Better Characterization of Subambient Transitions Using Power Compensated DSC

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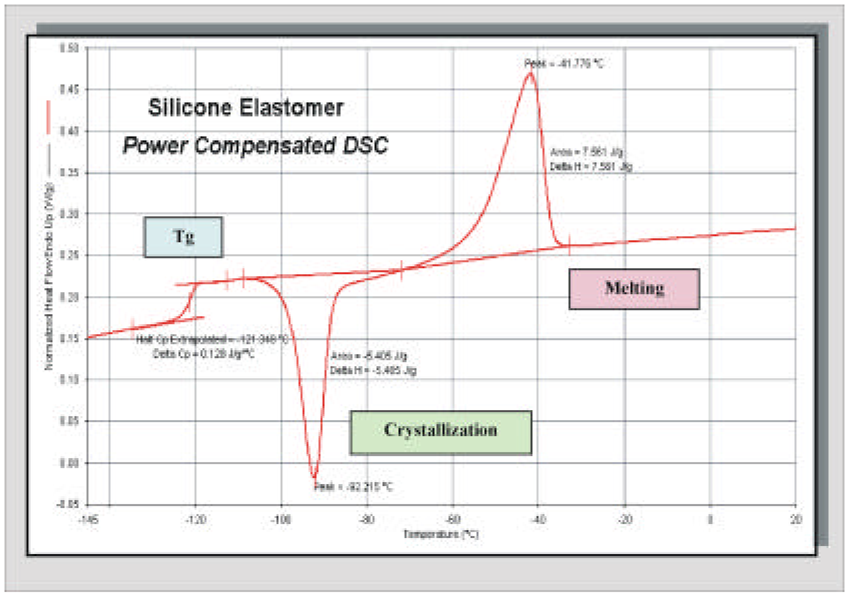
One of the most demanding applications of a DSC instrument is the assessment of low temperature, subambient transitions, especially those below –100 C. The successful measurement of very low temperature transitions, such as Tg, crystallization or melting, requires that a stable baseline be obtained in a temperature interval before the desired transition is encountered during heating. A stable sample heat flow response ensures that a better assessment will be obtained for the desired transition, as this provides a better integration baseline for melting and crystallization events, and a more well-defined heat capacity response in the glassy regions for Tg.

The generation of a stable, linear sample heat flow response at very low temperatures can be difficult for many DSC instruments as this requires that the device quickly achieves a quasi-equilibrium state once the experiment is initiated. For a heat flux DSC, which has a large massive furnace, the instrument does require significant time before a stable and linear heat flow response is obtained. This may mean that, if the experiment is initiated at –150 C and the transition occurs at –130 to –120 C, the sample baseline response may not be completely equilibrated or linear before going into the transition. This will not give a good assessment of the low temperature transition.

Figure 1. Power compensated DSC results obtained on silicone potting compound at a heating rate.

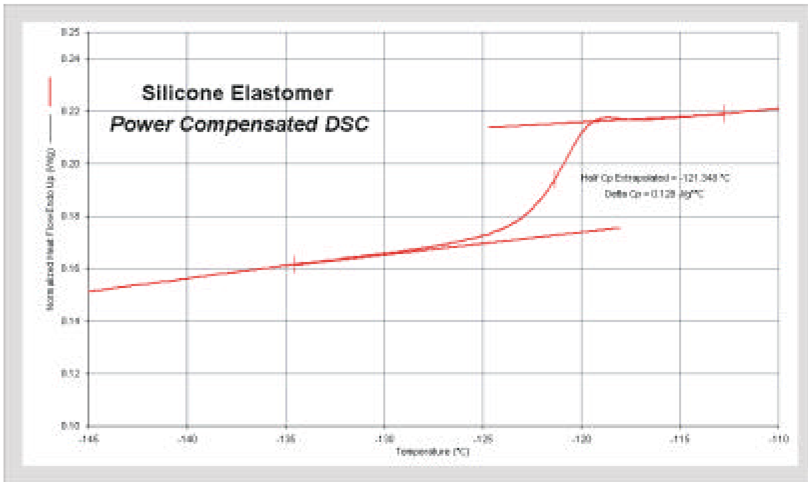


In contrast, the power compensated DSC from PerkinElmer provides separate and low mass furnaces (1 g) for both the sample and reference sides. This not only provides true calorimetric measurements, but also yields rapid thermal equilibration once the experiment is initiated. The power compensated DSC, under the same experimental conditions, will thermally equilibrate significantly more rapidly than a heat flux DSC device. This permits the power compensated DSC to obtain a stable and linear sample heat flow response quickly during a DSC experiment. Even samples, which have very low temperature transitions (-130 to –120 C), will yield excellent and well-defined results with the power compensated DSC.

As an example, a traditionally difficult application for many DSC instruments is the successful measurement of the glass transition or Tg of a silicone elastomer or potting compound. The glass transition event of cured silicones occurs at approximately –120 C. To adequately observe the Tg of silicones, and obtain a flat sample response going into the

PerkinElmer™ instruments.

Figure 2. Very low temperature Tg of silicone elastomer at heating rate of 20 C/min.



Tg, many heat flux DSC instruments require the use of slower heating rates (10 C/min or less) rather than the 'standard' heating rate of 20 C/min. The faster rate of 20 C/min gives an experimental time of only 1.5 minutes if the DSC experiment is initiated at –150 C before the Tg at –120 C is encountered. The use of a heating rate of 10 C/min provides 3 minutes of experimental time and 5 C/min gives 6 minutes. However, the true test of performance is the ability of a DSC to obtain a well-defined Tg at a rate of 20 C/min for a silicone elastomer.

A sample of a cured silicone potting compound sample was analyzed using the PerkinElmer Pyris 1 power compensated DSC instrument. A 25 mg specimen was heated at a rate of 20 C/min from –150 C to 20 C and the results of this experiment are displayed in Figure 1.

These DSC results demonstrate that the power compensated DSC yields a linear sample heat flow response between –145 and the onset of the Tg at –125 C. This provides a more accurate assessment of the mid-point Tg value since this requires that linear responses be obtained below and above the actual Tg.

The silicone elastomer yields two additional transitions: an exothermic crystallization event at –92 C and the melting of the crystalline phase at –42 C. The power compensated DSC provides excellent data on all three subambient transitions associated with the silicone potting compound at a rate of 20 C/mn.

The excellent results obtained for the very low Tg of the silicone sample are shown in an enlarged view in Figure 2.

These results demonstrate that the power compensated DSC achieves rapid thermal equilibration from the starting temperature of –150 C, even at a fast heating rate of 20 C/min. A stable and linear heat flow response is obtained in the regions below the onset of the glass transition event. This also means that the power compensated DSC would yield excellent quantitative heat capacity results in the very low temperature regions.

Summary

The PerkinElmer power compensated DSC yields excellent results in the very low temperature regions (i.e., below –100 C). The power compensated DSC instrument has very low mass furnaces (1 g) for both the sample and reference sides, which provides rapid equilibration. In contrast, heat flux DSC devices have a single large mass furnace (200 g mass) which yields a more sluggish response. Even at a fast heating rate of 20 C/min, the power compensated DSC has the ability to provide a linear and stable sample heat flow response in the very low temperature regions. This is critical for the accurate assessment of low temperature Tg's of silicone elastomers or potting compounds.

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