BETTER RESOLUTION OF DSC TRANSITIONS USING COOLING

When performing DSC experiments, overlapping transitions are sometimes observed. Better resolution, or separation, of the overlapping transitions can be obtained using the following suggestions:

- Slower heating rate (5°C/min or less)
- Lower sample mass
- Use of helium purge gas rather than nitrogen
- Flatter, thinner samples
- Use of cooling rather than heating experiments

Typically, most DSC experiments are performed in the dynamic heating mode. But, oftentimes, more informative data on certain samples, can be obtained by dynamic cooling rather than heating. For the case of overlapping transitions observed during heating, the use of cooling experiments can sometimes provide better resolution of the transitions because of supercooling.

Supercooling refers to the sample existing in an unstable thermodynamic state as it is cooled from the melt. The sample may not nucleate and crystallize during cooling and remain in an unstable liquid state. Eventually, however, with enough cooling, the sample will either crystallize or form an amorphous glass. Water, for example, will not immediately crystallize at 0°C during cooling by DSC. Water will remain in the liquid state well below 0°C. If the water droplets are tiny enough, water can be cooled to -40°C before it finally crystallizes and turns into ice.

If two overlapping first order transitions are obtained during heating, one of the transitions may undergo supercooling to a greater extent than the other. The greater amount of supercooling may then provide better resolution or separation of the two transitions during DSC cooling experiments.

The Seiko DSC6200 is ideally suited for performing cooling experiments as it provides the following ideal features:

- unparalleled subambient baseline performance
- exceptional baseline stability for prolonged experiments
- high sensitivity
- ease of operation

As an example of the benefits of performing cooling experiments, a metal alloy was analyzed using DSC to measure its physical properties. The alloy exhibits two severely overlapping first order transitions near 20°C during heating.

Figure 1 shows the DSC results generated on the metal alloy using both heating and cooling experiments. During heating, the two transitions are highly overlapped with peak temperatures of 16.7 and 19.4°C. However, during the cooling segment, one of the transitions undergoes

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supercooling to a large extent and separation of the two transitions is obtained. The transitions are well resolved and observed at 15.4 and -45.7°C during cooling.

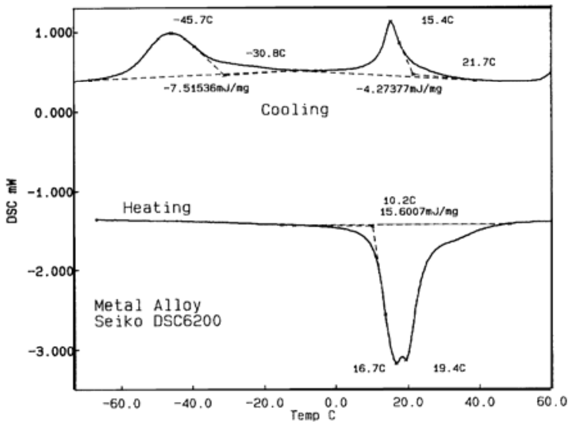

Figure 1

The Seiko DSC6200 yields highly reproducible results on the metal alloy even under prolonged subambient conditions. Displayed in Figure 2 are the data generated from 5 consecutive heating and cooling experiments on the metal alloy sample. The design of the DSC cell is such that an exceptionally stable baseline is obtained even when operating at temperatures well below 0°C. This is an important feature when attempting to obtain precise and accurate temperatures and heats of transitions of materials. Figure 3 shows an enlarged view

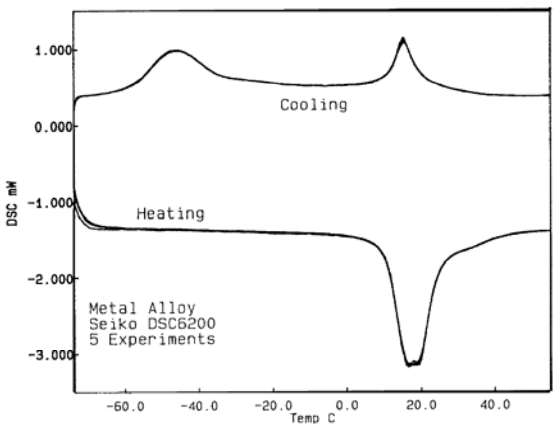

Figure 2

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of the 5 sets of cooling results obtained on the metal alloy and the data is remarkably reproducible.

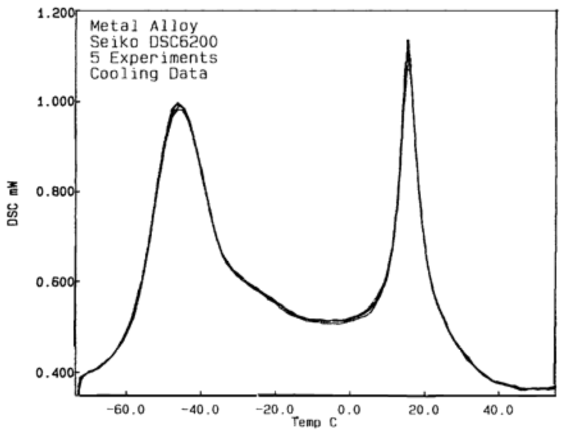

Figure 3

Summary

For some samples, better resolution of overlapping DSC transitions can be obtained by performing cooling, rather than heating, experiments. Some transitions will undergo significant supercooling which causes the transition to be significantly depressed in temperature. If one transition supercools to a much greater extent than the other, DSC cooling experiments can help to provide better separation of the two events and provide enhanced data interpretation.

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