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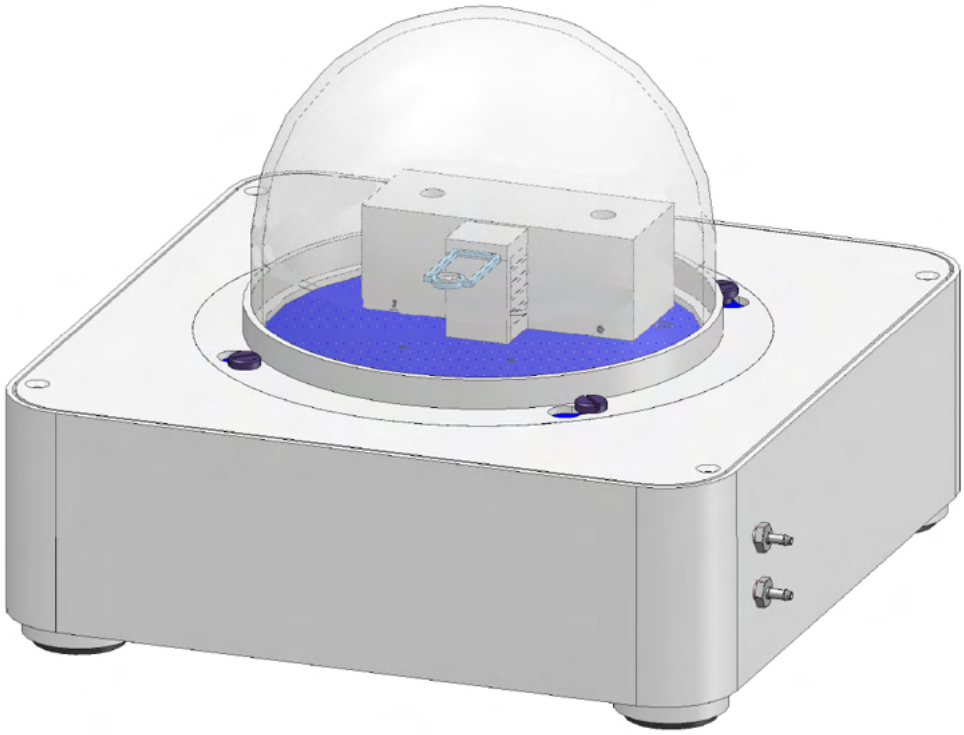
pushing boundaries

How To
Determine crystallinity at polymers with Chip-DSC



Linseis Messgeräte GmbH
Gerlach
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Index

General

Crystallization of polymers *is a process associated with partial alignment of their molecular chains. These chains fold together and form ordered regions called lamellae, which compose larger spheroidal structures named spherulites. Polymers can crystallize upon cooling from the melt, mechanical stretching or solvent evaporation. Crystallization affects optical, mechanical, thermal and chemical properties of the polymer. The degree of crystallinity is estimated by different analytical methods and it typically ranges between 10 and 80%, with crystallized polymers often called "semi-crystalline". The properties of semi-crystalline polymers are determined not only by the degree of crystallinity, but also by the size and orientation of the molecular chains.*

[From Wikipedia *"Crystallization of polymers"* 04.11.2019]

Measuring

Requirement of the measuring system

- You need a well-functioning Chip DSC
- The chip DSC need to be temperature and enthalpy calibrated
- The temperature range of your Chip DSC must be sufficient for the desired effect

Samples

- Your sample need to have a flat surface or a good cutting surface
- The weight should be around 10-30 mg (depends on the enthalpy of the melting point)
- The weight must be determined very accurately for reliable enthalpy values
- To measure the initial crystallinity of your sample you have to use the first heating of the sample so sample preparation is very important

Temperature profile

- It is important to think carefully about which temperature profile you use before you start measuring
- The heating rate of the first heating cycle should be ~10 to ~30 K/min
- The cooling rate depends on your polymer (some crystallize faster, others slower)
- The heating rate of the second heating cycle (only needed if you want to compare the crystallization between different cooling rates) should be the same like the first heating rate

Evaluation and calculation

Evaluation

- If your measurement is finished start the Evaluation software and load the curves you want to evaluate
- If necessary, arrange the curve until melting peak is clearly visible (check other HowTo's to see how you can optimize the view of your measurements)
- Use the peak evaluation tool and determine the enthalpy of the melting peak

Calculation

- The degree of crystallinity is the percentage ratio between the enthalpy of the actual sample and a 100% crystalline sample (commonly known by literature values)
- The formula for this is $X_c = \frac{\Delta H_M - \Delta H_C}{\Delta H_0} \times 100\%$

- X_c is the crystallinity you want to determine
- ΔH_M is the melting enthalpy of the real sample, ΔH_C is the cold-crystallization enthalpy of the real sample and ΔH_0 is the enthalpy of an 100% crystalline sample
- Insert the measured enthalpy and the literature value in the formula and your result is the crystallinity of your sample

Literature values

- The following table contains some literature values about crystallinity of polymers

Polymer	**ΔH_0 100% [J/g]**	**Typical crystallinity [%]**
PA 6	230	35 … 45
PA 66	255	35 … 45
PE – HD	293	70 … 80
PE – LD	293	45 … 55
PET	140	30 … 40
POM homopolymer	326	90
POM copolymer	326	75
PP isotactic	207	70 … 80
PP syndiotactic	207	~30 … 40
PP ataxic	207	~0
PTFE	82	60 … 80