SII NanoTechnology Inc.

DSC AS PROBLEM-SOLVING TOOL: DETECTION OF Tg OF BIAXIALLY ORIENTED PET FILM

Problem

A customer is involved in the characterization of biaxially oriented polyethylene terephthalate (PET) films using thermal analysis. He reports difficulty in measuring the glass transition temperature (Tg) of the 'as received' films using differential scanning calorimetry (DSC). On the initial heating experimental, the customer obtains an endothermic peak, but no clear Tg. On the second heat, nothing is observed with the customer's existing DSC until the melting point of the PET polymer at 250°C. The customer wishes to obtain a well-defined Tg on the PET film, preferably in the 'as received' state, since this information is important to the further processing of the film into magnetically coated tapes.

Solution

The Seiko Instruments DSC220C provides the two necessary features required to obtain the well-defined glass transition of the 'as received' biaxially oriented PET films:

- high sensitivity
- Oscillating DSC capabilities.

The high sensitivity aspect is required since the PET films are highly crystalline and oriented. Both of these cause the intensity of the glass transition event to decrease making it difficult to observe. The degree and type of orientation can have a major effect on the temperature of

the PET glass transition event. Under very high orientation, the Tg of PET can increase from 75°C to over 100°C.

During the production of the film, it is stretched in two directions which places a good deal of stress on the polymer. Since the Tg of PET is above room temperature, these applied stresses are frozen into the film. Upon heating, the stresses are released and this causes an endothermic event to appear in the DSC data. This endothermic peak makes it difficult to clearly observe the weak glass transition event for the film specimen.

Thermal Analysis & Surface Solutions GmbH
Pfingstweide 21
61169 Friedberg - Germany

+49-6031-16223-1
+49-6031-16223-29
Info@thass.net
http://thass.net

SII NanoTechnology Inc.

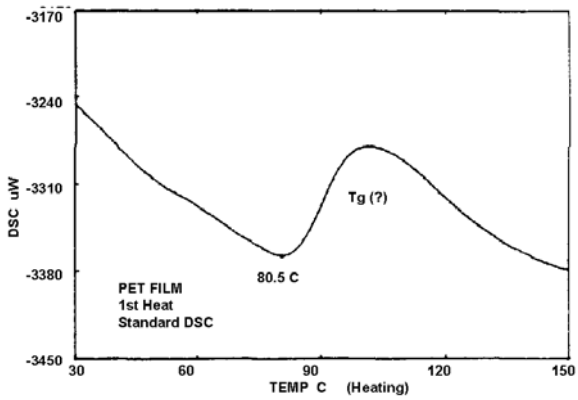

Figure 1

Displayed in Figure 1 are the results obtained on the DSC220C for the 'as received' PET film. The stress relief associated with the film is observed as an endothermic peak at approximately 80°C.

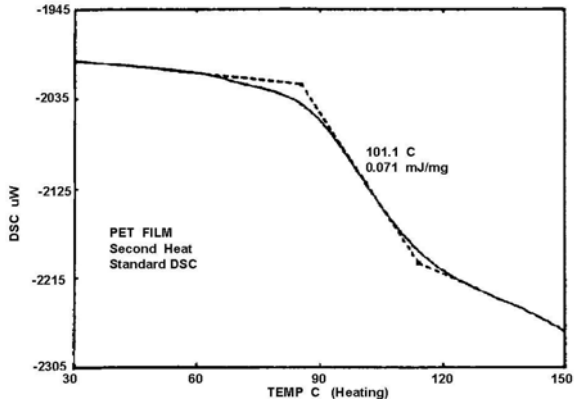

Figure 2

This stress relief peak can be eliminated by heating above Tg, cooling and then reheating. This was done for the PET film and the data obtained from the second heating experiment is displayed in Figure 2. The stress relief endotherm now disappears, and the Tg of the film is observed as a small step wise change in the heat flow at 101°C. These results demonstrate that the DSC220C has the necessary high sensitivity required to detect the weak Tg associated with the highly crystalline and highly oriented film.

There is, however, a more direct means of obtaining the glass transition information on the 'as received' PET film without the need to heat, cool and reheat. This may be accomplished using Oscillating DSC (ODSC). ODSC involves superimposing an oscillating

Thermal Analysis & Surface Solutions GmbH
Pfingstweide 21
61169 Friedberg - Germany

+49-6031-16223-1
+49-6031-16223-29
Info@thass.net
http://thass.net

time-temperature wave over a constant underlying heating ramp. The resulting data yields three separate data sets:

- Cp component which reflects the reversible aspects of thermal events, such as Tg
- kinetic component which represents the irreversible nature of transitions, such as stress relief at Tg
- parent signal, which is equivalent to the standard DSC data.

Through the separation of the parent signal into the reversible and irreversible data sets, a well-defined Tg can be obtained on the 'as received' film during a single heating experiment.

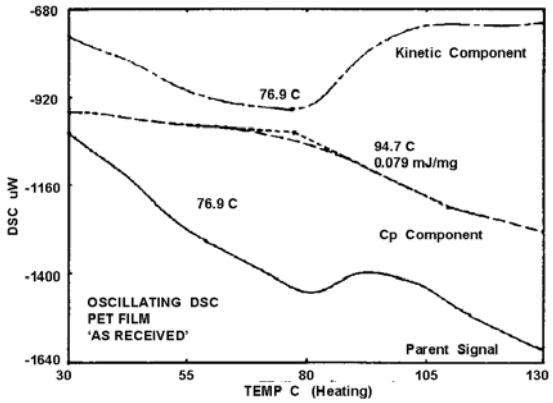

Figure 3

This may be seen in Figure 3 which shows all three ODSC data sets for the 'as received' PET film. The interfering effects of the endothermic stress relief peak are relegated to the kinetic component data set permitting a clear observation of the step wise change at Tg in the Cp component.

Summary

The advantages of the ODSC approach, in the analysis of highly crystalline, highly oriented films are:

- only a single heating experiment is required resulting in a greater degree of lab efficiency
- there is no chance that the sample's original structure or morphology could be disturbed or altered as could happen during a heat, cool, reheat type of experiment
- the high sensitivity of the DSC220C permits the weak Tg event to be detected.

Thermal Analysis & Surface Solutions GmbH
Pfingstweide 21
61169 Friedberg - Germany

+49-6031-16223-1
+49-6031-16223-29
Info@thass.net
http://thass.net

SII NanoTechnology Inc.

Thermal Analysis & Surface Solutions GmbH
Pfingstweide 21
61169 Friedberg - Germany

+49-6031-16223-1
+49-6031-16223-29
Info@thass.net
http://thass.net