ESEM examinations for assessment of damage mechanisms in short glass fiber reinforced thermoplastics

Marcus Schoßig[1], Christian Bierögel[1], Armin Zankel[2], Peter Pölt[2], Wolfgang Grellmann[1], Thomas Mecklenburg[3]

[1] Martin-Luther University Halle-Wittenberg, Center of Engineering Sciences, D-06099 Halle/Saale, Germany
[2] Institute for Electron Microscopy, Graz University of Technology, A-8010, Austria
[3] LyondellBasell Industries, D-65926 Frankfurt, Germany

marcus.schossig@iw.uni-halle.de
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The aim of this work was the characterization of damage mechanisms during the quasi-static loading of fiber reinforced thermoplastic materials. For this purpose, different test methods were combined: environmental electron scanning microscopy (ESEM), mechanical testing and acoustic emission (AE) analysis. The ESEM enables the investigation of electrically insulating specimens, such as polymeric materials, without the necessity of an electrically conducting coating. Thus, mechanical tests can be performed using special equipment, which enables the direct observation of the occurring damage mechanisms. On the other hand, the frequency analysis of the recorded AE, enables the determination of damage mechanism in fiber reinforced materials like debonding, fiber pull out with and without matrix yielding, friction processes of fibers in the polymeric matrix or fiber breakage [1]. By the simultaneous recording of the AE during a tensile test in an ESEM, a correlation of characteristic frequency ranges with existent deformation and damage mechanisms is possible.
In this study, polypropylene (PP) and polybutene-1 (PB-1) reinforced with 20 wt% short glass fibers were investigated. Due to the nonpolar character of PP, maleic anhydride was used to improve the bonding of the fibers in the polymeric matrix. For PB-1, no coupling agent was used and so, different bonding conditions were realized. The used E-glass fibers have a fiber diameter of 10 µm and a length of approx. 370 µm for PP/20 and approx. 270 µm for PB-1/20. The specimens used for the *in situ* tensile tests have the dimensions of 60x10x4 mm^3. By polishing the injection-molded specimens with silicon carbide sandpaper, the characteristic flash was removed. A notch with a radius of 0.1 mm and notch depth of 2 mm was made to achieve a stress concentration. Furthermore, this notch offers the possibility for a direct observation of the damage mechanisms during the loading of the specimens. The tensile loading was realized by a MT5000 Tensile Stage from Deben (Suffolk, UK), mounted on the stage of the ESEM Quanta 600 FEG equipped with a Schottky emitter (FEI, EINDHOVEN, THE NETHERLANDS) [2] and a load cell of 5 kN. For the clamping of the specimens a torque of 1 Nm was used. The AE measurements were realized with the 3 channel AMSY-4 measuring system (VALLEN-SYSTEME GMBH, ICKING, GERMANY) with the preamplifier AEP-3 and a broadband sensor AE204A. The bandwidth of the preamplifier and the AE sensor are 95–1000 kHz and 100–780 kHz, respectively. For a suitable coupling of the broadband sensor to the specimen surface, beeswax was used.

In the following, some results for the PP/20 material should be discussed. The load–time curve from the tensile test and the energy distribution from the AE analysis are shown in Fig. 1a. The load–time curve can be separated in four regions, (1, 2) linear-elastic, linear-viscoelastic, (3) non-linear viscoelastic and (4) crack propagation. The energy distribution in the regions (1, 2) shows only a low acoustic emission activity. In the region (3), a continuous

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increase of acoustic emissions occurs. After 434 s at the load maximum, the acoustic emission energy increases significantly due to the stable crack propagation. The maximum of energy is reached at the point where unstable crack propagation (at 484 s) occurs. The reason is the accumulation of the damages during the fracture of the specimen. Fig. 1b shows selected ESEM images at different times (SE-images). After a time of 116 s, blunting of the sharp crack (pre-existing damage due to the polishing) (**i**) as well as the formation of a void at a lateral glass fiber (**ii**) are recognizable. By increasing the load, widening and blunting of the notch (**iii**) as well as exposure of separate glass fibers (**iv**) and the smashing of a transverse glass fiber (**v**) occur. At 396 s, the formation of voids in front of the notch (**vi**) with drawing of matrix material into it (like formation of crazes) is predominant. In addition to this, also formation of dimple structures (**vii**), pull-out of glass fibers (**viii**) as well as the orientation of near-surface fibers (**ix**) in the direction of the load line could be seen. Based on the results from fractography investigations of PP/20 at defined times and the corresponding AE signals, correlations between the appearing damage mechanisms and frequency domains could be found. The basis for these correlations is the functional interrelationship between frequency, time and wavelet (WT) coefficient. For the PP/20 material, 3 typical frequency ranges are determinable:

120–160 kHz: glass fiber breaking
160–400 kHz: drawing of matrix material into voids with motion of glass fibers and friction into the matrix
300–400 kHz: debonding and pull-out with matrix yielding

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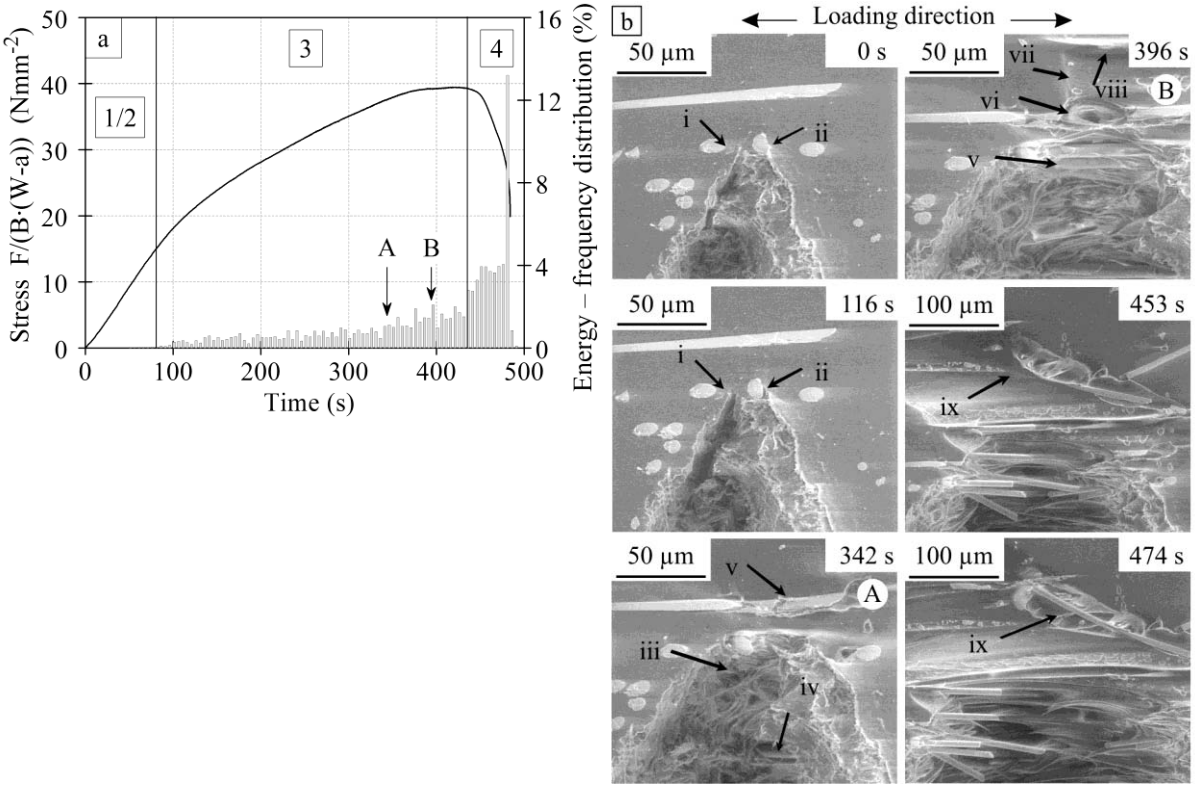

Figure 1. a) Stress–time and energy–time diagram and b) ESEM images at different times

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