Determination of the crystalline conformity of composite samples by D.S.C

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Introduction

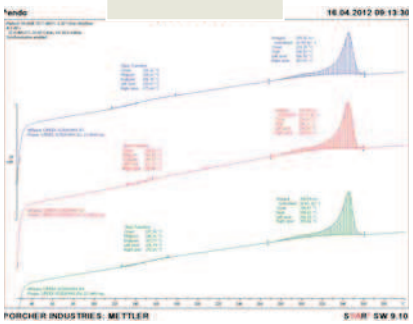
The Expertise & Innovation Center of Porcher Industries develops composite products for the aeronautic industry. Composite plates are composed of a matrix of PEEK (PolyEtherEtherKetone = thermoplastic resin), with reinforcements in carbon. Part of the production control is carried out in the Analytical Laboratory to determine the crystallinity degree in relation to the melting temperature, the melting energy and the sample weight. The glass transition is also controlled. The crystallinity degree depends also on the resin rate in the sample, which is given by another analysis. Production control consists in thermal analysis, by Differential Scanning Calorimetry (DSC). This technique measures the differences in heat flow between the sample and the reference during a cycle of temperature. We measure here differences of energy, not of temperature. The analysis by DSC shows different endo and exothermic phenomena like melting, polymerization, or glass transition, that we can see on a thermogram.

Experimental methods

The experiment needs to be performed in specific conditions:
Flow : nitrogen, to have an inert gas
Ramp Temperature : from 25 to 400 °C, at 20°C per minute.
Samples are conditioned in aluminium capsules which are tapped manually of two holes. The sample weight must be between 18 and 24 mg. The sample and the standard (an empty capsule) are placed in the same furnace. The temperature of heating, provided by an electrical resistance, varies linearly. A probe allows to control and record changes of temperature in the apparatus.

Figure A :Thermograms of a production control of composites plates C/PEEK, 3 conclusives results.
Figure B : Tabular of experimental values and limits values of glass transition, and melting temperatures, and crystallinity degree given by the thermograms in the Fig. A

A)



B)

	Glass transition temperature (°C)	Melting Temperature (°C)	Crystallinity degree (%)
Experimental results	143.8 ± 3.4	343.5 ± 0,3	29.8 ± 0.4
Specifications according to standard.	—	343 ± 15	32 ± 10

Results and discussion

To determine the crystallinity degree and complete the control paper, it is necessary to analyze 3 samples of the same batch. The thermogram (Fig. A) shows us two phenomena: the glass transition at the beginning, and the melting at the end. Results are given in the Fig.B. We observe that the standard deviation is weak for the melting temperature and the crystallinity degree. The standard deviation of the glass temperature is higher. Moreover, melting temperature and crystallinity degree are included between high and low limits of the norm.

Conclusion

The difference of deviation standard between the glass transition and the two other measurements means that the glass transition is a phenomena a little bit less regular than the melting. The results of melting temperature and crystallinity degree are closed to the standard. Consequently, the batch is conform and can be delivered to the customer.

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