DIFFERENTIAL SCANNING CALORIMETRY (DSC)

Introduction

DSC is one of the analytical techniques belonging to a group called Thermal Analysis (TA). They involve meausuring changes of various physical quantities with changing temperature *e.g.*:

technique	measured quantity
DTA – Differential Thermal Analysis	temperature difference (between sample and reference)
DSC – Differential Scanning Calorimetry	heat difference (between sample and reference)
TG - Thermogravimetry	mass
TMA – Thermomechanical Analysis	dimensions

The applications of DSC include:

- phase transition definition including melting point, glass transition, Curie point
- determination of crystallinity
- kinetic studies
- material fingerprinting

Theory

The idea of a DSC experiment is to heat up or cool down a sample and a reference according to a set temperature programme. The sample and reference are maintained at the same temperature during the whole experiment. When a thermal event occurs in the sample, certain, additional amount of energy has to be supplied to or withdrawn from the sample to maintain zero temperature difference between the sample and the reference. Therefore, the reference should not undergo any physical or chemical changes at the temperature range of the experiment. The sample and reference are placed in identical environments – metal pans on individual bases, each of which contains a platinum resistance thermometer (or a thermocouple) and a heater (Fig. 1). An empty pan is very often used as a reference. The temperatures of the two thermometers are compared and the electric power supplied to each heater adjusted so that the temperatures of the sample and the reference remain equal *i.e.* any temperature difference which would result from a thermal event in the sample is compensated for. If an exothermic change occurs in the sample, more heat has to be supplied to the reference (which is equivalent to withdrawing energy from the sample). During an endothermic process, additional amount of energy has to be supplied to the sample heater. The difference in the heat supplied to the sample and the reference is recorded as a function of temperature. This signal is proportional to the sample specific heat, which determines the amount of heat that is necessary to change the sample temperature by a given amount.

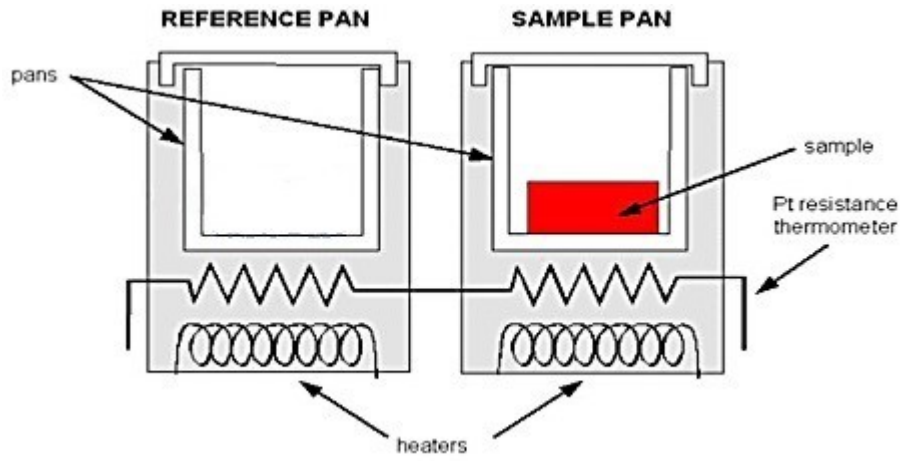

Fig. 1. DSC experimental setup
(adapted from http://www.evitherm.org/Files/982/TherAna_Depository_MeasurementMethods_DiffPowerScanCalorimeter.jpg)

Any transition that is accompanied by a change in specific heat produces a variation in the power signal. Exothermic and endothermic processes give peaks with areas proportional to the total enthalpy change of the events (Fig. 2).

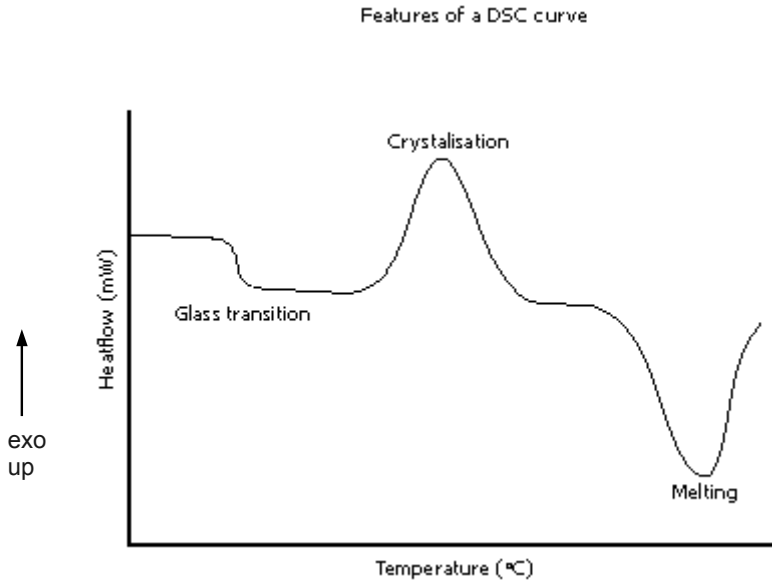

Fig. 2. A typical DSC curve
(source: http://en.wikipedia.org/wiki/File:InterprettingDSCcurve.png)

Summing up, the measuring principle in DSC is to compare the rate of heat flow to a sample and to a reference. Both the sample and the reference are heated or cooled at the same rate and their temperatures are maintained at the same level. Changes in the sample that are associated with absorption or evolution of heat cause variations in the differential heat flow which are then recorded as peaks, The area of an individual peak is directly proportional to the enthalpy change of the sample and the direction of the peak indicates whether the thermal event is exo- or endothermic.