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Measurements of the full 3D orientation tensor of an injection molded fiber-filled polymer.

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INTRODUCTION

The processing route has a strong influence on the fiber orientation distribution in a short-fiber reinforced composite, then highly influences its mechanical and physical response. In the particular case of injection-molded polymers, three stages are successively followed: polymer filling into the mold cavity, post-filling and cooling. A typical shell-core structure of the fiber distribution induced by the flow results, where the fibers tend to be aligned in the injection direction at skin and lie perpendicular to it at core. To predict the properties (especially thermo-elastic properties) of these composite materials, homogenization methods for the unidirectional composite can be used, but a full 3D fiber orientation characterization is needed. We will see that the use of orientation tensor is a good and sufficient way for obtaining a concise (and not cumbersome) description of orientation and the aim of the present work is precisely to be able to measure orientation tensors.

REPRESENTATION OF FIBER ORIENTATION

The orientation of an individual fiber is defined by 2 angles θ and ϕ (Figure 1), or by Cartesian components of a vector \vec{p} aligned parallel to the fiber:

$$\vec{p} = \begin{pmatrix} \sin \theta \cos \varphi \\ \sin \theta \sin \varphi \\ \cos \theta \end{pmatrix}$$

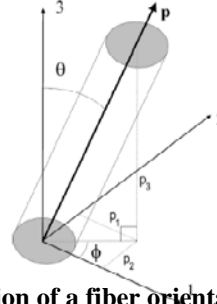


Figure 1: definition of a fiber orientation.

For millions of fibers, it is impractical to specify the orientation of each fiber and an alternative is to use the probability distribution function $\psi(\theta, \phi)$ [1]. However this probability density function holds considerable information and any calculation based on these data are highly computationally intensive, that is why the tensor description is more widely used for fiber orientation characterization.

The second and fourth-order tensor are calculated by forming dyadic products of the vector \vec{p} : $[a_2] = [pp']$ and $[a_4] = [pp'pp']$, the order being a way of changing the detail of the distribution description.

For an individual fiber k, the coefficients of the second order orientation tensor $[a_{ij}^k]$ are equal to:

$$\begin{cases} a_{11}^k = \sin^2 \theta^k \cos^2 \varphi^k \\ a_{22}^k = \sin^2 \theta^k \sin^2 \varphi^k \\ a_{33}^k = \cos^2 \theta^k \\ a_{12}^k = \sin^2 \theta^k \cos \varphi^k \sin \varphi^k \\ a_{13}^k = \sin \theta^k \cos \theta^k \cos \varphi^k \\ a_{23}^k = \sin \theta^k \cos \theta^k \sin \varphi^k \end{cases} \quad (1)$$

The global orientation tensor $[a_{ij}]$ of the sample is obtained by averaging all individual fiber orientation, so for each component:

$$a_{ij} = \frac{\sum_{k=1}^n F^k a_{ij}^k}{\sum_{k=1}^n F^k} \quad (2)$$

where a_{ij}^k is the component defined for an individual fiber k.

F^k is a correction factor necessary for fibers of finite length: $F^k = 1/Q^k$ where $Q^k(\theta_k) = L \cos \theta_k + D \sin \theta_k$ (3) is the probability that the fiber k of length L and diameter D intersects a section plane at the angle θ_k .

ORIENTATION MEASUREMENT

If a cross section is considered, the intersection of a fiber with a section plane is an ellipse. The use of an image analysis software (such as *Micro Vision System*® or *Aphelion*®) enables to identify each fiber as an ellipse and determines the ellipse center coordinates (x_c, y_c) , its major and minor axis a and b respectively, as well as its in-plane fiber orientation ϕ . However an ambiguity remains as for the orientation of the fiber, which can have two positions (Figure 2): a fiber oriented at $+\theta$ and $-\theta$ yields the same cutting ellipse, so the sign of the angle θ stays undetermined.

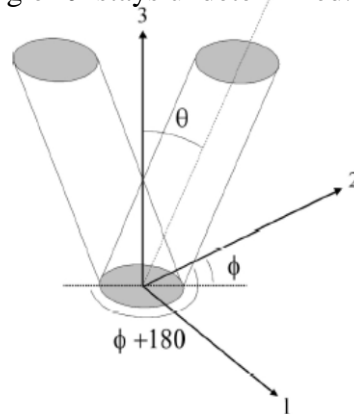


Figure 2: ambiguity concerning the fiber orientation.

Consequently the components a_{13} and a_{23} of the orientation tensor remain undetermined.

To remove this ambiguity, several methods have been developed to determine the orientation tensor among the use of ultrasonic methods, complex optical microscopy methods [2], or confocal microscopy [3,4]. We propose to solve this problem by using Scanning Electronic Microscopy images. Very well polished samples are observed via a source of retro-diffused electrons. A quite binary image is obtained, where the fibers appear in white and the matrix in black.

If a high voltage tension is applied (typically 20kV), it comes out that a track appears in grey at one of the extremities of the ellipse (Figure 3), due to the low amount of matrix covering the fiber: it thus corresponds to the fiber, which penetrates the material. Then the method consists of observing the same SEM image by the mean of an image analysis system, with two different thresholds: a « low » threshold to include the track in the fiber image, and a « high » one to exclude it.

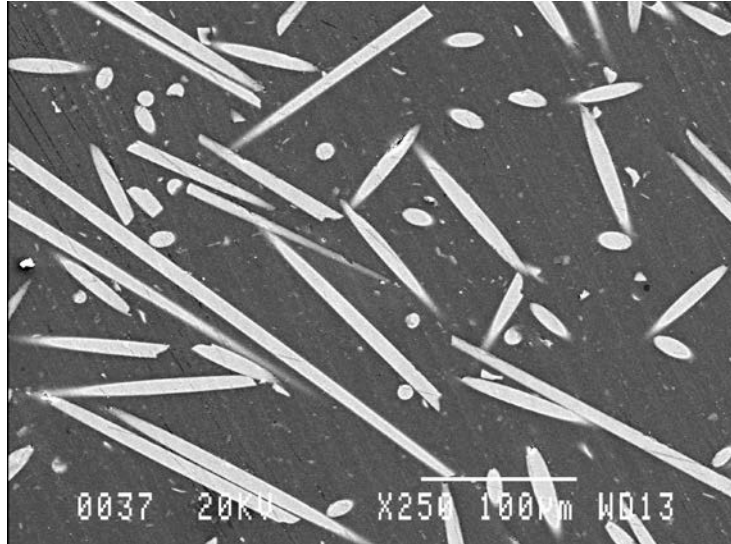


Figure 3: a local SEM micrograph of a 30wt% glass fiber reinforced polyarylamide .

Orientation is determined by choosing in which condition angle θ is positive and from the shift of centers \overline{GbGh} : the sign of θ is given by the sign of the scalar $\overline{GbGh} \cdot \overline{\varphi x}$ (see in Figure 4).

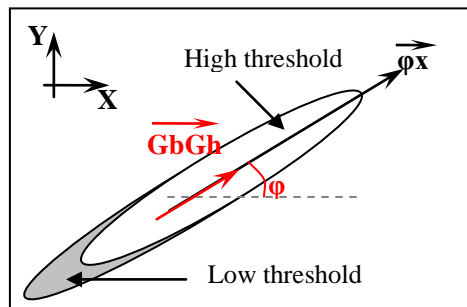


Figure 4: determination of angle θ sign.

It is now possible to remove the ambiguity of fiber orientation. However for hundreds of fibers, it is impractical to specify the orientation of each fiber and an alternative is to automate the orientation determination. A by-house program has then been written with the use of MATLAB software, to automate the procedure of collecting data on many fibers efficiently. Small surfaces ($0.15 \times 0.23 \text{mm}^2$) are successively analyzed to eventually scan the whole sample.

Knowing that in injection-molded parts, fibers are mainly oriented in the flow direction in the part skin, the precision of the orientation determination is improved when sectioning is performed at 45°C from the flow direction. Indeed as the fibers are circular in cross-section, they will appear as ellipses on the section plane when oriented at an out-of plane angle θ by preferential sectioning, typically in the range $30^\circ \leq \alpha \leq 80^\circ$ [5]. This technique appeared to be very effective in reducing angle measurement uncertainty. Nevertheless the reliability of fiber orientation data highly depends upon the quality of the surface preparation (polishing) of the sample.

RESULTS

1- and 3-mm thick plates have been mold-injected and the method has been applied to measure the a_{ij} components of the orientation tensor. Direction 1 is the flow direction and direction 3 corresponds to the thickness. To get comparable results, both thicknesses have been normalized, so that their values vary from -1 to 1.

Figures 5 and 6 underneath depict the evolution of a_{11} and a_{22} components through the thickness:

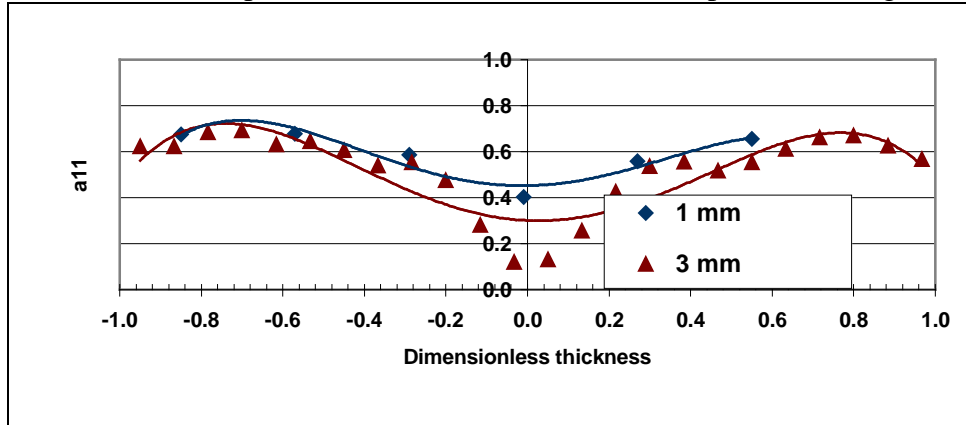


Figure 5: evolution of a_{11} through the thickness of 1mm- and 3mm-thick injected plates.

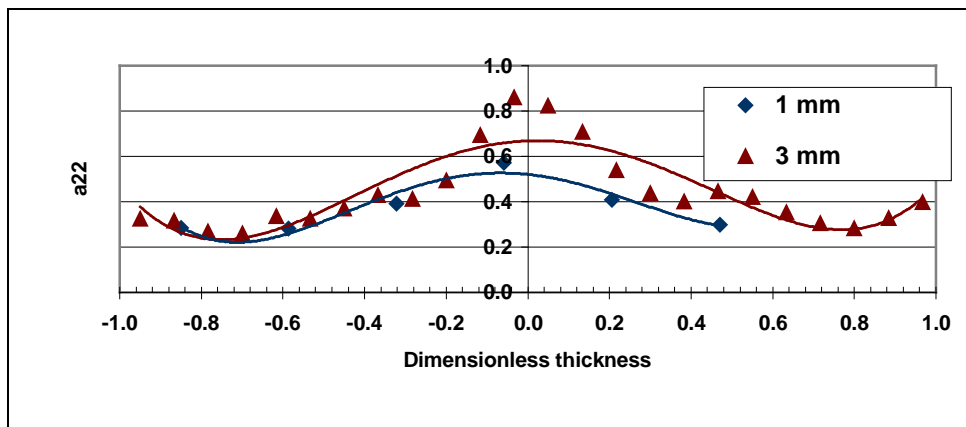


Figure 6: evolution of a_{22} through the thickness of 1mm- and 3mm-thick injected plates.

The shell-core structure of the distribution clearly appears and is more pronounced for the 3mm-thick sample. Evolutions of a_{11} and a_{22} are of course complementary.

CONCLUSION

The method enables the efficient determination of fiber orientation: there is no longer any uncertainty as to the direction of one fiber and a complete tensor description can be formed. The procedure, applicable to any fiber-reinforced polymer composite, has been fully automated, making it possible to measure a sample's orientation, even for large areas.

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