Thermal Analysis

TGA – Thermogravimetric Analysis
DSC – Differential Scanning Calorimeter

Dr. Aseel B. AL-Zubaidi

Basic Principle

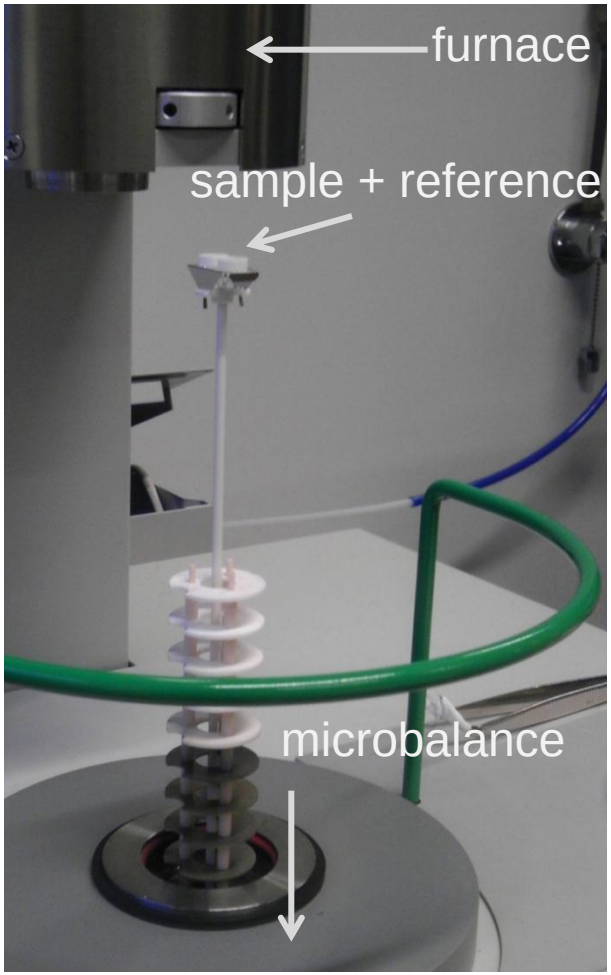
Sample is heated at a constant heating rate

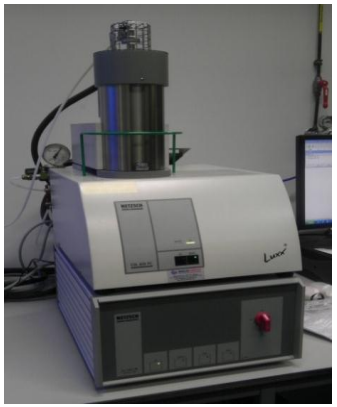
	Sample's Property Measured
TGA	Wt
TMA	Size
DSC	Heat Flow
DTA	Temp
TPD	Gas evolved

Measurement conditions

Netzsch STA 409 PC Luxx:

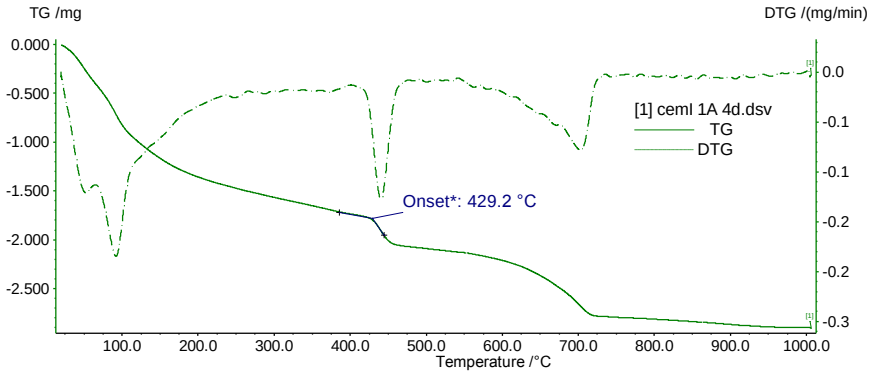
- T [25 – 1000 °C]
- heating rate 10 °C/min
- N_2 atmosphere (60 ml/min)
- alumina crucibles
- simultaneous measurement of mass change (TG) and heat flow (DSC)
- 1 analysis (25→1000→25 °C) = 3 hrs





Thermal analysis (TGA/DSC)

TG analysis – mass (T)



Examples of thermal reactions resulting in **mass change**, measured by **TG** analysis:

loss of free water-

loss of bound water-

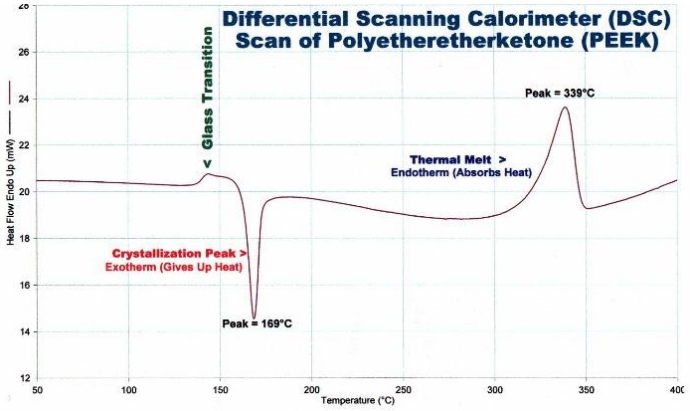
decomposition-

TG applications:

classification and quantification of sample components -

thermal stability studies-

DSC analysis – heat flow (T)



Examples of thermal reactions resulting in **heat flow**, measured by **DSC** analysis:

- crystallization
- melting
- glass transitions

DSC applications:

- thermodynamic characterization of pure substances
- quality control: sample purity
- thermal stability studies

Sample

Requirements:

- no reaction with alumina crucible
- no expansion or creep during thermal decomposition

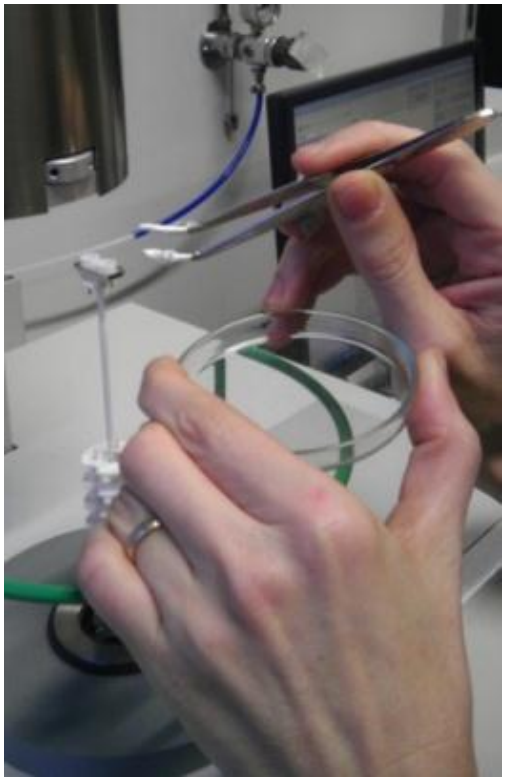
well-matched materials:

- clays and other geological materials
- cements
- slags

limits:

- polymers can be dangerous, as they can foam at high temperatures,

Sample preparation



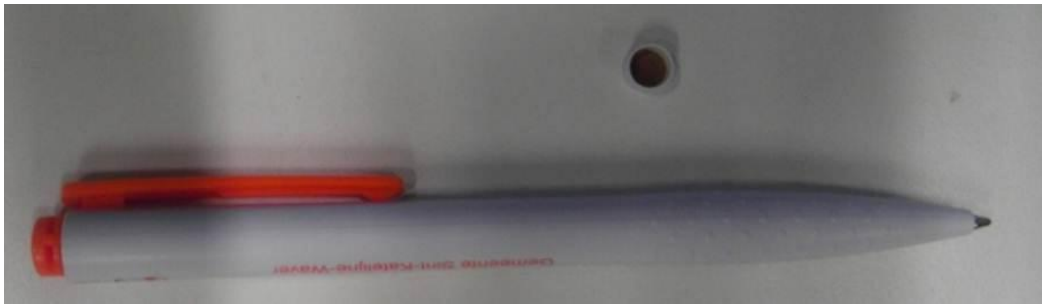
Sample form:

- fine powders
- compact solids
- films, fibers

Ensure good thermal contact between sample and heat flux-sensor:

- powders: evenly distributed at the bottom of the sample crucible, gently tamped

Always use the same sample mass (~ 10-30 mg)

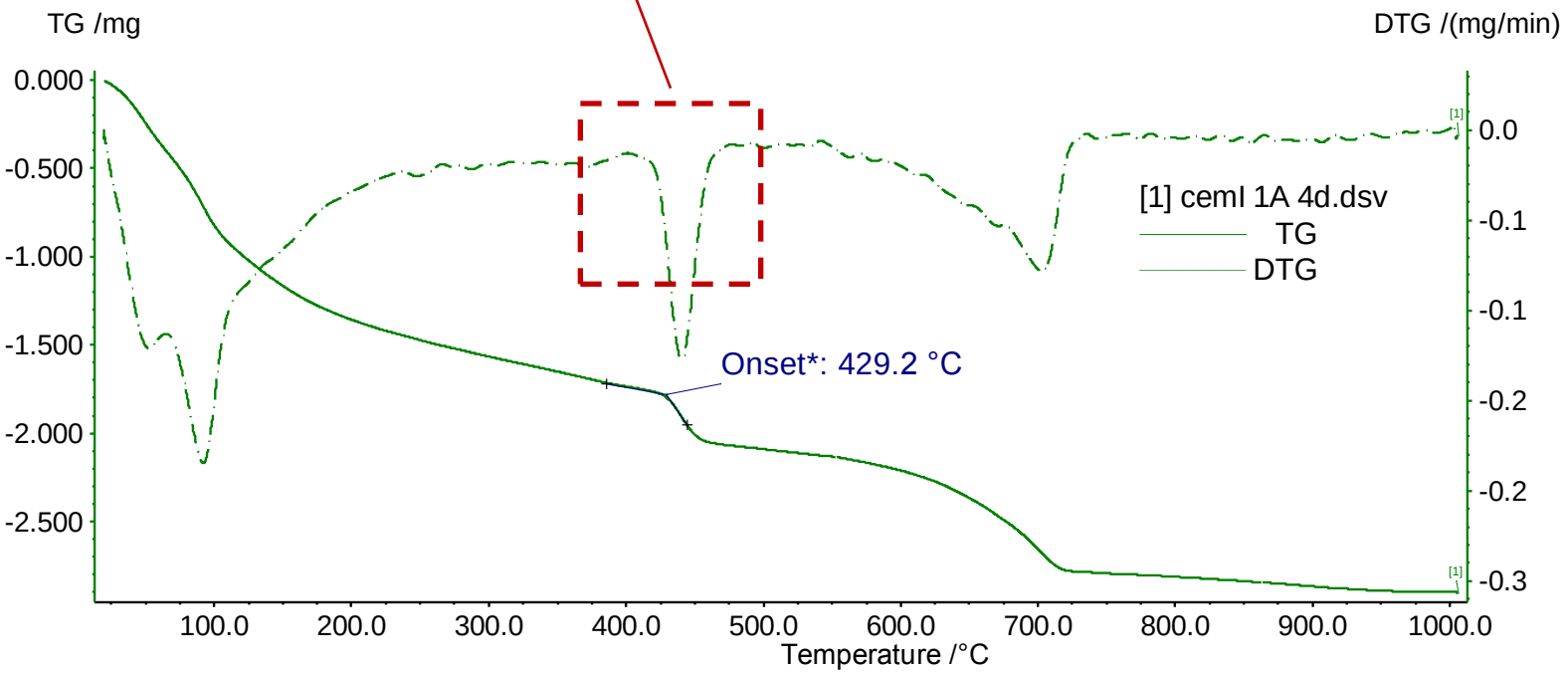


Analysis of results

Software: NETZSCH Proteus® (Marsh procedure)

Quantification of portlandite ($Ca(OH)_2$) content in cement

~430°C: $Ca(OH)_2$ -> $CaO + H_2O\uparrow$



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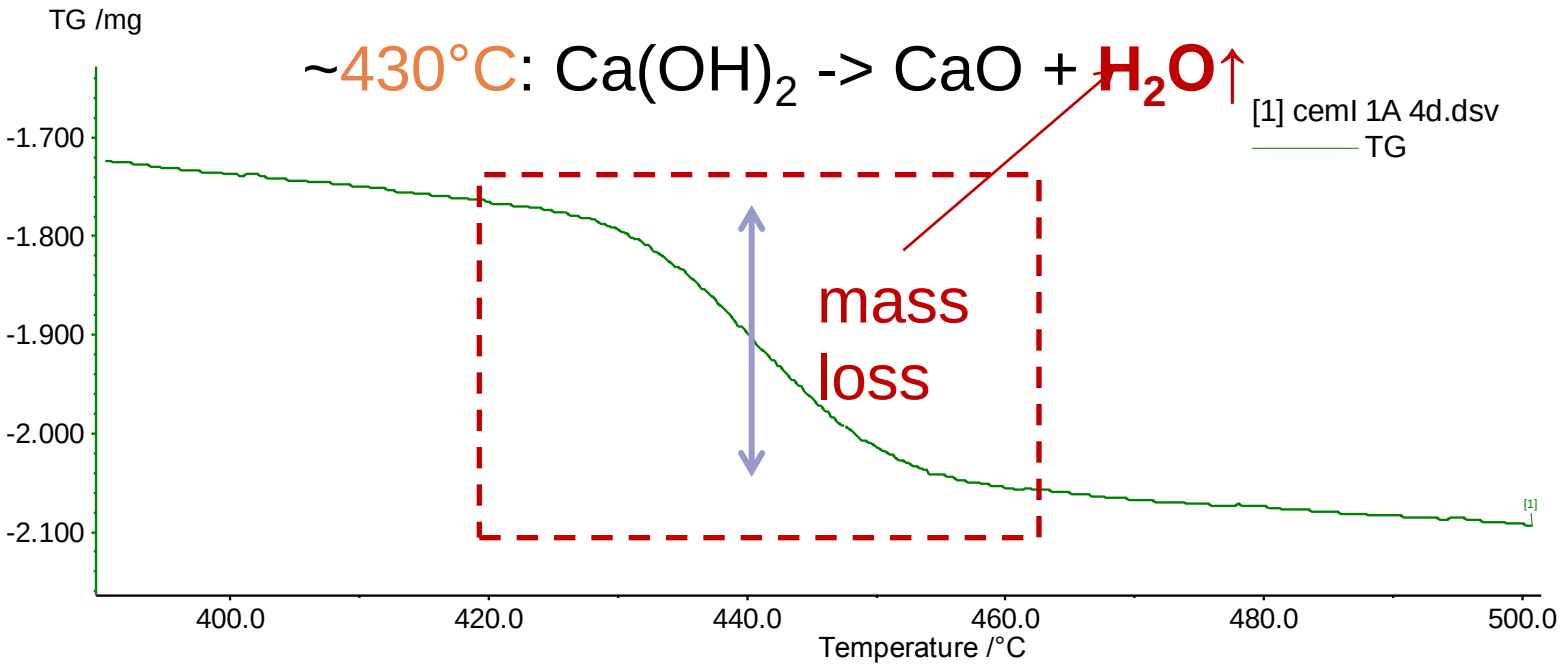

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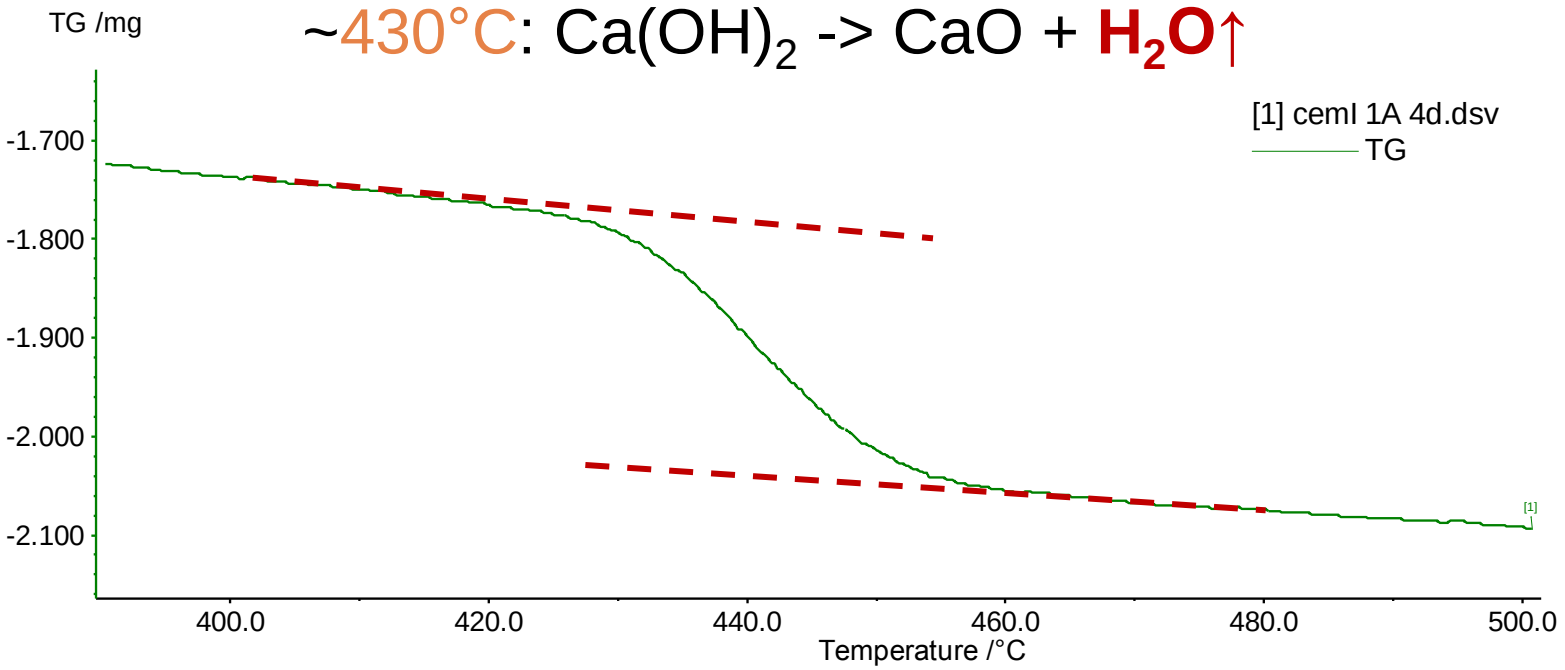


DSC

Constant Heating R

Initial Temp

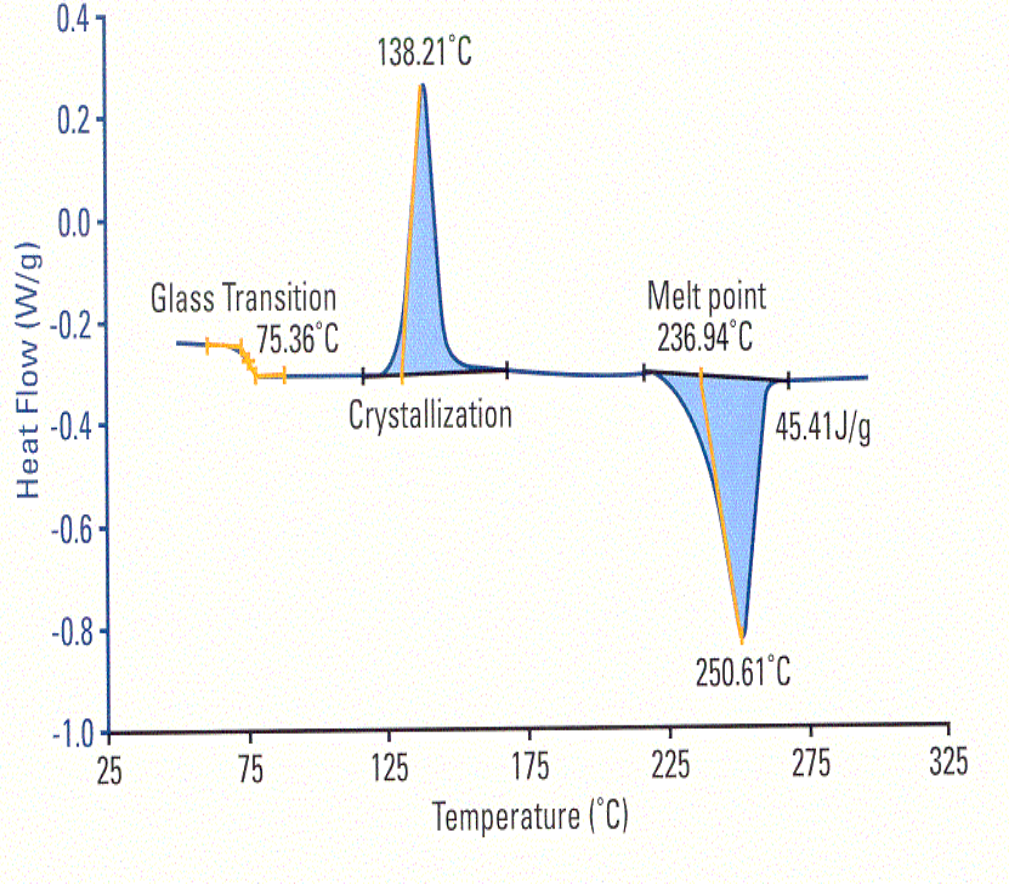
Final Temp

Heating Rate (°C/min

D

Heat flow to sample minus
Heat flow to reference vs
Time (Temp.)

Measures hea
crystallizatior



Polymer without weight change in this temperature range

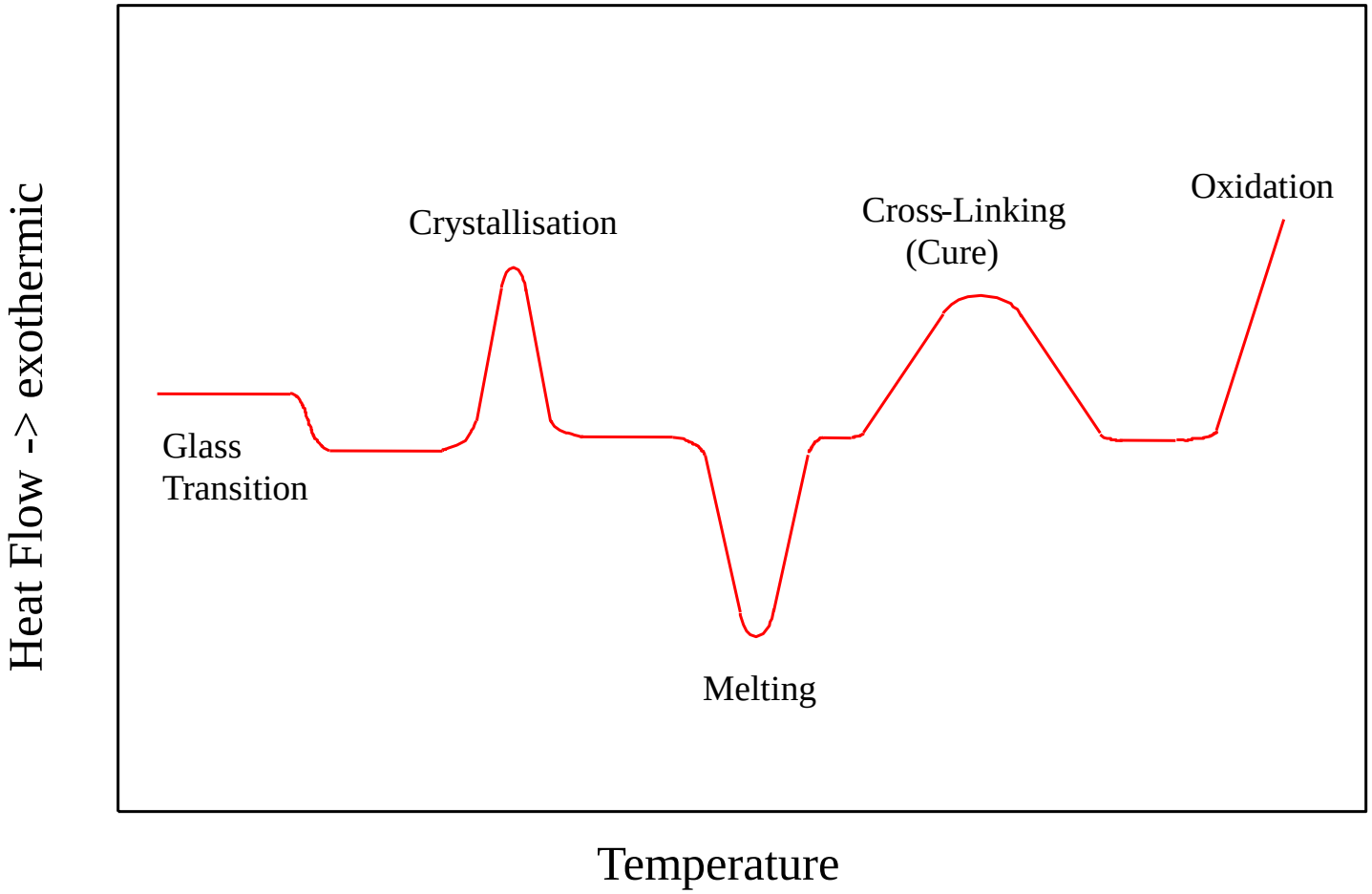
Definitions

- A ***calorimeter*** measures the heat into or out of a sample.
- A ***differential calorimeter*** measures the heat of a sample relative to a reference.
- A ***differential scanning calorimeter*** does all of the above and heats the sample with a linear temperature rang.
- ***Endothermic*** heat flows into the sample.
- ***Exothermic*** heat flows out of the sample.

What can DSC measure?

- **Glass transitions**
- **Melting and boiling points**
- **Crystallisation time and temperature**
- Percent crystallinity
- **Heats of fusion and reactions**
- Specific heat capacity
- Oxidative/thermal stability
- **Rate and degree of cure**
- Reaction kinetics
- Purity

DSC Thermogram



Influence of Sample Mass