Heat Capacity and Latent Heat

Objective

The objective of this laboratory is for you to explore the heat capacity of materials due to atomic vibrations and the latent heat of phase transitions and how these properties can be used to provide storage of thermal energy.

Preparation

Read the chapter from Haines on differential scanning calorimetry and the introductory sections of the review on thermal storage materials.

Equipment

- Station for differential scanning calorimetry (DSC)

- Samples of graphite, SIC, paraffin, Bi, stearic acid, $MgCl_2 \cdot 6H_2O$

- DSC Al cans and press for loading

- Computer, data-acquisition software, plotting and analysis software

Introduction

Knowledge of the heat capacity of materials and the latent heat of phase transitions is needed to understand the thermodynamics of materials and their phase transitions. For example, for measurements at constant pressure, the change in enthalpy is given by the temperature integral of the heat capacity and the change in entropy is given by the temperature integral of the heat capacity divided by temperature. For most solids, at temperatures away from any phase transitions, the heat capacity is controlled by the vibrations of the atoms. At a first order phase transition, the latent gives the difference in enthalpy and entropy of the two phases.

Heat capacity and latent heat also have a practical application in the storage of thermal energy from sunlight. The basic idea is to use the sun to create heat that can be stored for later use, either for heating or for conversion to electricity.

Session 1: Debye heat capacity and the latent heat of phase transitions

- Measure the heat capacity of graphite and SiC as a function of temperature; analyze to determine Debye temperature or Einstein frequency

- Measure the latent heat of the solid-liquid transformation of Bi, paraffin, stearic acid and $MgCl_2 \cdot 6H_2O$ for both heating and cooling. Choose the upper temperature range sensibly.

Discuss the entropy and enthalpy of the first order phase transitions.   Measure the phase transition of $MgCl_2 \cdot 6H_2O$.

- Analyze the potential of each material for storing thermal energy on a per mass basis and a per volume basis.

Session 2: Accuracy and precision in scanning calorimetry

- For measurements of the latent heat of first-order phase transitions, how does changing the scanning rate change the results? Why? Are the uncertainties different for Bi compared to stearic acid? Are the results for latent heat the same in heating and cooling? Why or why not?

- For measurements of heat capacity, how does changing the scanning rate change the results? Do some materials or loading methods give more accurate results than others?

Instrument procedures

DSC data acquisition

1. Log on to the computer station using your NetID and password.

2. Under Start -> All Programs -> PYRIS Software, launch Pyris Manager.

3. Click

4. In the Method Editor, enter Sample ID, Operator ID, and any comments. Enter the sample mass. If a baseline (used for heat capacity analysis) is being run, leave this box blank.   Under File Name, click browse. Go to C:\Program Files\Pyris\Data and click MSE 307. Go into the folder for your lab day and group. Choose an appropriate file name and click Save.

5. Under the Initial State tab, change the Initial Temp to a value appropriate for your experiment. We will discuss this in lab. Leave the Y initial at 20.00 mW.

6. In the Program tab, tailor your program to obtain the desired data. Typically, you will want to begin with a 2 minute isothermal hold, then begin heating.   Minimum and maximum temperatures are -120 and 450°C, respectively. The range of heating and cooling rates is 5-100°C/minute.

7. Remove the inner and outer lids of the DSC using tweezers. Make sure a reference pan is present in the furnace. Add the sample pan, making sure the sample pan is centered on the small circular platform. Replace the inner and outer lids.

8. If everything is set the way you want it, click to start the run.

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Analysis of heat capacity data

1. To determine heat capacity of a material on this DSC, you must first perform a baseline run. A baseline run will determine the small errors in the differential measurements of the heat capacity of the system (furnace, pans, air, etc.) and will be subtracted from the sample data. Place an uncrimped pan and lid in the furnace, along with a reference pan.

2. Using the Method Editor, set up your program exactly how you plan on setting up the sample program. Save the method file by clicking File-> Save Method As and selecting your group's folder. Give the file a name such as Graphite_baseline and save. Then, run the program.

3. After the program has run, take out the empty pan and lid and weigh 10-20 mg of material into the pan. Place the lid on, crimp the sample and place it back in the DSC. Execute exactly the same program, but add the sample mass and change the file name.

4. After your sample program has been run, a Data Analysis window should be open with your data curve. In the upper toolbar, click the Add Curve button ( ) and add your baseline data.

5. With the sample data curve highlighted, go to Math -> Subtract and click the baseline curve. The resulting curve is the data for just the material.

6. Click Calc -> Delta Y. Change the X location of the right limit to the temperature at which you want to know the heat capacity.

7. Calculate the heat capacity using $C_p = \Delta Y/(m\ \beta)$, where m is the mass of the sample in milligrams and β is the heating rate of the sample in degrees C per second.

8. Save your data file. Also, click File -> Export Data -> ASCII format and save. This will create a .txt file that can be opened with any spreadsheet program.

Analysis of latent heat data

1. No baseline is required for latent heat measurements. Weigh 10-20 mg of material into an empty pan. Place the lid on top, crimp and place in the DSC along with a reference pan.

2. In the Method Editor – Sample Info tab, insert sample info as before. For latent heat analysis, heating runs should begin about 50°C below the melting point of the material, hold at that temperature for 2 minutes, and then begin heating. Heating should continue to 50°C above the melting temperature, and then hold again for 2 minutes. For best results, cool the sample at the same rate as heating, again to 50°C below the melting point.

3. One curve will be present after data acquisition is complete, but only one can be analyzed at a time. Save your data file before analyzing. Right-click on either the heating or cooling half of the curve and choose remove curve. Click Calc -> Peak Area. Change the left and right limits to a point on either side of the endo/exothermic peak. Choose these points such that they lie on a flat portion of the curve, before the peak. Check the onset and end boxes, and if the molar mass is known, check the kJ/mole box and insert the value.

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4. Click Curves -> Heat Flow, and select the other curve for analysis. Remove the first curve and repeat the process.

5. When finished, load both curves back onto the plot and export to ASCII data.

Shut down

When you are done using the DSC for the day, exit out of Pyris Manager and tell the TA. Do not turn off the DSC, intercooler, or gas regulator. Transfer all of your ASCII files and any screen shots or other files either onto a USB memory stick or via email, netfiles, etc. Take your sample out of the DSC and place it in the provided sample trash bin.

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