THERMAL ANALYSIS

application note

Characterization of EPDM Elastomers Using DSC

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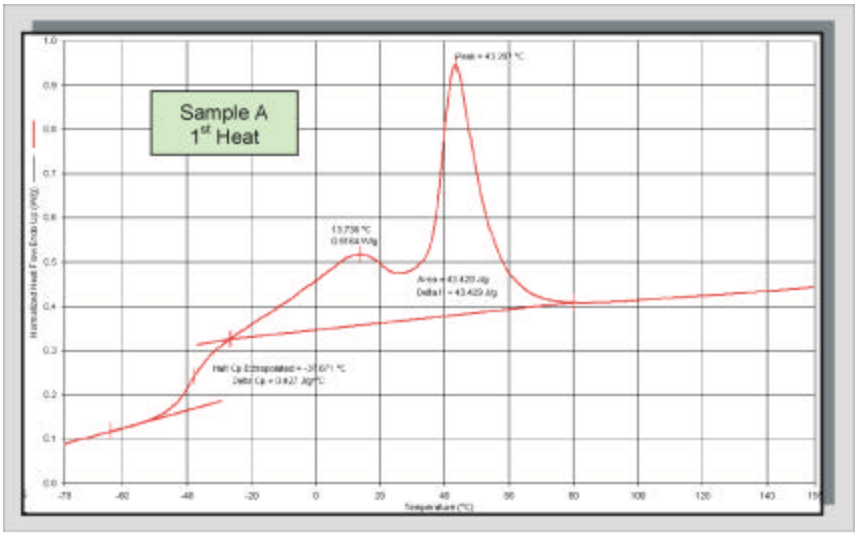
Introduction

Thermoplastic elastomers (TPE) are a class of polymeric materials that combine the ease of processability of a thermoplastic with the properties of conventional crosslinked elastomers. The use of TPEs has been steadily increasing over the past decade because of their unique processing and end-property characteristics.

One of the most widely used thermoplastic elastomers is EPDM, which contains a meltable, ethylene component. The largest market for EPDMs is automotive because of the material's low cost, low specific gravity or density, ease of processability, paintability and weatherability. It is widely used for bumper fascias, grilles, air dams and rub strips. Non-automotive uses for EPDMs include swim fins, handle grips, wire and cable jacketing, and weatherstripping.

The amount of ethylene in the EDPM elastomer will affect the material's end use characteristics. A high concentration of ethylene imparts a more crystalline nature to the EPDM and will increase its glass transition temperature, Tg, and melting temperature. It is desired to have an easy means of characterizing the compositional and thermo-physical properties of EPDM elastomers and one of the best techniques is differential scanning calorimetry (DSC).

Figure 1. DSC results on EPDM sample A during 1 st heating segment.



Experimental

DSC measures the heat flow into or from a sample as it is heated, cooled and/or held isothermally. The technique provides valuable information on softening temperatures (or Tg), melting temperatures, heats of melting, percent crystallinities, and recrystallization (temperatures and heats).

PerkinElmer offers a variety of different DSC instruments and technologies for various applications and needs.

Two EPDM thermoplastic-elastomeric samples were received for analyses and were labeled as A (high ΔH) and B (without oil and low ΔH). The following experimental conditions were utilized to characterize the weight loss properties of the samples:

Sample container:	Crimped standard aluminum pan
Sample mass:	Approximately 9 mg
Initial temperature:	-85 C
Final temperature:	160 C
Heating rate:	20 C/min
Purge gas:	Nitrogen at a flow rate of 20 mL/min

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Figure 2. DSC results for sample A during the 2nd heating segment.

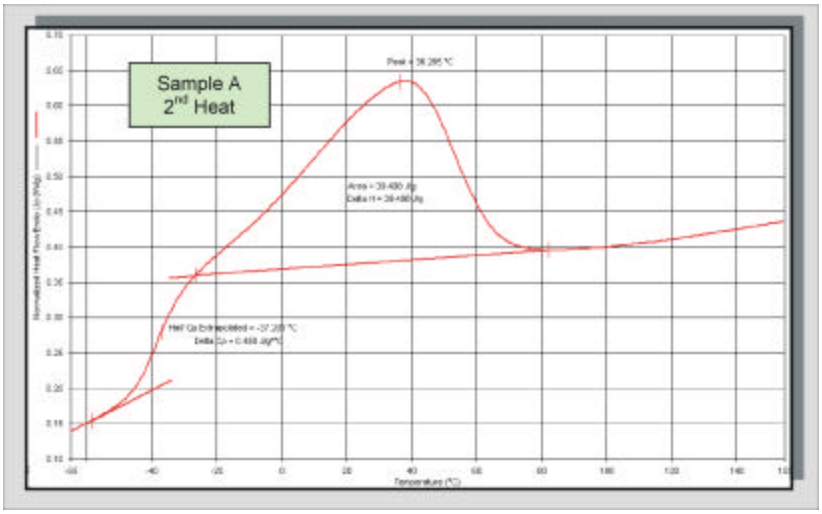
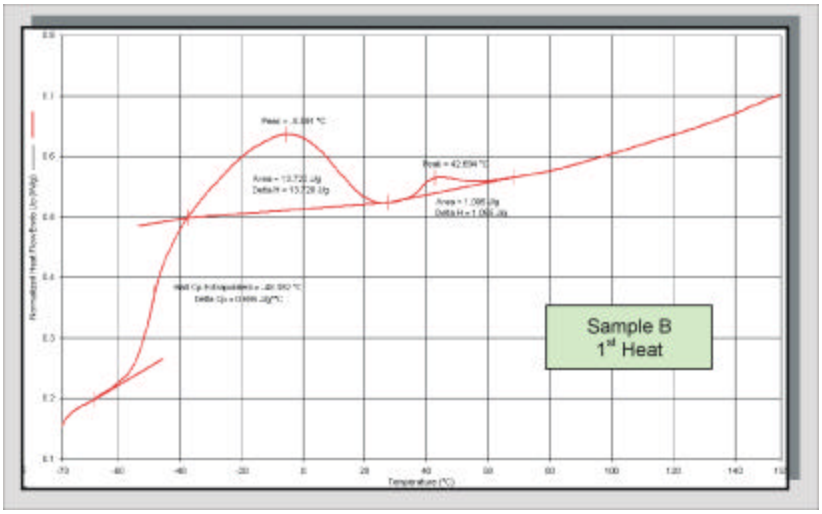


Figure 3. DSC results for EPDM sample B during 1st heating segment.



The samples were heated to 160, held for 10 minutes, and cooled at a rate of 10 C/min back to –85 C. The samples were then reheated to 160 C at a rate of 20 C/min to provide the 2nd heating information.

DSC Results

Displayed in the following figure are the DSC results on EPDM Sample A generated during the 1st heat (as received). The plot shows the sample heat flow as a function of sample temperature.

During the 1st heating segment, sample A yields its glass transition (Tg) at –37.7 C. This followed immediately by the melting of the ethylene component at 13.7 and 43.2 C with a total heat of melting of 43.3 J/g. The Tg of the EPDM is best defined by extrapolating the linear heat flow results of the liquid phase (above 80 C) back down through the melting of the ethylene component. This extrapolation gives a good approximation for the end point of the Tg of the EPDM for data analysis purposes.

After heating sample A though the melting of its ethylene component, the sample was cooled back to –70 C and then reheated. This places a new thermal history into the EPDM sample and helps when comparing the characteristics of two different samples or batches. The results generated for sample A during the 2nd heating segment are displayed in Figure 2.

During the reheat experiment, the EPDM sample yields its Tg at –37.2 C with melting occurring at 36.3 C and ΔH of 39.5 J/g. The melting characteristics of the ethylene component change significantly between the 1st and 2nd heating experiments and this reflects the differences due to thermal history. The as received EPDM material apparently undergoes crystalline perfection and/or thermal fractionation as it sits at room temperature. This may allow different molecular weight components to selectively crystallize out resulting in the occurrence of two different melting peaks during the 1st heating segment. During the reheat, only a single, broad melting peak is observed. If the EPDM were

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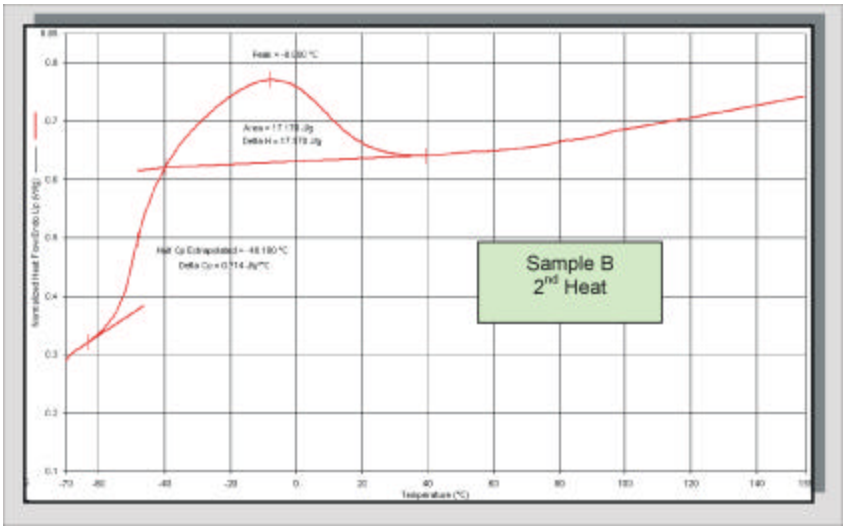
Figure 4. DSC results for EPDM sample B during 2 nd heating segment.



permitted to remain at room temperature for a prolonged period, the sample will progressively redevelop the two melting peaks due to selective crystallization and/or perfection.

EPDM sample B was analyzed using the same conditions as for the other material and Figure 3 shows the results generated during the 1st heating segment.

Sample B exhibits its Tg at –48.1 C which is significantly lower than sample A. Immediately upon passing through its Tg, sample EPDM B undergoes melting of its crystalline ethylene component at –5.6 and 13.7 C. The heats of melting for the two transitions are 13.7 J/g and 1.1 J/g, respectively. The total heat of melting of B is much less than that of sample A indicating a significantly lower crystalline component for EPDM B.

Displayed in Figure 4 are the results generated for sample B during the 2nd heating segment.

During its reheat, sample B yields its Tg at –48.2 C and undergoes melting at –8.0 C with a heat of melting of 17.2 J/g. The 2nd ΔH of melting is much less for EPDM sample B as compared to sample A (17.2 J/g versus 39.5 J/g). This is consistent with the fact that EPDM B has a significantly lower ethylene component in comparison to A.

Summary

The PerkinElmer Pyris 6 DSC yielded excellent results on the two EPDM thermoplastic elastomeric samples and was able to show distinct and significant differences between the two in terms of crystalline content. The EPDM sample with the greater concentration of ethylene had a significantly higher Tg, melting temperature and heat of melting. This is valuable information, as the differences in the thermo-physical properties will affect the end use characteristics of the EPDM elastomers.

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