 'slow' processes taking place during the experiment. The enthalpic relaxation event, which can occur on physically aged samples at Tg, will show up in the IsoK Baseline data set.

Because the StepScan DSC approach requires rapid DSC response times, the technique is only feasible with the power compensated DSC, which allows for fast heating and thermal equilibration. The application of the StepScan approach to a large mass furnace, heat flux DSC instrument would be difficult or technically unfeasible due to the inability to rapidly respond and equilibrate. In addition, the StepScan DSC experiments are generally faster (by a three-fold improvement) as compared to equivalent TMDSC results generated on a slower responding, heat flux DSC device.

Polymer Blend Results by Standard DSC

Displayed in Figure 2 are the standard DSC results generated on a polymer tri-blend sample consisting of ABS, polycarbonate and amorphous PET. The sample was heated at a temperature rate of 10 °C/min.

From these results, the blend yields two Tg's: one at 80 °C for the amorphous PET component and one at 115 °C for the SAN (styreneacrylonitrile) component of the ABS polymer. The amorphous PET also yields a cold crystallization transition at 140 °C and the melting event at 255 °C. However, the Tg of the polycarbonate component, which should be around 155 °C, is not observed. The crystallization peak of the PET obscures the Tg of the polycarbonate component.

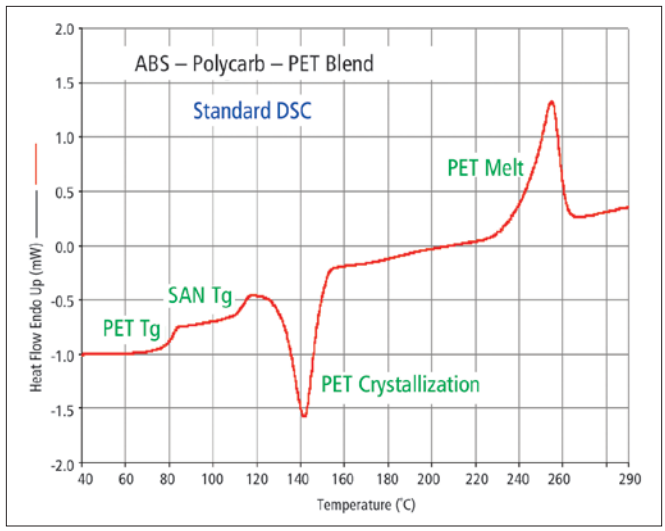

Figure 2. Standard DSC results obtained on polymer tri-blend of ABS, polycarbonate and amorphous PET.

StepScan DSC Results on Polymer Blend

The polymer tri-blend was analyzed using the StepScan DSC technique. The experimental conditions used are given in the following table.

Experimental Conditions	
Instrument	Pyris 1 DSC with StepScan DSC
Heating rate between steps	10 °C/min
Step isothermal hold time	20 seconds
Temperature increment between steps	2 °C
Initial temperature	0 °C
Final temperature	280 °C
Sample mass	20 mg in standard crimped Al pan
Cooling system	Cryofill liquid nitrogen
Purge gas	Helium

Displayed in Figure 3 are the StepScan DSC results obtained for the polymer blend sample.

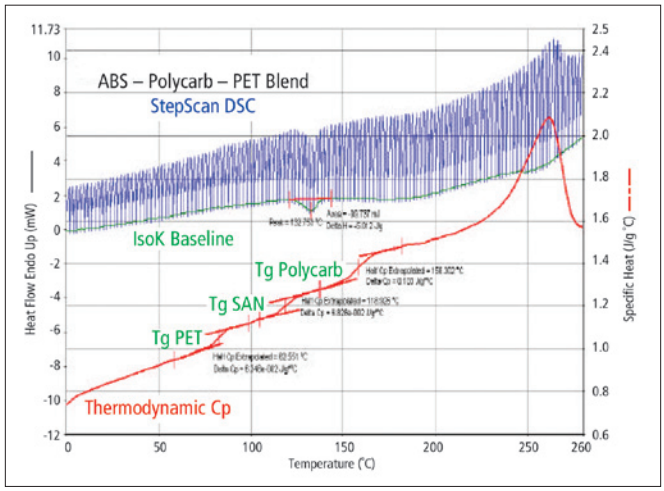

Figure 3. StepScan DSC results on polymer tri-blend.

From the StepScan DSC Thermodynamic Cp results, the polymer blend shows three distinct glass transition events – Tg for the amorphous PET at 82 °C, Tg for the styrene-acryolinitrile (SAN) of ABS at 115 °C, and the Tg of the polycarbonate component at 158 °C. With StepScan DSC, the polycarbonate Tg is separated out from the interfering and irreversible crystallization of the amorphous PET component. The use of the StepScan DSC technique allows for the detection and analysis of the hidden polycarbonate Tg, which provides valuable characterization information on the polymer tri-blend.

Summary

The high performance Pyris 1 DSC provides outstanding results on materials, such as polymer blends. The use of the StepScan DSC technique yields enhanced characterization information by separating out the reversible and irreversible thermal events. The StepScan DSC approach allows for the observation and quantitative analysis of transitions that might be hidden or obscured. The polycarbonate Tg, masked by the crystallization of the amorphous PET component, was clearly identified and analyzed with the StepScan DSC technique. The high resolution of the Pyris 1 DSC coupled with the StepScan DSC technique yielded all three Tg's of each of the polymer tri-blend components.

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