



Research Article

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Comparing Test Methods for the Intra-ply Shear Properties of Uncured Prepreg Tapes

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Abstract: To achieve cost-efficient manufacturing and a high part quality in Thermoset Automated Fiber Placement (TS-AFP), knowledge about the interaction between material and process parameters is of special interest. Material properties of prepregs are well known at the cured state of the resin. However, there are no standardized test procedures for the mechanical behavior of the uncured prepreg tapes. To investigate the intra-ply shear deformation behavior of uncured unidirectional prepreg tapes, we compared several measurement procedures and conducted experiments for rheometer based tests using 8552/AS4 material. We identified a rotational parallel platens rheometer test method and a torsion bar rheometer test method to be suitable. Experiments using both methods revealed that the Torsion Bar Test has a higher repeatability and the analysis is less complex. Furthermore, first results show that changes in material properties caused by aging can be analyzed using this method. In the future, we will use the Torsion Bar Test to characterize changes in deformation behavior due to material aging as well as material modifications. By this, we will be able to provide data for the material modeling thus enabling the prediction of lay-up defects such as buckling due to steering.

Keywords: automated fiber placement; prepreg aging; rheometer

1 Introduction

During Thermoset Automated Fiber Placement (TS-AFP) several preimpregnated (prepreg) slit-tapes are laid up onto a mold by a placement head. The head is manipulated with a defined rate by an industrial robot while applying compaction pressure and processing temperature to the material [1, 2]. The use of narrow tapes – usually 1/8", 1/4", or 1/2" – enables lay-ups along non-geodesic paths to achieve variable stiffness laminates as well as lay-ups on complex geometries [2–4]. This is possible because a smaller tape width leads to a smaller minimum steering radius [5]. However, in these lay-up scenarios the tapes are deformed both in-plane (bending, shear) and out-of-plane (bending) potentially causing defects like out-of-plane buckling, bridging, or tape peel off [3–6]. In order to minimize these defects and to achieve optimal lay-up rates, knowledge about the material process interaction is of special interest – in particular since changes of the material properties due to aging have a strong influence on the occurrence of lay-up defects [7].

Several authors have investigated the in-plane and out-of-plane deformation behavior of uncured prepreg materials such as woven fabrics and cross-ply stacks – see reviews in [8, 9], and [10]. However, many of these test methods have limitations regarding the characterization of uncured unidirectional (UD) prepreg tapes. Hence, there are no test standards for this type of material. To define a characterization strategy for the intra-ply shear and the out-of-plane bending behavior, we evaluated several test methods for uncured UD prepreg tapes. The objectives were finding test methods with a high reliability of results, which require only standard test equipment and a low amount of material. Furthermore, the test methods should be suitable to detect changes in material properties due to aging or material modifications.

This paper shows the results for longitudinal intra-ply shear deformation. For this, we compared several test principles regarding the above mentioned objectives and conducted experiments with HexPly 8552/AS4 [11] at different out times using the Thin Plate Torsion Test [12] and the Torsion Bar Test [9, 13].

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2 Methods and materials

The following section presents the assessment of the test methods and the description of the conducted experiments – Thin Plate Torsion Test and Torsion Bar Test – followed by the description of the material preparation.

2.1 Test Methods for Longitudinal Intra-Ply Shear

The intra-ply shear encompasses the movement of fibers past one another within a ply – either parallel (longitudinal) or orthogonal (transverse) to the fiber direction [14, 15]. The longitudinal intra-ply shear is generally seen as the most important forming mechanism [9, 16] while the transverse intra-ply shear is of lower importance when investigating UD prepreg tapes [9].

To find the most suitable test method for uncured UD prepreg tapes, we evaluated four different test principles known in literature – see Figure 1: a) picture-frame test setup, b) off-axis tension test setup, c) rotational parallel platens test method, d) torsion bar test method.

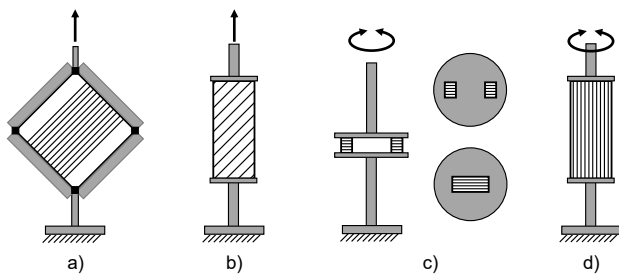


Figure 1: Test principles for longitudinal intra-ply shear characterization (as described in [9, 10], and [13]): a) picture-frame test setup; b) off-axis tension test setup; c) rotational parallel platens test method; d) torsion bar test method.

We did not consider the picture-frame test setup suitable as strong wrinkle formations cannot be avoided in this method. By using a picture frame in which all edges of the specimen are clamped, there is no slippage of the material and the shear properties can be measured via the axial force and the angle of the stretched frame [15]. Leutz [17] conducted picture frame test experiments comparing single tape, multi tape / single layer, and multi tape / multi layer uncured UD prepreg specimens. He only obtained results for the latter since twisting of the tapes impeded any measurements with the single layer specimens. However, even for the multilayer specimens results were not satis-

factory. Due to the clamping, the fibers cannot rotate and are forced to bend leading to out-of-plane wrinkling and therefore invalid results [13, 17].

The principle of off-axis tension – Figure 1 b) – is used in the bias extension test for cross-ply specimens [18, 19] and in the off-axis test for multilayer UD specimens [20, 21].

We did not consider the bias extension test suitable for the characterization of longitudinal intra-ply shear properties because of its limitations for the use of uncured UD prepreg tapes. In this test principle, a bidirectional specimen is loaded with a tension force that is non-parallel to the direction of the reinforcing fibers [22–24]. By measuring the displacement and the load, shear properties can be determined. This test method has been used by several authors for cross-ply specimens of uncured UD prepreg material [18, 19, 25]. However, slippage between the layers leads to interply shear, which impedes a reliable measurement of the intra-ply shear properties. Additionally, uncontrolled distortion of the flexible specimens leads to wrinkle formation and therefore invalid results before the critical load is reached [9].

The off-axis test introduced by Potter [20] and subsequently used by Wang *et al.* [21] is a suitable test method for the characterization of uncured UD prepreg tapes. Here, a multilayer UD specimen is loaded off axis using a universal test machine. The shear stress is determined by the load-displacement result and the off-axis angle – see [21]. Using this test principle, Wang *et al.* were able to investigate the influence of test rate and test temperature on the shear properties. The needed test equipment – universal testing machine, digital image correlation, thermal chamber – however, is disadvantageous. Also, the combination of thermal chamber and digital image correlation requires a very accurate test setup and calibration in order to obtain reliable images recorded through the safety glass of the thermal chamber.

We considered the Thin Plate Torsion Test [12], which is based on the principle of rotational parallel platens – see Figure 1c) – a suitable test method. Here, single layer specimens are subjected to a rotational load by parallel platens using a rheometer. By varying the applied shear angle and aspect ratio of the specimens, the shear properties can be determined – see [12]. The use of a rheometer allows for an accurate setting of shear rates and test temperatures. In addition, the possibility to analyze single layer specimens is a unique characteristic of the Thin Plate Torsion Test compared to the above mentioned test methods.

Furthermore, we considered the Torsion Bar Test – see Figure 1d) – a suitable test method for the characterization of UD prepreg material. In this test principle, which was developed by Haanappel and Akkermann [9], a prismatic

Table 1: Test parameters – thin plate torsion tests

Aspect ratio (L/H) [mm/mm]	Shear rate $\dot{\gamma}$ [s^{-1}]	Shear angle θ [$10^{-3} \cdot \text{mrad}$]
20/15; 25/12; 30/10	0.004; 0.002; 0.0008	40; 20; 10; 5; 2.5; 1.25; 0.625

bar is loaded torsionally in a rheometer. By using UD specimens with a square cross-section and aligning the fiber direction parallel to the rotational axis, the shear properties can be determined – see [9]. Again, the use of a rheometer allows for an accurate setting of shear rates and test temperatures. Moreover, previous studies have demonstrated the applicability of the test to uncured UD prepreg material [13].

Since the Thin Plate Torsion Test and the Torsion Bar Test fulfill the requirements of a simple, reliable test setup, using standard equipment, and requiring a comparably low amount of material, we conducted experiments with both principles. Both test methods are based on a rotational load application, which enables a direct comparison of test results. The following sections detail the procedures for both experiments.

2.2 Thin Plate Torsion Test (TPT)

The Thin Plate Torsion Test is based on Rogers' method for the rheological characterization of anisotropic material [26]. The analysis procedure for uncured UD prepreps is explained in [10] and [12].

The equipment for the experiments is a rheometer (here: Anton Paar MCR 302) with a plate-plate configuration. During the test, a single layered specimen is put in the middle of the lower plate. The upper plate applies a minimum pressure onto the specimen and rotates oscillating, thereby loading the specimen in shear within the Linear Viscoelastic (LVE) region. Here, the material behaves linearly when a sinusoidally applied deformation results in a purely sinusoidal stress response [27]. The analysis is done using mean values of the torque M_{TPT} and the phase lag δ measured by the rheometer. According to Rogers [26], it is possible to correlate between the torque M_{TPT} , the shear angle θ , the distance between upper and lower plate d , and the longitudinal and transversal shear moduli G_L and G_T with their respective storage and loss moduli parts G' and G'' for viscoelastic material behavior using the following equations:

$$\frac{3 M_{TPT} \cos(\delta) \cdot d}{4 LH^3 \theta} = G'_L + G'_T \left(\frac{L}{H} \right)^2 \quad (1)$$

$$\frac{3 M_{TPT} \sin(\delta) \cdot d}{4 LH^3 \theta} = G''_L + G''_T \left(\frac{L}{H} \right)^2 \quad (2)$$

where L represents the specimen length and H the specimen width. The equations are arranged as a first order polynomial regression of the form $m \cdot \left(\frac{L}{H} \right)^2 + n$, where the slope m corresponds to the transverse shear modulus and the intercept with the ordinate n to the longitudinal shear modulus. By repeating the experiment with specimens of the same area but different aspect ratios, the storage and loss moduli can be determined. Furthermore, the relaxation modulus G_r can be approximated as a linear combination of the frequency dependent storage and loss moduli using equation (3) [9, 28, 29]:

$$G_r(t) = G'_L(\omega) - 0,528G''_L \left(\frac{\omega}{2} \right) + 0,112G''_L(\omega) + 0,0383G''_L(2\omega) \quad (3)$$

If measurements are performed at a constant shear rate $\dot{\gamma}$, G_r can be approximated with a power law function – equation (4) – and the relationship between shear stresses σ_{12} and shear strains γ_{12} can be determined using equations (5) and (6) [9]:

$$G_{r,L}(t) = G_\infty + at^{1-b} \quad (4)$$

$$\gamma_{12}(t) = \dot{\gamma}_{12} \cdot t \quad (5)$$

$$\sigma_{12}(t) = \dot{\gamma}_{12} \left(G_\infty t - \frac{a}{b-1} t^{1-b} \right) \quad (6)$$

where a , b , and G_∞ are approximation coefficients from the power law function – see equation (4). These can be determined with the function of the regression line of G_r with respect to the time t .

In previous experiments, a lower plate made out of aluminum was used which was slightly deformed due to the clamping. Therefore, we replaced it with a stiffer plate made out of stainless steel. In order to assure a material characterization within the LVE region, we performed amplitude sweeps from 10^{-4} mrad to 10 mrad at a constant frequency of 1 Hz. The end of the LVE region was determined by the deviation of the storage modulus by more than 10% from the initial plateau [30]. By this, we found a maximum amplitude of 0.04 mrad. We conducted the experiments at room temperature with the material having no additional out time. We repeated each experiment three times using the parameters listed in Table 1 for the same full factorial experimental design as Margossian *et al.* [12].

Table 2: Test parameters – torsion bar tests

Temperature T [°C]	Glass transition temperature T_g [°C]	Angular frequency ω [rad/s]
27; 40	-2.8; -1.8; 6.9	0.1... 500

2.3 Torsion Bar Test (TB)

Haanappel and Akkermann developed the Torsion Bar Test for the characterization of fiber reinforced thermoplastic melts [9] and Margossian applied it to uncured UD thermoset prepregs [13]. The approach is based on the correlation between the torque M_{TB} and the rotation angle ϕ for a prismatic bar with the fiber direction parallel to the rotation axis and linear elastic material behavior:

$$M_{TB} = G_{L,TB} J \frac{\phi}{L_{TB}} \quad (7)$$

where $G_{L,TB}$ corresponds to the elastic shear modulus, J to the torsional constant, and L_{TB} to the free specimen length. For the measurements, a multilayered specimen with a square cross section is positioned inside the rheometer using the standard torsion clamps – see Figure 1 d). The specimen is subjected to an oscillating torsional load within the LVE region by the upper clamp while the lower clamp remains fixed. Frequency sweeps are conducted and the generated data for the storage modulus and the loss modulus are used to calculate the relaxation modulus and the stress-strain behavior analogous to the Thin Plate Torsion Test with equation (3) to (6) [9].

We conducted the experiments with an amplitude of 0.003% strain – determined by amplitude sweeps in accordance with the procedure in [30] to find the limit of the LVE region. The specimen geometry was: length $L_{TB} = 60$ mm, width $W = 12.5$ mm, and thickness $T = 12.03$ mm. We repeated each experiment three times and used the parameters listed in Table 2 for a full factorial experimental design. For the test parameters, we defined two different temperatures within the range of the process temperature during TS-AFP. We chose 27°C since a process temperature just above ambient temperature can be seen as a lower limit for TS-AFP to obtain sufficient tack for the lay-up. The second test temperature – 40°C – represents a typical elevated process temperature which is used to further increase the tack while staying below temperatures that lead to undesired curing during lay-up. In order to investigate the influence of material aging, we conducted the experiments with three different material conditions defined by its out time. We quantified the material condition by the glass transition temperature – see section Material below.

2.4 Material

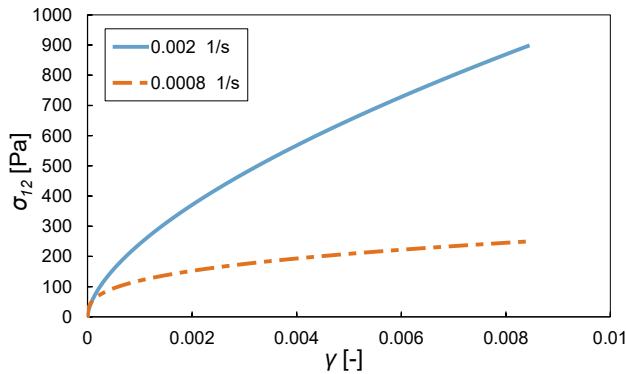
The material used in the experiments was HexPly 8552/AS4 with an aerial weight of 194 g/m² and a resin weight content of 34% [11]. We prepared 60 layer specimens for the Torsion Bar Test to achieve a total thickness of 12 mm. The cross section has to be close to square to ensure reliable results while the clamp restricts the width to 12.5 mm. To achieve a homogenous bond between the layers without artificially aging the material, we compacted large layers applying vacuum at room temperature for 35 min. We measured the glass transition temperature T_g using Differential Scanning Calorimetry (DSC) before and after the process to examine the aging of the material. The T_g rose by 0.17°C, which we considered negligible. In comparison, we also investigated the effect of compaction with an elevated temperature by applying 40°C for up to 70 min. This led to a not negligible rise in T_g by 2.18°C. Micrograph images showed that there was no improvement in layer bond due to the temperature application proving that the above parameters of 35 min vacuum at room temperature are appropriate. After the compaction, we cut the large plies into the specimen size using an NC cutting machine which helped to avoid localized specimen deformation and led to a significant improvement in specimen preparation compared to previous experiments [13].

To verify whether the tests can be utilized to detect changes of the material properties, we stored a number of specimens at 60°C for two hours (equivalent to 1.5 days at room temperature) and others at ambient temperature for fourteen days. The resulting material conditions are listed in Table 3.

Table 3 includes two examples of the degree of cure α calculated from T_g – one simple linear approach adopted from [31] and one non-linear approach as proposed by [32]. Since there are several different cure models for Hexcel 8552 – see review in [33] – we evaluated the experimental results with reference to the glass transition temperature.

Table 3: Tested material conditions

Equivalent out time at room temperature [d]	Glass transition temperature T_g [°C]	Degree of cure α_{lin} [%] (linear approach [31])	Degree of cure α_{nonlin} [%] (non-linear approach [32])
0	-2.8	0.0	0.0
1.5	-1.8	0.5	0.8
14	6.9	4.7	7.2

**Figure 2:** Results – Thin Plate Torsion Test: longitudinal shear stress σ_{r2} vs. shear strain γ for two different shear rates.

3 Results and discussion

3.1 Thin Plate Torsion Test (TPT)

Figure 2 presents the results of the Thin Plate Torsion Test excluding the results for the shear rate of 0.004 s^{-1} for which problems with the measurement setup led to invalid results. Figure 3 gives an example of the analysis procedure.

The experiments revealed that there are various limitations when using the Thin Plate Torsion Test. There are several sources for possible error. First, the values for torque M_{TPT} and phase lag δ used for the analysis are averages of the measured values – see Figure 3 (left). There is scatter in the results of the repeated measurements of up to 20% – see Figure 3 (middle). One reason for this is the slight buckling of the lower plate because of the clamping, which still happened with the stiffer steel plate. Other causes for uncertainties are the approximations, which have to be done in order to comply with equations (1) to (6). Figure 3 (middle) and (right) indicate that both the regression lines for the determination of G' and G'' as well as a , b , and G_∞ can deviate from the measured values. The use of single layer specimens and the low material demand are advantages of this test method. However, the number of measurements that have to be performed with the different aspect ratios

and shear angles represent another drawback of the test method.

Because of the above-mentioned limitations, we did not consider the Thin Plate Torsion Test for the detection of changes in material properties due to aging.

3.2 Torsion Bar Test (TB)

Figure 4 summarizes the results of the Torsion Bar Test. For better readability only the results computed at a shear rate of 0.005 s^{-1} are depicted. Figure 5 gives an example of the analysis procedure.

The Torsion Bar Test measurements revealed less scatter and the analysis procedure is less susceptible to inaccuracies compared to the Thin Plate Torsion Test. The data for storage and loss modulus are directly determined by the rheometer software so that no approximations for the determination of G' and G'' are needed. The results of the repeated measurements revealed comparably low scatter with all values of the coefficient of variation being less than 5%. In addition, the deviation of the regression line for G_r from the measurement points is comparatively low – see Figure 5. One drawback is the specimen thickness of 60 layers, which makes the specimen preparation more time consuming and does not allow for a direct characterization of the single layer properties. Another drawback is the low maximum shear strain – see comparison below.

Yet, because of the above-mentioned advantages, we considered the Torsion Bar Test for the detection of changes in material properties. The results shown in Figure 4 underline that the test method is suitable for the characterization of changes in material properties due to aging. The property changes resulting from the different material conditions are clearly visible and the shear stresses increase as expected with the increase in T_g . Figure 4 also demonstrates that the expected material response at different temperatures is quantifiable using the Torsion Bar Test revealing a significant decrease in shear stress from 27°C to 40°C .

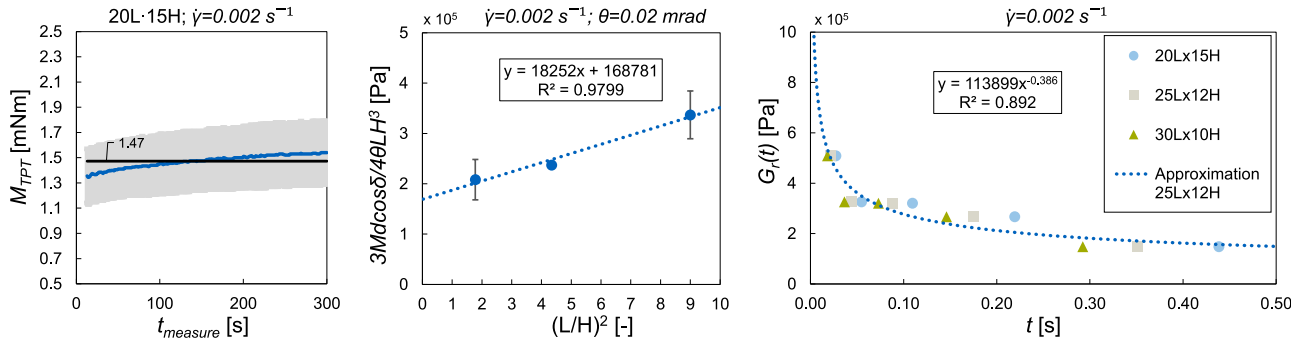


Figure 3: Analysis example – Thin Plate Torsion Test: measured torque M_{TPT} vs. measurement time $t_{measure}$ (left); $3Md\cos\delta/4\theta LH^3$ vs. $(L/H)^2$ (middle); relaxation modulus G_r vs. time t (right).

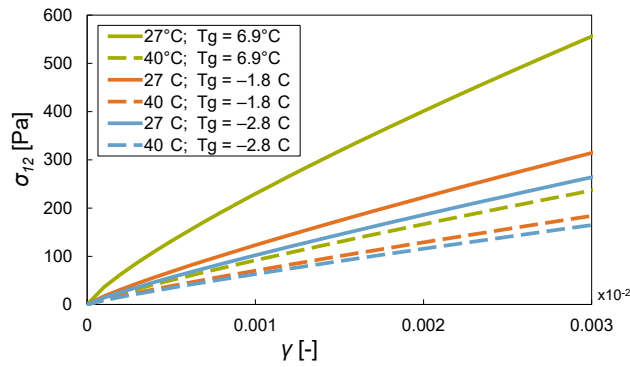


Figure 4: Results – Torsion Bar Test: longitudinal shear stress σ_{12} vs. shear strain γ at 0.005 s^{-1} for two different temperatures and three different material conditions.

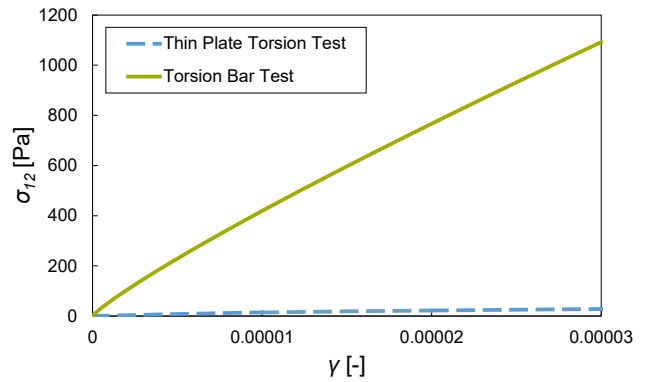


Figure 6: Results – Thin Plate Torsion Test and Torsion Bar Test: longitudinal shear stress σ_{12} vs. shear strain γ at 0.002 s^{-1} .

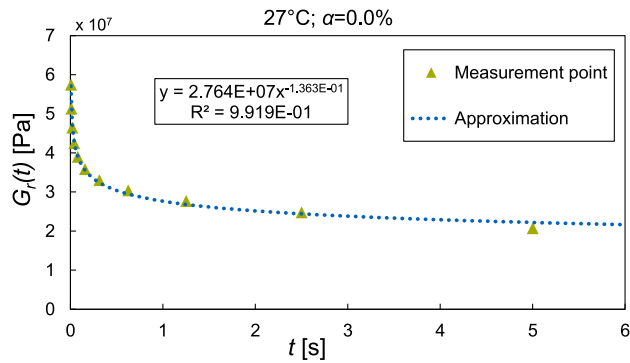


Figure 5: Analysis example – Torsion Bar Test: relaxation modulus G_r vs. time t .

4 Comparison of results

We consider the Torsion Bar Test suitable for the characterization of changes of the longitudinal intra-ply shear properties because of the above mentioned advantages regarding reliability and simplicity. Furthermore, the test method enables the application of a higher shear rate. The maximum strain rate during the TB experiments was

0.012 s^{-1} whereas we only obtained results up to a strain rate of 0.002 s^{-1} during the TPT experiments. In comparison, Wang *et al.* [21] calculated that the shear rate would be 0.035 s^{-1} to 0.089 s^{-1} in a typical AFP lay-up with a steering radius of 900 mm and a lay-up speed of 200 mm/s to 500 mm/s. They conducted experiments at rates from 0.001 s^{-1} to 0.05 s^{-1} . Therefore, the TB shear rate is in the same order of magnitude as during AFP deposition whereas the TPT shear rate is considerably lower. In contrast, the maximum strain is significantly lower in the TB test setup – 0.00003 – compared to the TPT test setup – 0.0084. In comparison, Wang *et al.* [21] plotted results up to 0.09 strain. This demonstrates a limitation of the TB test method. However, we still consider this test method suitable for the characterization of material changes and their effects on the intra-ply shear properties as shown in the presented results.

The comparison of stress-strain curves reveals further differences in the results – see Figure 6.

Both curves result from experiments with material with no additional out time and the same shear rate of 0.002 s^{-1} . The test temperature differs slightly since the

TPT experiments have been carried out at approximately 22°C while a temperature of 27°C was set during the depicted TB measurement. Surprisingly, there is a significant difference in the stress-strain response resulting from the two test procedures. The TB result reveals a stress curve several orders of magnitude higher than the TPT result. The reasons for this result are not yet understood. It cannot be ruled out that slippage instead of shear deformation occurred during the TPT experiments. In contrast to the TB test setup, the specimen is not clamped in the TPT test setup. Therefore, there might be slippage between the rotating plate and the specimen. Furthermore, the difference in specimen shape and rotational load application might impede a direct comparison of the two test methods. The TPT specimens comprise a single layer of material and the rotational axis is perpendicular to the fiber direction. The TB specimens comprise 60 layers of material and the rotational axis is parallel to the fiber direction.

As the causes for these differing stress-strain curves are not yet understood, future studies will focus on determining the reasons for the different results.

5 Conclusion

The Torsion Bar Test is capable of characterizing the longitudinal intra-ply shear deformation behavior of uncured unidirectional prepreg tapes. Using this test method, it is possible to analyze changes in material properties (degree of cure) and process parameters (temperature) within a range that is common for TS-AFP. Therefore, we will use it to characterize the shear deformation behavior as a function of material aging. The test will also be included in a characterization procedure for the development of new materials. In addition, future work will focus on the correlation between material properties and the occurrence of lay-up defects with the overall objective of predicting said defects without the need for lay-up trials.

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