TA Instruments

Thermal Analysis & Rheology

THERMAL SOLUTIONS

QUANTIFYING POLYETHYLENE TEREPHTHALATE / POLYCARBONATE BLENDS

PROBLEM

The blending of two or more polymers is becoming a common method for developing new materials for demanding applications such as impact resistant parts and packaging films. Since the ultimate properties of blends can be significantly affected by what polymers are present, as well as by small changes in the blend composition, suppliers of these materials are interested in rapid tests which provide verification that the correct polymers and amount of each polymer are present in the blend. Differential scanning calorimetry (DSC) has proven to be an effective technique for characterizing blends such as polyethylene/polypropylene where the crystalline melting endotherms associated with the polymer components are sufficiently separated to allow quantitation. However, many blends do not exhibit this convenient separation and thus are difficult to accurately quantify by conventional DSC.

SOLUTION

Conventional DSC is a technique which measures the total heat flow into and out of a material as a function of temperature and /or time. Hence, conventional DSC measures the <u>sum</u> of all thermal events occurring in the material. Modulated DSC™, on the other hand, is a new technique which subjects a material to a linear heating method which has a superimposed sinusoidal temperature oscillation (modulation) resulting in a cyclic heating profile.

Deconvolution of the resultant heat flow profile during this cyclic heating provides not only the "total" heat flow obtained from conventional DSC, but also separates that "total" heat flow into its heat capacity-related (reversing) and kinetic (nonreversing) components. It is this separation aspect which allows MDSC™ to more completely evaluate polymer blend compositions.

Figure 1. DSC of PC/PET BLENDS

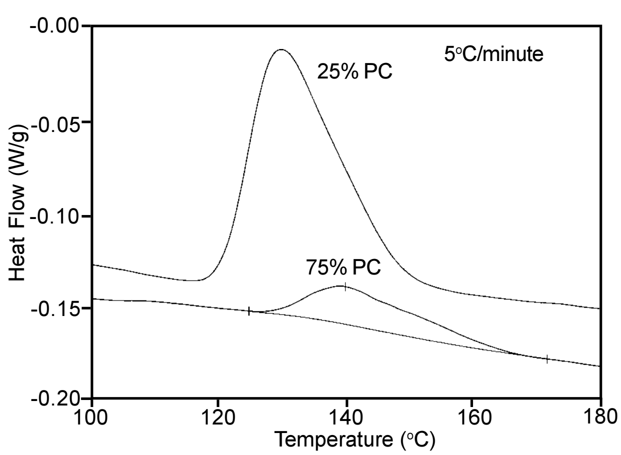
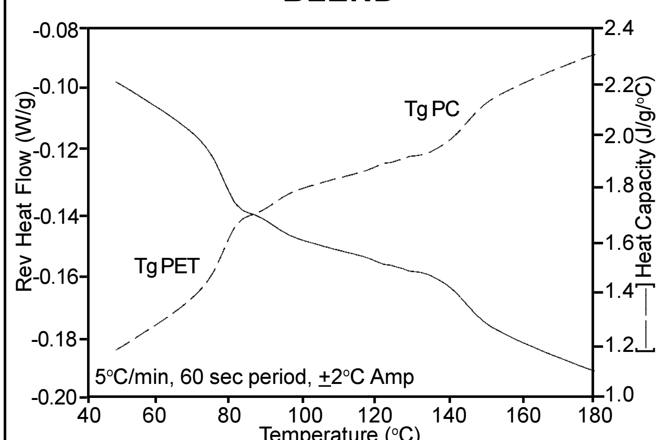


Figure 2. SPECIFIC HEAT of a PC/PET BLEND



Figures 1- 4 illustrate this capability for a series of polyethylene terephthalate (PET) / polycarbonate (PC) blends. Amorphous PC exhibits only one DSC transition - a glass transition in the range 130 to 150°C. Amorphous PET exhibits three DSC transitions - a glass transition at about 80°C, a cold crystallization peak in the range 120 to 150°C, and a melting peak above 200°C. In conventional DSC, separation of the PC glass transition and the PET cold crystallization is not possible, even in blends where the PC content is high (75%) as shown in Figure 1. On the other hand, since the PC glass transition is a reversing thermal event and the PET cold crystallization is a nonreversing thermal event, MDSC™ clearly separates the two. Figure 2 shows the heat capacity (Cp) curve for a typical PET/PC blend by MDSC. The ΔCp observed at 150°C is directly related to the PC content in the blend. By running a series of blends with known composition, a plot such as shown in Figure 3 can be generated. The straight line fit through the data in this case is described by the equation $\Delta Cp = 2.33 \times 10^{-3}\,(PC) - 1.9 \times 10^{-3}$ where ΔCp = the specific heat difference in mJ/g°C at 150°C and PC = the percent polycarbonate by weight. The correlation factor is 0.991.

The PET content can be determined in a similar fashion by measuring the ΔH of cold crystallization from the nonreversing heat flow. [This determination can also be performed from the total heat flow (conventional DSC) because the PC glass transition has only a small effect on the measured enthalpy. However, the nonreversing heat flow approach should be more reproducible since the heat capacity contribution is removed.] Figure 4 shows typical calibration plots. In either approach, the enthalpy of crystallization increases with increasing PET content with a correlation factor of 0.999. The straight line fit through the nonreversing heat flow results is described by the equation $\Delta Hc = 0.317\,(PET) - 0.132$ where ΔHc = the enthalpy of crystallization (J/g) and PET = the percent polyethyleneterephthalate by weight.

Figure 3. ΔCp vs PERCENT PC

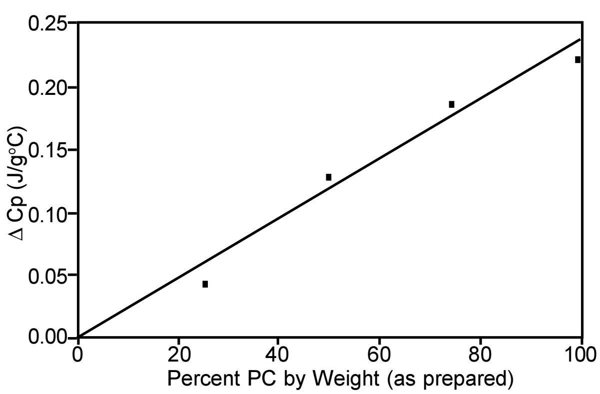
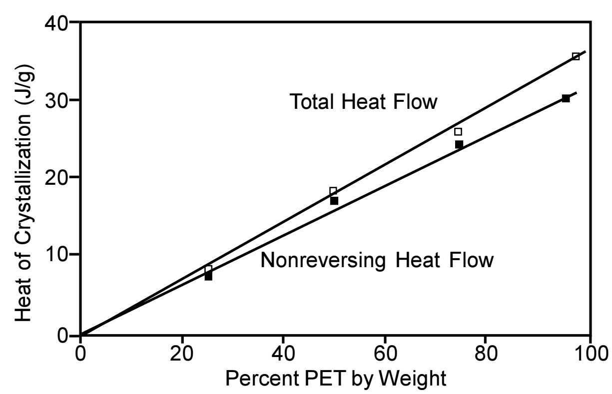


Figure 4. HEAT OF CRYSTALLIZATION vs. % PET



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