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NUCLEAR MAGNETIC RESONANCE (NMR) STUDY OF POLYETHYLENE TEREPHTHALATE CRYSTALLIZATION*

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THERE are several reports of crystallization studies [1-3] by classical methods, e. g. dilatometry etc. Here we describe a standing the crystallization of polyethylene terephthalate (PETP) by means of the NMR impulse method and show that it can give informational additional to the usual kinetic characteristics, i.e. about changes in molecular movement during crystallization.

EXPERIMENTAL

An industrial PETP sample with a number average molecular weight M_n 5000 was used. The isothermal crystallization was carried out from the glass-like state ("cold crystallization") in the range 100-180°C, or from the melt at 180-240°C in the NMR spectrometer cell. The temperature ranges were selected on the basis that 180°C is the temperature at which the rate of crystallization is at its maximum. Oriented and non-oriented samples were used in the cold crystallization (about 300 and 2000% elongation) and were in the amorphous state. The cell temperature was kept constant within 0.5% and the time taken to reach thermal equilibrium in the sample was about 2 min.

The examined parameters were the proton spin-spin relaxation time T_2 and the nuclear density of the amorphous and crystalline phases. The short relaxation period (≤ 50 μ sec) was estimated from the free induction signal behind the 90° impulse, or from the NMR

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