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Identification of Polymers with Differential Scanning Calorimetry

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Abstracts: Differential Scanning Calorimetry, commonly known as DSC, is a thermo analytical technique for polymeric and non- metallic materials. There are many application of differential scanning calorimetry such as, Measurement of plastic or glassy material glass transition temperatures or softing temperatures, which change depend upon the temperature history of the polymers or the amount and type of fill material among other effect. Determines crystalline to amorphous transition temperatures in polymers and plastics and the energies for inorganic compound. Differential Scanning Calorimetric is one of the most efficient and cost effective polymer test methods available. Differential scanning calorimetric can be used to study the melting of a crystalline polymer or the glass transition.

Keywords: Crystallization; crystallization temperature; Differential Scanning Calorimetry (DSC);enthalpy ; first –order transition; glass transition ; heat of fusion; heat of transition; melting temperature; polymer; transition temperature

INTRODUCTION

Differential Scanning Calorimetry, commonly known as DSC, is a thermo analytical technique for polymeric and non- metallic materials. Most commonly used for detecting glass transition temperature and other thermal properties, Differential Scanning Calorimetric is one of the most efficient and cost effective polymer test methods available. Differential scanning calorimetric can be used to study the melting of a crystalline polymer or the glass transition[1,2].

The technique was developed by E.S. Watson and M.J. O'Neill introduced commercially at the 1963. The first Differential Scanning Calorimeter that could be used in biochemistry was developed by P.L. privalov and D.R. monaselidze at institute of physics[4,5]

The term DSC was coined to describe this instrument. Which measures energy directly and allows precise measurement of heat capacity?

THE INDIAN STANDARD

ASTM D 3418 Standards:

E 473 Terminology Relating to Thermal Analysis and Rheology
E 691 Practice for conducting an interlabortry study to determine the precision of a test method
E 967 test method for heat flow calibration of differential scanning Calorimeters and differential thermal analysers
E 968 Practice for heat flow Calibration of DSC
E 1142 Terminology Relating to thermo physical properties
E 1953 Practice for Description of thermal analysis and Rheology Apparatus.

ISO Standard:

ISO 11357-1 plastics Differential Scanning calorimetric (DSC) Part 1: Gernal principles
ISO 11357 -2 plastics Difrerential Scanning Calorimetric (DSC) Part 2: Determination of Glass Transition Temperature Scaning Calorimetry (DSC) Part 3: Determination of Temperature
ISO 11357- 3 Plastics: Diffrential and Enthalpy of melting and crystallization

Table 1: Range of polymer

Material	Softening range
LDPE	105-117℃
LLDPE	117-127℃
HDPE	128-138℃
HMHDPE	138-139℃
PP	164-165℃
PP/CP	172-179℃
NYLON	220℃
PET	250-254℃

Typical DSC specification:

Temperature Range: 80℃ -500 ℃

Temperature accuracy: ± 0.2 k

Temperature precision: ± 0.02 k

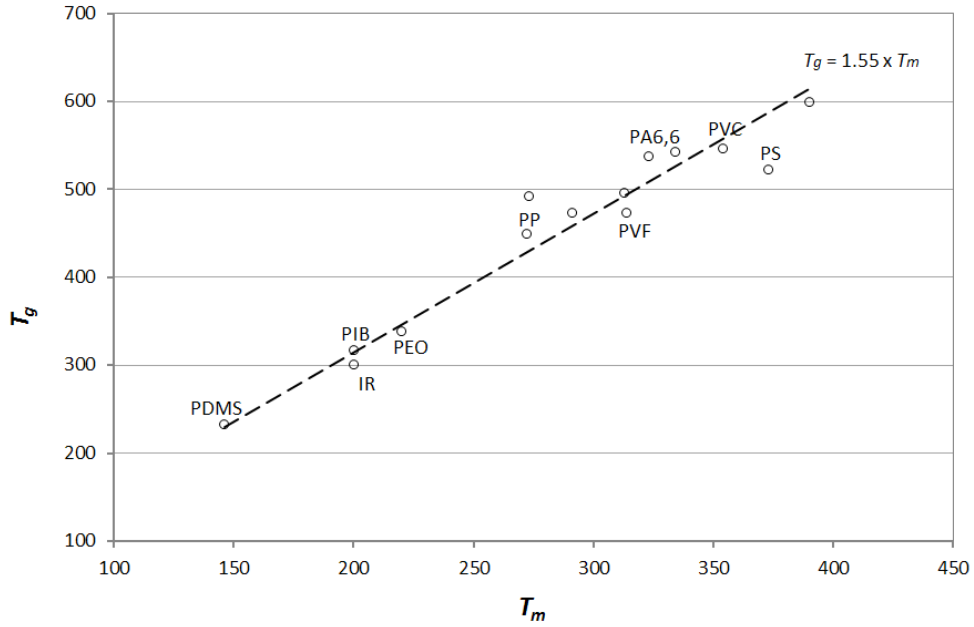
Furnace temperature Resolution: ±0.00006 k

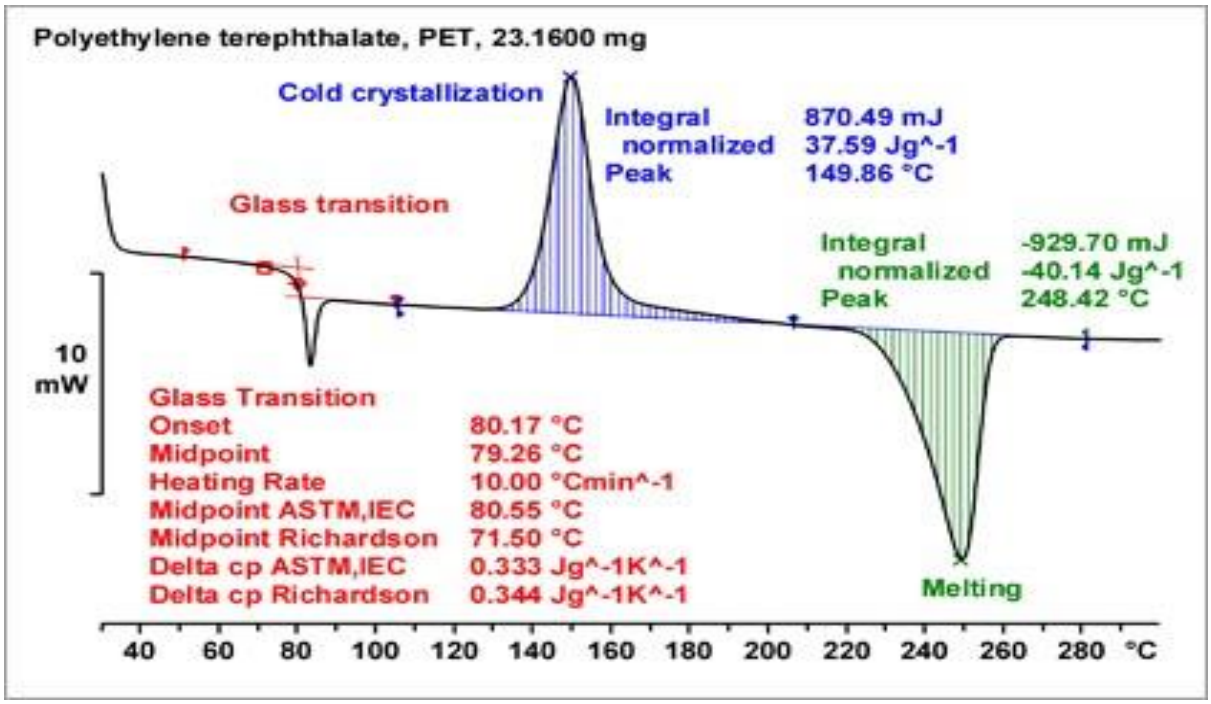
Heavy rate: 0.02.50k\min

Calorimetric Resolution 0.01 μ W

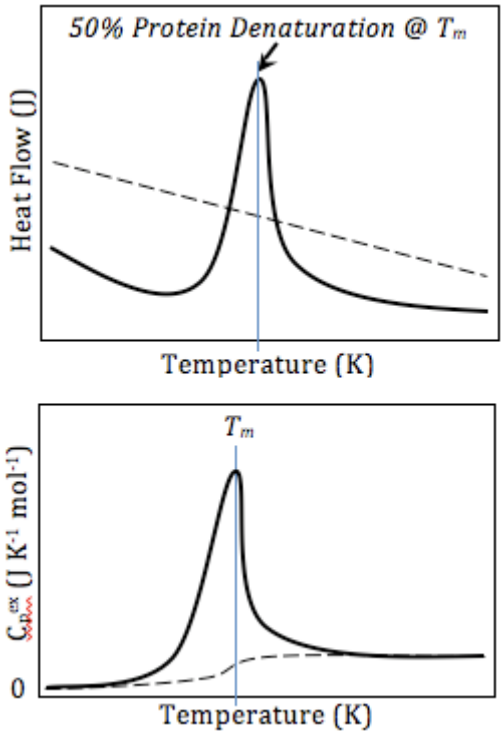
Measuring environment: Air, N_2, and O_2 (other possible)

Automation: Sample Robot, 42 positions





Melting: The polymer chains are able to move around freely at the melting temperature and thus do not have ordered arrangements. Melting is an endothermic process requiring the absorption of heat .the temperature remains constant during melting despite continued heating the energy added during this time is used to melt the crystalline regions and does not increase the average kinetic energy of the chain that are already in the melt in a plot of heat flow against temperature this appears as a jump discontinuity at the melting point as seen .it can be calculated from the area of a melting peak observed in a plot of heat flow against temperature after melting the temperature again increases with heating. However, the heat capacity of a polymer in the melt is higher than that of a solid crystalline polymer. This means the temperature increases at a slower rate than before.



SET-UP AND EXPERIMENT

Important:

1. Make sure the nitrogen is connected and flowing.
2. We clean sample pans. Make sure nothing contacts the heat surface .be sure the sample cannot flow out of the cell after melting.
3. In general, never heat aluminium pans above 500 oc.

Starting the system:

1. Log into the computer with the username DSC and password DSC
2. Starting the DSC by pressing the power button the instrument.
3. Make sure the purge nitrogen is connected and the flow rate is correct. Set the floometer to match the two black marks.
4. Allow the machine to warm up for 30 sec. and then start the control program by clicking the TA controller icon.

Encapsulating the sample:

1. Practice making a few nonthematic sample pan to become familiar with the procedure before trying with your sample
2. Wight the sample pan then place the lid on the pan.
3. Place the pan in the well of the crimping dry.
4. Pull the press lever forward until the handle hits the stop.
5. Raise the lever and remove the pan with tweezers. Ask for assistance if the pan is stuck. Inspect the pan .the bottom should be smooth and the side should appear rolled down.
6. If quantitative work is to be done, weigh the pan with encapsulated sample to determine the sample weight.
7. Prepare an empty nonthematic pan with lid to use as a reference.

8. Use tweezers to remove the cell cover and silver lid from the heating chamber.
9. Carefully place the sample pan on the raised platform in the front and the reference pan on the platform in the rear.
10. Centre to the pans on the grid to end use they we cantered on the platform
11. Replace the silver lid and cell cover.

REPORT

1. Complete identification and description of the material tested, including source, manufacturer's code.
2. Description of instrument used for the test.
3. Statement of the mass dimention, geometry and materials of specimen container and the heating rate.
4. Description of temperature calibration procedure.
5. Identification of the Sample atmosphere by purge gas flow rate purity and composition including humidity, if applicable.
6. Heat of fusion or crystallization, or both.

Application of Differential Scanning Calorimetry:

1. Metal alloy melting temperatures and heat of fusion.
2. Metal magnetic or structure transition temperatures and heat of transformation.
3. Intermetallic phase formation temperature and exothermal energies.
4. Oxidation temperature and oxidation energy.
5. Exothermal energy of polymer cure (as in epoxy adhesives) allows determination of the degree and rate of cure.
6. Measurement of plastic or glassy material glass transition temperatures or softing temperatures, which change depend upon the temperature history of the polymers or the amount and type of fill material among other effect.
7. Determines crystalline to amorphous transition temperatures in polymers and plastics and the energies for inorganic compound.
8. Oxidative induction period of an oil and fat.
9. May be used as one of multiple techniques to identify an unknown material or by itself to confirm that it is the expected material.
10. Determine the thermal stability of a material.
11. Determine the reaction kinetics of a material.

REFRENCES

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