

# Polymer orientation and crystallinity measurements by FT-IR and IR dichroism

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# Polymer Orientation and Crystallinity Measurements by FT-IR and IR dichroism

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## Introduction

Final properties of a polymeric product are determined by its morphology that was developed as a consequence of processing conditions. Measurement of crystallinity and orientation is of importance for process optimization [1].

## Experimental: FT-IR spectra analysis

Three IR spectra have to be collected (Fig. 1): non-polarized, polarized parallel and perpendicularly to the flow direction.

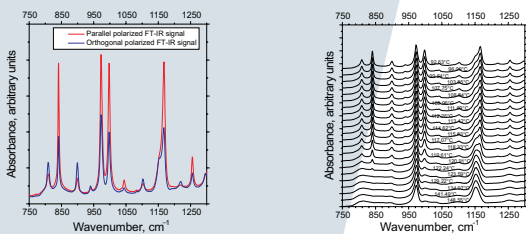


Figure 1 Left: polarized FT-IR spectra from an iPP sample, Right: un-polarized FT-IR spectra collected during cooling

## Crystallinity

Considering a crystalline peak and a peak insensitive to phase content, and starting from Lambert and Beer's law, crystallinity  $X_c$  can be evaluated as follow:

$$X_c = (a_{av}/a_{cr}) (A_{cr}/A_{av})$$

$A_{cr}$ ,  $A_{av}$ : absorbancies,  $a_{cr}$ ,  $a_{av}$ : absorptivities of crystalline fraction and of a peak insensitive to phase content respectively, (for iPP  $a_{av} = 973 \text{ cm}^{-1}$ ,  $a_{cr} = 841 \text{ cm}^{-1}$  and  $a_{973}/a_{841} = 0.79$  [2]).

## Orientation

The orientation factor can be obtained from Fraser's theory (dichroic ratio  $D_\nu = (A_\pi/A_\sigma)_\nu$ , for iPP  $K_{973} = K_{841} = 1$  [2]):

$$f = \left[ \left( \frac{D-1}{D+2} \right) \left( \frac{D_0+2}{D_0-1} \right) \right]_\nu = K_\nu \left( \frac{D-1}{D+2} \right)_\nu$$

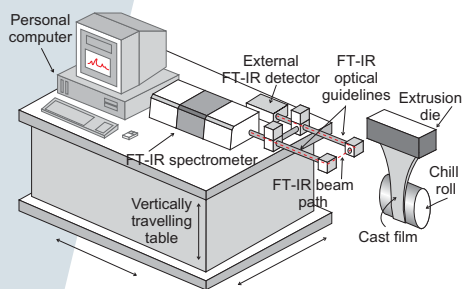


Figure 2 The system developed at University of Salerno to gather transmission spectra during polymer film casting /department of mechanical engineering

## Case histories

- Quenched polypropylene films [2] and film casting products [1] were analysed off-line (UNISA).
- On-line measurements were performed during film casting by a dedicated apparatus (UNISA, Fig. 2) [1].
- Injection moulded samples have been investigated by a FT-IR microscope (TUE, Fig. 3) [3].

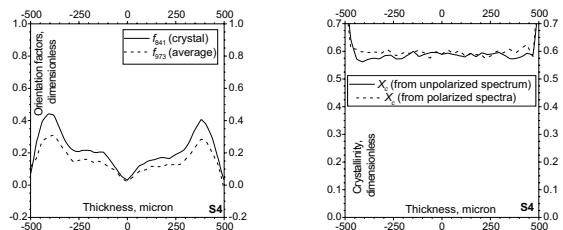


Figure 3 Left: orientation distribution, Right: crystallinity distribution, both are obtained from injection moulded sample

## Future work

In principle, the techniques can be applied on a rheometer. Rheological responses ( $\eta$ ,  $G'$ ,  $G''$ ) can directly be related (being measured during the same experiment) to morphology ( $f$ ,  $X_c$ ). A set-up like the one sketched in Fig. 4, is under development.

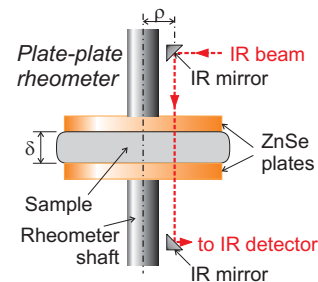


Figure 4 The system under development at MATE/TUE to gather transmission spectra during rheology experiments

## Problems

- Two opposite needs on sample thickness: (i) Rheology:  $\delta > 300 \mu\text{m}$  to avoid excessive forces, (ii) FT-IR:  $\delta < 150-200 \mu\text{m}$  to avoid saturated absorbancies
- Little room for IR mirror system positioning ( $\rho \approx 1-2 \text{ cm}$ )
- No reliable optical fibres available to gather the spectral region between  $750-1000 \text{ cm}^{-1}$

## References:

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