COMPARISON OF METHODS TO CHARACTERIZE DAMAGE ONSET IN SHORT GLASS FIBER FILLED POLYPROPYLENE

A.M. Hartl1,*, W. Balasooriya1, M. Reiter2, M. Schossig3, M. Jerabek4 and R.W. Lang1
1 Institute of Polymeric Materials and Testing, Johannes Kepler University Linz, 4040 Linz, Austria, 2IPPE, Johannes Kepler University Linz, 4040 Linz, Austria, 3IPW Merseburg, 06217 Merseburg, Germany, 4Borealis Polyolefine GmbH, St.–Peter Str.25, 4021 Linz, Austria
* Corresponding author (anna.hartl@jku.at)

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1 Introduction and Scope
Damage onset and evolution are essential factors governing failure and lifetimes in composites such as short glass fiber reinforced polypropylene (sgf-PP). The main micro-mechanisms of failure can either be attributed to the matrix (i.e. shear yielding, crazing and micro-cracking), the matrix-fiber interface (debonding and fiber pull-out) or the fiber itself (fiber breakage). Several experimental approaches are suggested in literature to characterize these damage micro-mechanisms in terms of the onset and the kinetics of damage evolution. These include acoustic emission measurements, volume strain determination, modified versions of tensile/compression experiments (multi-cycle tests, isocyclic stress-strain curves), SEM in-situ experiments, fractography and combinations thereof (i.e. [1-3]).

As a detailed understanding of the micro-mechanisms governing damage evolution in sgf-PP materials is of prime importance for failure and lifetime assessment utilizing modern simulation techniques based on finite element methods and micro-mechanics concepts, various experimental methods were used in this paper to detect and study the micro-mechanisms of failure in sgf-PP under tensile loading. Hence, acoustic emission experiments and volume strain measurements in a monotonic loading mode and two-cycle tensile tests were performed, and the results are compared and interpreted based on fracture surface observations by SEM. The specific focus in this paper is on the effect of fiber orientation on the micro-modes of failure.

2 Experimental
Polypropylene (PP) with 32 m% fiber content and three fiber orientations (0°, 45° and 90°) was investigated. The specimens were injection molded in a specially designed glass fiber multi-tool allowing for a high and uniform fiber orientation. The fiber orientation function and fiber length distribution were assessed by computer tomography. First, two-cycle tensile tests were performed (see Fig. 1). During this test the specimen is loaded to a certain pre-strain in the first cycle, then unloaded and held at zero force for 6h, and then again loaded to ultimate failure in the second cycle. The moduli of the two cycles are compared and the residual strains evaluated.

Second, while performing tensile tests, acoustic emission measurements were conducted with a 3-channel measuring system of the type AMSY-4 (Vallen-Systeme; Icking, D). In the data analysis, all signals below 40 dB were filtered out. Details on test set-up and data recording are given elsewhere [2]. The data were evaluated in terms of the cumulative energy of the acoustic signals, which is a function of the amplitude and the signal duration. Here a predefined threshold value of 5% was taken as an indicator for damage onset. Furthermore, the distribution of the amplitude of the detected signals was analyzed.

Third, volume strain measurements during tensile tests were performed with a 3D Digital Image Correlation (DIC) system (Aramis; Gesellschaft für optische Messtechnik mbH; Braunschweig, D). The volume strain can be calculated by the combination of the Hencky strains in the three principal deformation axes. Details on data analysis are provided in [3]. For the 0° specimen, identical values for the contraction may be assumed in the width and the thickness direction of the specimen. For the 45° and 90° specimens, all three major strains were assessed.
All tensile tests were carried out at room temperature and at a strain rate of 0.001 s\(^{-1}\). For selected fracture surfaces, SEM investigations were performed to illustrate the main micro-modes of failure.

**3 Results and Discussion**

As expected, values for modulus and the tensile strength were found to decrease with increasing angle between loading axis and fiber orientation (see Fig. 2). The exact fiber orientation tensors and corresponding orientation ellipses are given in Fig. 3(a).

In terms of ultimate failure, different mechanisms in the micro-modes of failure are visible on the fracture surfaces for the three orientations investigated (see Fig. 3(b)). Specifically, matrix deformation on the 90° fiber orientation fracture surface is significantly more ductile, probably due to void formation at the fiber-matrix interface and fiber-matrix debonding in early stages of deformation and subsequent local stretching of the interfiber matrix regions in a plane stress mode [4]. By comparison, matrix failure in the 0° and 45° orientated specimens appears considerably more brittle, with the 0° fracture surfaces exhibiting a certain amount of fiber pull-out, while failure in the 45° specimens occurs in planes along the fiber orientation most likely shear. A closer look on these fracture surfaces at the mechanisms of fiber-matrix interfacial failure reveals that the mechanical separation actually takes place at some distance away from the fiber surface, as all of the fibers are still covered with a thin layer of polymer (Fig. 4). This is, of course, a sign of good fiber-matrix adhesion.

![Fig. 1: Illustration of two-cycle test method.](image)

![Fig. 2: Mechanical properties as a function of the fiber orientation.](image)

![Fig. 3: (a) SEM pictures of 0, 45 and 90° oriented sample (200x) and (b) the corresponding orientation tensors and ellipses.](image)
While the tensile test results, described above, were generated in a single monotonic loading mode up to failure, specifically designed 2-cycle tests offer an opportunity to better assess the damage evolution prior to ultimate failure. In this context, several authors suggested using the changes of macroscopic properties (i.e. modulus, Poisson’s ratio or the dimension) as a function of the loading history (i.e. prestrain) to investigate damage onset and evolution [5-7].

The results of the 2-cycle test are shown in Fig. 5(a) in terms of the relative E-modulus, defined as the ratio $E_{\text{cycle 2}}/E_{\text{cycle 1}}$, and the residual strain after relaxation as a function of the applied prestrain (curves in this diagram correspond to a polynomial fit of the data). Considering the inherent data scatter, a reduction in E-modulus of 5% corresponding to a relative E-modulus of 0.95 was defined as damage threshold criterion, resulting in strain onset values of 0.8, 1.6 and 1.1 % for the 0°, 45° and 90° specimen, respectively. Apparently, the 0° and the 90° normalized moduli seem to deteriorate at significantly lower values of deformation than 45° moduli. A similar trend can be observed for the residual strain. In interpreting these findings, one must consider that the ultimate strain values of the 45° specimens are also higher than for the 0° and 90° specimens.

Hence, the data of Fig. 5(a) are replotted in Fig. 5(b) as a function of the normalized strain defined as the ratio $\varepsilon_f/\varepsilon_u$, where $\varepsilon_f$ is the prestrain of the first loading cycle and $\varepsilon_u$ corresponds to the average strain-at-break from standard tensile tests. Interestingly, in terms of relative modulus values the 90° specimens depict the most substantial deterioration, while in terms of residual strains the 45° specimens exhibit the highest values. With regards to micro-mechanisms of deformation this presumably reflects the enhanced tendency for micro-voiding, debonding and/or micro-cracking in the 90° specimens, while the 45° specimens may be more prone to plastic deformation modes up to higher strains prior to the occurrence of debonding and/or micro-cracking.

In concluding this section it should be remarked, that independent of the fiber orientation irreversible deformation mechanisms (plastic matrix deformation, void formation, debonding and micro cracking) are observed even at very small values of prestrain of about 0.5-1%. In other words, the inhomogeneous inner material structure and the modulus mismatch of fiber and matrix apparently lead to local strain magnifications initiating these deformation mechanisms even at low values of overall specimen strain.
In good agreement with the mechanical 2-cycle experiments described before, acoustic emission tests also provided a clear indication of damage mechanisms at early stages of deformation in tensile tests. Figure 6(a) depicts the stress-strain curves for the three specimen orientation along with the corresponding cumulated acoustic signal energy also versus the mechanical strain. The 90° specimens are characterized by a more or less continuous increase in the AE signal energy up to strains of 3% above which the signal energy increases more sharply. In contrast the 0 and 45° specimen reveal a more moderate increase in the AE signal energy at low strain levels, but then rise more sharply at strain levels of 1.5% in the case of the 0° specimens and 2.2% in the case of the 45° specimen.

By applying a 5% threshold criterion for the total accumulated acoustic energy as significant damage initiation criterion, the onset values for the different orientations can be compared in terms of strains and stresses (see also Fig. 6(a)). As to the onset strain values, the materials rank in the order 0° and 90° specimens being nearly equivalent at around 1.4% strain while the 45° specimens reach about 2% strain. Unfortunately these strain values in these experiments were measured as nominal strains (using the crosshead displacement) and can thus not be directly related to the strain values in the 2-cycle (discussed above) and the volume strain tests (to be discussed below). For this reason, the 5% AE energy threshold was also transformed into the corresponding stress levels, which allow for a more direct comparison with the other two experiments. As indicated again in Fig. 6(a), the 5% AE energy threshold is reached in the 90°, 45° and 0° specimens at 10, 35 and 75 MPa, respectively.

The cumulated overall amplitude distribution up to final failure is shown for the three orientations in Fig. 6(b). For all orientations, the highest signal intensity was detected for amplitudes below approximately 55 dB, a range which is attributed to matrix related deformation and cracking modes [8]. Considering the total distribution for each orientation, this range is particularly dominating for the 90° specimens, as one would expect. By comparison the signal distributions for the 0° and especially for the 45° specimens are considerably broader, indicating a more pronounced overall contribution of fiber-matrix debonding and fiber pull-out.

In other words at least in general terms these observations also corroborate the findings and conclusions of the above described purely mechanical 2-cycle experiments.

Fig. 6: (a) Cumulated AE signal energy as a function of mechanical specimen strain and stress-strain curve with indication of 5% cumulated energy value (■), and (b) overall detected amplitude distribution up to final failure.
Figure 7(a) depicts the total volume strain and true stress values as a function of the true longitudinal strain. The total volume strain may be composed of a dilatational (pure Poisson’s ratio effect), a deviatoric (pure shape change without volume change e.g. shear yielding) and a cavitational (e.g. void formation, crazing, debonding, microcracking) component [9]. The dilatational part, which is predominant in the low deformation regime, is due to hydrostatic tension in the stressed material and is directly related to Poisson’s ratio effects. For the 0° and 45° specimen, a linear dependence was found for the total volume strain versus the longitudinal strain up to about 2% (0.4% volume strain), indicating a pure Poisson controlled dilatational volume change. Above 2% true strain, non-linearity is observed particularly in the case of the 45° specimens as these are deformed to significantly higher true strain values, which also implies significantly higher total volume strain values prior to fracture. Thus total volume strain values prior to failure in 45° specimens of up to 1.6% are reached, whereas the corresponding value of the 0° specimen is about 0.6%. The 90° specimen reveals a very different behavior exhibiting non-linearity even at lower values of true strain, which becomes more pronounced as the deformation increases. Prior to fracture, at about 3.8% longitudinal strain, a total volume strain value of about 2.6% is reached.

By subtraction of the calculated dilatational volume strain (based on pure Poisson’s ratio effects) from the measured total volume strain, the cavitational volume strain was determined, and the results are plotted in Fig. 7(b). In agreement with the previous remarks, hardly any cavitational component can be observed for the 0° specimen at least up to 1.5% true strain. In other words, here the deformation is indeed mostly governed by pure dilatational effects. This also agrees with the fracture surface observations in Fig. 3(a), which reveal only little indication for deviatoric or cavitational deformations in the matrix (fiber pull-out, as is observed in the fracture surfaces, is believed to occur in the very late stages just prior to or at fracture).

For the 45° specimens the behavior up to 1.5% longitudinal strain is apparent rather similar to the 0° specimens, again indicating a nearly pure dilatational volume change. Above 1.5% the cavitational contribution is seen to rise up to about 0.6% at ultimate failure. The underlying mechanisms may be shear induced matrix-fiber debonding and the development of shear microcrack in the matrix. Indications for both mechanisms are to be observed on the fracture surface of Fig. 3(b).

In contrast, the deformation behavior in the 90° specimen is governed by dilatational deformations only up to 1% longitudinal strain. At higher longitudinal strains significant cavitational deformation modes seem to develop which may even be disguised by the already occurring deviatoric response of the PP matrix at least at very high strain levels prior to ultimate failure. The latter assumption is based on the fracture surface observations in Fig. 3(b), which reveal a high degree of local ductility in the matrix deformation.

![Fig. 7: True stress and (a) volume strain and (b) deviatoric and cavitational volume strain as a function of true strain.](image-url)
Summary and Conclusions

In the present paper three techniques to determine damage onset by cavitational deformation mechanisms were investigated and compared as to their applicability and limitations for sgf-PP of various fiber orientations. The experimental determination of damage onset depends on the technique applied, the damage onset criterion defined for the specific technique and on the inherent scatter of the experimental data. Thus it is not surprising that it is difficult to define accurate values for damage onset particularly in terms of their relevance towards longer structural integrity issues. Nevertheless, depending on the technique and the associated sensitivity criterion for damage onset selected, the various techniques allow for a more or less sound definition of damage onset strain or corresponding stress ranges.

A comparison of the damage onset results of all techniques is depicted in Fig. 9 for the various fiber orientations. Thus the damage onset ranges were found to be roughly at 1-1.5% for the 0° specimens, around 1.5% for the 45° specimen and approximately 0.5-1% for the 90° specimen.

Finally, further work is needed to investigate the above findings in terms of their relevance for component design and structural integrity assessment.

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