Analysis of Fibre Orientation using μCT Data

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Summary: Integrative simulations are based on a calculated fibre orientation from which the local material properties can be derived in several ways. For instance the micro-mechanical model proposed by Tandon and Weng may be used coupled with an orientation averaging approach to include the fibre orientation. This approach then gives the elastic properties of the fibre matrix compound with a strong dependency, of e.g. the elastic modulus, on the fibre orientation. Modeling failure for finite element simulations, e.g. crash, also requires knowledge of the fibre orientation, because the failure strains and energy dissipation also depend on the orientation of the fibres [1, 2]. The accuracy of the calculated fibre orientation depends on several simulation input parameters, which are not necessarily physical properties. The most important example is the fibre interaction coefficient (fic). This parameter allows the user to modify the calculated fibre orientation from isotropic to transversely isotropic [3]. In this paper a new experimental method to determine the fibre interaction coefficient is presented. The classical approach to validate the calculated fibre orientation would be the usage of optical microscope images of cut surfaces of the specimen and the calculation of the fibre orientation by measuring the cut ellipsis dimensions. This method is very time consuming and with respective to the necessary magnification not very accurate, because not all fibres can be accounted for. The new method is using a model based algorithm to analyze three-dimensional micro computer tomography measurements. This enables the identification of up to 90% of the fibres within the specimen and calculate a second order orientation tensor and the fibre length distribution in any arbitrary space. Due to the fact that a model based algorithm is used, the fibre detection can also be performed, if the density of the matrix polymer is near to the density of the fibre material. This is a novelty to existing fibre orientation measurements with computer tomography. To obtain reliable data which can be directly compared with injection moulding simulations, several steps had to be taken. First of all a representative volume must be defined, in which the fibre orientation will be evaluated. This representative volume must be the same in the injection moulding simulation and the μCT measurement. As the injection moulding model is already discretized, the representative volume is set as a stack of finite elements over the part thickness. To calculate the second order orientation tensor in exactly the same geometrical space and the same coordinate system as in the injection moulding simulation, it was necessary to develop a method which allows a reconstruction of the original part from which the μCT specimen was taken. A special painting and evaluation procedure were implemented into the existing method to recalculate the original position and orientation of the specimen, enabling us to achieve the desired measurements. At the moment the determination of the fibre interaction coefficient requires still many injection moulding simulations, which then are compared to the measured values. This allows for a more

realistic fibre coefficient in comparison to the default parameter. The next steps are to automate the described procedure and to correlate the measured fibre orientations directly with the fibre interaction coefficient to avoid unnecessary simulations.

Keywords: Composite structures, fibre orientation, integrative simulation

1 Introduction

The mechanical properties (e.g. stiffness, strain at failure, yield stress) of short glass fibre reinforced polymer parts depend on the orientation of the reinforcement fibres. The resulting fibre orientation in injection moulded parts, depends on several parameters. These are the material properties of the reinforced polymer on one hand (e.g. pvT, viscosity), and the machining parameters (e.g. velocities, pressures, temperatures) on the other hand. The geometry of the part also has an important influence on the fibre orientation. In shell like parts, fibres are oriented in layers [4]. It can be said that the fibres in the boundary layer are mostly oriented in flow direction and the fibres in the core layer are oriented perpendicular to the boundary layer. This is illustrated in figure 1.

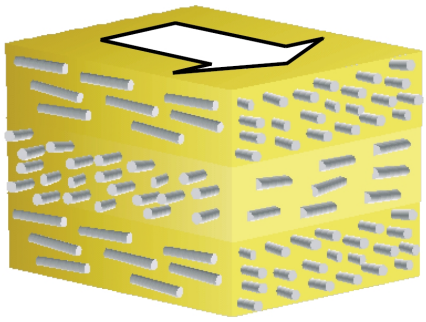

Figure 1: Schematic fibre orientation

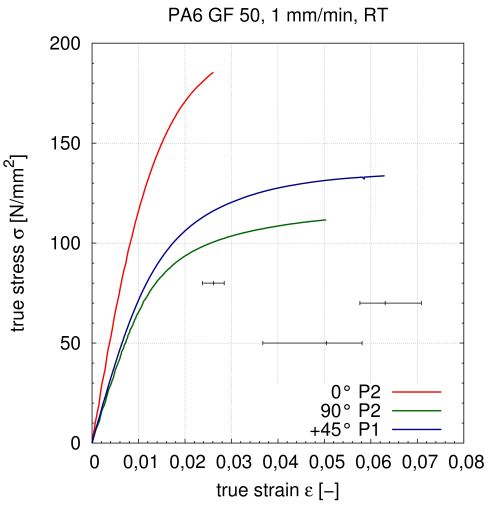

Figure 2: PA 6 GF 50% under different loads

The mechanical properties of a shell like structure are demonstrated in figure 2. These results were obtained from uniaxial tensile tests (see also [5, 6, 7, 8, 9]. The test specimens (Beck-erzugstab [10]) were milled out of a 80 mm x 120 mm x 2.5 mm plate and then tested under three different angles. The 0° specimens correspond to the flow direction of the polymer and the 90° specimens are oriented perpendicular to it. The area in which the results were measured is the same in all tests. The graph shows a difference in strength that is nearly factor two as well as a significant difference in stiffness. If a new thermoplastic part is developed and there is no knowlegde of the fibre orientation in the part, the constructor must use the lowest material strength and Young's modulus. This results in an overdimensional part, which leads to higher material usage, longer cycle times and makes it therefore harder to compete against a standard metal part.

To elude this problem the fibre orientation must be taken into account during the part design phase [11]. This can be done by simulating the whole injection moulding process and then transfer the calculated fibre orientations into the structural simulation of the part. In figure 3 the desired process chain is shown. The part and mould are designed in a 3D CAD environment to use the geometry in an injection moulding simulation, during which, all typical parameters can be calculated. Two other important parameters, which must be given by the user, are the

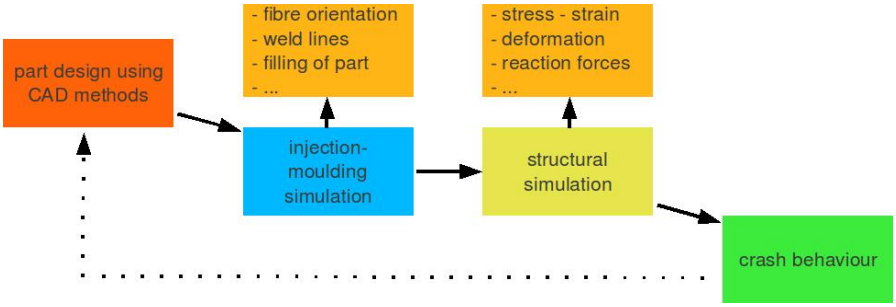

Figure 3: Processchain "integrative simulation"

fibre length or fibre length distribution and the volumetric fraction of fibres. The results from the injection moulding simulation can then be transfered to the structural simulation, which will use the material properties of the fibres and the matrix polymer combined with a calculated fibre orientation to predict the compounds mechanical properties.

As we have seen in figure 2, the influence of the calculated fibre orientation is tremendous and has to be validated before going on with the structural simulation.

2 State of the Art

To validate a simulated fibre orientation the state of the art technique is a microscopical evaluation of polished cut surfaces from reference parts [12]. The fibre length distribution and volume fraction can be obtained by incineration and likewise microscopical analysis of the remaining fibres. These two experiments will be described in this chapter to show the need of a new validation method.

The first step to validate a calculated fibre orientation is to choose a representative volume in which the real orientation will be analyzed. This could be a volume of a finite element stack taken from the simulation, because this saves the trouble of recalculating a mean tensor over a new representative volume. As we expect the fibre orientation to be layer wise, the analysis of the thickness of single layers and their corresponding fibre orientation is the most interesting.

After a representative volume is chosen, it needs to be prepared for analysis. This preparation takes several steps. First the specimen must be cut out of the reference part and then very carefully placed into a small rubber cup which will be filled with a thermo-set resin (see figure 4). During this step it is of essential importance to align the specimens under the correct angle, because the following analysis is very sensitive to this parameter. This is manually very difficult and requires experience. If the specimen is aligned correctly, the rubber cup is placed in a vacuum container and is slowly filled with thermo-set resin to avoid bubbles. After the curing period of the resin, the specimen is taken out of the rubber cup and then subjected to a series of grinding and polishing steps.

At first the specimen is clamped in a device to ensure a parallel grinding process. Then the specimen is grinded down to the desired position. This takes three different types of coarseness of the grinding paper as well as different grindforces applied to each specimen. After the grinding process the surface must be polished. This again takes three different polishing parameter sets varying in size of grains used in the polish and force applied to each specimen. The surface has

to be polished to a degree where the specimen shows no grooves from the grinding process and the fibres are clearly visible. Different states of a specimen are shown in figure 4.

Figure 4: Different states of the specimen

To achieve such results, the clamping device must be cleaned thoroughly after each step. First under running water to remove all grains from the previous grinding or polishing run. Secondly in an ultra sonic bath to remove all grains that might be in grooves in the specimen itself. And third the specimens must be rinsed with ethanol to clear any remaining dirt away. Subsequent polishing steps differ in the applied force, the time and grain size of the polish.

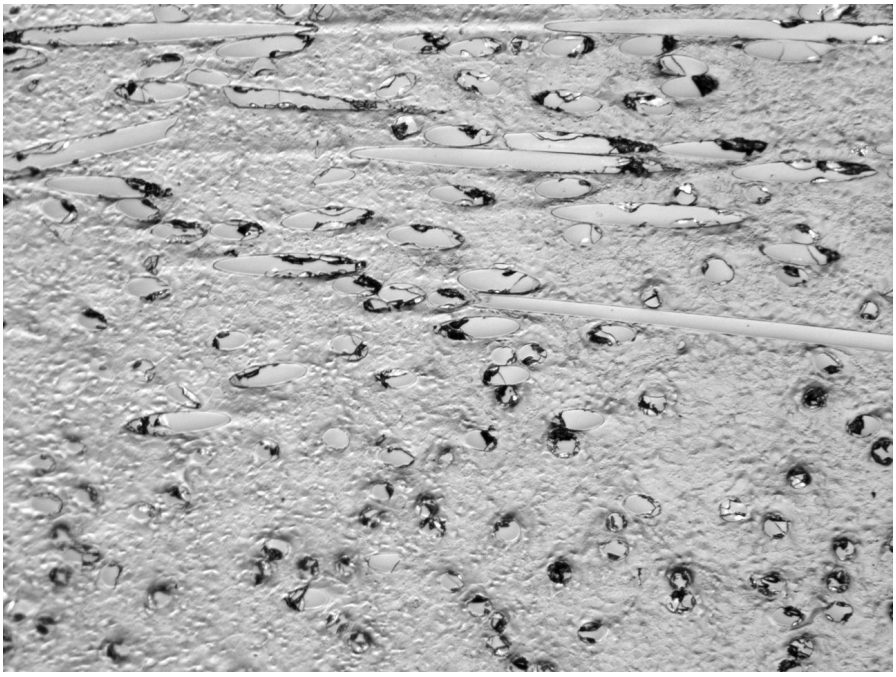

Figure 5: Glas fibre ellipses in a test specimen

If the specimen preparation is finished the specimens are analyzed under an optical microscope. Here, fibres that penetrate the cut surface become visible as ellipses with different ratios of

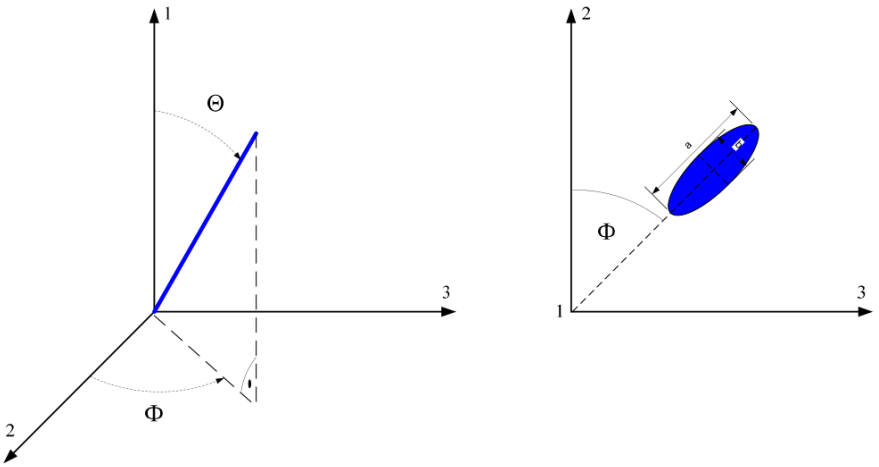

Figure 6: Angular correlation in cutsurface

semimajor axis to semiminor axis. This can be seen in figure 5. Using a specialized computer software to measure the length and angle of the semimajor axis and semiminor axis the second order orientation tensor can be calculated from the results [13, 14, 15].

Problems that arise during the preparation process can be comprehended by looking at figure 5, which shows a polished surface of a Polyamide 6 with 30% glass fibres (PA6GF30). Many fibres are pulled out at the edges or even crushed in a way, that polish remains in the cracks. This makes it very difficult to measure the semimajor and semiminor axis and therefore the error in the measured orientation tensor is comparatively high.

The next problem with this method is the fact, that a fibre with an orientation (Θ, Φ) is indistinguishable from a fibre with an orientation of $(\Theta, \Phi + \pi)$. The only solution is to prepare a second cutsurface of the same specimen, some micrometer below the first and to analyze the specimen again [16]. Then the identification of fibre orientation is unique, because of the correlation of the ellipses in both cutsurfaces.

To analyze fibre length distribution and volume fraction, a representative volume of the reference part must be incinerated. This experiment starts with the preparation and weighing of the specimen. Then the specimen is put into a porcelain cup and incinerated. The remains are only the fibres. The fibre length distribution can then be determined by measuring the fibres under an optical microscope.

This shows that for a precise and significant evaluation of the fibre orientation, a lot of manual work has to be done. The specimen preparation is time consuming, difficult and the quality depends strongly on the skills of the preparator. As the development process of a new technical part becomes ever more faster, this validation method is inappropriate [17]. A new validation method needs to be established.

3 Measurement with a Micro-Computer-Tomograph

In this chapter the new measuring equipment will be introduced and then a novel measuring procedure for fibre detection will be explained [18]. In chapter 3.1 the technical properties and

functional principle of the used μCT will be shown. Sample preparation and the implemented algorithm will be explained in chapters 3.2 and 3.3.

3.1 Micro-Computer-Tomograph Equipment

In figure 7 the used μCT is shown, and the functional principle is schematically constituted in figure 8. The technical properties of the SkyScan 1072-100 μCT are shown in table 1. The maximal specimen size depends on the desired resolution. In order to analyze a glass fibre reinforced polymer, the maximal resolution is needed. This results in a maximal specimen size of Ø 4 mm × 4 mm. The specimen is mounted on the specimen holder (see figure 10), which then moves towards the detector. During the scan of the specimen, the specimen holder rotates by either 180° or 360° depending on the user settings. Fibre analysis only needs 180° rotation. After each rotation, the specimen is lifted upwards and the process starts again. The μCT computer reconstructs from the layerwise density distribution a 3D density distribution, which then is subjected to the evaluation algorithm.

Figure 7: SkyScan 1072-100 at the DKI

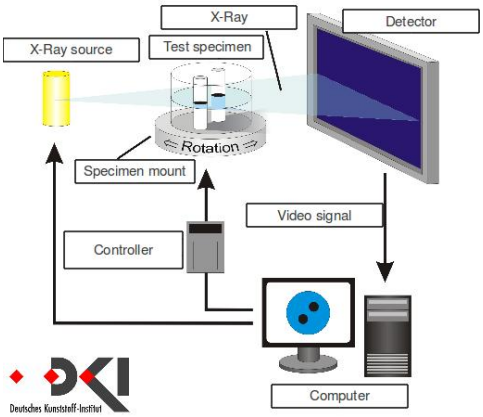

Figure 8: Schematic functional principle

Table 1: SkyScan 1072-100 properties

x-ray source	20kV - 100kV, 0 - 100μA
detector	1024×1024×12Bit CCD (cooled)
magnification	12× to 163×
best resolution	1.8μm
specimen mount	180°/360° turntable w. hight adjust
max. specimen size	40mm×40mm
scanning time	ca. 1h
reconstruction time	ca. 10min

3.2 Specimen Preparation

The specimen preparation is by far simpler than the process for optical analysis. To extract a Ø 4mm × 4mm sized specimen, a simple core hole drill is used. This drill can be positioned directly over the desired position of analysis and provides a clean cutting of the specimen. After preparing the specimen out of the part, the exact original position of the specimen is no longer obvious. This refers mainly to the rotation of the specimen. When the specimen is drilled out, the plain surface and round geometry makes it impossible to tell which direction the specimen had before. This circumstance is illustrated in figure 9. As the second order orientation tensor depends on the local coordinate system, a method to identify the original rotation of the specimen within the μCT was needed, thus enabling us to correlate the measurement directly with the simulated data. It became also apparent, that the z-position of the specimen can not be adjusted precisely enough trough the μCT controller. This led to the proposed painting and reorientation procedure, which can be seen on top of the specimen in figure 10.

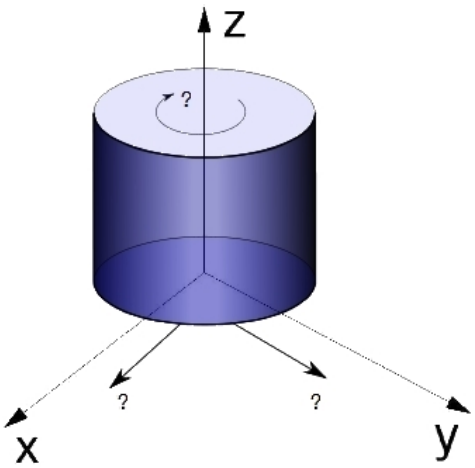

Figure 9: Unclear orientation of specimen

Figure 10: Ø 4 mm specimen on mount

The first step to prepare a specimen for fibre analysis with respect to the coordinate system of the injection moulding simulation, is to make the top of the specimen perfectly visible in the μCT. As the image quality of the μCT depends on density differences, a very dense paint is applied to the top of the reference part. This special paint absorbs most of the x-rays and produces a clearly visible black line in the μCT images. This line is set as the desired z-coordinate. To identify the rotation of z and the absolute position of x and y, paint flakes with a stochastic shape are applied on top of the fresh layer of dense paint. Thus the paint flakes become visible themselves in the μCT image. In figure 11 the dense paint is visible as the black band in the middle of the figure. The structures on top of the dense paint are the paint flakes, which are also visible.

The stochastic nature of the paint flakes is used to identify the μCT image in an original photo of the reference part. The method of reorientation and positioning is directly coupled with the single fibre analysis and returns the second order orientation tensor with respect to an arbitrary coordinate system (e.g. coordinate system of injection moulding simulation). Figure 12 shows the work flow necessary to reorient and position the μCT images in the photo of the original reference part. First (position 1.) the reference part, which is already processed with the dense

Figure 11: μCT picture, dense paint and paint flakes clearly visible

paint and the paint flakes, is either photographed or scanned. On position 2, the μCT images are added by image fusion to receive a summed picture of the paint flakes, which equals the picture from position 1. To shorten calculation time, the summed up pictures are then searched in a picture of the specimen (position 3), which itself is then searched in the picture from position 1. This procedure allows a very precise positioning of the μCT images in the picture of the reference part and makes it possible to calculate the second order orientation tensor with respect to the coordinate system from injection moulding simulations.

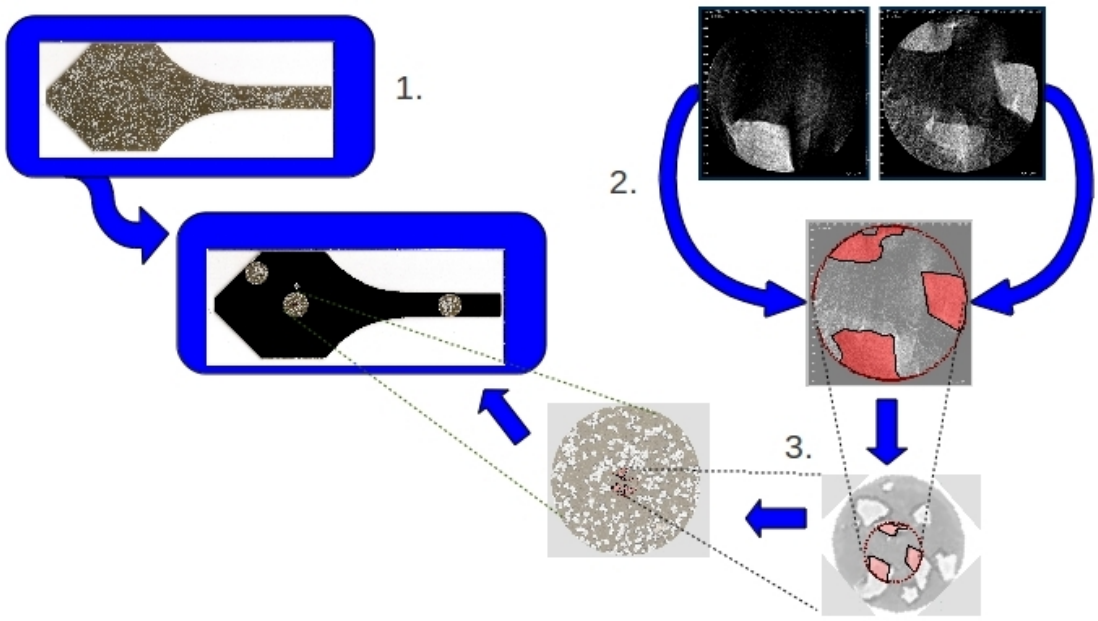

Figure 12: Positioning and reorientation using special paint

3.3 Single Fibre Analysis

The aim of the model-based fiber detection, proceeding from a three-dimensional density distribution information, is to characterize each fibre of a sample with regard to position, length and orientation in order to obtain accurate information about the fibre structure of the sample. The heuristics of the process are derived from assumptions about geometry and symmetry properties of the fibres: constant diameter and small curvature without turning points. The quality of μCT-based density distribution information is fundamentally dependent on voxel size, fibre content of the sample and density ratio between matrix and fibre material. Figure 13 shows an example of an image area within which an assignment of voxels to individual fibres without model assumptions is impossible.

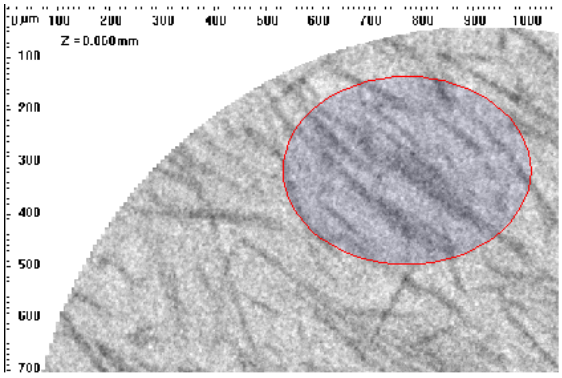

Figure 13: Problematic area for approaches without model assumptions

The approach developed at the DKI consists of four functional units (see figure 14). In the initialization phase, the raw data from the μCT are processed (filtered and binarized) and random numbers for the Monte-Carlo method are accumulated. After that, an iteration of parallel Monte-Carlo pattern recognition processes and merging of results (controller-process) is repeated until the recognition quality falls below a certain limit.

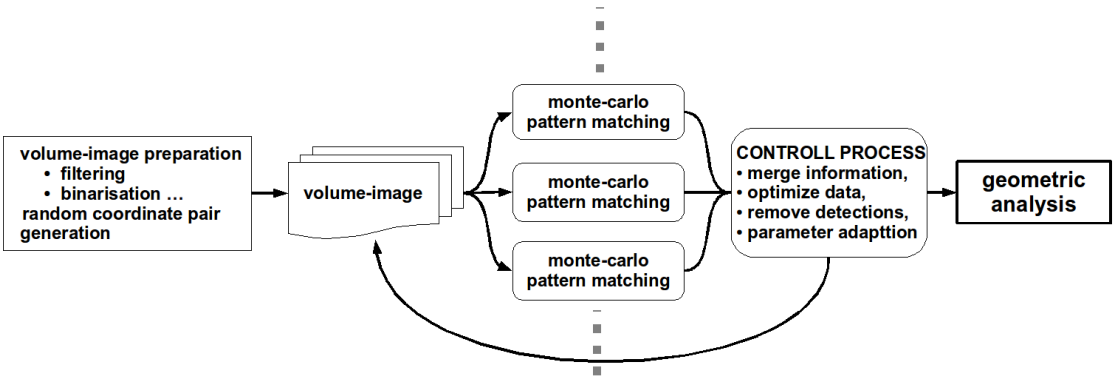

Figure 14: Flow chart of the new fibre analysis algorithm

Each of the parallel executed Monte-Carlo pattern recognition processes evaluates spherical integrals at random positions ($r_{ball} \gg r_{fibre}$) to identify centers of separate fibre pieces. At these

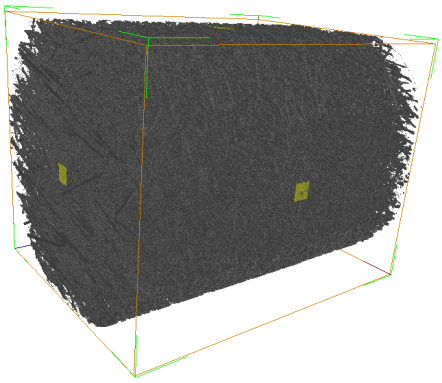

Figure 15: 3D scan results of PA6GF30 (some matrix removed for visibility)

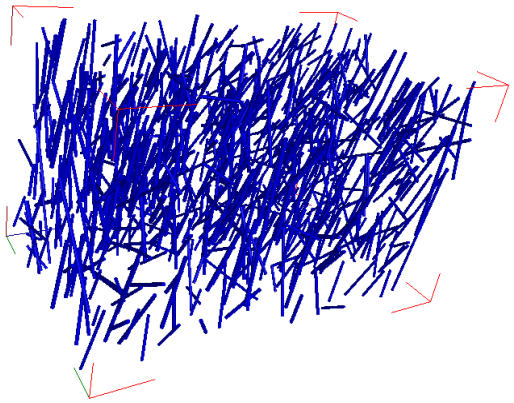

Figure 16: Detected fibres in PA6GF30 (only 10% shown)

centers main eigenvalues of the second order moments are used to determine first approximations for the orientation n of the fibre. Then cylinder integrals for different directions $n^* = \pm n \pm \varepsilon$ are calculated to determine exact orientations and length. This information is collected and sent to the control process. The control process recognizes detected fibers (duplicates), optimizes detection data, adjusts the parameters of the parallel Monte-Carlo pattern recognition and deletes voxel of well detected fibers from the volume image so that the pattern recognition only uses the residual image of yet not identified fibers. The control process evaluates the detection accuracy of the last iteration and eventually terminates the iteration. Upon completion of the iteration, the fiber vector data is analyzed geometrically (local distribution, local distribution of lengths, local alignment tensors, local longitudinal and transverse distances).

Figure 15 and 16 show a 3D density distribution and a fraction of 10% of identified fibres from the same volume. In figure 17 detected fibres are overlaid with the original μCT data.

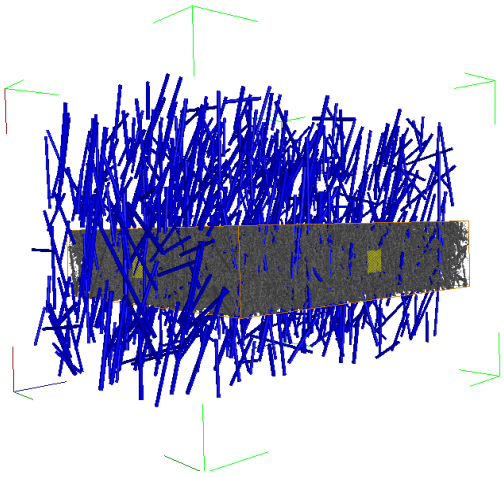

Figure 17: 3D scan results of PA6GF30 overlaid with detected fibres

4 Results

In this chapter the measured results from a Polyamide 6 with 30% short glass fibres will be presented. Volumes in which detected fibres where evaluated as second order orientation tensor equal the precise position of nine finite elements from an injection moulding simulation in which the filling and fibre orientation of the reference part was simulated. These elements build a stack through the part thickness. The fibre orientation was analyzed at two positions in the reference part.

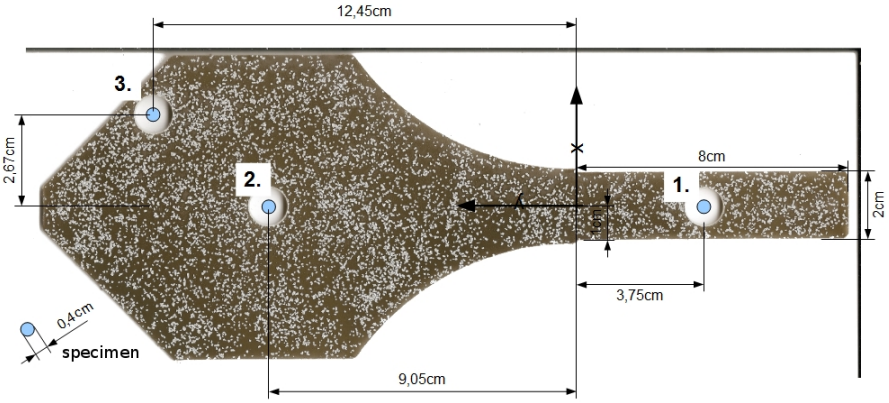

Figure 18: Specimen positions 1,2 and 3 in the part

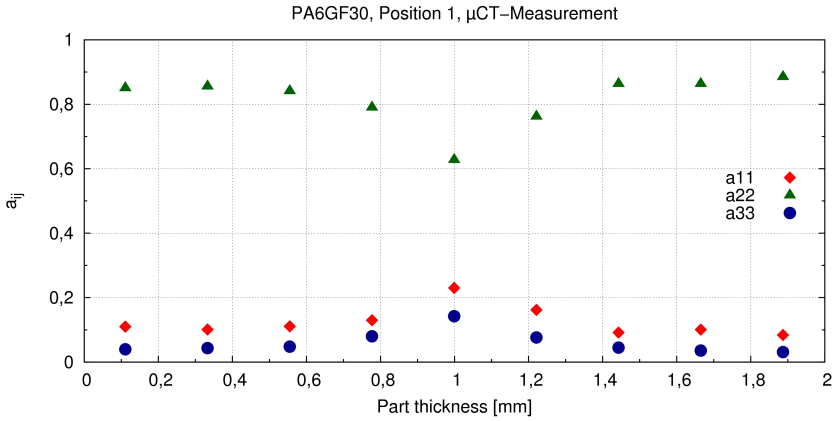

Figure 19: Analysis results Position 1

Position one is the area where the standard test specimen for uniaxial tension test is extracted [10]. The second position is shell like with a thickness of 2.5 mm (see figure 12). The y-direction corresponds with the flow direction of the polymer. Therefore a high value in the tensor component a_{22} represents a high fibre orientation in direction of the polymer flow. With $a_{22} = 1$ the material would be transversely isotropic with the y-plane as the isotropic plane [19, 20].

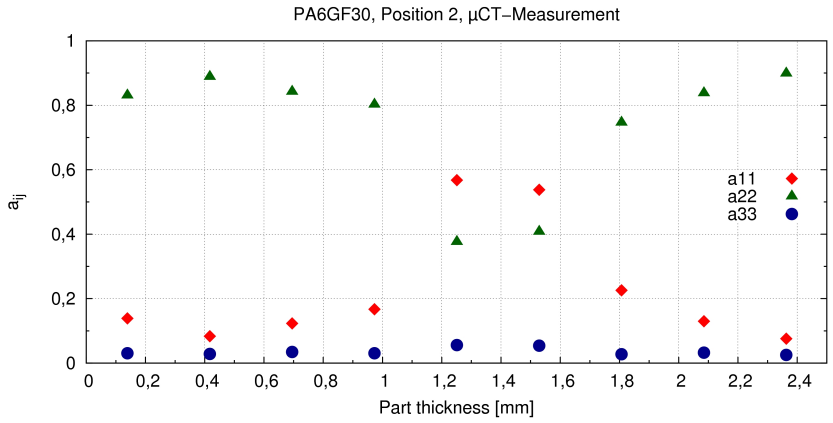

Figure 20: Analysis results Position 2

Figures 19 and 20 show the analysis results. The figures show on the axis of abscissa the part thickness and on the axis of ordinates the tensor components a_{ij}. Both figures show a high orientation of fibres in flow direction of the polymer at the top and at the bottom of the reference part. The boundary layers are as expected, oriented in the flow direction of the polymer. Figure 19 shows a very slight decrease of a_{22} in the middle of the part thickness. Contrary to this are the results in figure 20. Here a decrease of a_{22} and an increase of a_{11} in the middle of the part clearly show a core layer where the fibres are oriented perpendicular to the flow direction.

5 Summary and Outlook

Analysis of fibre orientations in reinforced thermoplastic parts is of immense importance for a light weight construction, because mechanical properties depend highly on this parameter. To take fibre orientations into account during a finite element analysis, the injection moulding process must be simulated. As the fibre orientation, which can be calculated during a injection moulding simulation, is of such importance, a validation is necessary. The state of the art method is complex, inaccurate and depends on the skill of the operator. Furthermore a second experiment is needed to validate the fibre length distribution.

This paper shows, that there is a powerful new alternative to existing methods of fibre orientation analysis. The algorithm and method developed at the DKI allows a fast and accurate analysis of fibre orientation, fibre length distribution and volume fraction with a single μCT measurement. Because of the mathematical representation of each single fibre, the second order orientation tensor can be evaluated in arbitrary subvolumes of the measured volume. This allows direct comparison of injection moulding simulation results and measurements. The whole preparation process is independent from the operators skill and takes much less time than the preparation for the optical method. This assures a constantly high level of measurement quality.

The next steps are to integrate all developed software into a graphical user interface and to fine tune the manual work process.

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