DSC Laboratory Experiment – Determining the Thermal Transitions of Semi-crystalline Polymers

Purpose

The purpose of this laboratory is to introduce the thermal transitions of thermoplastics. This lab will include:

1. Introduction to the two classes of common thermoplastics (Semi-Crystalline and Amorphous).
2. Techniques to determine the glass transition (T_g), crystallization temperature (T_c), and the melting temperature (T_m) of polyethylene
3. Calculating the percent crystallinity for polyethylene from the heat of fusion obtained (ΔH_{fusion})
4. Demonstrate the relationship between the molecular weight of polystyrene and its T_g

Materials

Polyethylene (4000 and 35000 M_w) from Sigma Aldrich

Procedure

A Q250 DSC is utilized under a nitrogenous flow at 20 cm^3/m to prevent oxidation of the samples. In order to obtain sufficient results, 5 to 15 mg of each sample is weighed on an analytical balance. The samples are weighed at room temperature and placed in standard DSC pans. The pans are then crimped to seal the sample. The samples are ran under a heat/cool/heat cycle at 20°C/min (50°C to 300°C) to erase the sample's thermal history. After the completion of the last thermal cycle the samples are then subjected to a temperature ramp of 20°C/min (50°C to 300°C) at which point the data is collected.

Analysis

The thermal transitions in each system will be determined qualitatively by properly identifying which transition is occurring. This will be accomplished through comparison of experimental data with figure 1.

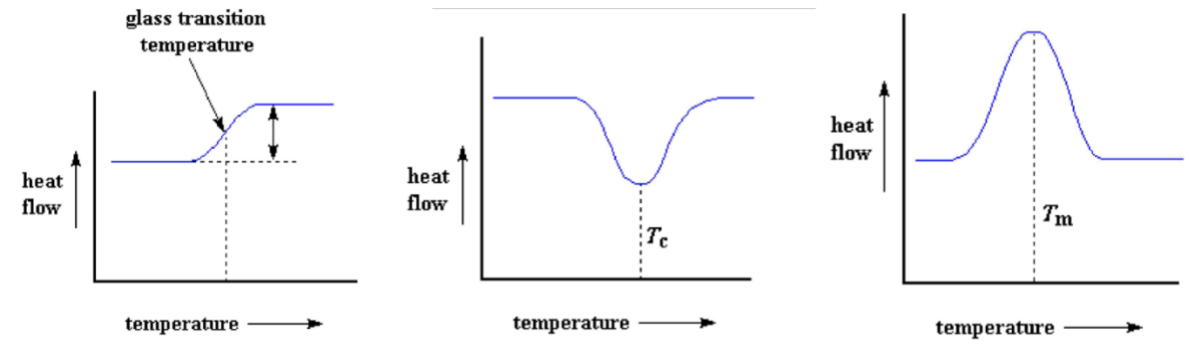

Figure 1. Illustration of polymer transitions.

Through data analysis, each transition temperature will be determined. Finally the heat of fusion will be obtained by utilizing the software to determine the area under the curve corresponding to the melting of the polymer.

Homework

1. If the molecular weight of polyethylene was increased to 70,000 what would be the effect on the T_g, T_m, and T_c.
2. If the heat of fusion for 100% crystalline polyethylene is 68.4 cal/g (Bernhard Wunderlich, 1967) what would be the degree of crystallinity for the polyethylene used in the lab. The degree of crystallinity can be calculated as:

$$\%Crystallinity = \frac{\Delta H_{experimental}}{\Delta H_{theoretical}}$$