MODELLING THE DAMAGE BEHAVIOUR OF SHORT FIBRE REINFORCED COMPOSITES USING A NON-DESTRUCTIVELY EVALUATED FIBRE ORIENTATION DISTRIBUTION AND MICRO CRACKING

V. Trappe^{1*}, S. Günzel¹, J. Goebbels¹

¹Federal Institute for Materials Research and Testing (BAM), Unter den Eichen 87, 12205 Berlin, Germany * volker.trappe@bam.de

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Abstract

Short fiber reinforced thermoplastic materials were investigated non-destructively in different damage states by X-ray computer tomography (CT) and small angle X-ray scattering (SAXS) to analyze the damage behavior due to mechanical loading. First the orientation distribution of the short fiber in a polyamide matrix were determined by high resolution CT as basis to separate the micro cracking in the matrix and fiber matrix debonding quantitatively by X-ray refraction topography (using SAXS-technique) and a specific model. Finally based on the model and perpendicular X-ray-refraction-topography scans, the materials damage state could be stated efficiently.

1 Introduction

Short fiber reinforced plastics are increasingly used for automotive applications. Due to high tooling costs it is of interest to calculate the in service strength numerically in the design-process. Therefore the fiber orientation distribution could be predicted by several numerical tools [1, 2]. However, a sufficient model for the prediction of the fatigue behavior with respect to the local fiber orientation is still an open question. Finally, the geometry of the mold and hence of the component has to be designed with respect to the fiber orientation due to the injection process, the desired stress level, and the in service strength.

The strength of short fiber reinforced plastics was investigated more than 20 years ago [3, 4]. Additional the fatigue strength was determined at high oriented material [5] in and of axis to the fiber filament orientation and can be summarized in normalized S-N-curves [5]. However modeling a material with a more statistical fiber orientation is difficult. In former work the increase of micro cracking and fiber matrix debonding was investigated non-destructively by X-ray-refraction topography in different damage states [6, 7]. But only in materials with high oriented short fibers the two damage mechanism can be separated by perpendicular measurements.

In the present work a basic material investigations were done to understand quantitatively the X-ray-refraction signal with respect to the fiber orientation distribution. This is the basis for a

nondestructive analysis of the damaging processes due to mechanical loading, as static-, long term- and fatigue loading.

2 Materials and testing methods

The investigated material was polyamide 6 with 30% per weight short glass fiber reinforcement with a fiber length distribution between about $20\mu m$ up to $1000\mu m$. The peek value was $200\mu m$ and the mean fiber filament diameter of about $11\mu m$. The presented investigations were done at standard tensile specimens according to ISO 527, type 1A.

The X-ray refraction topography was used to measure the increase of inner surface in the material due to micro cracking and fiber matrix debonding. Therefore the plane projection of the signal as function of the inclination of the sample length direction with respect to the plane of collimation has to be investigated to understand the signal due to the inner surface of the perfect bonded fibers and fiber-ends.

X-ray refraction topography [8] is caused by the effect of refraction at the interface of materials of different refractive index as well known from visible light passing glass lenses. In the case of X-rays the refraction angle is below half a degree and in opposite direction due to the dispersion function of isolators. In the experimental set-up (s. fig. 1, right) a collimated X-ray beam passes the sample. At a fixed angle the refracted signal is measured and additionally a signal proportional to the absorption. A characteristic refraction value C is determined, which is proportional to the surface per unit volume. It can be calculated from the scattering I_R and transmitted intensities I_A and the thickness d of the sample in relation to the zero values (without sample):

$$C = \left[\frac{I_R/I_{R0}}{I_A/I_{A0}} - 1\right] \cdot \frac{1}{d} \tag{1}$$

The intensity of the refracted beam will increase, if a difference of the refractive index occurs at the observed interfaces. Hence, the intensity will be higher for materials with de-bonded fibres or pores than without (s. fig. 1, left). By calibration the absolute as well as the relative inner surfaces C are measured. In most cases the relative increase is sufficient. Scanning the whole area of the sample gives a topographic map of inner surfaces.



Figure 1. left – X-ray-refraction effect, right – experimental set up of SAXS

With the inclination angle θ each fiber filament affects as cylindrical lens a cos²-shaped signal [9]. Respecting the total reflection of the fiber ends the signal can be described as:

$$\frac{C_m(\theta)}{C_m(\theta)_{\max}} = A + (1 - A) \cdot \cos^2(\theta) = f(\theta)$$
⁽²⁾

A is a parameter respecting the number of fiber ends per volume and do not change if the fiber length distribution remains for different materials.

In the next step the fiber orientation distribution was measured with high resolution computer tomography at the BAM-line at BESSY II with synchrotron radiation in a parallel-beam experimental set-up (s. fig. 2, left.). At a sample volume of 1mm³ a sub-micro-meter resolution was reached (s. fig. 2, right).





Figure 2. left – experimental setup of high resolution X-ray computer tomography at BESSY (synchrotron), right – high resolution (voxel size 0,4μm³) CT-image of short fibers in tensile specimen

However, to determine the fiber orientation frequency per inclination angle θ a resolution with a voxel size of about $2\mu m^3$ is sufficient [s. fig. 3]. Cuts of the skin layer and the middle layer were analyzed (s. fig. 3). The fiber orientation in injection direction in the skin layer is higher than in the middle layer. However, the mean value of the distributions has to be taken hence the X-ray-refraction technique takes the inner surfaces as a mean value over the thickness.



Figure 3. high resolution (voxel size 2 µm³) CT-image of short fibers in tensile specimen for fiber orientation analysis

A further indication was that mostly an inclination in the h1-h2-plane occurs due to the injection molding process and a tilting of the fiber filaments in the third dimension can be neglected. Hence the fiber orientation frequency over the inclination angle can be approximated as two dimensional elliptically function (s. fig. 4):

$$g(\theta) = \frac{h_2}{\sqrt{1 - \frac{h_1^2 - h_2^2}{h_1^2} \cdot \cos^2(\theta)}} \quad \text{and} \quad g(\theta) = B \cdot G(\theta) \quad (3)$$

B is a calibration factor respecting the experimental set-up of SAXS (e.g. scattering angle,..).



Figure 4. determined fiber orientation frequency along the specimens

Final all signals of each fiber filament (s. eq. 2) have to be superposed respecting the fiber orientation frequency (s. eq. 3) which results in:

$$F(\theta) = \frac{1}{T} \cdot \int_{\alpha}^{\alpha+T} f(\tau) \cdot G(\theta - \tau) d\tau$$
(4)

Again this is a \cos^2 -shaped function. The two value C and D can experimentally easily determined by two perpendicular measurements in 0° and 90° with respect to the collimation plane.

$$F(\theta) = C \cdot \cos^2(\theta) + D \tag{5}$$

In fig. 5 is shown graphically the mathematical approach. With the measured fiber orientation frequency by CT and the cos²-shaped signal of each fiber filament the measured refraction value at different inclination angles can be described by an cos²-shaped funktion. The offset value results in defraction effects due to the semi cristalline matrix.



Figure 5. fiber orientation distribution

3 Discussion and results

The main task of this paper was to show how to understand the X-ray-refraction measurements as a plain projection of inner surfaces due to the fiber filament orientations caused by the injection molding process. An analytical formulation was shown which enables to understand the signal variation when inclining the specimen with respect to the fiber orientation frequency.

The increase of inner surfaces due to mechanical loading was already investigated in previous work [7, 8, 9]. However, the fiber filament orientation is not perfect in length direction and an approach to distinguish between fiber matrix debonding and micro cracking is missing. Comparing the undamaged and damaged state the cos²-shaped function keeps still valid however an increase of all values over the inclination angle can be observed [s. fig. 6]. Though there is not a constant increase. More over the increase of inner surfaces has to be understood as increased fiber-matrix debonding and micro cracking at the fiber ends. Therefore a further degradation model was developed, respecting the fiber orientation and the direction of the stress vector. This is going to be published soon.



Figure 6. undamaged and damaged state of PA-GF 30

4 Conclusions

Short glass fiber reinforced polyamide is increasingly used in automotive applications. It is of high interest to understand the degradation mechanism due to mechanical and fatigue loading. With the present approach the efficient method of X-ray-refraction topography can be used to analyze the fiber orientation distribution and the increase of inner surfaces due to mechanical loading. With this NDE-technique the evolution of materials degradation could be investigated. Additional CT-measurements were done to analytically show the correlation between elliptical fiber orientation frequency and cos²-shaped X-ray-refraction results as a function of the inclination angle. In further work an approach to distinguish between micro-cracking and fiber matrix debonding will be shown. Finally this gives a better understanding of the degradation processes due to static and fatigue loading respecting the load ratio.

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