

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

Citation for published version (APA):

Lamberti, G., Titomanlio, G., & Peters, G. W. M. (2002). *Polymer orientation and crystallinity measurements by FT-IR and IR dichroism*. Poster session presented at Mate Poster Award 2002 : 7th Annual Poster Contest.

Document status and date:

Published: 01/01/2002

Document Version:

Publisher's PDF, also known as Version of Record (includes final page, issue and volume numbers)

Please check the document version of this publication:

- A submitted manuscript is the version of the article upon submission and before peer-review. There can be important differences between the submitted version and the official published version of record. People interested in the research are advised to contact the author for the final version of the publication, or visit the DOI to the publisher's website.
- The final author version and the galley proof are versions of the publication after peer review.
- The final published version features the final layout of the paper including the volume, issue and page numbers.

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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.

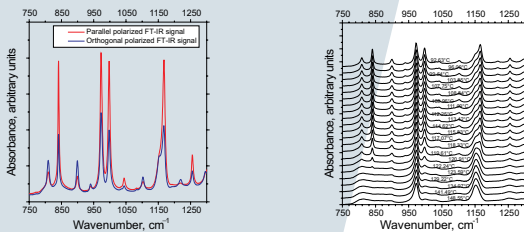


Figure 1 Left: polarized FT-IR spectra from an iPP sample, Right: un-polarized FT-IR spectra collected during cooling

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]):

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

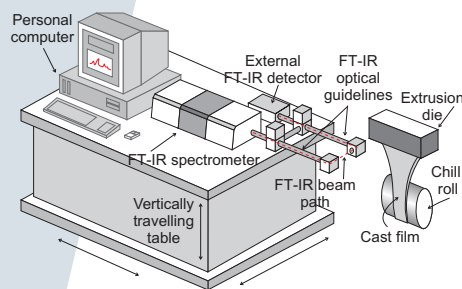


Figure 2 The system developed at University of Salerno to gather transmission spectra during polymer film casting /department of mechanical engineering

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].

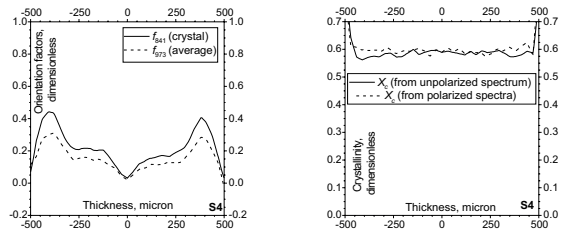


Figure 3 Left: orientation distribution, Right: crystallinity distribution, both are obtained from injection moulded sample

Future work

In principle, the techniques can be applied on a rheometer. Rheological responses (η , 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.

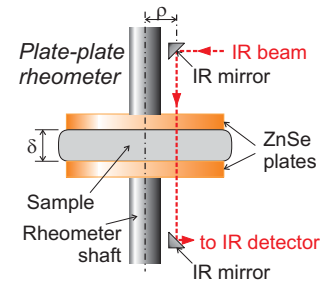


Figure 4 The system under development at MATE/TUE to gather transmission spectra during rheology experiments

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 $750-1000 \text{ cm}^{-1}$

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