CHARACTERIZATION OF DAMAGE MECHANISMS IN GLASS FIBRE REINFORCED POLYMERS USING X-RAY COMPUTED TOMOGRAPHY

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Keywords: X-ray computed tomography, short fiber reinforced polymers, fiber orientation, damage characterization.

Abstract

A laboratory X-ray computed tomography (XCT) device was used for the quantitative 3D characterization of damage mechanisms in short glass fiber reinforced polymers. Geometry for test specimens was developed that allows for high resolution and tensile testing within the XCT device. Interrupted in-situ tensile tests on specimens with perpendicular main fiber orientations were performed. Fiber orientation and damage mechanisms were analyzed on single fiber basis using XCT data analysis tools.

Damage mechanisms that were identified are fiber pull-out, fiber breakage, detachment and matrix cracks. Depending on the main fiber orientation pull-out or detachment is predominant. The determined fiber orientations for different regions showed a correlation to the region of final fracture.

1 Introduction

1.1 Short fiber reinforced polymers

Mechanical properties of injection molded filled polymeric materials are influenced by filler content and filler arrangement. For fibers, orientation and length distribution are of importance. Both are determined by the production procedure as well as by the kind of material and fiber content.
Most components that are produced by injection molding are thin walled. Due to inhomogeneous solidification within the mold, different fiber orientations develop across the walls. Fiber orientation is also influenced by the geometry of the component. To be able to produce fail safe componentss, knowledge about fiber orientation in the final components is necessary. [1, 2, 3]

Simulation tools are available and work well for simple geometries. At positions with high shear rates or turbulences the prediction of orientation is difficult. [4] Measurement of fiber orientation is standardly done by grinding and optical measuring of the elliptical shape of the fibers. This is destructive and time consuming. Usually only a small volume can be analyzed.

1.2 3D characterization by means of X-ray computed tomography

A powerful tool to determine fiber orientation and length distribution is X-ray computed tomography (XCT). [5, 6] Parts of a specimen can be analyzed non- destructively and three dimensionally. Laboratory X-CT systems are available that allow for reasonable high resolution characterize single fibers. Using commercially available software tools it is possible to determine fiber orientation on voxel level. More effort has to be made for the characterization of single fibers. [7]
Another advantage of XCT compared to optical methods is the very good statistic. One analysis can contain several 100.000 fibers while optical analysis is usually done for a few hundred fibers.
In this study both quantitative as well as qualitative analysis is performed on high resolution data. For quantitative analysis of damage mechanisms no automatic tool was available which made visual analysis necessary.

2 Materials & Methods

2.1 Materials

As model material system polypropylene filled with 30 weight % glass fibers (PP-GF30) was tested. The nominal mean fiber diameter was 12 µm. From a sheet that was produced by injection molding by Zizala, small test specimens were milled. An inset ensured that a highly oriented meld flow developed. This led to a high degree of orientation for defined regions.
Figure 1 shows the geometry and the positions where the specimens were milled. Main fiber orientations of 0 ° and 90 ° could be realized by this approach. 0° implies fiber orientations in tensile direction.

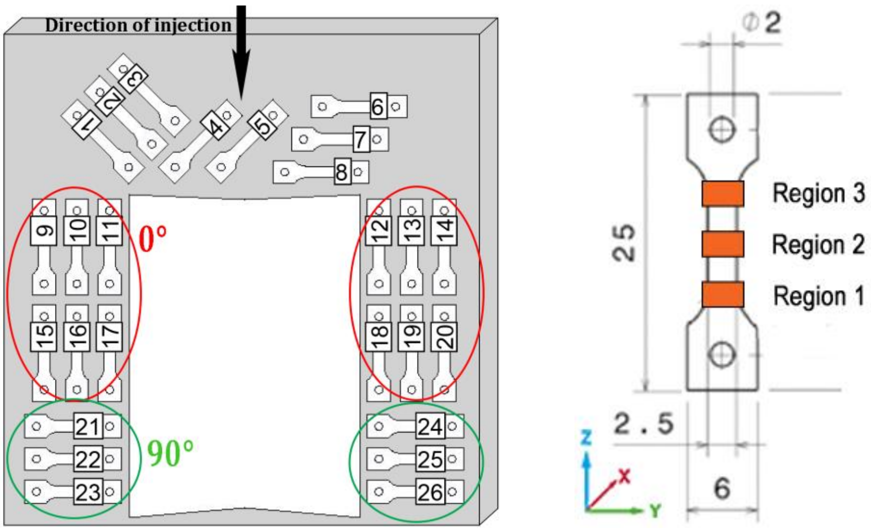

Figure 1. Sketch of the injection molded sheet with regions where test specimens were milled out of the sheet (left), miniaturized tensile test specimen geometry with position of three regions for data analysis (right, dimensions in mm). The sheet thickness was 2.5 mm.

2.2 XCT Scans and interrupted tensile tests

The XCT scans were performed at a laboratory system Nanotom 180 NF (GE Phoenix|x-ray, Wunstorf, Germany). Since a matrix detector (Hamamatsu 2300 x 2300 pixels) was used the system works in cone beam geometry. Together with the fact that the specimen has to be

projected completely onto the detector in horizontal direction the consequence is that the resolution is depending on the size of the specimen.
To be able to scan a bigger volume containing 6 specimens, the resolution was reduced to 5 µm voxel edge length. One single specimen was scanned at a resolution of 2 µm voxel edge length mounted on the tensile device.
The scan parameters are listed in Table1. The source parameters voltage (U), current (I) and Mode were optimized for the material system to achieve optimal contrast and resolution. Mode 0 means that the focal spot of the X-ray tube was depending on the tube power. The readout time for one image at the detector was set to T_{int}. To reduce noise and motion artifacts 6 images at each angular position were read out with the first image to be skipped and the other 5 images to be averaged and saved to disc. ZD denotes to the distance between source and detector.

U [kV]	I [µA]	T_{int} [ms]	Projections	Scantime [min]	Voxel-size [µm]	Target	Mode	Average	Skip	ZD [mm]
80	200	500	1700	88	5	Molybdenum	0	5	1	330
80	130	1200	1900	230	2	Molybdenum	0	5	1	400

Table 1. XCT scan parameters

Within the XCT device a tensile testing device was mounted onto the rotation table. The maximal load that can be applied is 500 N. The tensile tests were performed at a movement speed of 0.2 mm/min.
After mounting the specimen between the clamps without applying any load the first XCT scan was performed to document the initial condition of the specimen.
Three additional XCT scans were performed after increasing the load. The load levels were determined by applying tensile tests until break outside the XCT device. Since the material behavior is brittle and detectible damage occurs only shortly before break the levels were chosen such as 90 % and 95 % of tensile strength. The final scan was performed after break and after a time delay to reduce remaining movements within the specimen.
The maximum load for 0° specimens was approx. 400 N and for 90° specimens 190 N.

2.3 3D data analysis

For the determination of fiber orientation an in house software-development was used that allows for the characterization of every single fiber [7]. The approach uses the information of local orientation and the fact that the gray value in the center of the fiber is a maximum to extract the medial axes. After thinning the lines of voxel describe fiber segments which can be connected. To split up the connections into single fibers local angles and angles between segments are analyzed. The final result is the start and endpoint of each fiber.
For predefined sub-volumes the second order orientation tensor was calculated. The diagonal elements of the tensor are indicated as a11, a22 and a33.
The degree of orientation O_D can be calculated by analyzing eigenvalues λ_n of the orientation tensor according to equation (1).

$$O_D \left(\lambda_n - \frac{1}{3}\right) * \frac{3}{2} \tag{1}$$

The parallel length of the miniaturized tensile test specimen was divided into three regions as shown in Figure 1 along the Z-axis. Each region was analyzed separately and orientation

tensor and degree of orientation was calculated. Along the X-axis, which corresponds to the thickness of the sheet, each region was divided into 20 sub-regions to show the variation of orientation over sheet thickness.
Damage was analyzed qualitatively by visual inspection of slice images in initial state and after fracture.

3 Results

3.1 Fiber characterization

The chosen regions that were cut out of the sheet were pre-characterized by XCT scans with lower resolution of 5 µm voxel edge length. These results were used to select specimens with best homogeneity with respect to fiber orientation.
The following results were realized for specimen number 11 (0° orientation) and number 24 (90° orientation). The specimen numbers are shown in Figure 1.

Specimen	Volume	Voxelsize	number of fibers	O_D	a11	a22	a33	
0°	complete	5	187789	0,69	0,04	0,16	0,80	
	Region 1	5	36072	0,72	0,03	0,15	0,82	
	Region 2	5	36489	0,69	0,03	0,17	0,79	fracture
	Region 2	**2**	35568	0,74	0,02	0,15	0,83	fracture
	Region 3	5	36042	0,70	0,03	0,16	0,80	
90°	complete	5	247072	0,62	0,04	0,74	0,22	
	Region 1	5	40850	0,68	0,03	0,79	0,18	fracture
	Region 1	**2**	33077	0,73	0,01	0,82	0,16	fracture
	Region 2	5	42732	0,61	0,03	0,74	0,23	
	Region 3	5	43236	0,56	0,03	0,69	0,28	

Table 2. shows orientation tensor elements a11, a22, a33, degree of orientation O_D and number of fibers for different regions of two specimens (0°, 90°). Additional results with higher resolution (2 µm) for region of break.

Quantitative results of fiber orientation for the separated regions and also for the complete specimens are shown in Table 2.
The region, where final fracture occurred was scanned with higher resolution of 2 µm voxel edge length. This was region 2 in the middle of specimen number 11 (0°) and region 1 at one end of specimen 24 (90°).

Homogeneity of orientation can be revealed from the degree of orientation values and also from the plots of O_D and the biggest element of the orientation tensor shown in Figure 2.

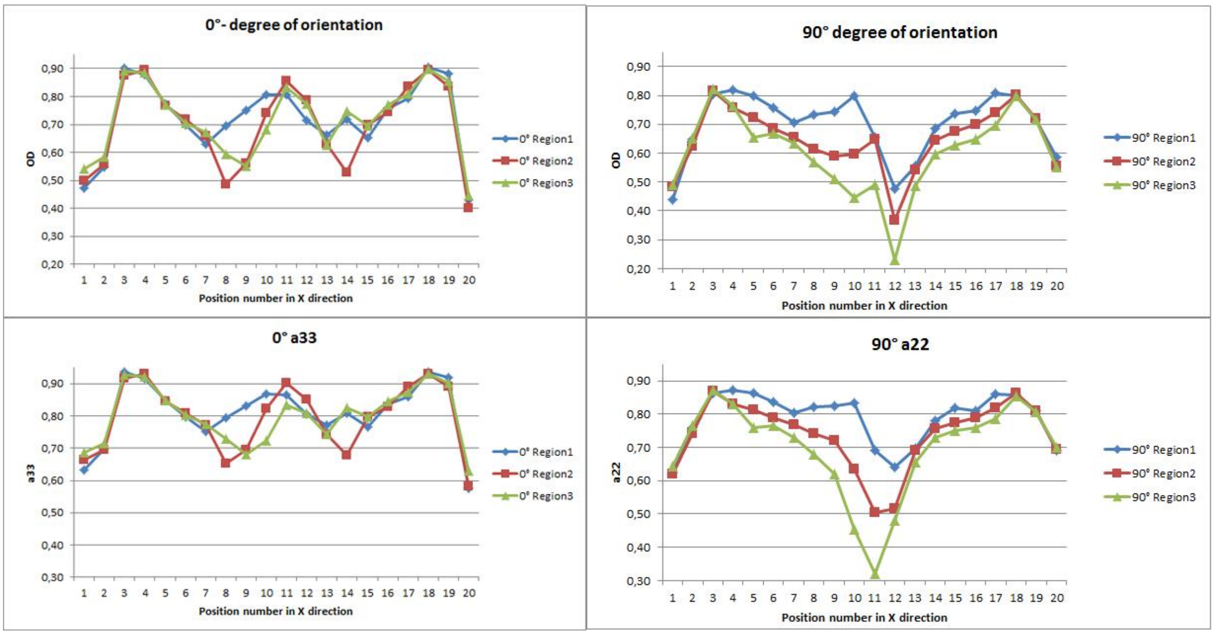

Figure 2. Degree of orientation and biggest tensor element across the sheet thickness for specimens 0° and 90°

3.2 Damage characterization

The main damage mechanisms that can be detected by XCT analyses are fiber pull-out, fiber breakage, fiber-matrix detachment and matrix cracks. Slice images parallel to the fibers in Y-Z plane are shown in Figure 3 to show these different kinds of damage.

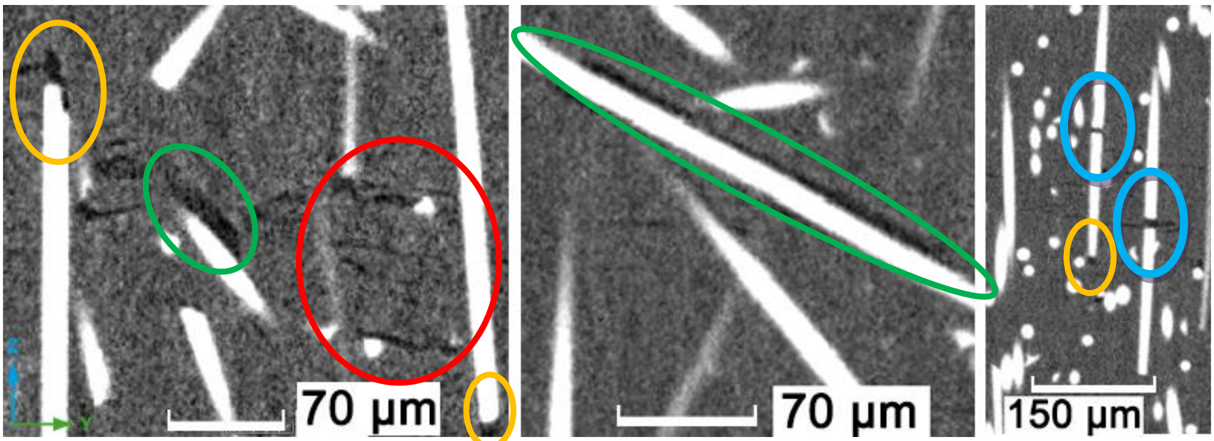

Figure 3. Vertical slice images showing different kinds of damage: fiber pull-out (orange), matrix cracks (red), detachment (green) and fiber breakage (blue)

By visual inspection of slice images at different load conditions it is possible to follow single fibers. Comparisons of the material before load and after fracture are shown in Figure 4 and 5. Due to necking it is not possible to find exactly the same plane containing the same fibers. A complete 3D analysis is necessary. Nevertheless it is possible to look at the region around the final fracture and compare fibers there.

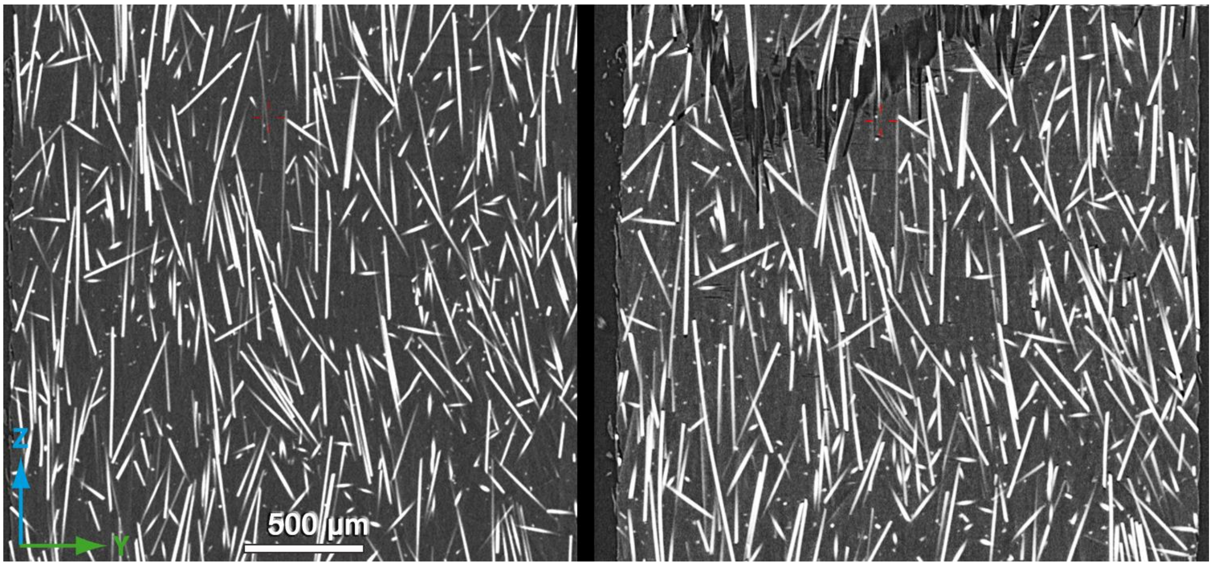

Figure 4. Vertical slice images of 0° specimen before loading (left) and after fracture (right). Voxel edge length 2 µm

The crack in the 0° specimen is surrounded by many pulled out fibers. Also broken fibers can be detected by comparing the two images in Figure 4. The pathway of the crack is not linear throughout the cross section but changes direction often. The polymer crazes within the crack show irregular shape.
The crack in the 90° specimen is determined by many detached fibers, only a few are pulled out of the matrix vertically. The crack moves through the specimen mainly in horizontal direction which is seen in Figure 5.

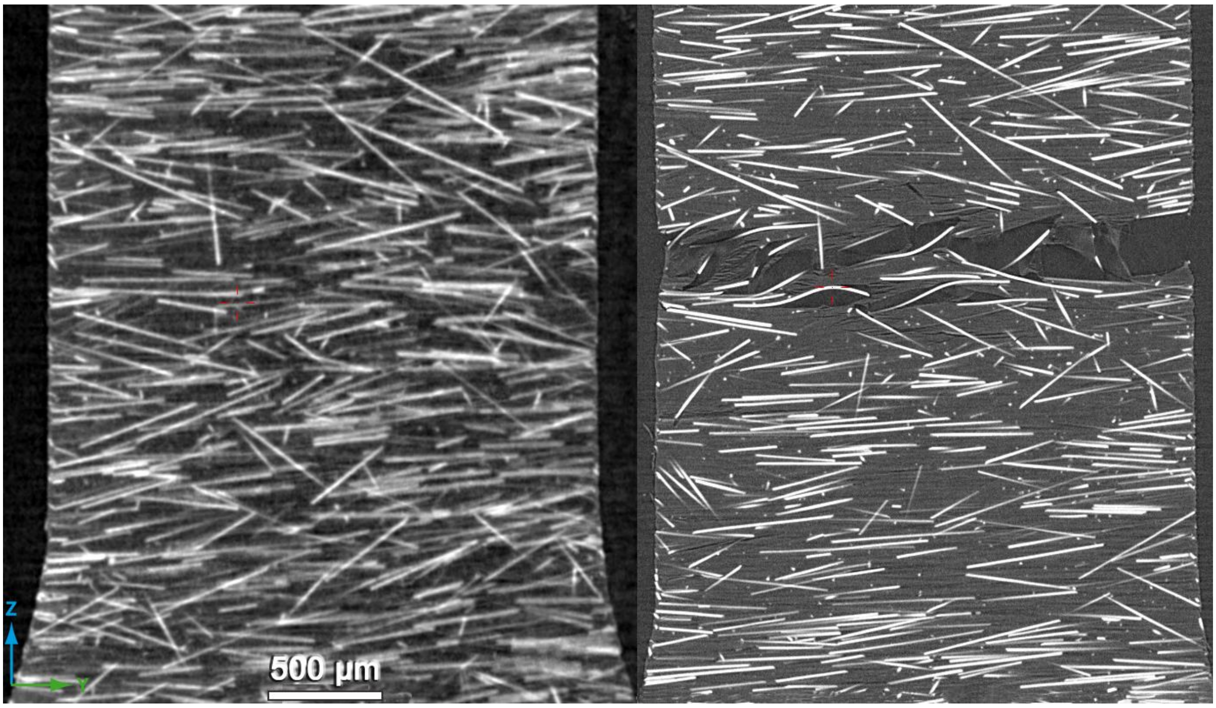

Figure 5. Vertical slice images of 90° specimen before loading (left) and after fracture (right). Voxel edge length 5 µm (left) and 2 µm (right)

The initial scan position for the 90° specimen with high resolution was not at the position of fracture but in the center of the specimen. Therefor the comparison of initial state and after fracture in Figure 5 was done with the overview scan with lower resolution.

Figure 6 shows horizontal slice images of the fracture for the 0° and the 90° specimen. The images consolidate the findings of the vertical image analyses. Fracture surface is much bigger in one slice for the 90° specimen which means that the crack extension is mainly horizontal. A distinct orientation of remaining polymer within the crack in Y-direction can be detected at the 90° specimen.

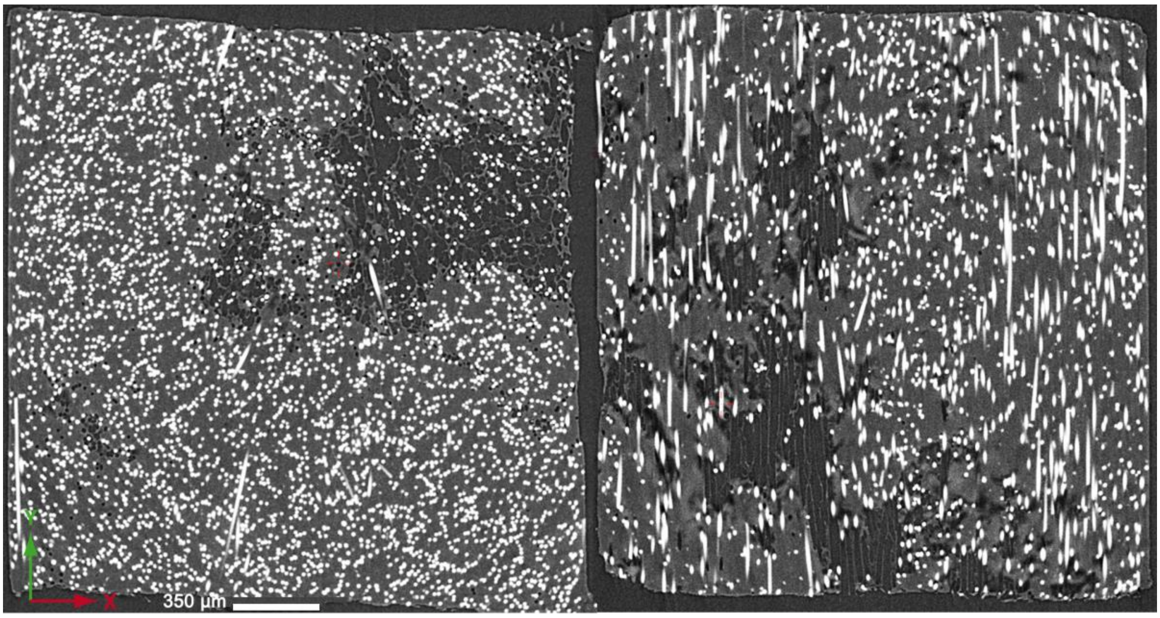

Figure 6. Horizontal slice images of 0° (left) and 90° (right) specimen. Voxel edge length 2 µm

4 Discussion

In this study a voxel edge length of 5 µm (approx. fiber diameter * 0.41) was used to determine orientation tensors. Comparing these results with scans at 2 µm voxel edge length in table 2 slightly lower fiber alignment is determined. Previous studies have shown that at 2 µm voxel edge length also fiber length distribution can be determined accurately. [7]

The usually well-known effect of inhomogeneous fiber orientation throughout sheet thickness (layered structure) can also be seen for the investigated material. Due to the inset this effect could be minimized especially for the 0° specimens. The values for the complete specimens in table 2 show a higher degree of orientation for the 0° specimen as for the 90° specimen.
For all specimens and regions a layered structure is found. The graphs in Figure 2 show basically 2 regions with strong orientation near the surface of the sheet. The 0° specimen shows a more homogeneous orientation distribution than the 90° specimen where the orientation in the middle of the sheet is changing to almost 0°.

Comparing the 3 regions of each specimen, differences in degree of orientation and also in the direction can be found. There is a correlation between degree, direction and final fracture. Both specimens break in a region where more fibers are oriented transversely to the loading direction. For the 0° specimen this is region 2 where degree of orientation and the value of a33 are lowest. For the 0° specimen the fracture is located in region1 where the degree of orientation and the value of a22 are highest.

For the characterization of damage it is necessary to use high resolution. For both main orientations damage of fibers, fiber pull-out, detachment and matrix cracks were detected. Since the maximum load was much lower for the 90° specimen and because of the orientation of the fibers transversely to the load, less fiber breakage and pull-out was detected. The main damage mechanism for the 90° specimen was detachment in combination with matrix cracks.

5 Conclusions

The method of XCT allows for the quantitative analysis of fiber reinforced materials. For the analysis on single fiber basis, data quality has to reach a reasonable level. One of the main influencing parameters is the voxel edge length but also parameters of the scanner are of importance.

The pre-characterization of the complete specimen using lower resolution gives the information where the final failure will occur if the orientation distribution is not homogeneous throughout the whole specimen.

For fibers oriented parallel to the tensile load the damage starts with fiber pull-out and breakage. Near the region of the final failure also detachment can be observed. Matrix damage is located around the fiber end since there the stress is highest.
For fibers oriented perpendicular to the tensile load the damage starts with detachment. The final crack can jump from one detachment to the other which leads to a small fraction area and a fast moving crack.

Future work will take the real fiber arrangement as basis for finite element modelling which can also give the information where and at which load the material will fail.

Acknowledgements

This work is part of the K-Project "Non-Destructive Testing and Tomography" supported by the COMET-Program and the project "3D-SFC" of the Austrian Research Promotion Agency (FFG) and the Province of Upper Austria (LOÖ).

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