MSE-S310 Lecture 3

Differential Scanning Calorimetry (DSC)

Recap

In **thermogravimetric analysis**, the change in weight in relation to a change in temperature in a controlled environment is measured.

Heat is used in **TGA** to force reactions and physical changes in materials.A thermocouple is used to accurately control and measure the temperature within the oven.

A TGA analysis is performed by gradually raising the temperature of a sample in a furnace as its weight is measured on an analytical balance that remains outside of the furnace.

Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) is a thermal analysis technique in which the heat flow into or out of a sample is measured as a function of temperature or time, while the sample is exposed to a controlled temperature program.

It is a very powerful technique to evaluate material properties such as glass transition temperature, melting, crystallization, specific heat capacity, cure process, purity, oxidation behavior, and thermal stability.

PRINCIPLE

When a sample undergoes a physical transformation such as a phase transition, more or less heat will need to flow to it than to a reference to maintain both at the same temperature whether more or less heat must flow to the sample depends on whether the process is endothermic or exothermic.

For e.g. as a solid sample melts to a liquid it will require more heat flowing to the sample to increase its temperature, at the same rate as the reference. This is due to the absorption of heat by the samples as it undergoes the endothermic phase transition from solid to liquid.

PRINCIPLE

Likewise, as the sample undergoes exothermic processes (such as crystallization) les heat is required to raise the temperature by observing the difference in heat flow between the sample and the reference. Differential Scanning Calorimeter is able to measure the amount of heat absorbs or release during such transition.

Differential Scanning Calorimeter measures the energy absorbed or released from a sample as a function of time or a temperature profile.

Instrumentation and Working of DSC

A small amount of sample (1-15 mg) was contained within a closed crucible and placed into a temperature-controlled DSC cell. A second crucible without sample was used as a reference.

A typical DSC run involves heating/cooling the sample at a controlled steady rate, and monitoring the heat flow to characterize the phase transitions and/or cure reactions as a function of temperature.

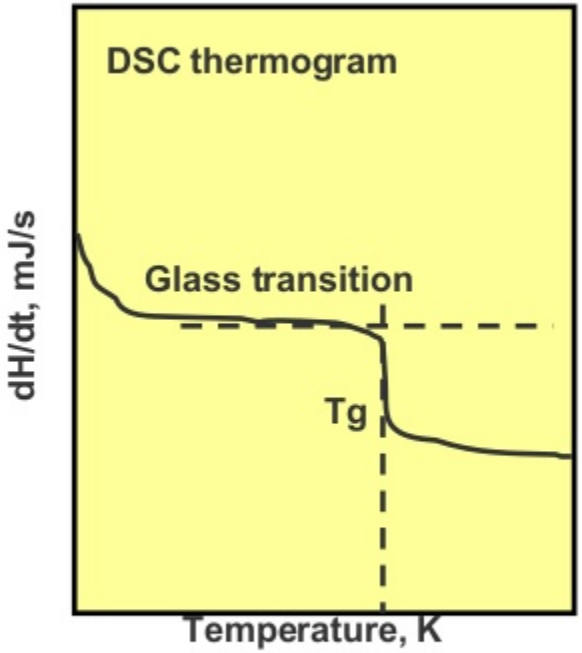
DSC is useful to make the measurements for melting points, heat of reaction, glass transition temperature, and heat capacity. A calorimeter measures the heat into or out the sample. A differential calorimeter measures the heat of sample relative to a reference. Calorimeter does all of the above and heats the sample with a linear temperature ramp.

THE GLASS TRANSITION TEMPERATURE

This temperature may occur as the temperature of an amorphous solid is increased. It appears as a stack in thermo gram. Glass transition is the transition from disordered solid to liquid. It is observed in glassy solids such as polymers. It is denoted as $T_{g.}$

Glass Transition - DSC

- Step in thermogram
- Transition from solid to liquid
- Transformation of a glass-forming liquid into a glass
- Tg, glass transition temperature



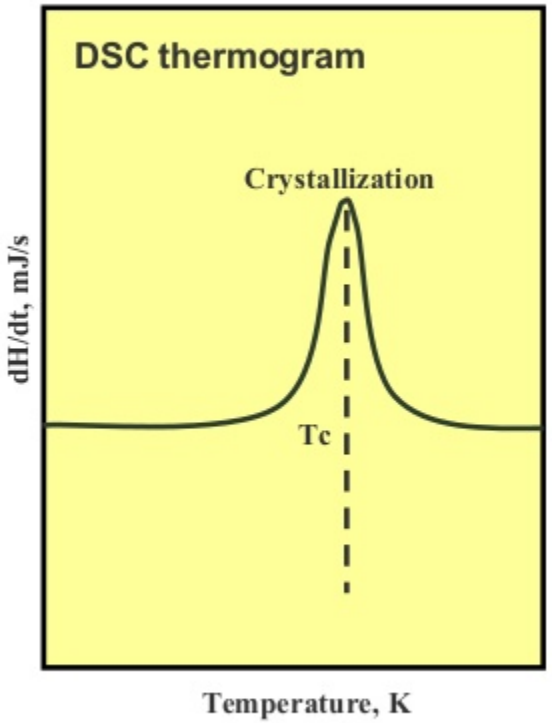
THE CRYSTALLISATION TEMPERATURE

As the temperature further increases an amorphous solid will become less viscous at some point, the molecules may obtain enough freedom of motion to spontaneously arrange themselves into a crystalline form. This is known as the CrystallizationTemperature.

It appears as a sharp positive peak. It is an exothermic process. This is the transition from disordered to ordered transition.

Crystallization

- Sharp exothermic peak
- Disordered to ordered transition
- Material can crystallize
- Observed in glassy solids, e.g., polymers
- Tc, crystallization temperature

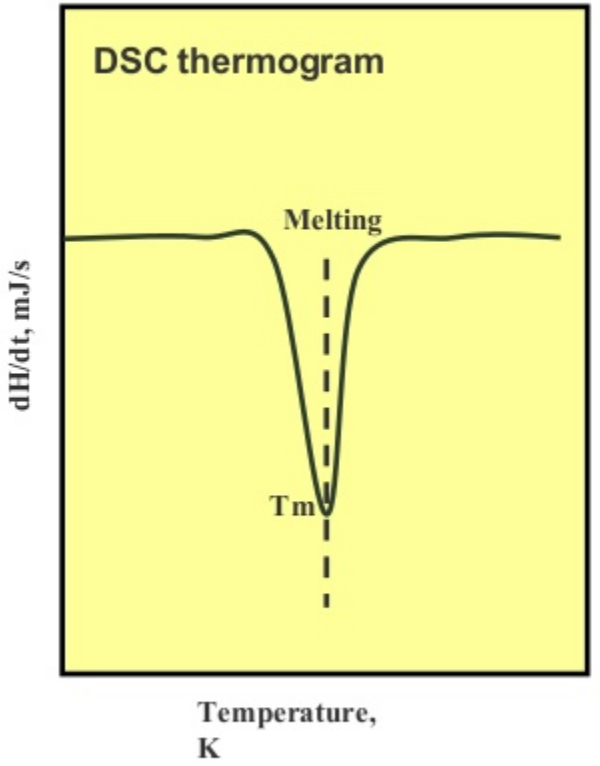


THE MELTING TEMPERATURE

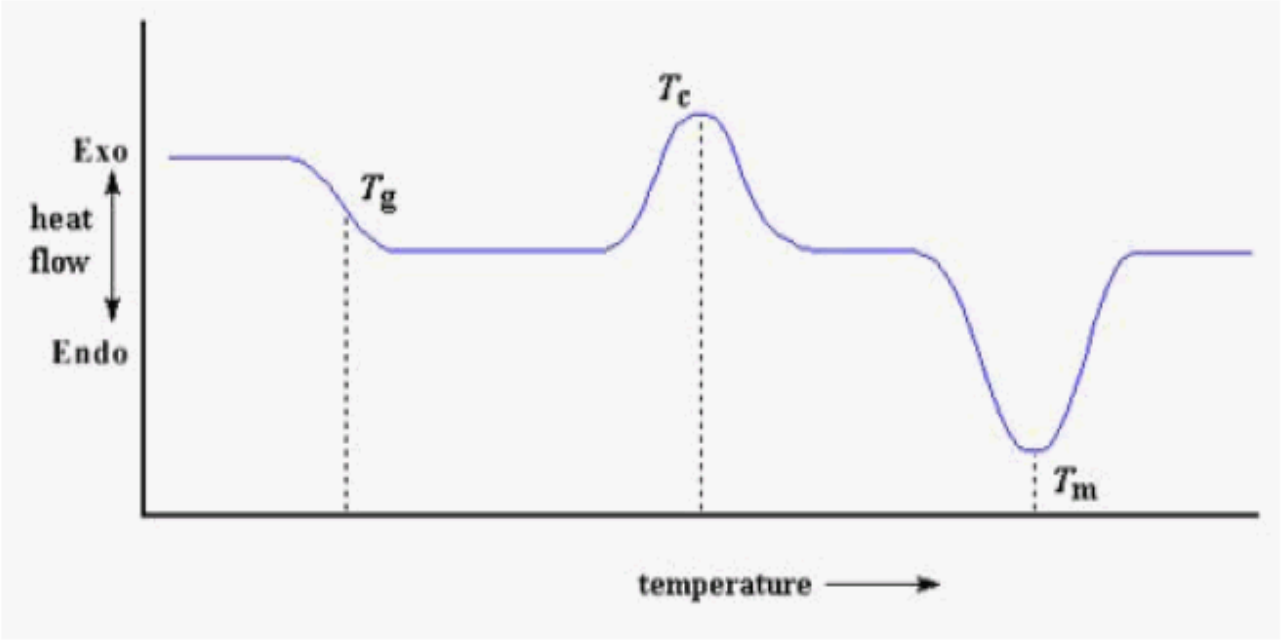
On further increasing the temperature the sample eventually reaches its Melting Temperature. The melting process is an endothermic process and it appears as a negative peak in the thermo gram. This is the transition from ordered to disordered transition. It is denoted as Tm.

Melting

- Endothermic peak
- Ordered to disordered transition
- Tm, melting temperature
- Tm > Tg



DSC Curve



These notes have been prepared with the help of Materials Characterization books by Sam Zhang, Lin Li, Ashok Kumar.