

# DSC

## Differential Scanning Calorimetry

Polymer reaction towards temperature can be studied by performing DSC measuring (**Differential Ethalpic Analysis** or **Differential Calorimetric Analysis**).

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### ▲ Principle

The apparatus is equipped with two measuring cells: one referential cell (generally empty) and another in which the sample to be analysed is placed. Each cell has its own heating and temperature control system. An identical temperature programme is applied to both cells.

When a transition phenomenon occurs in the sample, the latter can no longer respond to the temperature programme and a difference in temperature is detected between the two cells. The apparatus then supplies enough heat to keep both cells at the same temperature. These are the calorific compensations which are recorded according to time and temperature. The calorific capacity of the observed material can then be deduced.

### ▲ Accessible sizes

- Vitreous transition temperature (Gt/ glass temperature): Vitreous transition temperature can be observed by abrupt variation of the calorimetric capacity (Cp jump).

- Fusion temperature (Ft) and Crystallisation temperature (Ct), Fusion enthalpies and crystallisation enthalpies, crystallinity rate (xc).

When a certain amount of favourable conditions are gathered (structural regularity, gradual cooling...), Certain polymers can crystallise (semi-crystalline polymers). For a temperature above the vitreous transition temperature, the macromolecules have enough thermal energy to move and self-order.

This creation of order sets off heat and the cell's heating system, where the sample is, will need to supply less energy. This decrease in heat supply will give access to the crystallisation temperature (Tc).

When a semi-crystalline polymer is heated, its supply in thermal energy allows a rupture with the established order and for crystalline zones to melt. The fusion phenomenon is a first order transition. This means that when the fusion temperature is reached, the polymer's temperature will no longer increase until all samples have melted (isothermal transformation.)

The heating system will thus supply more heat to the sample cell for it to remain it at the same temperature as the referential cell. Thanks to this increase in heat-flow, the **fusion temperature (Ft)** can be determined.

**Latent fusion and crystallisation heat** can also be determined as well as the **crystallinity rate**.

- **chemical reactions** (reaction heat) : Polymerisation, decomposition, crosslinkability or oxidation reactions can be analysed in DSC, in temperature or isothermal scanning.

-**Constant pressure thermal capacity (Cp).**

CRITT Matériaux Alsace has recently acquired a thermo-modulated DSC, latest upgrade of the conventional technique of the NETZSCH brand.

The thermo-modulated DSC (TM-DSC allows the sensibility and resolution of the measuring to simultaneously be increased by subjecting the sample to two heat-flow speeds, a slow speed at medium heat-flow (under-lying) and an instant high heat-flow speed.

This particular configuration enables to detect low energy transition in highly crystalline or highly charged polymers and to separate reversible phenomena (Gt...) and irreversible phenomena (chemical reactions...).

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The laboratory at CRITT Matériaux Alsace is equipped with a Netzsch brand thermo-modulated DSC (DSC 204 F1 Phoenix®). This apparatus performs in a range of temperature going from **-180° to 700° with a heat speed between 0.001 and 100°C/min**