Mechanical Characterization of Talc Particle Filled Thermoplastics

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Summary:

In this paper, we present the experimental part of the development for an integrative simulation with Moldex3D and LS-DYNA for talc particle filled polypropylene.

The properties of thermoplastic polymers considerably depend on the process of moulding irrespective of the geometry of the part and the raw polymer used. Both, the polymer structure resulting from moulding and formation of weld lines may show a large influence on mechanical properties.

For analysing the influence of injection moulding on the resulting structural part properties, plates with different processing conditions were fabricated with Hostacom XBR169G, a polypropylene (PP) filled with rubber and talc. Afterwards, different test samples, longitudinal and lateral to flow direction, were milled out from the fabricated plates. Using this extracted test samples, the true deformation and failure behaviour were measured by uniaxial tensile tests and shear tests at small and high strain rates. The complex deformation behaviour was determined by grey-scale correlation strain measurement.

The analysis of quasistatic uniaxial tensile tests shows anisotropic effects depending on the preparation direction of the specimen in stress behaviour as well as in Poisson's ratio. This also can be seen at different testing temperatures and at high strain rates. The reason of the anisotropic effects might be an orientation of the talc particles. In all experiments, the strain at failure is not influenced by testing direction. The measured data provide the basis for a material model, which is implemented in LS-DYNA. The model is validated by a three-point bending test.

Keywords:

anisotropic material properties, injection moulding, input data for material models

1 Introduction

The application of plastic materials in technical fields is becoming more and more important. Therefore modelling and numerical simulations, in particular for safety-related components, become highly relevant. Actually car door panels made of PP are considered in the dimensioning of a car, to fulfil the requirements of the Euro NCAP crash test.

The deformation and failure behaviour of polymers is affected by its structure of long-chain, entangled or networked macromolecules [1, 2, 3]. In typical production processes as injection moulding, the microstructure of a plastic material is subjected to major changes. Mechanical properties like yield stress and strain at failure of the same polymer can fluctuate considerably depending on the production history. This is also well known for fibre reinforced polymers, where, amongst others, the fountain flow during filling causes an orientation of the fibres. Nearby, a surface layer and an intermediate layer is generated [4, 5]. The fibres in the surface layer are oriented parallel, whereas the fibres in the intermediate layer are orientated perpendicular to flow direction. This leads to an anisotropic deformation behaviour [6, 7].

Furthermore, the flow of polymer melts in the filling process can cause an orientation and a stretching of the macromolecules and may change the phase morphology. In recent years, fillers like talc or rubber are used in parts made from polypropylene. In contrast to fibre reinforced polymers the talc or rubber filled polymers are naturally counted like the unfilled polymers to the class of isotropic materials. But recent investigations show at least some anisotropic orientation of talc filled polypropylene [8].

For an accurate finite element modelling, the knowledge of the anisotropic deformation behaviour of filled polypropylenes is therefore important. Without these considerations uncertainties are involved and it's necessary to make high factors of safety in a constructional element. For a better material utilization and a reliable design of components, it is therefore of great importance to implicate the impact of process conditions taking effect on the material at a certain position of a part, to understand its mechanical properties and incorporated those in the material description.

Finally, the aim of the presented work is the development of an integrative simulation, see [9, 10]. Injection moulding simulation can calculate the local orientation of talc particles in the sample. This could be transferred to the finite element analysis to affect the inhomogeneities like anisotropy in the simulation. The experiments shown in this paper are the necessary part to calibrate the finite element simulation in order to develop an accurate integrative simulation for the field of polypropylenes filled with rubber and talc.

2 Experimental Methodolgy

In the following section, the preparation of the samples and the following mechanical tests like tensile or shear tests are described in detail.

2.1 Preparation of the Sample

First, plates made of Hostacom XBR169G (producer: LyondellBasell), a polypropylene filled with talc and rubber, were produced on the injection moulding machine FX-75 (producer: Klöckner-Desma Ferromatik). The size of the plates was 80 mm x 80 mm x 2,5 mm (see figure 1), the triangular gate ensures a parallel flow front in the plate. The process parameters of injection moulding are shown in table 1.

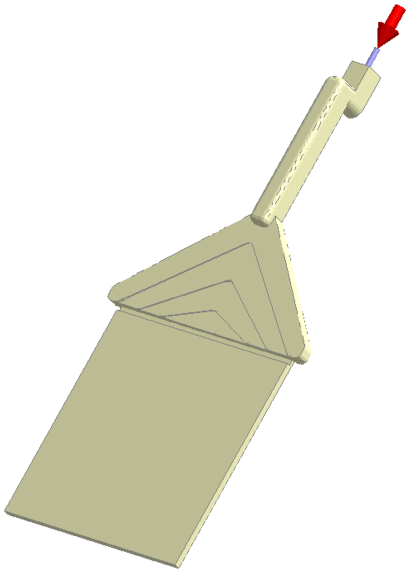

Figure 1: Injection moulded plate with delta gate

2.2 Sample Geometry

Proper material data from tensile, shear and bending tests are important to consider the deformation behaviour of filled polypropylene as precisely as possible in the FE analysis. Therefore, prior to the tests the focus was set on the selection of suitable samples that could be milled of the prepared plates.

The selection of the sample's geometry for tensile tests is described in detail in [11] and [12]. The development of the tensile bar and the geometry for shear test can be found the work of Becker [13]. Becker's tensile bar for tensile tests and the z-sample for shear tests are used both for quasi-static and dynamic tests. The three-point bending tests are carried out on bars, where the width remains constant over bar length, i. e. not tailed. All three geometries can be easily milled out of the injection moulded plates. The exact geometry of the samples shows figure 2: on the left hand side, the geometry for tensile tests is shown. In the middle, the shear geometry can be found, whereas the right image shows the geometry of the bending samples.

To determine the effect of anisotropy of the sample on the deformation behaviour, the test

Table 1: Process parameters of Hostacom XBR169G

melt temperature	[°C]	240
mould temperature	[°C]	40
injection time	[s]	4
holding pressure	[MPa]	40
holding time	[s]	30
cooling time	[s]	20

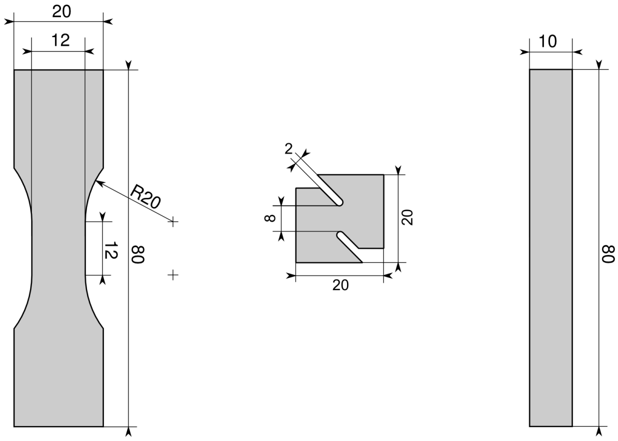

Figure 2: Different sample geometries

specimens are taken longitudinal and lateral to flow direction. The scheme of the withdrawal position is shown in figure 3. Analogous to the tensile specimens, shear and bending samples are milled out at the same positions.

2.3 Mechanical Characterization

In the analysis of the crash behaviour of plastic materials, high strain rates should be realized in the testing region. Neglecting the effects described above, the resulting strain rate depends on the haul-off speed and the geometry of the test specimen. Both parameters can not be changed arbitrarily. Machines and measurement technology restrict the haul-off speed and the geometry of the specimen, which also limits the theoretically achievable strain rate.

The tests at constant or variable strain rate are executed with a high speed servo-hydraulic testing machine [14]. Using proper test specimens and testing devices, different stress states can be achieved. The dynamic uniaxial tensile tests were performed using the servohydraulic testing machine HTM5020 from Zwick (max. 20 m/s, 50 kN) at $T = 23\,°\mathrm{C}$. The quasi-static tesile, shear and bending tests were performed on the Zwick universal testing machine Z020 (max.

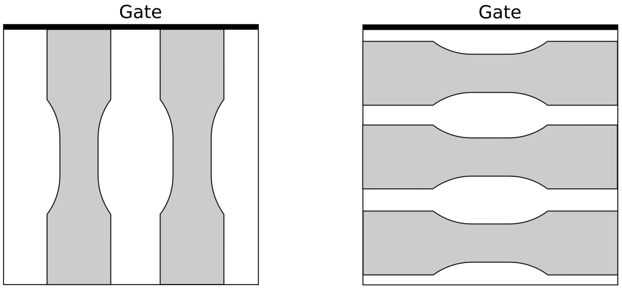

Figure 3: Withdrawal positions of samples

Figure 4: Tensile bar with grey scale pattern

750 mm/min, 20 kN) at $T = -35\,°\mathrm{C}$, $T = 23\,°\mathrm{C}$ and $T = 80\,°\mathrm{C}$. Three retries per test series were performed.

2.4 Strain Measurement

Effects, such as the necking of the tensile bar in the plastic region can lead to high local differences in the deformation of the sample. Therefore, a local strain measurement is required. The strain measurement via the crosshead displacement is not appropriate, since in this method an average strain value over the entire sample length is determined and local effects are neglected.

One method to measure the strain throughout the entire test locally is the grey-scale correlation [3, 14]. Thereby a stochastic grey-scale pattern is applied to the sample (see figure 4) and filmed with a digital camera during the test. After the test, displacements and distortions of the pattern based on the stored images are calculated using a cross correlation algorithm [15].

From the calculated displacements of the sample surface between two images, the strain on the entire surface can be calculated. After the image correlation, the local displacement and strain distribution on the sample surface for each captured image is available, as shown in figure 5.

Hence, local strains that occur during the test can be determined. The two-dimensional strain information can be converted by averaging a section in a scalar strain value for each time step. The length of the averaged section can be chosen either absolute (e. g. 5 mm) or relative (e. g. 95 % of the maximum strain.) In the results shown later, a strain section of 2 mm and the maximum strain in direction of loading were selected.

For the dynamic tests, the force signal is filtered, since the impact load created during the test by the design of the testing machine and the position of the load cell generates strong oscillations in the system. A detailed description of the filtering methods can be found in the work of Becker [13].

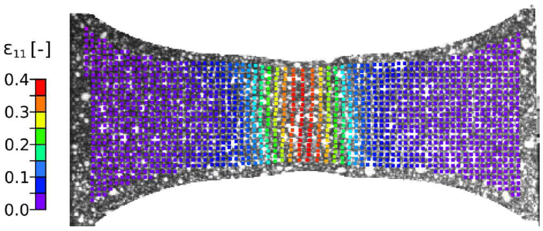

Figure 5: Correlated single picture including facets [13]

3 Test Results

In the following chapter the experimental results are presented. The influence of test temperature, strain rate and stress state to the deformation behaviour is displayed. The measured material data are the basis for a material model for crash simulations, which were presented by Hempel and Seelig in [16]. The validation of the model is performed via bending tests.

3.1 Influence of the Extraction Direction of the Sample

First, the dependence of test direction on the deformation behaviour of Hostacom XBR169G is shown. Figure 6 shows for both testing directions, longitudinal and lateral to flow direction, a small tendency for strain hardening of Hostacom XBR169G. In addition the material shows difference in the stress level of 20 % between samples taken longitudinal and lateral to flow direction. The differences in Poisson's ratio between these samples are at more than 60 % (see figure 7). Interesting in the results is the fact that the considered class of materials (polypropylene filled with talcum and rubber) usually is counted to the group of isotropic materials. In the simulation, therefore, usually no directional parameters have been taken into consideration.

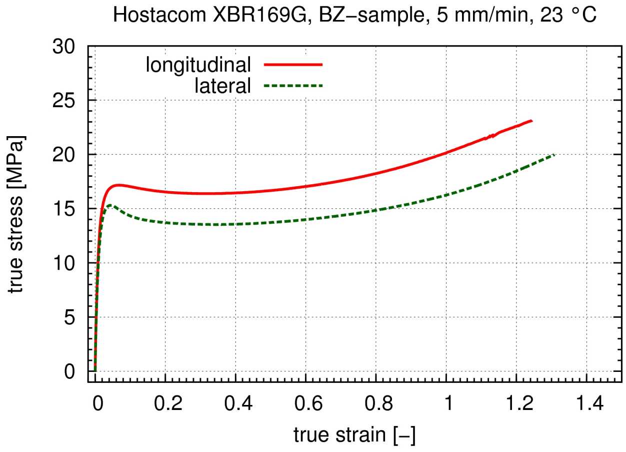

Figure 6: Stress-strain-behaviour of Hostacom XBR169G

The different degree of anisotropy in different polymers, can have several reasons. An orientation of the talc particles could occur in Hostacom XBR169G, which align themselves along a preferred direction in the filling process during injection moulding. This would result in a reinforcing effect, if the loading direction coincides to this preferred direction. Similarly an alignment of the EPDM would be possible; the rubber spheres could deform during the filling process by the high shear forces and also align along a preferred direction. To clarify this issue, images with a scanning electron microscope were made. In figure 8 the fracture surface of a tensile bar made of Hostacom XBR169G is shown, which was produced by cold shortness. As expected, a preferred orientation of the talc particles can be observed in the flow direction. Similar results are shown by Zhou and Mallick [17]: they present anisotropic effects which are created by injection moulding. The injection moulding process influences skin-core morphology exhibiting different orientation of talc particles in the skins than in the core.

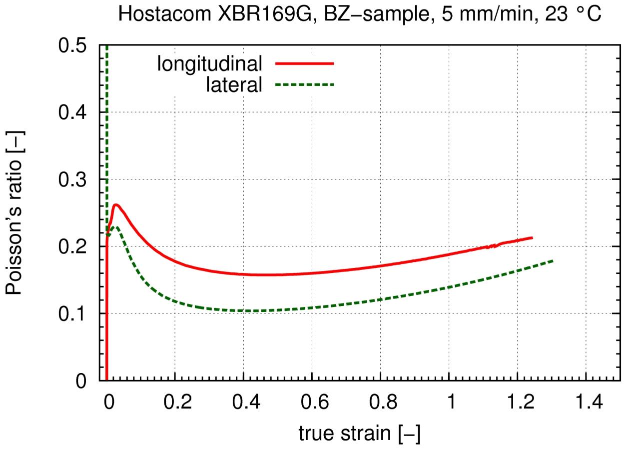

Figure 7: Poisson's ratio of Hostacom XBR169G

Besides the tensile tests also shear tests were performed. Figure 9 shows the shear stress as a function of shear strain for samples taken longitudinal and lateral to flow direction. Under shear load the talc and and rubber filled polypropylene shows no anisotropic effects. There is no difference in the shear stress level recognizable. It should be noted, that the end of the curves does not indicate the failure in shear. At this point, the distortions are too high. The evaluation of grey-scale correlation can not be made appropriate. Furthermore, at large deformations it is not a simple shear state any more.

3.2 Influence of Testing Temperature

Another important effect on the deformation and failure behaviour is the test temperature. These influences must also be properly considered in a material model. Relevant temperatures for the automotive sector are $-35\,^\circ\mathrm{C} < T < +80\,^\circ\mathrm{C}$. Therefore, quasi-static tensile tests were performed at the test temperatures $T = -35\,^\circ\mathrm{C}$, $T = 23\,^\circ\mathrm{C}$ and $T = 80\,^\circ\mathrm{C}$. The deformation behaviour of

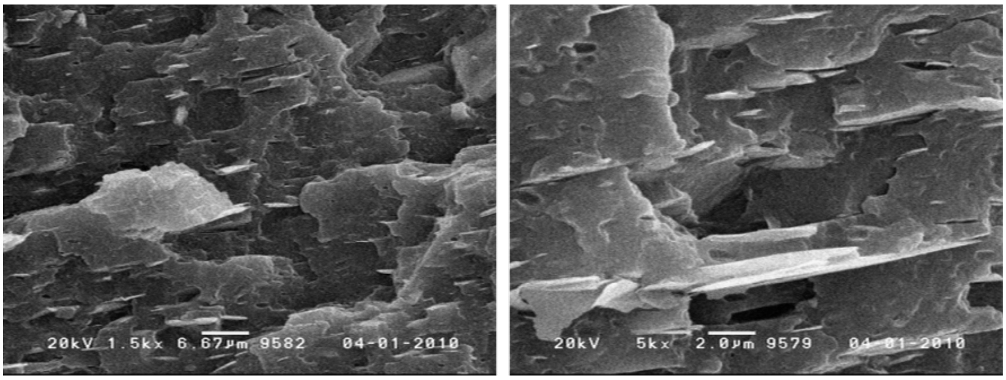

Figure 8: SEM pictures with magnification 1500 (left) and 5000 (right)

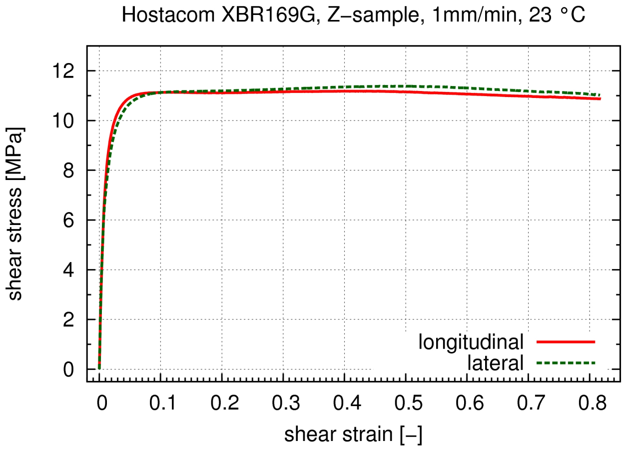

Figure 9: Shear behaviour of Hostacom XBR169G

Hostacom XBR169G is shown in figure 10.

For all temperatures, a significantly higher stress level occurs in samples taken longitudinal to the flow direction. The differences measured between $T = -35\,°C$ and $T = 23\,°C$ are at about 20 %, whereas the difference at $T = 80°C$ turns out lower, but the differences in stress level increase with ongoing strain.

3.3 Influence of Strain Rate

For a complete material data set, apart from the temperature dependency, the influence of the strain rate on the deformation behaviour plays also an important role. This is illustrated in

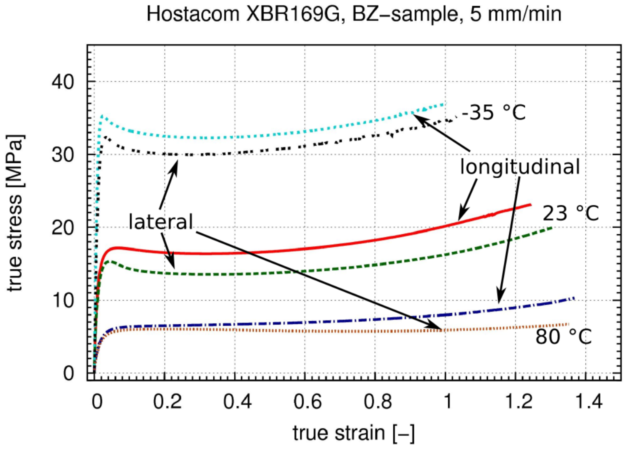

Figure 10: Influence of testing temperature of Hostacom XBR169G

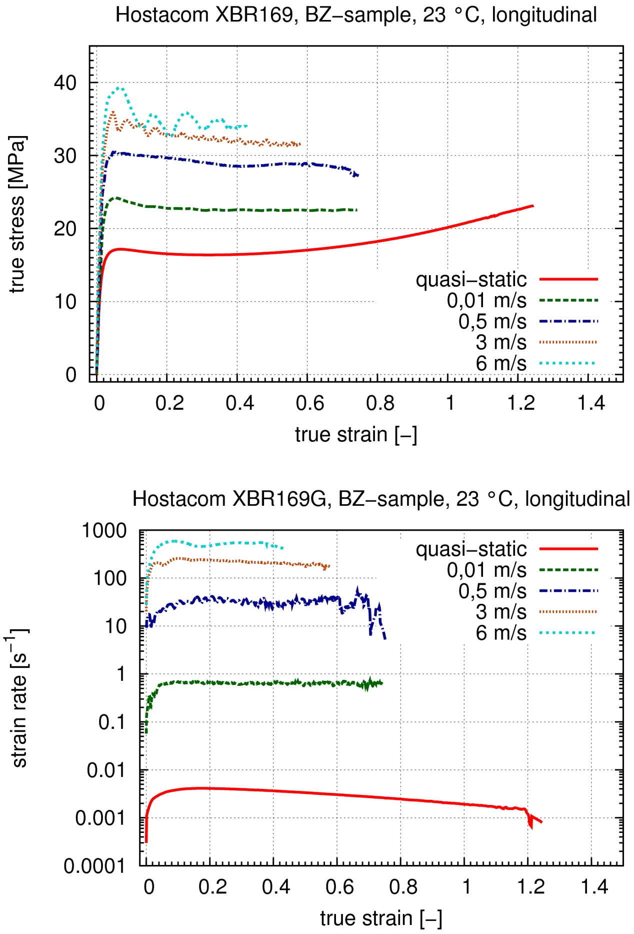

Figure 11: Strain rate dependence of Hostacom XBR169G

figure 11 for Hostacom XBR169G for samples which were taken longitudinal to the flow direction. In the top chart the true stress, in the lower graph the strain rate is shown as a function of the true strain. Therein, a significant strain rate hardening is recognizable. Strain hardening exists however only in the quasi-static test. The diagram of strain rate over the strain shows, that the strain rates developing at a constant haul-off speed of the testing machine are not constant with strain.

To demonstrate the distinctive anisotropic effects, the values strain rate and yield stress are charted from figure 11. Thereby, the withdrawal direction of the tensile specimens, longitudinal and lateral to flow direction, is considered. The measured data shows figure 12. In the double logarithmic plot yield stress (maximum stress before necking) and strain rate show a linear correlation. This relationship depends on the extraction direction of the tensile specimens. The samples taken longitudinal to the flow direction always show a higher yield stress than the lateral taken tensile specimens; the average difference is about 15 %.

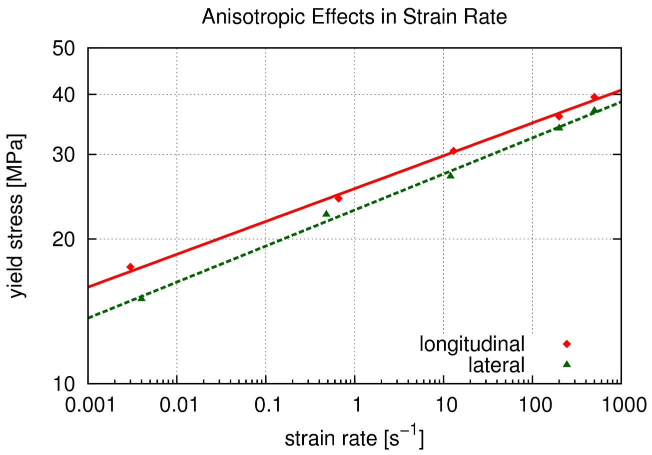

Figure 12: Anisotropic and strain rate dependent yield stress of Hostacom XBR169G

3.4 Summary of Mechanical Characterization

Hostacom XBR169G, generally counting as isotropic material, showed an interesting and complex deformation behaviour in the mechanical characterization. Therefore tensile- and shear tests were carried out at various test temperatures and test speeds. It appeared

- no distinct strain hardening for higher strain rates,
- a pronounced strain rate hardening,
- anisotropic effects in stress level according to testing direction in tensile tests,
- no anisotropy in shear tests,
- a resultant anisotropic and strain rate dependent yield stress,
- an anisotropic lateral strain behaviour
- and an increase of volume with increasing true strain.

These characteristics of Hostacom XBR169G have been considered in a material model that was developed by HEMPEL and SEELIG. Details of the material model can be found in [16] and are not explained explicitly here.

4 Validation via Three Point Bending Test

After developing the material model in [16] finally the validation of three-point bending tests should be described briefly. The bending tests were performed with the samples of figure 2. Figure 13 shows the experimental setup for bending tests.

The bending tests were performed at constant test speed of 2 mm/min. The experimental setup bases on DIN EN ISO 178 [18]. Figure 14 shows the behaviour of Hostacom XBR169G at the bending test. Just like the tensile tests the force displacement diagram shows anisotropic effects of approximately 20 %.

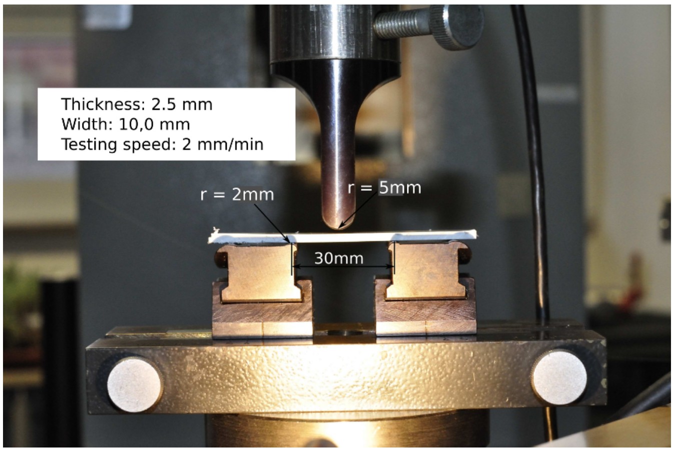

Figure 13: Eperimental setup of bending tests

In return the three-point bending test was simulated in LS-Dyna with the developed material model. The shell model contains of 3.200 elements and 10 integration points in the direction of thickness. Figure 15 shows the differences between measured and simulated behaviour of the force displacement diagram for the bending tests for longitudinal and lateral samples. The graphs show a good conformity with differences of only 10 %. Consequently the developed material model describes the deformation behaviour of Hostacom XBR169G in the three-point bending test very well. The description of the uniaxial tensile test for different (quasistatic and dynamic) strain rates fits also very well. This is shown by Hempel and Seelig in [16]. The material model will probably be implemented in LS-Dyna in 2011.

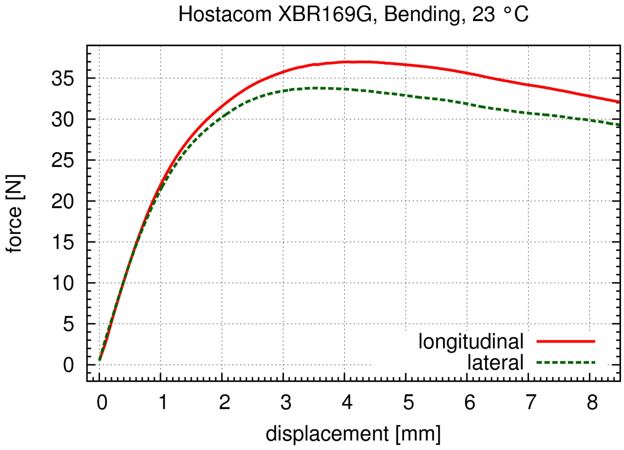

Figure 14: Three point bending test results of Hostacom XBR169G

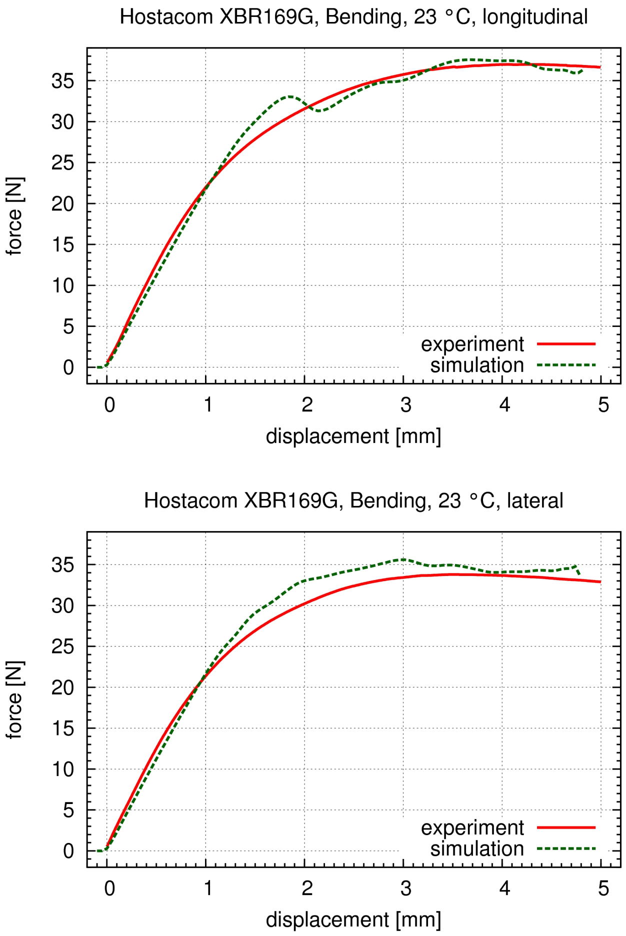

Figure 15: Validation of the material model of Hostacom XBR169G

5 Summary and Outlook

The aim of this study is to create a solid experimental basis for a material model for crash simulation, taking into account an anisotropy induced by injection moulding. The researches shows that this anisotropy in Hostacom XBR169G, a PP filled with talc and EPDM, shows pronounced differences in stress levels both at high strain rates and at different test temperatures. These differences for tensile bars, which are taken longitudinal and lateral to the flow direction of injection moulded plates, do not occur in shear tests.

The measured stress-strain curves in combination with the measurement of strain depending lateral extension at different strain rates and test temperatures are used by Hempel and Seelig [16] as a basis to create a material model for Hostacom XBR169G and its application in crash simulation. This material model was validated successfully in LS-Dyna via three point bending tests an will be implemented in LS Dyna in the future.

Furthermore, the prepared samples will be analysed using SEM and TEM to explain the occurrence of anisotropy depending on injection moulding settings. Approaches for an alignment of the talc particles have already been explained in literature. Furthermore, the influence of injection moulding setting is analysed in more detail using other plates produced under different injection moulding settings. Through these researches it will be possible to explain the measured stress differences depending on orientation.

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