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MDSC™ Applications ..... Solutions to Materials Characterization Problems

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DSC has been a very useful analytical technique for characterizing the physical and reactive properties of materials. However, limitations of the technique are obvious when it is used to characterize complex materials such as polymer blends or to study the effect of processing conditions on polymer morphology.

Modulated DSC (MDSC™) is a new approach to making DSC measurements and is rapidly becoming the preferred technique because of six (6) significant advantages over traditional DSC. Examles of these advantages are illustrated below.

1. Separation of Complex, Overlapping Transitions

Because MDSC separates the total heat flow signal into its heat capacity and kinetic components, it greatly simplifies interpretation of overlapping transitions. **Fig.1** shows how the glass transition of the ABS component in an ABS/PET blend can be seen in the MDSC Reversing signal but not in the Total signal which is the only signal available to traditional DSC.

2. Increased Sensitivity for Measuring Weak Glass Transitions

MDSC baselines are far superior (flat and straight) to DSC baselines due to the unique way in which the Total heat flow is deconvoluted into its heat capacity and kinetic components. This permits the study of materials with weak glass transitions. **Fig.2** shows how the glass transition of Nylon 6 shifts with moisture content.

3. Increased Resolution Without Loss of Sensitivity

With traditional DSC it is not possible to optimize both resolution and sensitivity in the same experiment. Resolution increases as the heating rate decreases while sensitivity increases with increased heating rates. MDSC solves this problem by having two independent heat-

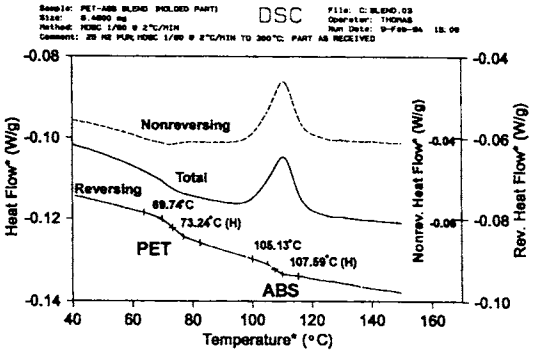

Fig.1 Separation of complex.

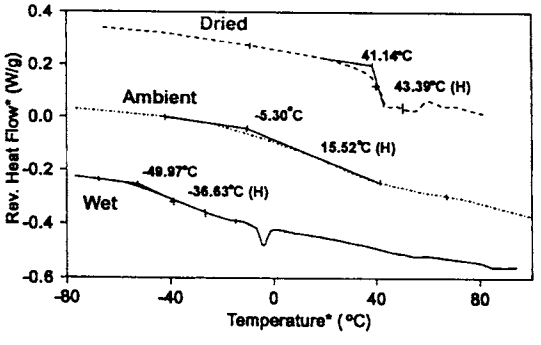

Fig.2 Measuring weak glass transition.

TA Instruments, 109 Lukens Drive, New Castle, DE 19720-2795

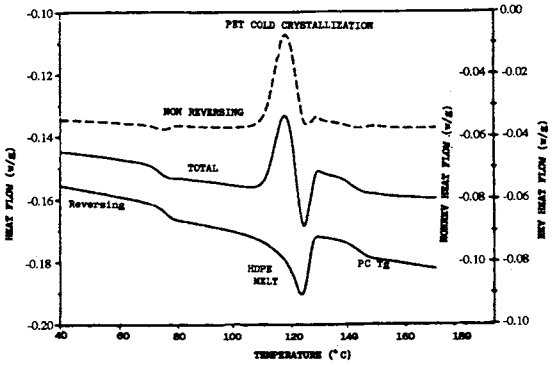

Fig.3 Polymer blend characterization.

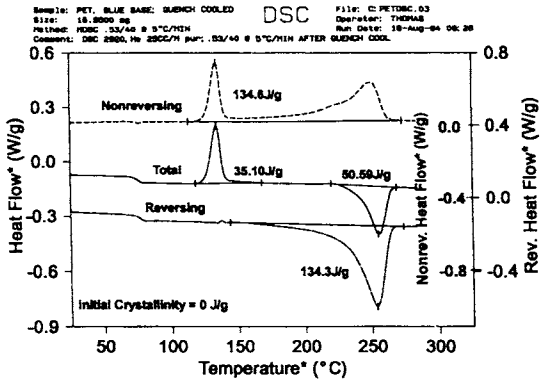

Fig.4 Determine the crystallinity.

ing rates. The average rate can be set very slow (as low as 0.01 ℃ · min^{-1}) while the instantaneous heating rate could be set as high as 50 ℃ · min^{-1}. **Fig.3** shows the separation of three overlapping transitions in a polymer blend over the temperature range of 110-140 ℃.

4. Ability to More Accurately Determine the Crystallinity of Semicrystalline Polymers

Polymers often change (anneal, crystallize etc.) as they are heated in a DSC experiment. Since DSC can only measure the sum (Total) of all thermal events, it is often very difficult to determine how the changes in the sample have contributed to the final result. MDSC solves this problem by providing a separate signal (Nonreversing) to monitor the kinetic processes (changes) occurring in the sample. Using the sum of

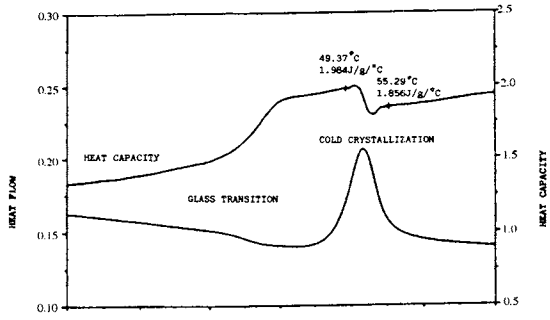

Fig.5 Measurement of heat capacity.

the Reversing (endothermic) and Nonreversing (exothermic) signals, MDSC shows that quench cooled PET has no crystallinity (−134.3 + 134.6 = 0.3 J · g^{-1}) while interpretation of the Total signal would imply it had 15 J · g^{-1} (50.59 - 35.10).

5. Measurement of Heat Capacity and Heat Flow in a Signal Experiment

Reactions, such as the cure of a thermoset, crystallization of a thermoplastic, cause structual changes in the material which result in a change in the heat capacity of the sample. This change in heat capacity can easily be measured by MDSC, as seen in **Fig.5**, but is very difficult to measure by DSC due to the heat of reaction which occurs simultaneously with the heat capacity change.

6. Measurement of Thermal Conductivity Insulating Materials

DSC instruments have been physically modified in the past in order to make thermal conductivity measurements. However, this is time-consuming and also reduces the productivity of the DSC for routine measurements. No modification of the instrument is required for MDSC measurement of thermal conductivity since it is based on differences in the apparent heat capacities of thick (3.0mm) and thin (0.4mm) samples.

The equation used to calculate thermal conductivity is shown in **Fig.6** along with a comparison of typical precision from other measurement techniques.

EQUATION

$K=LC/mAC_pP$

where K = thermal conductivity
L = sample length
C = apparent heat capacity
m = sample mass
A = cross section area
C_p = specific heat
P = period

PRECISION

MDSC	5 %
ASTM1225	7 %
ASTM4351	6-11 %

Fig.6