SII NanoTechnology Inc.

DSC AS PROBLEM-SOLVING TOOL: EVALUATION OF GOOD AND BAD PVC PELLETS FOR WIRING APPLICATIONS

Problem

A chemist has a need to characterize the properties of flexible PVC (poly vinyl chloride) polymers used as a jacket material for electrical cables. Some of the PVC has produced a jacket that failed prematurely and it is suspected that the discolored or 'Bad' polymer pellets are causing the failures. The customer desires an analytical test to easily characterize and make distinctions between the 'Good' and 'Bad' PVC pellets and to determine the possible cause of the problem.

Solution

Differential scanning calorimetry (DSC) provides a straightforward and easy to use means of examining the thermal characteristics associated with polymeric materials, such as PVC. DSC measures the heat flow into or from a sample as it is either heated, cooled or held isothermally, or a combination of any of these.

Cyclic DSC provides an enhanced means of testing or examining the properties of materials since the total heat, reversible and irreversible aspects can be elucidated. Cyclic DSC is performed by heating a sample at a relatively fast rate (20°C/min) through the temperature range of interest, cooling it back to the starting temperature, and then reheating. A direct comparison can be made between the data obtained during the first (as-received) and second (reversible) heating segments to

establish the nature of the transitions taking place within a sample as it is heated. Using cyclic DSC, thermal transitions can be better identified as to whether they are reversible, such as Tg, or irreversible, such as thermal history effects, loss of water, crosslinking or crystallization.

Cyclic DSC experiments can be performed relatively quickly, by heating and cooling at a rate of 20°C/min. Advantages offered by cyclic DSC over temperature modulated DSC (TMDSC) are the time savings, reliability of the results, and ease of use. The maximum heating rate that can be used with TMDSC is only 5°C/min. If a sample is heated from -50 to 175°C, the total time of the TMDSC experiment would be 45 minutes. Using cyclic DSC, the total time would only be 34 minutes to obtain equivalent information.

The Seiko EXSTAR DSC6000 system offers the following major advantages for the study of materials:

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- high sensitivity (the DSC6100 offers the highest sensitivity of any DSC on the market)
- very stable baseline performance for highly reproducible results
- exceptional subambient performance for the study of Tg's near or below 0°C
- state-of-the-art robotic accessory for reliable, unattended operation
- 20 point temperature calibration for unsurpassed accuracy
- 10 point enthalpic calibration for highly accurate heat capacity measurements and heats of transitions

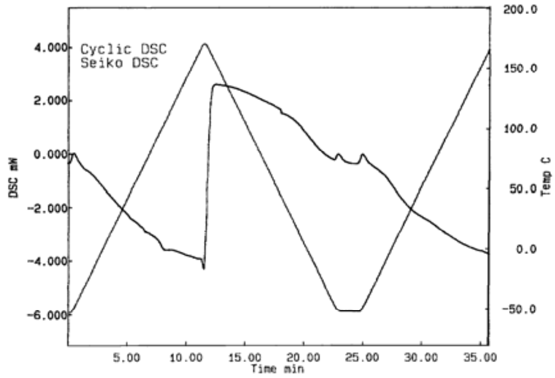

Figure 1

Displayed in Figure 1 are the cyclic DSC results generated on a PVC pellet specimen. The plot shows the heat flow and the time-temperature profile as a function of time. The first heating segment represents the thermal properties of the as-received pellet while the second heating segment reflects the reversible aspects of the sample.

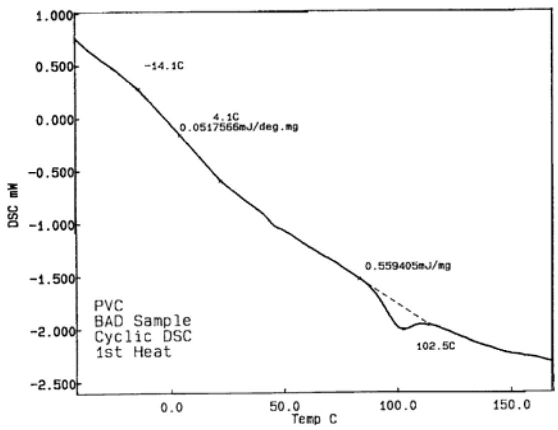

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Figure 2

The DSC results obtained during the 1st heat are displayed in Figure 2 for the 'Bad' PVC pellet specimen. The as-received sample exhibits a glass transition event (Tg) beginning at -14.1°C with a mid-point temperature of 4.1°C. The polymer yields an small endotherm at 102.5°C, which is most likely due to processing or thermal history conditions. The heat of transition is 0.56 mJ/mg for the 'Bad' polymer.

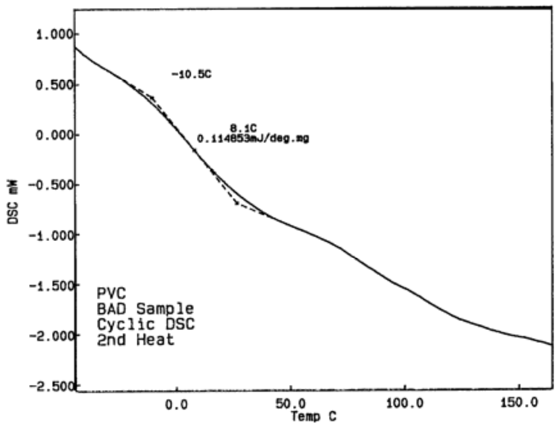

Figure 3

Displayed in Figure 3 are the DSC results obtained during the 2nd heating segment for the 'Bad' pellet specimen. A well-defined glass transition event is obtained at 8.1°C. The endotherm at approximately 100°C is no longer observed indicating that the transition was irreversible in nature and, most likely, due to processing conditions.

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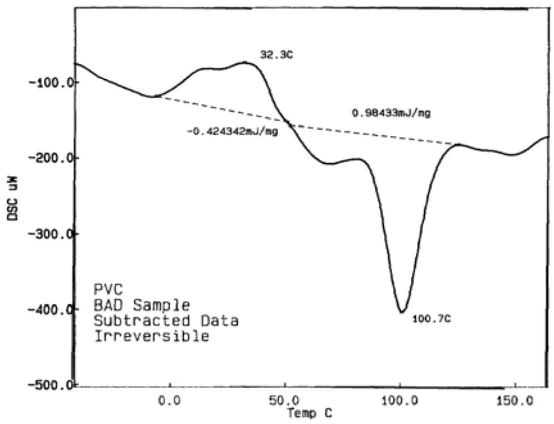

Figure 4

By subtracting the 2nd heat results from the 1st heat data, a subtracted data file is obtained which then reflects the irreversible aspects of the sample. The subtracted file for the 'Bad' pellet sample is displayed in Figure 4. The sample yields an exothermic response in the vicinity of Tg, which represents the irreversible transformations which occur at Tg. An endothermic peak is obtained at 101°C which probably reflects the processing conditions that the 'Bad' pellet specimen was exposed to.

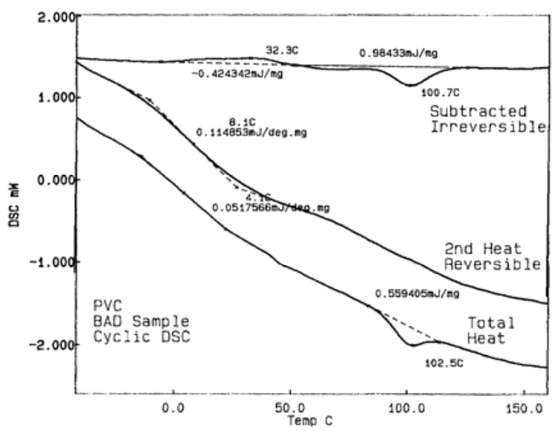

Figure 5

Shown in Figure 5 is a direct overlay of the three data files obtained from the cyclic DSC experiment on the 'Bad' pellet. The lower curve represents the thermal properties of the as-received pellet; the center trace is that of the 2nd heat or reversible properties, and the upper curve reflects the irreversible characteristics of the sample.

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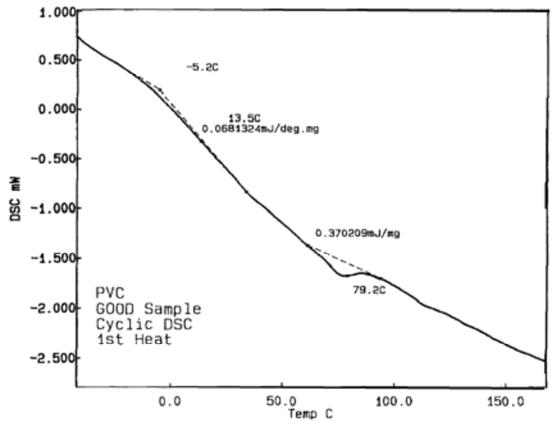

Figure 6

The 1st heat data generated on the 'Good' pellet sample is shown in Figure 6. The as-received specimen yields its Tg at 13.5°C along with a small endotherm at 79°C with a heat of transition of 0.37 mJ/mg. The endotherm associated with the 'Good' pellet sample occurs at a much lower temperature as compared to the 'Bad' (79°C versus 102°C). The heat of transition is significantly greater for the 'Bad' pellet as compared to the 'Good' (0.56 mJ/mg versus 0.37 mJ/mg). This indicates that the 'Bad' pellet has been subjected to more severe processing conditions and may account for its poorer performance in end-use applications.

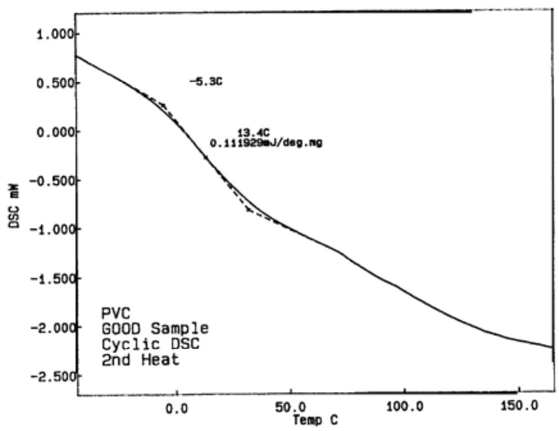

Figure 7

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The 2nd heat results obtained for the 'Good' PVC pellet are displayed in Figure 7. A well-defined Tg is observed at 13.4°C for this sample. The Tg of the 'Good' pellet is significantly higher than that of the 'Bad' material (13.4°C versus 8.1°C).

The subtracted, or irreversible, data file for the 'Good' pellet specimen is shown in Figure 8. The sample exhibits a significant endotherm at 74°C which most likely reflects the processing conditions that the pellet was exposed to during production.

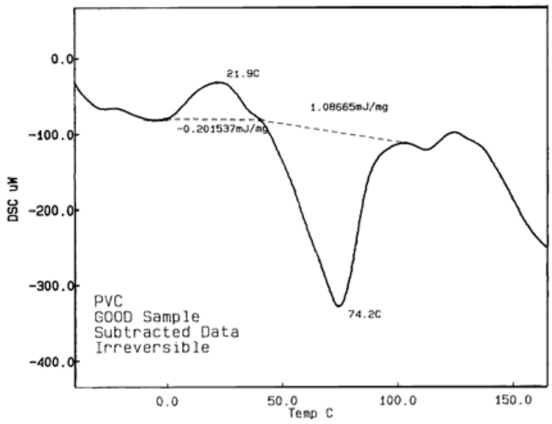

Figure 8

A complete overlay of the three separate data sets (total heat, reversible and irreversible) is displayed in Figure 9 for the 'Good' pellet material.

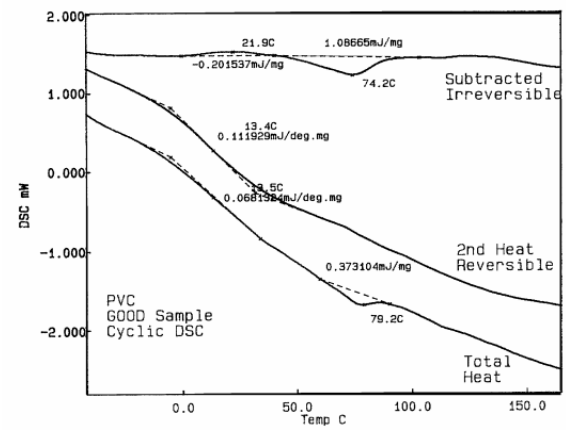

Figure 9

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Summary

Cyclic DSC was used to characterize the thermal properties of flexible PVC pellets utilized to produce jacket materials for electrical cables. One set of pellets was classified as 'Bad' while the other set was considered satisfactory or 'Good' with respect to its end-use performance characteristics. The 'Good' pellet exhibited a heat history endotherm at a much lower temperature than the 'Bad' pellet (74°C versus 102°C). The reversible or reheat Tg was significantly higher for the 'Good' sample as compared to the 'Bad' (13.4°C versus 8.1°C). The cyclic DSC approach provided information on the total heat characteristics, as well as the reversible and irreversible aspects of the PVC samples. The Seiko DSC instrument provides excellent data on the materials.

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