MULTIAXIALLY LOADED SHORT FIBRE POLYAMIDE: A CONTRIBUTION TO NON-DESTRUCTIVE EVALUATION OF MICRO CRACKING AND DAMAGE EVOLUTION

K. Metzkes¹, Y. Hentschel¹ V. Trappe¹*

¹ BAM Federal Institute for Materials Research and Testing, Division 5.3 – Mechanical Behavior of Polymers, Berlin, Germany
* Corresponding author (volker.trappe@bam.de)

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1 Abstract
Short fibre reinforced thermoplastics are increasingly used in automotive applications because of their potential for lightweight design and cost-efficient manufacturing by injection moulding. The fibre orientation, tuneable in the production process, defines the degree of anisotropy which causes different damage behaviour depending on the multiaxial stress state. Therefore, the multiaxial damage behaviour, based on micro cracking, is analysed by combining mechanical loading tests and non-destructive X-ray refractometry.

2 Introduction
There is an increasing interest in short fibre reinforced polyamide in automotive applications due to its potential for lightweight design and cost-efficient manufacturing by injection moulding. The simulation of the injection moulding process allows to predict the fibre orientation and an accordingly designed anisotropy. Besides material strength and elastic properties, fatigue behaviour and damage mechanisms are also dependent on the load direction in relation to the fibre orientation distribution.

In order to describe the structural durability of short fibre reinforced polyamide, it is necessary to understand the damage behaviour under different components of load. Since the damage of short fibre reinforced polyamide is based on micro cracking, the non-destructive method of X-ray refraction analysis provides the potential to characterize the evolution of damage.

The damage mechanisms of short glass fibre reinforced polyamide in uniaxial tensile and compression tests have been studied by GÜNZEL [1] and TRAPPE et al. [2, 3] at BAM Federal Institute for Materials Research and Testing. In this study, the X-ray refraction analysis was used to measure the plane fibre orientation distribution and the directional micro cracking evolution. A micro damage model, developed by GÜNZEL [1], enables to quantitatively understand the X-ray refraction signal and to separate the micro cracking phenomena of fibre matrix debonding and matrix micro cracking with respect to fibre orientation distribution.

In the following the damage behaviour due to axial as well as torsion loadings will be focused on.

3 Theory
3.1 Damage mechanisms in short fibre reinforced polyamide
The damage in short glass fibre reinforced polyamide is ascribed to the micro cracking phenomena of fibre-matrix debonding and matrix micro cracking [4]. The occurrence of these phenomena depends on the stress state and in particular on the load direction in relation to fibre orientation.

Damage starts with fibre-matrix debonding and matrix micro cracking and results in cumulative damage either from further occurrence or from local concentration of these phenomena (Fig. 1).
3.2 X-ray refraction analysis

X-rays are refracted at interfaces of materials or media with different electron densities and respectively different refractive indices. This effect is used for X-ray refraction analysis based on the technique of small angle X-ray scattering (SAXS) which has been developed by HENTSCHEL et al. at BAM Federal Institute for Materials Research and Testing [5, 6]. The short wavelength of X-rays (Mo-Kα 0,7Å) allows for confident detection of inner surfaces from minimum size of 70 nm [7].

In Fig. 2 the used Kratky-SAXS measuring system with fixed scattering angle is shown. The X-ray beam, collimated to a rectangular square section ((2-3) mm x 50 µm), passes the sample. The two scintillation detectors measure the intensity of the refraction $I_R$ directly and the intensity of the absorption $I_A$ indirectly by a scattering foil. These intensities as well as the zero values $I_{R\theta}$ and $I_{A\theta}$, measured without a sample in the path of rays, enable the calculation of the refraction value $C$ (eq. 1).

$$C = \left[ \frac{I_R}{I_{R\theta}} \right] \cdot \frac{1}{d} \cdot \left[ \frac{I_A}{I_{A\theta}} - 1 \right]$$

The refraction value which depends on the thickness of the sample $d$ is proportional to the inner surface of the radiographed volume [8].

The absorption of the material is described by the Lambert-Beer law (eq. 2) with the absorption coefficient of the material $\mu$.

$$I_A = I_{A\theta} \cdot e^{-\mu d}$$

From the equations (1) and (2) follows the specific refraction value $C/\mu$ which is independent of the specimen thickness and directly proportional to the refractive intensity.

Scanning a significant area of the sample yields a spatial resolution of the inner surface integrated over the sample thickness.

Glass fibres are cylinder lenses on which X-rays are refracted in a plane perpendicular to the axis of the fibre. This effect is used by the rotational X-ray refraction topography in order to measure the orientation of fibres [9].

In composite materials three types of inner surfaces appear: Bonded fibre-matrix interfaces, debonded fibre-matrix interfaces and matrix-cracks (Fig. 1 and Fig. 3). Assuming that in the undamaged state the fibre claddings are bonded and the end faces are debonded due to the manufacturing process, the rotational X-ray refraction topography allows to detect the integral fibre orientation over the wall thickness.

Progressive damage includes debonding of fibre-matrix interfaces and matrix cracking. The intensity of refraction depends on the index of refraction resulting from the electron density change at interfaces. For light materials, the electron density change approximately correlates with the mass density change. Therefore, the intensity of refraction increases with an increasing state of damage.

4 Materials and test specimen

A polyamide 6 containing 30 weight per cent (respectively 15 volume per cent) of short glass fibres is analysed.

For uniaxial as well as multiaxial mechanical tests, an injection moulded tube specimen with a pin-point gate was designed and provided by a project partner [10]. This sample design enables high fibre orientation in the direction of the centre line, geometrical stability against buckling due to compression and torsion loads, minimized boundary effects in comparison to flat specimens and constant testing conditions for uniaxial as well as combined tension, compression and torsion tests.

The parallel measuring section with a length of 20 mm and a wall thickness of about 2 mm starts at a distance of 95 mm from the pin-point gate (Fig. 4).

Hence, the fibre orientation in the measuring section varies slightly, which is shown by X-ray refraction analysis and Young’s modulus according to the peripheral angle of the tube specimen (Fig. 5). A pronounced binding line is not detected. As a result of the thin walled tube, the fibre orientation in the measuring section shows a weakly distinctive centre layer.

The short glass fibres have an average fibre length of 180 µm and a mean fibre diameter of 11 µm.

The distribution of the fibre orientation in the φ-z-plain is measured by X-ray-refraction topography. This result is confirmed by a high resolution computer tomography measurement of a specimen
section ((2x1x1) mm³). Post processing via VGSTUDIO MAX by VOLUME GRAPHICS provides the symmetric second order orientation tensor of the spatial fibre orientation (eq. 3).

\[
a_{ij} = \begin{bmatrix} 0.03 & 0 & 0 \\ 0 & 0.12 & 0 \\ 0 & 0 & 0.85 \end{bmatrix} \quad (3)
\]

This tensor shows a high degree of fibre orientation in the direction of the centre line \( (a_{rr}) \). Since the degree of fibre orientation in the direction of the thickness \( (a_{ct}) \) is only 25 per cent of the circumferential direction \( (a_{eq}) \), a two dimensional fibre orientation frequency in the \( \varphi-z \)-plain, approximated by an elliptical function over the orientation angle, is applied.

5 Experimental setup

The experimental setup comprises mechanical loading tests and offline non-destructive X-ray refraction analysis to study the damage evolution. Additionally, destructive fractographic checks are carried out.

5.1 Mechanical loading tests

The mechanical loading tests were conducted at a servo-hydraulic tension-torsion testing machine which is equipped with a climate chamber to regulate the temperature and humidity. The quasi-static and the fatigue tests were performed at the climate state of 23 °C and a minimized relative air humidity of 10 %. To avoid thermal failure during fatigue testing, the temperature of the specimen surface was measured and the surrounding temperature was reduced to a minimum of 19 °C to keep the specimen temperature at a state of (23 ± 3) °C.

The strain was measured using the optical measuring system ARAMIS/IVIEW. For this purpose, the axial and the shear strain between two selected points on the specimen surface were analysed. The static and the fatigue test were operated in a load controlled manner. To reach axial and torsion damage in the static tests in the same time span, the load rates were adjusted to the particular strength. All further experimental data are summarized in the table below (Tab. 1).

After fixed load levels or fixed numbers of load cycles respectively, X-ray refraction analysis was performed. Each test procedure was accompanied by 6 to 9 non-destructive X-ray refraction cycles.

<table>
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<td>load level [-]</td>
<td>cycles/ fatigue step [-]</td>
</tr>
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<td>100000</td>
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Tab. 1 Experimental data

5.2 Damage evaluation

For damage analysis, an area of 6 mm arc length \( (\Delta \varphi = 20^\circ) \) and \( \Delta z=20 \) mm axial length was measured by X-ray refraction analysis. The tube specimen was scanned line-wise in axial direction on the diameter (Fig. 7). Afterwards, the specimen was rotated to the next scanning angle and scanned again. Due to the geometry of the tube specimen, the two opposite walls were scanned simultaneously. The inhomogeneity of the composite material causes a variance of the measured intensities. Therefore, a representative mean value of the refraction (eq. 4) is used.

\[
C_m = n^{-1} \sum_{i=1}^{n} C_i \quad (4)
\]

As a consequence of the mechanical loading, the wall thickness varies. Hence, the absorption coefficient \( \mu \) is calculated for the undamaged state because the initial wall thickness is known (eq. 5).

\[
\mu = -\ln\left(\frac{l_A}{l_{A0}}\right) / d \quad (5)
\]
At all further damage steps, the refraction value is independent of the wall thickness (eq. 6).

\[
C_m = \frac{\left(\frac{L}{L_0} - 1\right) \cdot \mu}{-\ln(L/L_0)}
\]

The calculated variation of the refraction (eq. 7) is proportional to the variation of the density of micro cracks.

\[
\Delta C_{mi} = \frac{C_{mi} - C_{m0}}{C_{m0}} \quad i = \text{load step}
\]

X-ray refraction analysis detects inner surfaces depending on their orientation relative to the collimation plane of the X-ray (Fig. 3 and 6). Hence, the rotation of the sample in the collimated X-ray beam provides information about the orientation of the inner surfaces (Fig. 3). In undamaged test specimens, the averaged fibre orientation over the thickness is measured [11]. Based on these measurements, the orientation of damages in damaged specimens can be determined by X-ray scanning at different angles relative to the collimation plane.

The axially loaded test specimens were scanned with the axis of the specimen and the load direction being orientated parallel (0°) or perpendicular (90°) with respect to the collimation plane.

The torsion loaded test specimens are additionally scanned at the diagonal orientations 45° and -45°. (Fig. 7)

6 Results and Discussion

This chapter shows experimental results on the damage behaviour of axially and torsionally loaded tube specimen from short fibre reinforced polyamide.

6.1 Damage evolution under static loading

The damage accumulation resulting from static tensile load is shown in the figures 8 and 9. Both the 0°-scan and the 90°-scan show a progressive increase of micro crack density. At the maximum value of refraction, the maximum of micro crack density is reached and failure occurs. With respect to non-linear elastic strain (eq. 8), the accumulation of the micro crack density increases linear (Fig 9).

\[
\varepsilon = \varepsilon_{\text{linear elastic}} + \varepsilon_{\text{non-linear elastic}}
\]

\[
\varepsilon_{\text{non-linear elastic}} = \varepsilon - \frac{\sigma}{E}
\]

The degree of damage that is measured in the 90°-scan is six times higher as compared to the 0°-scan. In the 90°-scan, inner surfaces orientated vertically to the tensile load direction and the preferred orientation of fibres are detected. In tensile tests, matrix micro cracking occurs vertically to the load direction which is even visible in stress whitening. Due to the high fibre orientation in axial direction, the increase of micro cracking detected in the 0°-scan is mostly caused by fibre-matrix debonding.

Regarding the static torsion load, (Fig. 10) the 90°-scan increases progressively whereas the 45°- and the -45°-scan increase regressively over shear strain. No relevant changes in refraction value are measured in the 0°-scan which indicates that static torsional loading along the z-axis does not cause fibre matrix debonding on fibres oriented in the z-direction.

Looking at the micro crack density as a function of the non-linear elastic shear strain (eq. 9) (Fig. 11), the 90°-scan shows a linear correlation whereas the micro crack densities in the ±45°-scans increase regressively. There is a linear correlation between the shear stress and the variation of micro crack densities measured in the ±45°-scans.

\[
\sigma_{\text{non-linear elastic}} = \varepsilon - \frac{\tau}{G}
\]

A comparison of the damage accumulation in static tensile and torsion tests shows a progressive increase of the micro crack density in the 90°-scans, vertical to the principal direction of the fibres.

In conclusion, the matrix micro cracking starting at the end faces of the fibres seems to be a leading damage phenomenon.

6.2 Damage evolution under fatigue loading

In the axial fatigue test (R = 0.1) at a load level of 0.65, failure occurs after 150000 cycles. The torsion fatigue test (R = 0.1, σf/σt = 0.75) reaches 525000 cycles.

Regarding the evolution of the micro crack density over load cycles, the 0°-, the 90°- and the 45°-scans
increase regressively. The 90°-scans, detecting inner surfaces with a vertical orientation to the principle direction of the fibres, posses the major growth in the micro crack density. As a result, the matrix micro cracking with a vertical orientation to the principle direction of the fibres even dominates the damage evolution of tension and torsion fatigue tests.

7 Conclusion
The evolution of the micro crack density caused by axial and torsion static and fatigue tests was analysed by X-ray refractometry. By scanning different orientations, (Fig. 7) micro mechanical damage effects were evaluated. Furthermore, it becomes clear that matrix micro cracking vertically aligned to the principle orientation of the fibres is a leading damage phenomenon under axial as well as torsion static and fatigue loadings. Due to the high degree of fibre orientation in the axial specimen direction, the directional scans allow for a first estimation of the damage mechanisms fibre matrix debonding and matrix micro cracking. A precise separation of these phenomena is introduced by GÜNZEL [1] for axially loaded short fibre reinforced polyamide flat specimens. In the next step, a model which separates these damage mechanisms with regard to the fibre orientation distribution and the load direction is going to be developed.

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Fig. 3 Configurations in composites effecting X-ray refraction
- debonded fibre
- mean intensity of refraction
- bonded fibre
- low intensity of refraction
- matrix crack normal to collimation plane
- no intensity of refraction
- matrix crack parallel to collimation plane
- high intensity of refraction

Fig. 4 Tube specimen
- 210 mm length
- 20 mm wall thickness
- configuration details

Fig. 5 Variation of Young's modulus $E_z$ according to the injection point
- $E_z = (9400 \pm 180)$ MPa

Fig. 6 Rotational X-ray refraction: The detected refraction intensity depends on the orientation of the surface
- Fibre orientations
- Beam expansion
- Collimation plane

Fig. 7 X-ray refraction: Scanning directions
- Scanning directions: 0°, 45°, -45°, 90°
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Fig. 8 Damage in static tensile test

Fig. 9 Damage in static tensile test: Increase of the refraction value $\Delta C_m$ as a function of the non-linear elastic axial strain $\varepsilon_{e,\text{ne}}$

Fig. 10 Damage in static torsion test

Fig. 11 Damage in static torsion test: Increase of refraction value $\Delta C_m$ as a function of non-linear elastic shear strain $\gamma_{\phi,\text{ne}}$
Fig. 12 Damage in static torsion test: Increase of refraction value $\Delta C_m$ as a function of shear stress $\tau_{\phi z}$

Fig. 13 Damage in tensile fatigue test: Increase of refraction value $\Delta C_m$ over load cycles

Fig. 14 Damage in torsion fatigue test

References


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