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Effect of flow on crystallization of polypropylene

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Introduction

Structure development during flow-induced crystallization determines to a high degree the properties of (semi-) crystalline polymer products. Processing parameters strongly affect the crystal nucleation and therefore the final morphology of polymeric products. Still the effect of flow on the nucleation phenomena is not fully understood and thus this is still an open subject for research [1-3]. The aim of this work is to study the influence of shear parameters on the flow-induced crystallization of isotactic polypropylene (iPP) by combining different experimental techniques.

Material and experimental protocol

linear iPP HD120MO (Borealis): M_w=365 000 g/mol
M_w/M_n =5.2; T_m =165 ^{0}C

Linkam shear cell CSS40

- $T_{cr,shear}$: 125 to 165 ^{0}C
- total shear strain: 30 to 360

three types of experiments:

- exp. I : constant shear rate
- exp. II : constant shear time
- exp. III: constant total shear strain

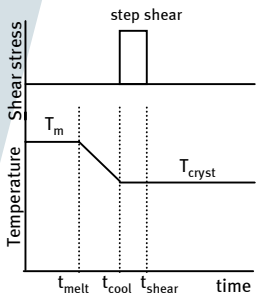

Figure 1. *Working protocol*

Experimental techniques

SAXS / WAXS : [nm÷Å]
Optical Microscopy: [mm-µm]
SALS : [µm]
DSC
ESEM : [nm]

in-situ | *ex-situ* → **crystal type, size, orientation, kinetics**

Results

Exp. I : constant shear rate $\dot{\gamma}$ =60 s^{-1} ; t_{shear}= 1 to 6 s

Optical microscopy

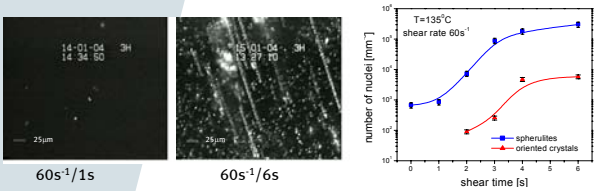

Figure 2. *Left -pictures of iPP taken 120 s after crystallization start; right - number of crystals at different shear time;* $T_{shear,\,cr}$ *=135 ^{0}C*

→ A longer shear time changes the type of crystals and increases their number

Exp. II : constant shear time t_{shear}= 6 s

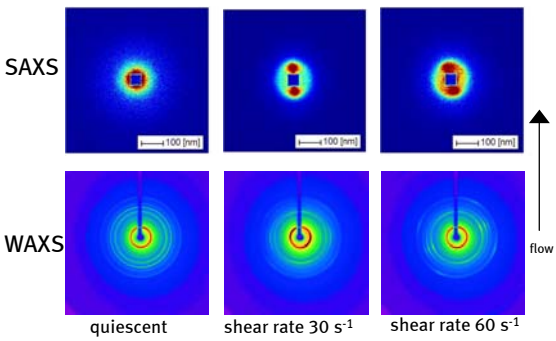

Figure 3. *SAXS/WAXS patterns of iPP during crystallization at* $T_{shear,\,cr}$ *=135 ^{0}C and different shear conditions, all images are taken 10 min after the beginning.*

→ Higher orientation and faster crystallization kinetics at higher shear rates

Exp. III : constant total shear strain γ **=60 to 360**

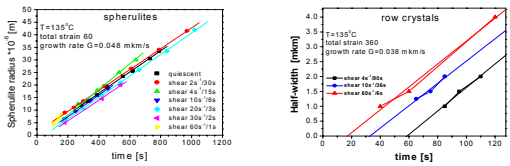

Figure 4. *Growth rate of the non-oriented spherulites (left) and oriented row crystals (right) at two different total shear strains.*

→ Flow strength doesn't influence the crystal growth rate

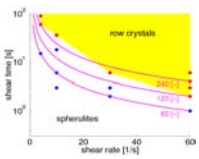

Figure 5. *Different type of crystals depending on shear parameters*

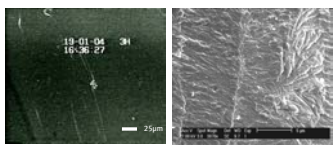

Figure 6. *Oriented row crystals observed during shear (left-OM picture) and after completed crystallization (right - ESEM picture).*

→ Increasing the shear rate lowers the critical strain level at which oriented crystals are observed

Conclusion

Only combined experimental work gives a full understanding of the complex influence of the shear parameters on flow induced crystallization of polymers

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