Dielectric Spectroscopy and Adiabatic Calorimetry during Early Stages of Polymer Crystallization

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Existence and formation of pre-ordered structures as the initial step of polymer crystallization are discussed controversially. Most of the findings and interpretations are based on scattering experiments, which test small density differences between the assumed precursors of the crystals and the surrounding melt. Because of the low contrast, the interpretation of experimental results becomes often speculative. Contrary relaxation experiments are probing motions in the sample and are therefore independent on density contrast. During crystallization, material is transformed from the liquid to the solid state. Consequently, motions (fluctuations) typical for a liquid become impossible and no longer contribute to the measured relaxing signal. For pre-ordered structures, we expect some changes in mobility too because of the changes in conformation on pre-ordering. Adiabatic calorimetry is suitable to detect temperature changes in the sample, which can be related to crystallinities below one thousandth. In this study, we compare crystallinity data from adiabatic calorimetry in the sub-percent range with complex dielectric permittivity of polycaprolactone (PCL) during isothermal crystallization.