DIFFERENTIAL SCANNING CALORIMETRY (DSC)

BY

Dr. Sanjay Shivram Ankushrao

(M.Sc., SET, GATE, PhD)

Assistant Professor,

Vivekanand College, Kolhapur (Autonomous)

Outline

1. General Introduction
2. Principle: What is DSC?
3. Various techniques of DSC
4. Instrumentation and Working of Heat Flux DSC
5. Factors Affecting to DSC Curve
6. Applications

1. General Introduction

- *It is fundamental tool in Thermal Analysis*
- *It is widely used in many industries from- Food, Pharmaceticals, Polymers, Agriculture, nanomaterials, etc.*

2. What is DSC?

It is thermal technique in which,

The heat or energy necessary to establish a zero temperature difference between sample and reference is measured as a function of temperature or time, when both are heated or cooled at predetermined rate.

It was developed by E. S. Watson and M. J. O'Neill (1962)

3. Various Techniques of DSC

A) Heat Flux DSC

- ***Single heater is used to heat Sample and reference***
- ***Allow tempt. Between Sample and Reference to vary.***
- ***This diff. in tempt is used to get required heat flow.***
- ***Less sensitive***
- ***Slower heating and cooling rates***
- ***Less accurately measures heat capacity and enthalpy.***

B) Power Compensated DSC

- ***Two identical and separate heaters are used***
- ***Tempt. Diff. during phase transition or reaction in the sample are compensated by varying electrical current supplied to heaters.***
- ***The supplied electrical current is directly proportional to heat absorbed during process.***
- ***Gives Better resolution***
- ***Rapid cooling and heating***

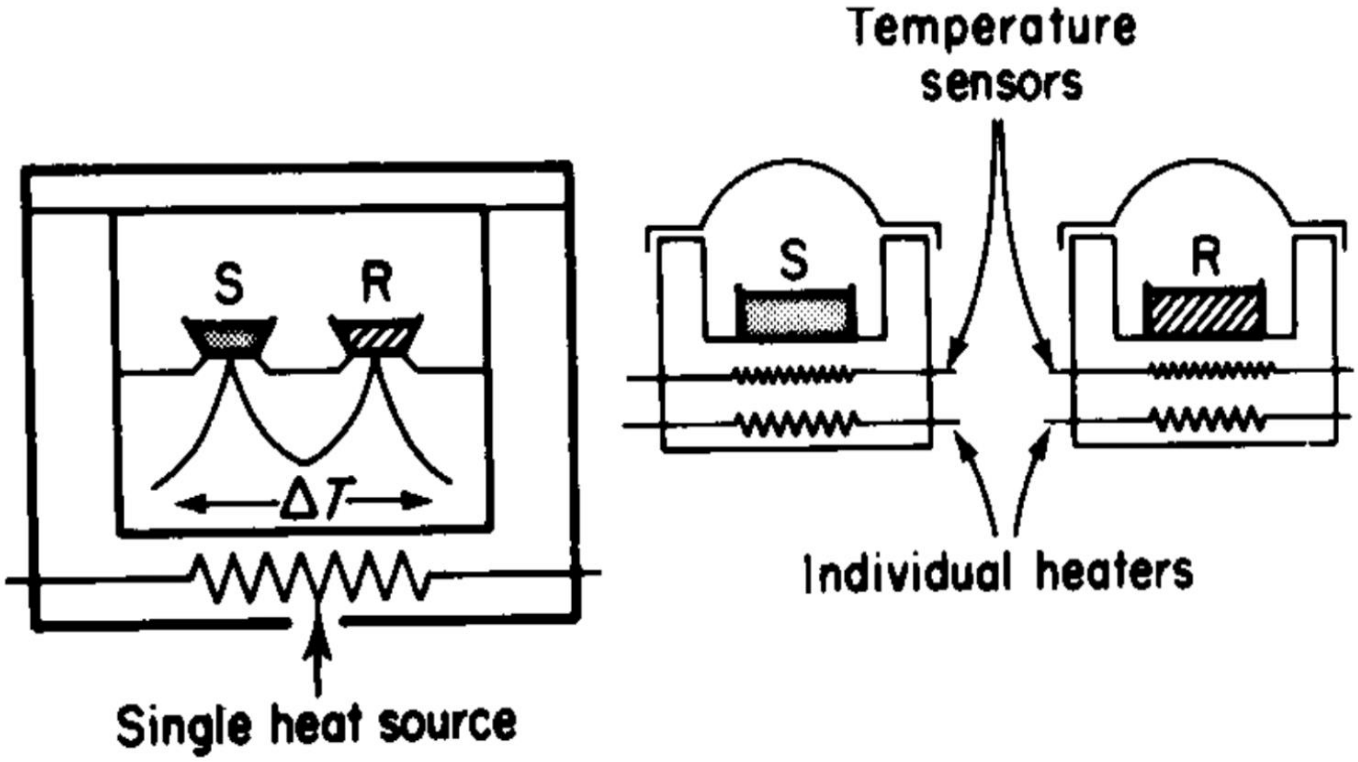
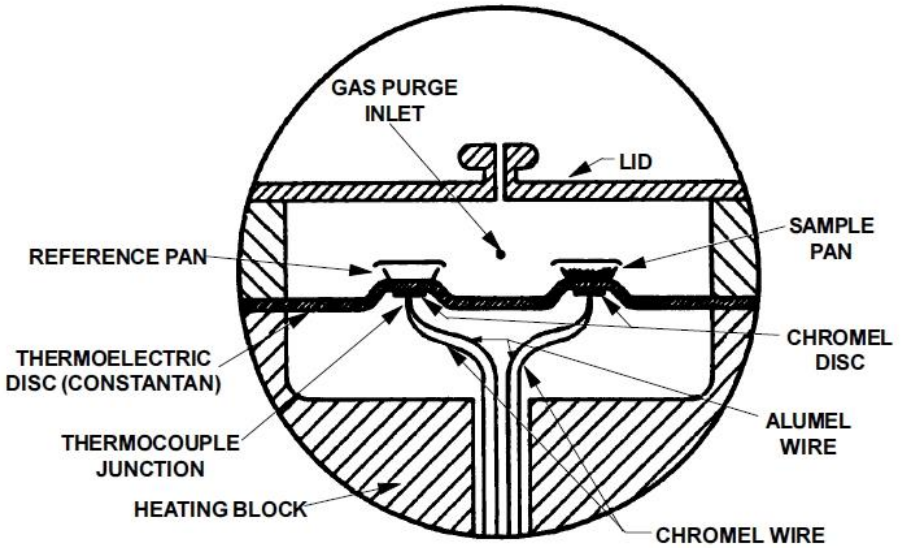


Fig. 1: (a) Heat flux DSC; (b) power–compensation DSC

4. Instrumentation & Working of Heat Flux DSc



- Consist of Cylindrical Silver heating block
- This block gives heat to the specimen via constantan thermoelectric disc
- The disc has two raised platfoms on which 'S' & 'R' placed
- Underside of each platform chromel disc is attached
- Chromel-constantan thermocouple used to measure differential tempt
- Alumel- Chromel thermocouple determines individual tempt of S & R
- Thermocouple embedded in Silver block, acts as tempt controller for programmed heating cycle.
- Inert gas passed at const. flow 40 ml per min.

Schematic Diagram of DSC

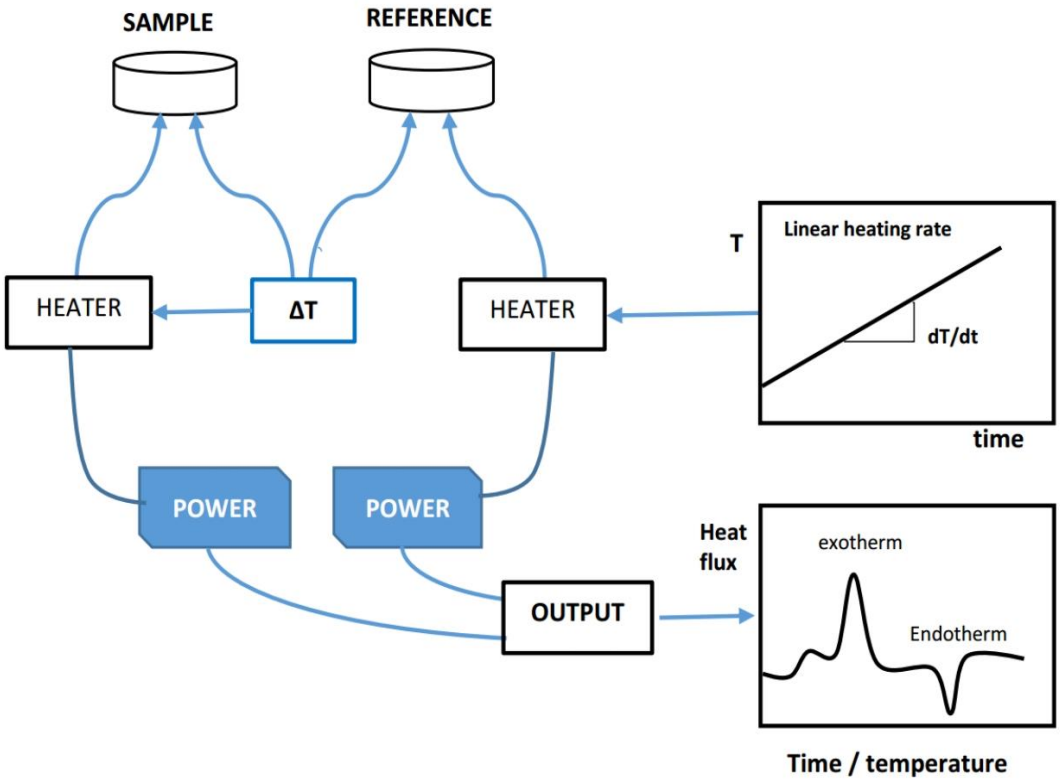
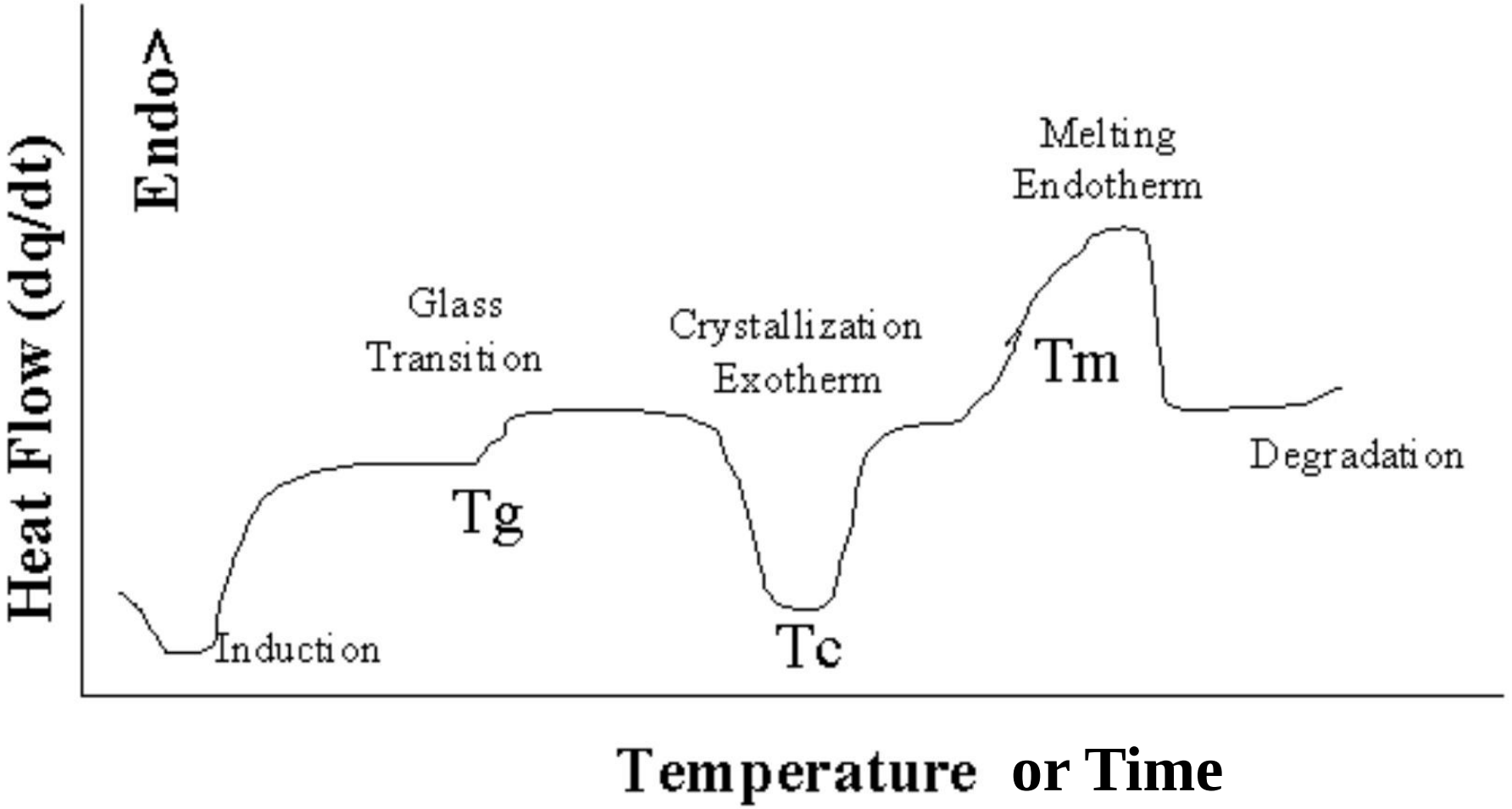


Fig. 2.2: Schematic diagram of a Differential Scanning Calorimetry (DSC)

Working of DSC

- *S & R sealed in Al or Cu pan*
- *Placed adjacent to each other*
- *Common heating applied to both*
- *Heater heats S & R at const rate and sensor senses tempt*
- *Now sample tends to absorb or release heat due to transitions & tempt varies*
- *To maintain constant tempt more or less heat supplied to sample*
- *The amt of heat transferred to sample is monitored by PC and output received in the form of thermogram.*

Nature of Curve and transitions obtained in DSC



5. Factors affecting to DSC Curve

A) Instrumental Factors

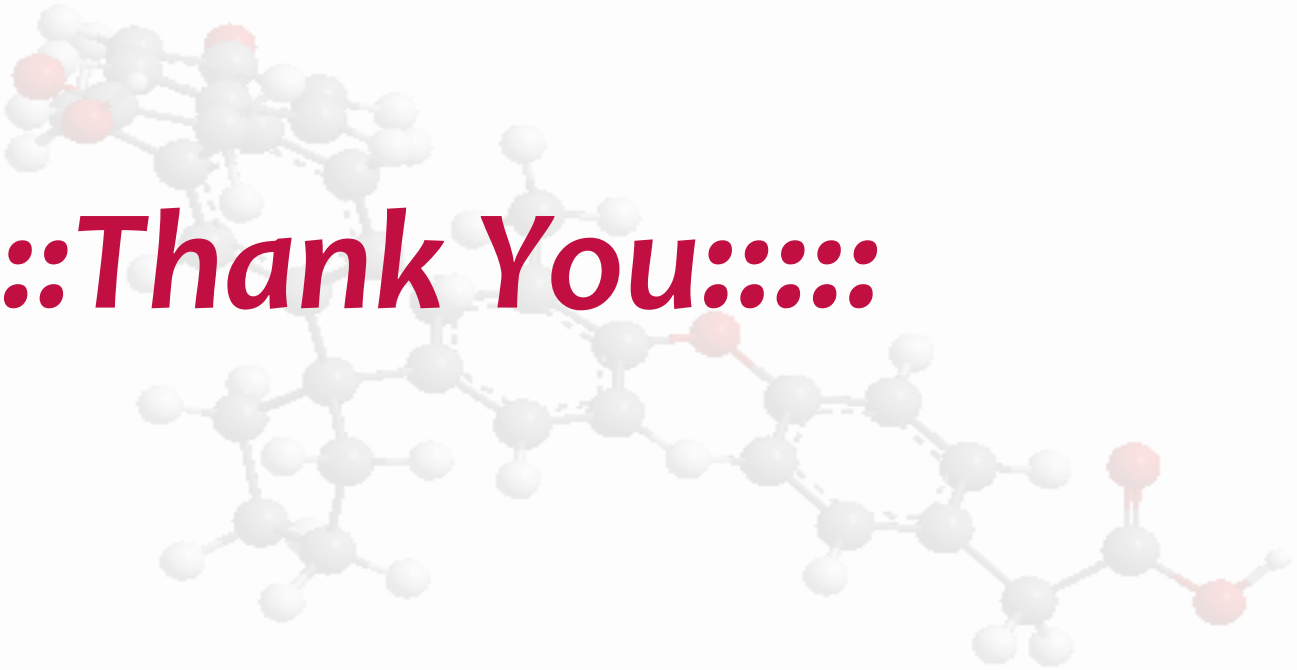
- Heating rate
- Geometry of Sample & Location of sensor
- Recorder or chart speed
- Sensitivity of recorder
- Furnace atmosphere
- Material of sample holder

A) Sample Characteristics

- Amount
- Size of Particle
- Nature
- Compactness
- Heat of reaction
- Thermal conductivity
- Solubility of evolved gases in sample.

6. Applications

Sr. No	Industry	Transitions	Purpose
1.	Pharmaceticals	Tg	Storage tempt, amorphous content
		Cp	Processing conditions
		Tm	Polymorphs, purity , QC
2.	Polymers	Tg	Processing, Material Property, effect of additives
		Tm	Polymer processing, heat history
		Exotherm	Reaction rate, curing of materials, etc.
		Cp	Energy needed to process
		Tc	Recrystallization time, kinetics
3.	Food	Tg	Storage tempt, other properties
		Tm	Processing tempt.



:::::Thank You:::::