ChE 529 Polymer Experimental Methods University of Rhode Island
Professor Greenfield Fall 2008

Lab 4, Differential Scanning Calorimetry (DSC)
October 6/7, 2008

Objective: The goal of this lab is to use Differential Scanning Calorimetry (DSC) to measure thermal transitions in several polymer systems. For well-characterized polymers, the results will be compared to literature values. For less well-defined polymers, the thermal scans will be used to identify the interpret reactive groups and phases that may be present.

Safety: The main risk in this experiment comes from the high temperatures reached in the experiment and the sharp tweezers used for handling the samples. The sample chamber covers on the DSC apparatus should be handled with the tweezers as a precaution. The main cover can be touched only after confirming on the computer interface that the sample chamber is near room temperature. The end of the tweezers shouldn't be near your hand. If you need to poke a hole in a sample holder, for example, put the sample pan onto a surface. DON'T hold it in your hand when poking a hole.

Instrument Preservation: The DSC instrument relies on measuring the relative amounts of heats required to change the temperature of the sample and a reference. The sample is heated in a pan (either a regular pan or a larger hermetic pan). **The sample chamber should be at its standard temperature (40°C) before opening the chamber** to add or remove pans. A chamber that is more hot has the risk of burns; a chamber that is cold has the risk of condensation forming on the inside of the instrument, which will damage it.

The reference is the same kind of pan as the sample. The pan lid need to be pressed down firmly before running the experiment; there is a press that is used for this. Pans are designed to be used with a single sample.

Procedure to perform Differential Scanning Calorimetry

The DSC instrument is located in Professor Bothun's lab in Chemical Engineering, 224 Crawford Hall. Prof. Bothun has prepared a Standard Operating Procedure (or SOP) for how the DSC is used, and it is posted next to the instrument. The instructions below complement the information in that SOP.

Prepare the instrument

1. Follow the standard operating procedure to begin equilibrating the instrument. Note that only the main valve on the nitrogen cylinder needs to be opened. Also note that RCS means Refrigerated Cooling System.

Prepare the sample

2. Use the tweezers to pick up a small pan and lid. Measure their mass on the balance. DON'T use the "tare" function, since we'll want to reweigh the sample after the balance has been used for other samples.
3. Remove the pan and lid from the balance. Use the tweezers to add a small piece of polymer sample.
4. Put the lid on the pan and weigh the total (pan+lid+sample). The desired mass range is 5-30 mg of sample.
5. Use the press to crimp the lid to the sample. See below for the proper die to choose. If the sample is expected to have vapor products (e.g. the floor samples), poke a hole in the pan (using the tweezers) so it doesn't explode.

6. Use the tweezers to place the lids in the sample compartment. Place the sample in the front right stand in the sample compartment. Place the reference (empty pan+lid of the same size) on the left rear stand. Replace the 3 lids.

Choose the instrument settings and Measure

7. The default instrument parameters can be changed for each run. Some will be modified as described below. Make the changes to "Run 1" in the instrument software.
8. Be sure to specify a new name for the results file. This is easier than changing it later.
9. Click the "play" button (right-pointing triangle) to start the sample equilibration and measurement.

View and use the results

10. The results are viewed using the TA Universal Analyzer program. Open your results file and use the mouse to scale the axes to your data.
11. The instrument software can help with interpreting a glass transition.

Changing the sample

12. Wait for the sample compartment to return to 40°C.
13. Open the sample compartment to remove the pan (and reference, if necessary) using the tweezers.
14. Remove your sample. You may need to reweigh it. If it will be used again, place it in the instrument and conduct the next measurement, or place it aside if a different measurement is being done first. Otherwise, dispose of the used samples+pans in the "used DSC pans" waste container box, which is near the DSC instrument and is indicated.
15. Prepare the new sample and put it in the instrument, as directed above.
16. Conduct the next experiment as directed above.

Finishing the experiments

17. Follow the instructions in the standard operating procedure.
18. Keep the software open and computer on.

Experiment 1: Results for a well-characterized polycarbonate sample

The goal of this experiment is to obtain the DSC trace for a polycarbonate sample.

Use a single polycarbonate pellet and regular pan to prepare the sample. Use the "cup die" for crimping the lid onto the pan in the press. Use a ramp program that heats the sample from 40°C to 180°C at 10°C/min.

Experiment 2: Results for a poly(ethylene terephthalate) sample

The goal of this experiment is to learn about how the DSC trace changes for different samples of PET.

Prepare the sample by cutting a small piece of PET from one of the lab samples. (Your group will work with either a bottle or a fruit container.) Use the standard pan and cup die, as with polycarbonate. Use a ramp program that heats the sample from 40°C to 280°C at 10°C/min. If time permits after experiments 3 and 4, use a ramp that starts the sample at 110°C (above the glass transition) and brings it to 160°C at 5°C/min. How do the results compare between the two heating rates?

Experiment 3: Functioning polyurethane from Mackal Gym

The goal of this experiment is to learn via the DSC trace about the transitions found in a polyurethane that "works" as a gym floor.

Prepare the sample using a small piece of the non-degraded floor. Note if the sample corresponds to the "2003 floor" or "2005 floor". The pan choice (regular or hermetic) will be decided in the lab, based on your sample. Heat it from -40°C to 200°C at 10°C/min. Weigh the sample afterwards, and repeat the experiment if time permits (not likely).

Experiment 4: Nonfunctioning polyurethane from Mackal Gym

Use a wooden toothpick to add the appropriate amount of the degraded floor to a hermetic pan. Note again if the sample corresponds to the "2003 floor" or "2005 floor". Heat it from -40°C to 200°C at 10°C/min. Weigh the sample, and repeat the experiment if time permits (not likely).