On the Generation of Highly Oriented Test-Specimens and the Influence of Preparation

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DOI: 10.5185/amp.2023.5587.1005

In the context of this contribution, a new injection molded unidirectional plate for specimen-extraction is introduced, and current investigations on the analysis of the sample preparation process are discussed. The injection molding (IM) manufacturing process leads to fiber orientation distributions, resulting in an anisotropic material behavior, which complicates the reliable design of components. The detailed determination of macroscopic, orientation dependent material data is a continuous subject of research. In this context, samples with a high fiber orientation distribution are required. Established and available methods for specimens generated by IM show limitations. In this work, a novel test-specimen with a homogeneously high fiber orientation is introduced. It enables the extraction of specimens under different angles with respect to the main fiber orientation. The fiber orientation simulation of the novel plate is compared to results from micro computed tomography. Furthermore, a detailed investigation of the influences of the milling process for sample extraction on the final specimen behavior is presented. It includes a qualitative characterization of roughness values and a correlation between these and the experimentally acquired mechanical parameters.

Introduction

Injection molding of plastic parts is a cost and time efficient process for mass production. To modify the physical properties such as stiffness and strength, often short fibers of length up to 1 mm are added to the processed polymers. These are then denoted as short fiber reinforced thermoplastics (SFRTP). The achievable strength-to-weight ratio is profitable for lightweight designs. Combining the properties of both, fibers, and matrix material, allows tailoring and optimization of material properties.

Injection molded parts of SFRTP show locally inhomogeneous fiber orientations (FO) due to rheological phenomena [**1**]. The anisotropic (direction dependent) mechanical material behavior of SFRTP complicates the computational design of components. In case of planar geometries, the fibers are generally oriented multi-directionally (MD) [**2**]. Karger-Kocsis et al. described the basic FO distribution in thickness direction as consisting of three main layers [**3**]. Each main layer has its own FO and consequently specific material properties. The edge layers show a high FO in injection direction, while in the core layer an orthogonal FO dominates. Hence, a tensile test with specimens made from MD plates will provide information on the convoluted mechanical behavior of all three layers [**4**]. However, a highly (unidirectionally) oriented plate would be ideal to provide a direct correlation of the mechanical properties and the underlying FO.

Experiments with a defined FO are essential to gain material data for structural simulations. To improve the quality of design, high quality data based on reliable and comprehensive mechanical material tests are needed. Mechanical tests are also more time- and money-intensive than simulations. It is not possible to fully replace mechanical tests by virtual simulation, but the number of tests can be dramatically reduced, especially if high quality data are available. So mechanical tests will be needed even if the quality of the mechanical simulations will be better in the future.

Current approaches for the processing of highly oriented specimens lack an adequate possibility for the testing of cross flow material properties. E.g., DIN EN ISO 527-2 introduces the specimen geometry A1 which possess a high FO. Also, at the Deutsches Kunststoff Institut DKI studies were performed regarding highly oriented specimens and injection molded plates with a highly oriented area to extract specimens [**5**]. All of them have one crucial aspect in common: their width is in comparison to other plates relatively small. Those UD-plates lead to an orthogonal specimen´s length of 20 mm, which makes transverse tensile testing impossible. Hence a novel approach is presented in this work.

Regarding the manufacturing process and the FO dependency, a crucial difference is that real components are directly injection molded, whereas test specimens are extracted via machining from injection molded plates. This is required to resolve anisotropic effects in different orientations. Mechanical machining is a discontinuously cutting process and typically induces surface damage, which may be addressed and quantified by roughness measurement.

Research work on the impact of the manufacturing process of specimens on the mechanical behavior of SFRTP is scarce, and guidelines are unspecific and vague. Eriksen studied short glass fiber reinforced thermoplastics and found, that theory and experience from other materials cannot be directly applied [**6**]. Eriksen calls for new guidelines to ensure a satisfactory result from machining [**7**].

In existing guidelines on mechanical testing, it is specified that test specimens made by machining must be manufactured very carefully. DIN EN ISO 2818 - 2019 Plastics Preparation of Test Specimens by Machining, as well as ASTM Standard D638 - 2014 Standard Test Method for Tensile Properties of Plastics, point out, that a material cannot be tested without validating the test-method itself. Regarding the surface condition both standards state that the specimen´s surface must be free of visible flaws, scratches or imperfections when viewed at low magnification. Due to these unquantified statements, it depends on the observer´s point of view, whether imperfections are noticed, and specimens are excluded or not. Both standards fail to present a measurable and quantifiable criterion for the status of the surface quality. However, the surface quality may have a significant influence on the material properties. Hence, investigations to get a deeper understanding regarding the surface quality are required.

To fill this gap of knowledge, the investigations presented here focus on the preparation of test specimens with a high fiber orientation. A novel injection molded plate with a predefined FO is introduced. It enables the extraction of specimens with unidirectional fiber orientations. Tensile tests at room temperature and -10 °C are evaluated. Finally, the impact of the surface roughness of these specimens on their mechanical properties is discussed. .

Experimental setup

Material

This research focusses on a representative short glass fiber reinforced thermoplastic material. Polybutylene terephthalate reinforced with 30 weight percentage short glass fibers (PBT GF 30) was chosen, as it is commonly found in automobile applications. Its trade name is BASF Ultradur® B4300 G6. The material is semicrystalline.

Development of plates with unidirectionally oriented fiber orientation

Out of the need for a highly oriented specimen that allows for the extraction of specimens in different directions, we designed an innovative injection molding tool with the DKI plate [**5**] serving as basis for the new design. The main requirements for the optimization of the geometry where set as follows:

- adequate fiber orientation: first principal component of the second order FO tensor should be larger than 0.75 (currently perceived as highly oriented)
  - o homogeneous FO across the entire thickness
  - o homogeneous FO in the testing area
  - o no multi directionality
  - o no core layer
- sufficient width to extract specimens
  - o possibility of extraction under arbitrary angles
  - o width should be as large as possible.

From a processing point of view the boundary conditions for the geometry were given by the pressure constraints of the available injection molding machine with a shadow image lower than 12 000 mm² and a closing force of maximum 800 kN.

Injection molding of the plates

Before using the granulate for injection molding, it is pre-dried. The temperature of the mold is set to 80 °C, the melt has a temperature of 260 °C and is injected within 1.3 s with an injection flow of 45 ccm/s. After injection a pressure of 300 bar is held for 25 s, cooling time takes further 25 s. From the molded plates, the inlet was cut off and the testing areas of the UD plates were stored at 23 °C in vacuumed bags until milling.

Specimen geometry

In **Fig. 1**, the dimensions of the applied BZ6 geometry are shown. It is based on the BZ12 geometry with a parallel evaluation area of 12 x 12 mm², introduced by Becker in [**8**] for the mechanical testing of polymers. BZ means Becker-Zugstab (German for tension rod). The BZ12 is applied in numerous research on short glass fiber reinforce polymers, see e.g. [**9–11**]. The BZ6 is a scaled version with half the size, having an evaluation area of 6 x 6 mm². The small sized specimen was chosen to allow the extraction of specimens in different angles from the plates injection molded in this study.

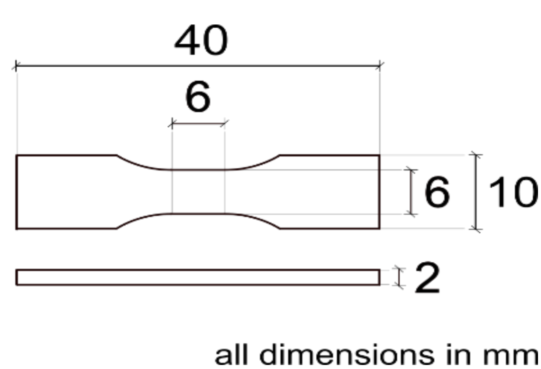


Fig. 1. Geometry of test specimen BZ6 for tensile tests, which is milled out of unidirectionally orientated plates which initially measure 40 x 80 mm².

All specimens were cut from unidirectional (UD) plates under different extraction angles with respect to the longitudinal axis of the plates which corresponds to the flow direction of the polymer melt. The UD-plate is shown in **Fig. 2**.

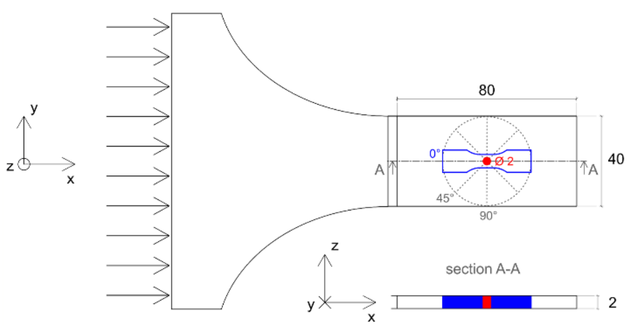


Fig. 2. Geometry of the novel plate to extract BZ6 specimens (blue) for tensile tests. Centric position of microcomputer tomography specimen (red).

Manufacturing of Specimens through Milling

A study with differing feed rates and rotational speeds was performed. The chosen parameters for two specifications are summarized in **Table 1**. After milling, the BZ6 specimens were again sealed in vacuumed bags and stored at 23 °C until tensile testing.

Table 1. milling parameters used for manufacturing of BZ6 specimens.

specification	feed rate mm min^{-1}	rotational speed mm^{-1}
mod0	500	6500
mod1	1250	4000

Analysis of Surface Roughness

To calculate the roughness values, topography studies are essential. The surface roughness is obtained with a triangulation method. The sample roughness is deduced from 2D-scans with a scan length of 6 mm and the measurements for this work are recorded with a profilometer FTR MicroProf CWL 600. The scanning direction is longitudinal on the machined surface. The scan position was set to one third of the thickness to avoid possible inhomogeneities in the core, such as potential core layers with transversely oriented fibers. To avoid possible edge effects, special care was taken in order to ensure that determined scan position was not too close to the edge. For the UD plates, preliminary studies showed that the roughness profile over the thickness is homogeneous.
The average surface roughness R_a was determined by averaging the height profile z along the full scan length L, which is 6 mm in this case. It can be computed by

$$\mathrm{R_a} = \frac{1}{\mathrm{L}} \int_0^{\mathrm{L}} \mathrm{z}\,(\mathrm{x})\mathrm{dx}\,.$$

The total height of the roughness profile is defined as roughness depth R_t. It represents the difference of the highest peak and the deepest valley within the full scan length and is described by

$$\mathrm{R_t} = z_{max} - z_{min}\,.$$

Local effects cannot be adequately resolved by the average roughness, whereas the roughness depth only depicts the maximum local peaks. Hence, both roughness values are included in the discussion of results.

Mechanical tests

All mechanical tests were performed at standard climate conditions of 23 °C and 50 % relative humidity. Right before testing, the specimen is extracted from the vacuumed bag. Its dimensions (width and thickness) are quantified in the evaluation area, onto which a random gray scale pattern is spray painted. The color is a black water-based primer coating with white spray paint and it´s thickness is 10-20 µm. This pattern is mandatory for the optical gray scale correlation of strains, which was performed with VIC2D from Correlated Solutions®. The strain correlation is based on the evaluation of subsequent deformation images of a gray scale pattern on the specimen. Images are recorded with 10 frames per second to determine characteristics such as break. To compare the mod0 and mod1 specimens, the following mechanical quantities are considered: true stress and Hencky strain.

The quasistatic tensile tests were executed on a servo-hydraulic Zwick Roell testing machine equipped with a 20 kN load cell at a constant velocity of 0.5 mm/min.

The average logarithmic Hencky strain values were extracted from the strain field in the evaluation area of 6 mm x 6 mm on the specimen surface. This averaged evaluation method of the strain field does not precisely resolve local effects such as failure. Alternatively, maximum strains could be evaluated, however, these would only determine effects on the specimen surface, which do not necessarily correspond to a possible crack initiation on the back side or inside of the specimens. Alternative methods to evaluate failure strains and a detailed account on the homogeneity of the strain distribution on the specimens should be considered in future. True stress values were calculated with force data from the load cell and the actual cross section of the specimen.

Results and discussion

Main results of these investigations are the mechanical response of the tensile tests. Prior to the mechanical analysis, fiber orientation analysis and topography measurements were performed.

Fiber orientation

The fiber orientation of the novel unidirectionally orientated (UD) plate is validated with PBT GF 30 and compared with a classical testing plate with multidirectional (MD) fiber orientation. As shown in **Fig. 3**, the FO of the UD plate is homogeneous over the entire thickness of the plates, whereas the MD plate shows a great decrease in FO in the core layer.

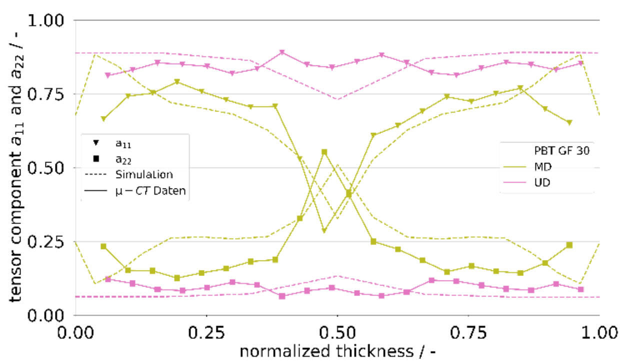
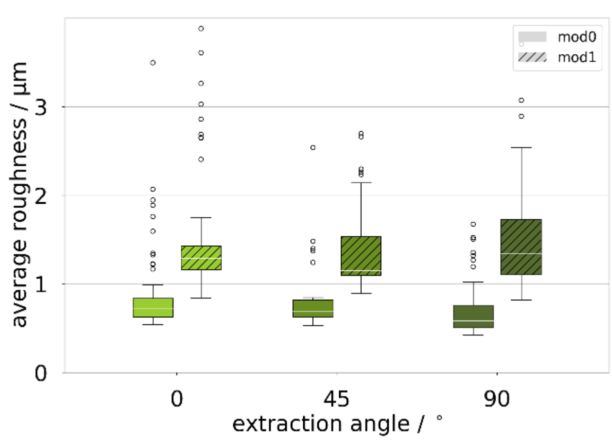


Fig. 3. Comparison of fiber orientation distributions.

Topography

The calculated roughness values for the average roughness and roughness depth are shown in **Fig. 4**. Values seem to be independent of the extraction angle. For a 90° extraction angle, the scattering is higher, which may be induced from the present fiber orientation.



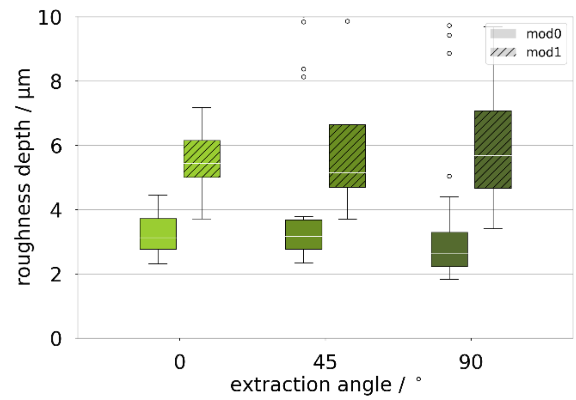
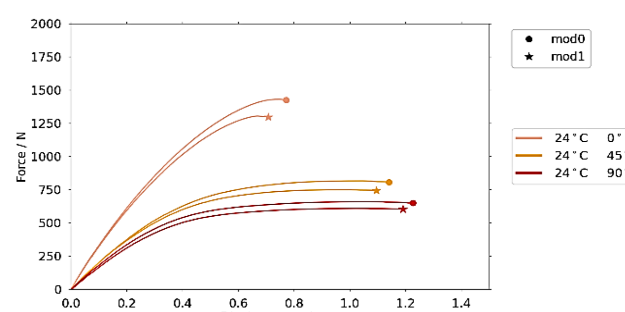


Fig. 4. average roughness and roughness depth for the milled surface of BZ6 test specimens.

Mechanical tests

Considering the force signal, shown in **Fig. 5 top**, the rougher specimens (mod1?) can take less force than the smoother ones (mod0?). This results from the smaller cross section of the rougher specimens, which measures 10.81 mm² whereas the smooth specimens´ cross section measures 11.75 mm².

If true stress and Hencky strain are compared, no differences in the stress levels are seen (**Fig. 5 down**). The dependency of mechanical properties on the extraction angle is evident. A 0° extraction angle leads to a fiber orientation aligned with the load direction in the tensile test and fibers are able to absorb most of the load. In contrast, at a 90° extraction angle the fibers are perpendicularly aligned with the load direction and the matrix material absorbs most of the load. Failure occurs at the same stress levels for rough and smooth surfaces.



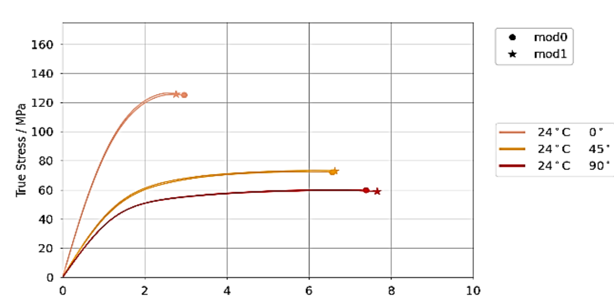


Fig. 5. force – displacement – diagram and true stress – strain – diagram of smooth (mod0) and rough (mod1) specimens at room temperature for different extraction angles.

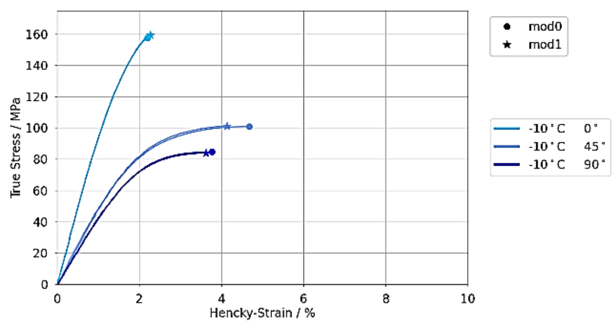


Fig. 6. true stress – strain – diagram of smooth (mod0) and rough (mod1) specimens at -10 °C for different extraction angles.

Under the assumption that more brittle materials would show a higher impact of roughness, the experiments are repeated at a lower temperature of -10 °C. With cooler testing temperature, the material behavior is stiffer and more brittle, as displayed in **Fig. 6**. Because of shear effects, the failure strain of specimens extracted at 45° is higher than that of specimens extracted at 90°. However, a clear influence of roughness impact is still not visible in the mean curves.

To get a better comparison of the failure points, the single measurements are considered and shown in **Fig. 7**. Influences of the surface roughness can be seen in scattering of the failure strain: at a 90° extraction angel, the scattering of the failure strain is wider that at a 0° extraction angle. Moreover, the width of the scatter band does not depend on the test temperature.

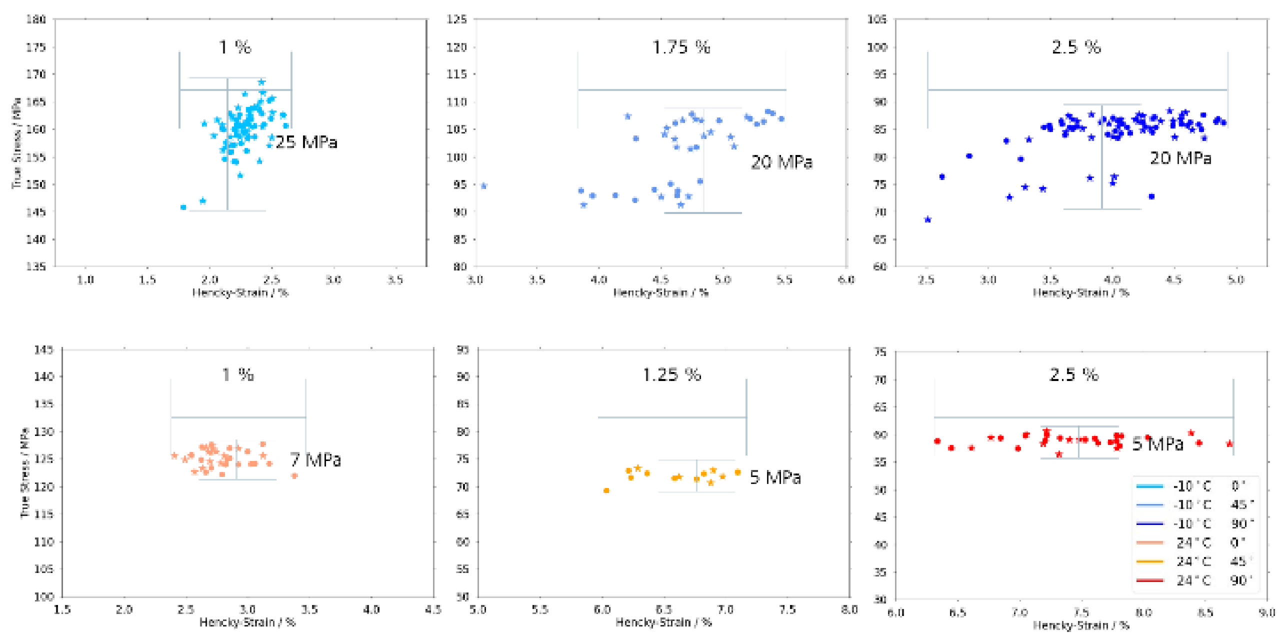


Fig. 7. True failure stress – failure strain – diagram of smooth (mod0) and rough (mod1) specimens at -10 °C and 24 °C for different extraction angles with indicated scatter band.

Conclusion

The present results show that the concept of strongly oriented plate was successfully implemented for the considered material. Fiber orientation analysis with micro-computed tomography shows that the fibers are homogeneously aligned over the entire thickness. Future studies will evaluated whether other reinforced materials show the same fiber orientation behavior.

Two different roughness levels of samples where achieved through different milling parameter sets. The average roughness and roughness depth seem to be independent of the extraction angle of the specimens. The scattering of the roughness values is slightly higher at an extraction angle of 90° as at 0° or 45°.

Mechanical tests were done with BZ6 geometries at room temperature (23 °C) and -10 °C. Material behavior is stiffer at low temperature. However, the scattering doesn't show any difference for the two temperatures.

Based on this database, no agglomerations of failure points regarding their roughness level can be stated. An increase of the extraction angle from 0° to 45° and 90° leads to a wider scatter of the failure strain, whereas the scattering of failure stress remains the same. An decrease of the test temperature from 23 °C to -10 °C leads to a wider scatter of the failure stress whereas the scattering of failure strain remains unchanged. Overall, the scattering of failure points regarding the stress seems to increase with a decreasing test temperature. The scattering of failure points regarding the strain seems to change in dependency of the extraction angle. However, regarding the number of tested specimens no significant differences of the averaged mechanical properties could be found for the different roughness levels.

It can be concluded that no dependency of roughness on mechanical values is present within the introduced test setup. Consequently, the impact of the surface roughness induced by milling is negligible for the presented tests. Hence the milling is a suitable process for the generation of specimens for mechanical testing.

Additional work

Deeper insight on this topic and further work can be found in the Journal of Reinforced Plastics and Composites 41 and the authors PhD dissertation.

Article: "On short glass fiber reinforced thermoplastics with high fiber orientation and the influence of surface roughness on mechanical parameters" by T. van Roo, S. Kolling, F.B. Dillenberger, J. Amberg, 2021.
Dissertation: „Einfluss der Oberflächenrauigkeit auf die mechanischen Eigenschaften hochorientierter kurzglasfaserverstärkter thermoplastischer Polymere" by T. van Roo which will be published by the end of 2023.

Conflicts of interest
There are no conflicts to declare.

Keywords

Fiber orientation, mechanical testing, fiber reinforced polymers

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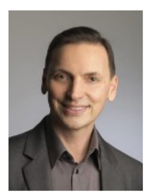
Authors biography

Tamara van Roo is a Mechanical Engineer and graduating for her PhD. Since 2017 she is both, a research assistant in the Group Mechanics and Simulation at Fraunhofer-Institute for Structural Durability and System Reliability and doctoral student at Graduate Centre for Engineering Sciences at the Research Campus of Central Hessen. In 2021 She was at Centre de mise en forme des matériaux, France for a research stay. There she was able to perform further experiments for her PhD thesis, which are discussed in this contribution. Her Expertiese is mechanical characterization of fiber filled plastic components.

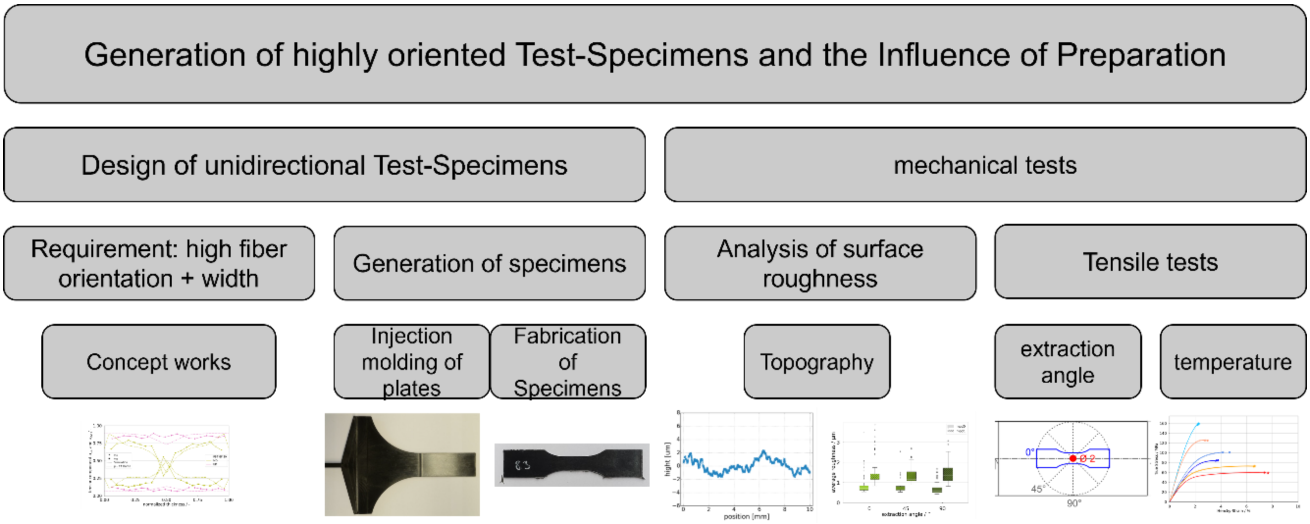


Stefan Kolling studied civil engineering at the Saarland University of Applied Sciences, Germany and mechanics at the Technical University of Darmstadt, Germany. From 2002 to 2008 he was an Engineer for vehicle development at Daimler in Sindelfingen, Germany and focused on crash simulation and development of material models for glass and plastics. Since 2008 he is Professor for mechanics at THM University of Applied Sciences in Giessen/Germany.

Felix Dillenberger studied mechanical and process engineering at Technical University of Darmstadt, Germany. From 2012 to 2018 he was research assistant in the Group Mechanics and Simulation in the Division Plastics, Fraunhofer Institute for Structural Durability and System Reliability LBF, Darmstadt, Germany. In 2018 he became manager of the Group Mechanics and Simulation and in 2023 he became deputy head of the Department Plastics Processing and Component Design. His fields of expertiese are mechanical characterization of plastics and composites, structural simulation of plastic components and material modelling.

Graphical abstract

To get information about the influence of preparation, specifically the surface roughness, on the mechanical behavior of short glass fiber reinforced thermoplastics is analyzed. A novel injection molding tool is introduced to generate highly orientated test specimens. The specimen´s surface roughness is analyzed. Mechanical tests are performed at different temperatures and extraction angles, the scattering of the values is discussed.


Generation of highly oriented Test-Specimens and the Influence of Preparation
Design of unidirectional Test-Specimens
mechanical tests
Requirement: high fiber orientation + width
Generation of specimens
Analysis of surface roughness
Tensile tests
Concept works
Injection molding of plates
Fabrication of Specimens
Topography
extraction angle
temperature
position [mm]
0°
45°
90°