

## Looking at polymer crystallization with SALS

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# Looking at Polymer Crystallization with SALS

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## Introduction

Crystallization of polymers during processing affects the properties of the final products. Recently, nucleation phenomena is subjected to an increasing interest. One of the promising techniques to study the evolution of the early stages of crystallization is Small Angle Light Scattering (SALS) [1, 2].

## Method and analysis

Samples of isotactic polypropylen iPP HD120MO (Borealis) with thickness of  $40\mu m$  are inserted in a LINKAM hot-stage at a certain temperature  $T_{cr}$  ( $125^{\circ}C - 145^{\circ}C$ ) and the crystal development during isothermal crystallization at  $T_{cr}$  is observed by SALS.

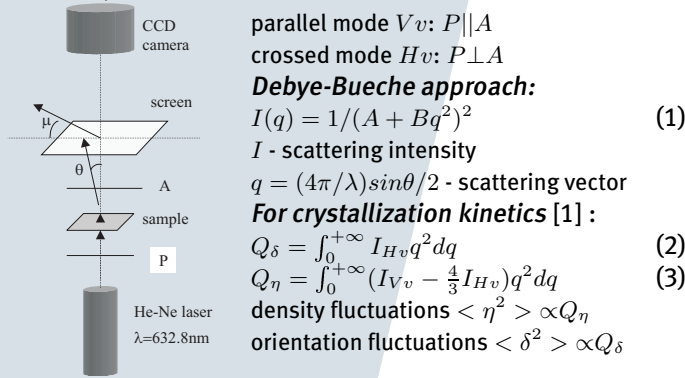


Figure 1. Small Angle Light Scattering set-up.

### Parameters to obtain:

- ⇒ time evolution of  $Q_{\eta}$  and  $Q_{\delta}$
- ⇒  $R_{sph}$  - from  $\theta$  at which  $I_{Hv}$  has a max
- ⇒ correlation length  $\xi = \sqrt{(B/A)}$  - from eq.(1)

## Experimental observations and Results

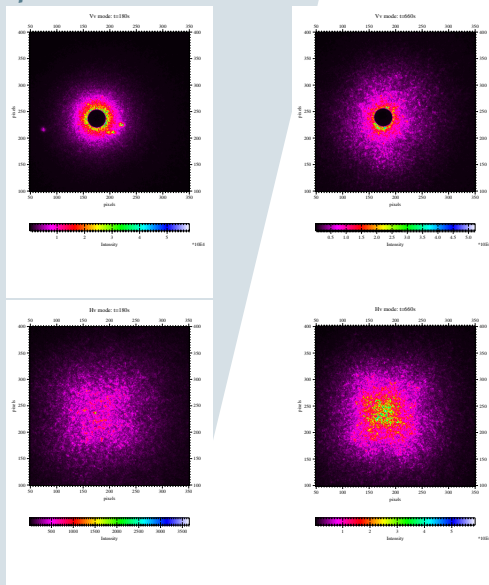


Figure 2. Scattering patterns obtained by  $P||A$  (top) and by  $P\perp A$  mode (bottom) at  $T_{cr} = 135^{\circ}C$ ;  $q$ -scale:  $100\text{pixels} = 1.5\mu m^{-1}$ .

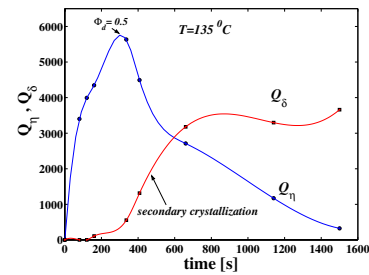


Figure 3. Time evolution of  $Q_{\delta}$  (for  $\mu = 45^{\circ}$ ) and  $Q_{\eta}$  (for  $\mu = 0^{\circ}$ ) invariants during crystallization of iPP at  $T_{cr} = 135^{\circ}C$ .  $\Phi_d$  is the volume fraction of growing crystals.

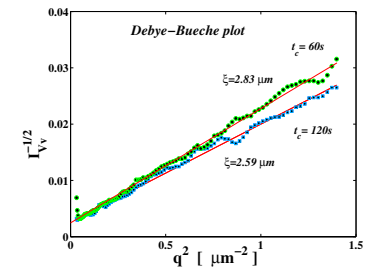


Figure 4. Debye-Bueche plots for parallel mode  $Vv$  at different times for  $T_{cr} = 135^{\circ}C$ , and calculated values of the correlation length  $\xi$ .

Table 1. Spherulite radius  $R_{sph}$  of iPP obtained by two techniques - SALS and hot-stage microscopy at different temperatures  $T_{cr}$  for a crystallization time of 360sec.

$T_{cr}, [^{\circ}C]$	$R_{sph}, [\mu m]$	
	SALS	hot-stage microscopy
125	43.1	44.4
135	27.9	31.6
140	12.5	-

## Conclusions

- ◆ The SALS patterns show the expected 2-fold (for parallel  $Vv$  mode) and 4-fold (for crossed  $Hv$  mode) symmetry.
- ◆ Density fluctuations  $\langle \eta^2 \rangle$  appear prior to crystal development and reach a maximum when volume filling  $\Phi_d$  is 0.5.
- ◆ Secondary crystallization is observed after spherulite impingement.
- ◆ The good approximation of  $I^{-1/2}(q^2)$  with a linear fit confirms the validity of Debye-Bueche approach to evaluate the data from SALS.
- ◆ SALS technique could be a very useful method for future investigations of flow-induced crystallization, where enhancement in crystal nucleation is expected.

## References:

[1] STEIN, R.S., CRONAUER, J., ZACHMANN, H.G.: *Journal of Molecular Structure*, 1996, 383, 19  
 [2] KUMARASWAMY, G., KORNFELD, J.A., YEH, F., HSIAO, B.S.: *Macromolecules*, 2002, 35, 1762