Application News

SHIMADZU

THERMAL ANALYSIS T113

Analysis of Isothermal Crystallization Process of High Polymers with DSC

Crystalline high polymers including polyethylene, polypropylene and nylon produce crystallized structures if they are left alone for a long time at a given constant temperature lower than that when they have been completely fused. This phenomenon is called isothermal crystallization.
Measurement of the isothermal crystallization process serves to determine a half-life (t1/2), which can be used as a yardstick for crystallization speed, evaluation of core substance of crystallization, and the study of bridging effect.
DSC, excellent in thermal sensibility, is an indispensable piece of equipment for measuring the process of isothermal crystallization where a specimen is rapidly cooled after fusion and kept under a strictly controlled temperature.
The following is a report on measurements of isothermal crystallization process of polyethylene and polypropylene with DSC.
A typical process of isothermal crystallization is presented in Fig.1. The curve shows that a fused specimen is rapidly cooled down to a given temperature, kept at a constant temperature, and produces an exothermic peak of crystallization after a lag time "tid."
DHc of whole crystallization calories can be known from the whole peak area, and crystallization calories at a given time t are calculated by the following equation.

$$1-\theta=\frac{\int_{tid}^{t}\frac{dH}{dt}\cdot dt}{\Delta Hc}$$

where θ : Ratio of non-crystallized portion,
$\triangle$Hc: whole crystallization calories.
Values of q for each point of t make an isothermal crystallization curve that can be drawn from the values θ determined by above equation for each t.

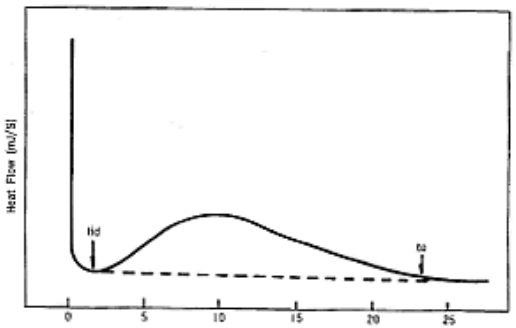


Fig.1
A Typical Curve of Isothermal Crystallization

Analysis of isothermal crystallization process of polypropylene

Figs.2-4 show results of analysis of isothermal crystallization process of polypropylene held at different temperatures. Each specimen weighing 10.62mg was rapidly cooled at a rate of -50˚C/min after fusion and held at constant temperature.

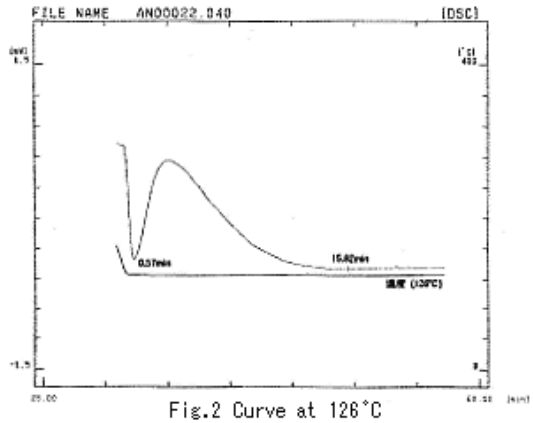


Fig.2 Curve at 126°C

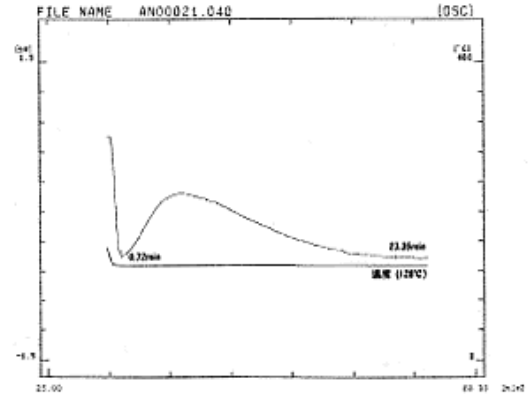
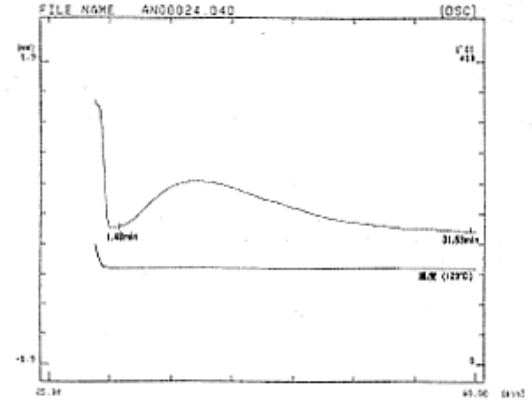


Fig.3 Curve at 128°C



Fig,4 Curve at 129°C

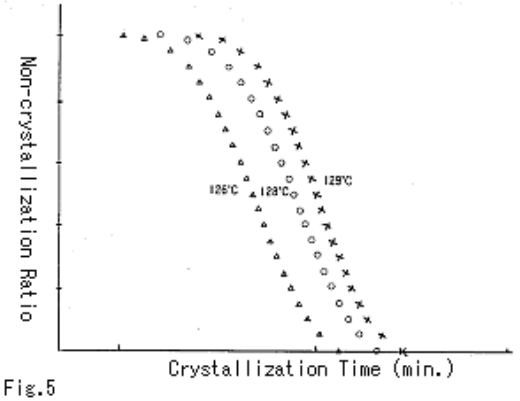


Fig.5
Isothermal Crystallization Curves of Polypropylene

Analysis of polyethylene

Similarly to polypropylene, each specimen weighing 10.84mg was rapidly cooled at a rate of -50°C/min after fusion, and held at a constant temperature.

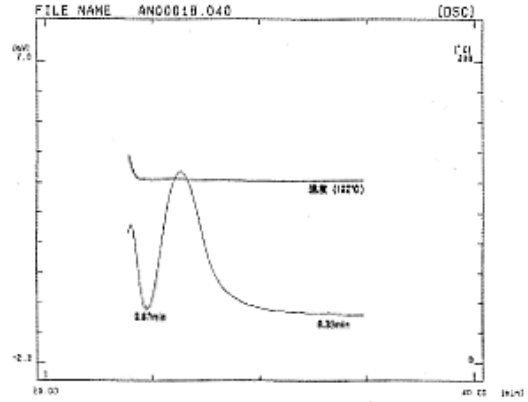


Fig.6 Curve for 122°C

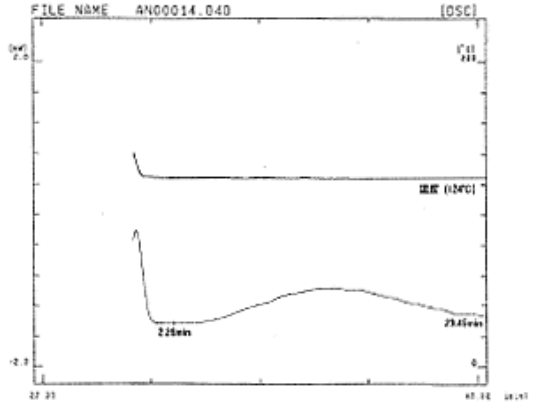


Fig.7 Curve for 124°C

Fig.8 shows isothermal crystallization curves of polyethylene.

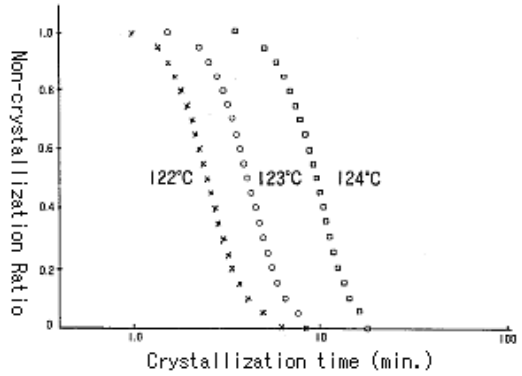


Fig.8 Isothermal Curves of Polyethylene

* Please be advised that data obtained before the implementation of the current Weights and Measures Law may be presented in terms of gravimetric unit.

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