

Dynamics of precursors of fibrillar crystals

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Dynamics of precursors of fibrillar crystals

L. Balzano, G.W.M. Peters



Introduction

Fibrillar crystals can be formed from melts of flexible macromolecules applying strong deformations [1]. Control over this phenomenon is a prerequisite to tailor properties of polymeric materials, like isotactic polypropylene (iPP). However, limitations arise from incomplete knowledge of the physics involved. For instance, it is known that precursors are formed [2], but how stable are they? And, how do they evolve?

Materials and Methods

- iPP homopolymer, $M_w=365$ kg/mol, $MWD=5.4$, $T_m=165$ °C (HD120M0, Borealis).
- Shear experiments performed using a slit flow cell allowing for in-situ X-ray observation, see Figure 1a.
- SAXS and WAXD performed at the beamline BM26B, ESRF (Grenoble, France), see Figure 1b.

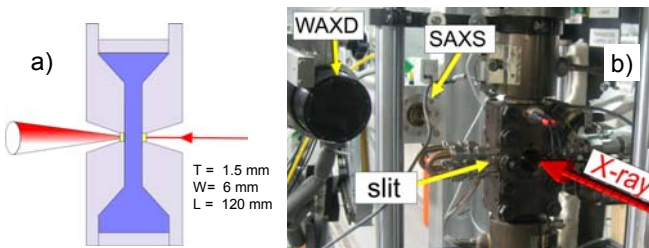


Figure 1: a) schematic of the slit; b) experimental set-up at BM26B.

Results

Short term shear experiments together with SAXS reveal that, at 165 °C, fibrillar crystal precursors are formed when the wall stress σ_w exceeds 0.12 MPa, see Figure 2.

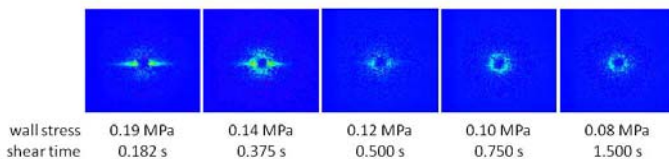


Figure 2: SAXS at early stages, $T=165$ °C. Formation of fibrillar precursors ($\sigma_w \geq 0.12$ MPa) is accompanied by an equatorial streak.

The time dependence of the equatorial streak intensity is shown in Figure 3. Experiments are grouped in two categories:

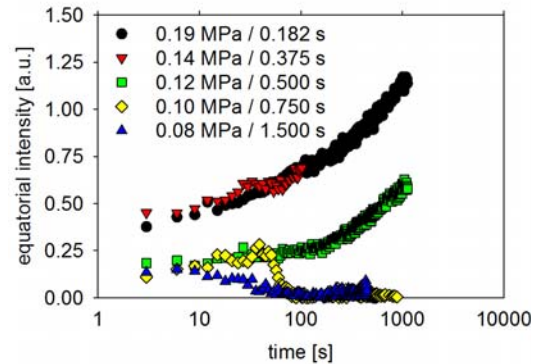


Figure 3: Intensity of the equatorial streak as a function of time.

- $\sigma_w \geq 0.12$ MPa: precursors are stable and crystallize (from WAXD) increasing the scattered intensity. During crystallization their length grows ~ 50 nm (see Figure 4).

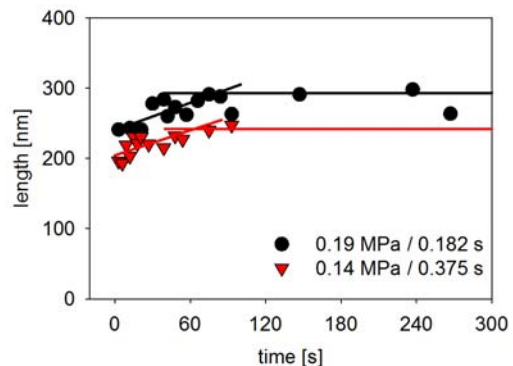


Figure 4: Length of fibrillar precursors/crystals from SAXS at 165 °C.

- $\sigma_w < 0.12$ MPa: precursors are not stable and dissolve reducing the scattered intensity.

Conclusions

Fibrillar structures are formed in a melt of flexible molecules, like iPP, when stress exceeds a critical threshold. The process starts with fibrillar precursors that crystallize and lengthen with time. When the critical stress is not exceeded, precursors dissolve back to the melt.

References

- [1] Kumaraswamy, G.; Kornfield, J. A.; Yeh, F.; Hsiao, B. S. *Macromolecules* **2002**, *35*, 1762-1769.
- [2] Balzano, L.; Kukalyekar, N.; Rastogi, S.; Peters, G. W. M.; Chadwick, J. C. *Physical Review Letters* **2008**, *100*, 048302.