

A COMPARATIVE STUDY OF THE STATIC AND CYCLIC ACOUSTIC EMISSION BEHAVIOR OF FIBER REINFORCED THERMOPLASTICS

C. Bauer^{1a}, T. Rief^{1b}, J. Hausmann^{1c} and T. Schalk^{2d}

¹Institute for Composite Materials, Design and Analysis, TU Kaiserslautern,
Erwin-Schrödinger-Straße, 67663 Kaiserslautern, Germany

²ZF Friedrichshafen AG, Graf-von-Soden-Platz 1, 88038 Friedrichshafen, Germany

^aEmail: constantin.bauer@ivw.uni-kl.de., Web Page: <http://www.ivw.uni-kl.de>

^bEmail: thomas.rief@ivw.uni-kl.de., Web Page: <http://www.ivw.uni-kl.de>

^cEmail: joachim.hausmann@ivw.uni-kl.de., Web Page: <http://www.ivw.uni-kl.de>

^dEmail: thomas.schalk@zf.com., Web Page: <http://www.zf.com>

Keywords: PA66GF30, short fiber, fatigue, acoustic emission, In-situ CT

Abstract

The investigation deals with the comparison of the acoustic emission behavior of fiber-reinforced thermoplastics in static and cyclic cases and how the information from static tests can be used for describing the fatigue behavior. In this case, short glass fiber reinforced polyamide 66 showed characteristic strain limits in static tensile tests, where the cumulative acoustic energy rises rapidly. In fatigue tests, these strain limits were tested and correlations to the static tensile tests were made. Additionally an in-situ micro-computer-tomography (μ CT) tensile test with a parallel acoustic emission analysis was performed to identify the damage mechanisms for the recorded acoustic frequencies. Furthermore, the free volume strain was calculated by a three dimensional digital image correlation to analyze the damage mechanisms at the characteristic strain limits.

1. Introduction

The aim of the present work is to draw conclusions from static tensile tests to the fatigue behavior of the investigated material, i.e. short glass fiber reinforced polyamide. The reasoning behind the experimental approach is to see if load limits of the fatigue behavior can be concluded from static tests, since fatigue tests are very time consuming due to the high number of load cycles needed. For one test specimen cyclic testing takes at least up to two weeks in time (depending on the load, frequency and cycles) while static tests can be conducted in a part of this time including the test setup. Therefore, static tensile tests have been conducted with the recording of the acoustic emission of the material. The acoustic emission provides information about damaging inside the specimen in consequence of the applied load. By distinguishing the recorded signals in their frequency, intensity and waveform, different damage states of the material can be detected [1–4]. As further investigations have shown, it is possible to relate significant acoustic events during static tests to the fatigue behavior of fiber reinforced thermosets [5, 6]. In the following sections similar investigations will be conducted for a thermoplastic material.

Additionally in-situ μ CT tensile testing with acoustic emission was set up to collect information of the material behavior inside the specimen and about possible material failure modes on the microscale and the coupling with the recorded acoustic events. Furthermore the μ CT-scans were investigated through volumetric image correlation analysis to acquire full field volumetric strain information. If this is successful, this information could be used to draw conclusions to the internal material behavior and failure and a correlation with the specific acoustic signal.

C. Bauer^{1a}, T. Rief^{1b}, J. Hausmann^{1c} and T. Schalk^{2d}

2. Material and methods

The following sections describe the materials used for the investigations as well as the specimens and the testing methods.

2.1. Specimens

The material investigated is a short fiber reinforced polyamide 66 containing 30 weight percent of glass fibers. The geometry of the specimens is defined in DIN EN ISO 527-2 [7] with type 1A (direct molded specimen) used for the static and fatigue tests and type 1BA (cut out of an injection molded plate according to DIN EN ISO 294-5 [8]) for the μ CT-tensile testing. Conditioning of the specimens took place at room climate.

The μ CT-test-specimen additionally contains a hole to specify the area of failure, as the volume for the CT scan is limited by optical as well as geometrical factors. The parameter of the hole are set according to ASTM D5766 [9]. This standard sets the diameter of the hole as one sixth of the width of the specimen. For the given width of 5 mm the diameter of the drilled hole was 0.8 mm.

2.2. Static tensile tests

The static tensile tests were conducted according to regulations in DIN EN ISO 527-1 [10] on an *Electroforce 3550* from *TA Instruments*. The testing speed is set to 2 mm/min and strain measurements were recorded with a *gom ARAMIS* optical deformation analysis system. The measurement system uses a speckle pattern on the specimen surface for digital image correlation and calculation of the strain values, thus offering a full field strain computation on the surface. A total of five valid tests to the standard [10] were conducted at room temperature. For the recording of acoustic events a *Vallen AMSY-5* system was used.

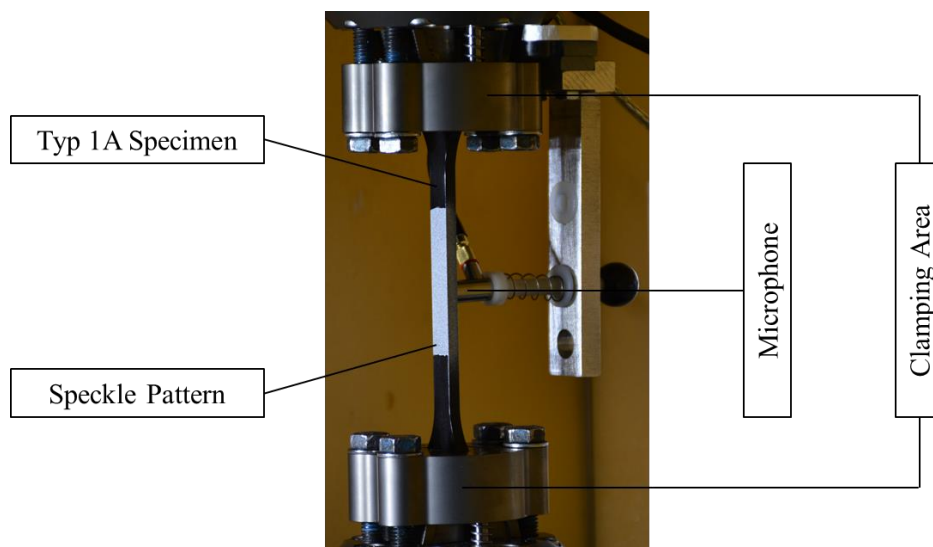


Figure 1. Static tensile test setup with acoustic emission recording

2.3. Fatigue tests

Fatigue tests were conducted using the same test setup as shown in Figure 1. The guidelines for the cyclic testing are provided by ISO 13003:2003 [11] and testing was conducted according to the standard with type 1A specimens. By means of consistent environmental conditions, testing took place in a temperature chamber. Additionally the specimens surface temperature is controlled with a thermal

camera system *OPTRIS PI400*. The maximum change in surface temperature of the specimen is limited to 10°C [11]. The tension-tension fatigue tests were performed using a stress ratio *R* of 0.1 at a variable frequency. Acoustic emission could not be recorded during the fatigue tests due to the noises of the testing machine during cyclic testing at higher frequencies.

2.4. In-situ CT test setup

For in-situ μ CT testing a test rig was developed to meet the requirements of the μ CT as well as the testing conditions. Limitations are given by the space in the μ CT. The test setup as construction and setup in the μ CT are shown in Figure 2.

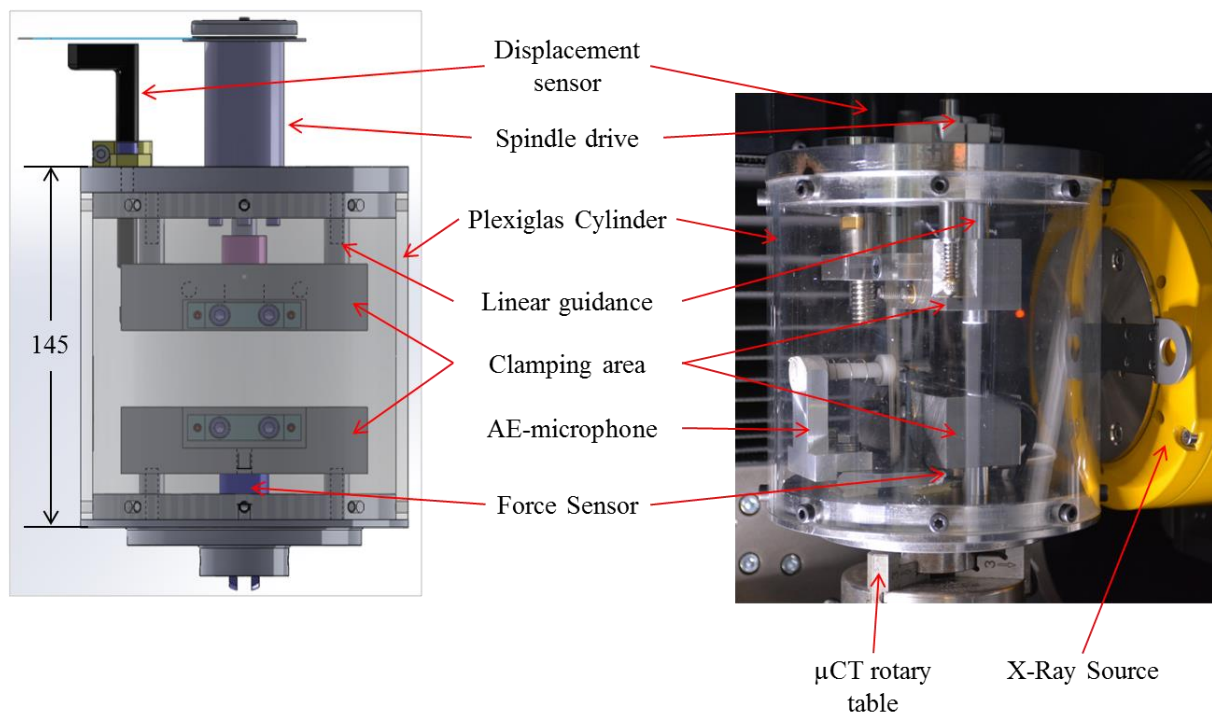


Figure 2. μ CT Test rig: construction (on the left) and test setup in the *Phoenix μ CT* (on the right side)

The test rig has to be free of metallic components in the central area to avoid measurement errors. As central part for the force transmission, a PMMA cylinder is used with the upper and lower part of the test rig mounted to it. This setup leaves the central area of interest, where the polymeric test specimen will be mounted, free of metallic components and thus applicable for the μ CT scans. The upper part of the test rig contains the spindle drive as well as the displacement sensor. With the linear guidance, a straight movement of the clamping area and in consequence of the specimen is given. At the bottom, the force sensor (2000 N maximum force) is mounted. At the right side of Figure 2 the test rig is shown mounted on the rotary table inside the μ CT and close to the x-ray source. Additionally, the setup features a microphone for recording acoustic emissions. The μ CT used for the investigation is a *Phoenix Nanotom 180NF* from *General Electric*.

The experimental procedure is conducted with a type 1BA specimen [7] with a hole in the area of interest (according to specifications in [9]) at a test speed of 0.005 mm/s. The testing is interrupted when significant acoustic events are recorded and the specimen held in place until a constant force is reached (the force drops due to creeping of the material). Then the μ CT scan is started with measurements each 0.2° at a resolution of 10 μ m per voxel.

2.5. CT-Strain computation

With the different μ CT-scans, a strain computation similarly to the strain measurements by digital image correlation can be conducted. The difference to strain computation through digital image correlation lies in the detail that not a speckle pattern applied to the surface is used, but the different contrasts inside the material (glass fibers and polymeric matrix). The computational software *VicVolume by Correlated Solutions* offers the possibility to calculate strains inside the scanned volumes. Therefore, a load change of the specimen is needed. Short fiber reinforced thermoplastics offer in this case automatically a random pattern of intensities, which is needed for successful digital image correlation.

3. Results and discussion

3.1. Acoustic behavior in static tensile tests and correlation to fatigue tests

The test results of the static tensile tests are shown in Figure 3. The graph shows the stress-strain-curves of the five valid test specimens in black with only low variations. For the acoustic events two different groups could be identified, one with frequencies of the acoustic waves between 50 – 100 Hz (light grey) and the other between 200 – 250 Hz (dark grey). The cumulated acoustic energy is noted in energy units (eu) a special *Vallen* unit due to the measurement setup. One eu equals 10^{-21} J for the actually used test setup. Both frequency groups show significant strain values for the first appearances of the acoustic events. The first group from 50 – 100 Hz shows a first significant acoustic event at 0.42 % strain with a standard deviation of 0.06 %. For the second group this strain lies at 0.78 % with a standard deviation of 0.14 %. Thus, the variation of the acoustic events is very low.

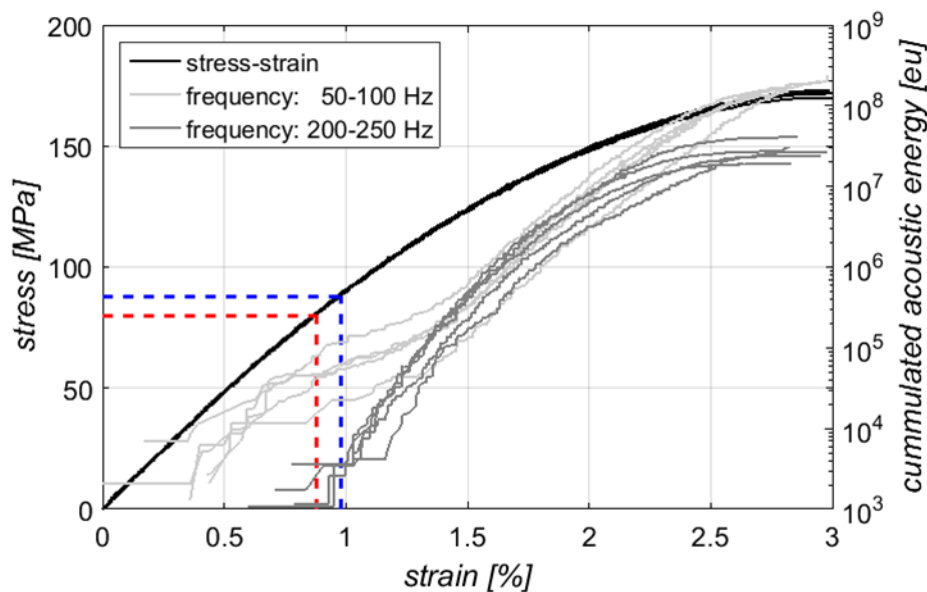


Figure 3. Stress-strain curve vs. cumulated acoustic energy in energy units (eu), 1 eu equals $1 \cdot 10^{-21}$ J

The results of the cyclic experiments are shown in Figure 4 for different stress levels as well as testing frequencies. Variations in test frequencies are given with respect to the testing standard which specifies that a heating up of the specimen due to the cyclic loading should not exceed 10 °C [11]. The maximum temperature difference during testing was 5 °C. For meeting these requirements, the test frequency was adjusted to 5 Hz at loads above 80 MPa, to 20 Hz at 80 MPa and to 45 Hz below 80 MPa. The recording of acoustic emissions was not possible due to noises of the testing machine at cyclic mode.

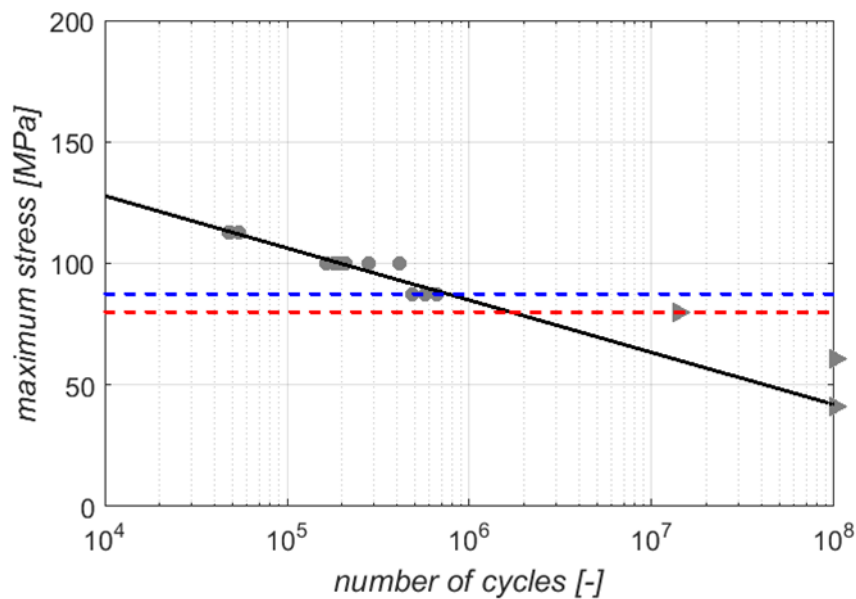


Figure 4. Cyclic testing results with load limit marked in red below second frequency group of the AE-signals and limit marked in blue for loads above; compare lines in Fig. 3 (triangles mark run-out specimens)

The first tests were performed at a maximum stress of 41 MPa which complies with the starting point of the first frequency group at a strain of 0.42 %. The second test was conducted at 61 MPa and a maximum strain of 0.6 %. In both cases no material failure after 10^8 cycles occurs. The third load level was chosen at 80 MPa and was still below the limit of 0.78 % where the second acoustic frequency group started (red lines in Fig. 3 and 4). This specimen also passed without failure, but for time reasons the test was aborted at $1.4 \cdot 10^7$ cycles. Above the limit for the second acoustic event, all specimens failed (marked with a dot) as shown at the blue line and above in Figure 4.

These results imply that with the acoustic events of the frequency group from 200 – 250 Hz major material defects start, which lead during cyclic loading to material failure. Below 80 MPa the material shows no fatigue failure up to 10^8 cycles.

3.2. Investigation of damage mechanisms by in-Situ- μ CT testing

As further investigation to acquire information about the damaging mechanisms at the different frequencies of the acoustic events a μ CT-test rig was set up with the microphone used with the static tensile tests (see also Figure 2 on the right side). Since it is hard to detect a material failure on microscale (matrix, matrix-fiber interface or fiber failure) in a full μ CT-scan the digital image correlation software *Vic-Volume* can be used to acquire information about regions with higher strains and thus of probable material defects. For this, a scan was conducted with no load applied to the specimen to give a reference for the following scans. In the beginning, testing was interrupted as soon as significant acoustic events were recorded. Further, on different load levels were investigated to see possible material failure.

The results of the static μ CT-tensile tests are presented in Figure 5, which shows the force-displacement-curve of the tested specimens as well as the different load levels a) to f) where μ CT scans were conducted. Results of the volumetric image correlation of these load levels at a cross section at 1.50 mm below the surface are shown for the vertical strain component. The scaling of the strain measurements is adjusted for the specific strain computations to see the main effects and differences of the loading inside the specimen.

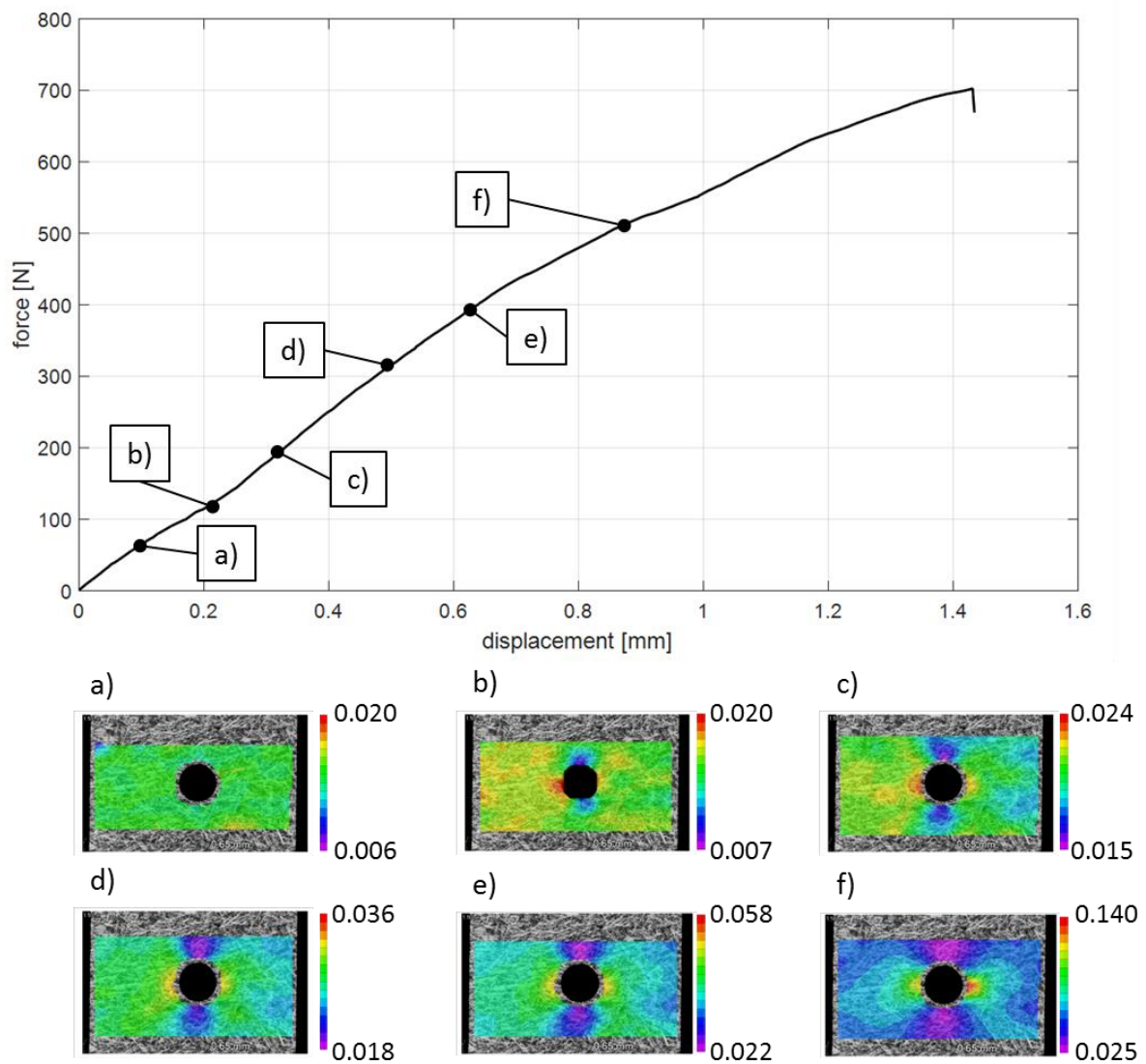


Figure 5. Force-displacement curve of the static μ CT-Tensile test with calculations of volumetric strains at different load levels a) - f) (vertical strain component displayed)

At point a) the tensile test was interrupted because a single acoustic event was detected. To see a possible source of the acoustic event a μ CT scan was conducted. Figure 5 a) shows a slice of the volumetric strain calculation. A correlation of the acoustic event to a special region of strain or deformation respectively was still not possible due to different reasons. For one reason the image shows only a part of the complete specimen volume, because of several limiting factors of the test setup as well as scanning specifications. Further reasons are (also due to geometrical and optical factors) the resolution of the CT-scans and measurement errors. The consecutive measurements b) to e) have the same problems but show the potential of the in-situ- μ CT testing with volumetric image correlation. A clear distribution of the strain can be observed with probable material failures. Measurement f) already shows a crack tip opening at the edge of the drilled hole where the strain reaches a maximum. A correlation of the acoustic events in the stadiums b) to f) was not possible due to a high range of acoustic signals of both observed frequency groups.

4. Conclusion

The presented work shows first correlations of the static behavior of a short fiber reinforced thermoplastic material to its fatigue behavior based on information of the acoustic emission of the specimens during static tensile tests. Two different groups of acoustic events could be observed which correlate with significant strain values where they start. The group of higher frequencies thereby has a direct correlation to the fatigue limits of the material. Loads below the limit imposed by this group are below the endurance limit of the material whereas loads above lead to fatigue failure.

Further investigations included in-situ μ CT tensile testing of a drilled hole specimen added with acoustic emission recording. The test setup shows high potential for investigations of internal material behavior and a correlation of the acoustic events although at this point no direct conclusions could be drawn. This should be possible with modifications to the test setup and further investigations.

Acknowledgements

The authors gratefully acknowledge ZF Friedrichshafen AG as well as Correlated Solutions INC, USA and isi-sys GmbH, Germany for the support and funding of this work.

References

- [1] P.J. de Groot, P.A. Wijnen, R.B. Janssen, Real-time frequency determination of acoustic emission for different fracture mechanisms in carbon/epoxy composites, *Composites Science and Technology*; 55:405–412, 1995.
- [2] R. Gutkin, C.J. Green, S. Vangrattanachai, S.T. Pinho, P. Robinson, P.T. Curtis, On acoustic emission for failure investigation in CFRP: Pattern recognition and peak frequency analyses, *Mechanical Systems and Signal Processing*; 25:1393–1407, 2011.
- [3] J. Karger-Kocsis, T. Czigány, Fracture behaviour of glass-fibre mat-reinforced structural nylon RIM composites studied by microscopic and acoustic emission techniques, *Journal of Materials Science*; 28:2438–2448, 1993.
- [4] A. Marec, J.-H. Thomas, R. El Guerjouma, Damage characterization of polymer-based composite materials: Multivariable analysis and wavelet transform for clustering acoustic emission data, *Mechanical Systems and Signal Processing*; 22:1441–1464, 2008.
- [5] D.S. Ivanov, S.V. Lomov, A.E. Bogdanovich, M. Karahan, I. Verpoest, A comparative study of tensile properties of non-crimp 3D orthogonal weave and multi-layer plain weave E-glass composites. Part 2: Comprehensive experimental results, *Composites Part A: Applied Science and Manufacturing*; 40:1144–1157, 2009.
- [6] S.V. Lomov, V. Carvelli, I. Verpoest, Correlations between damage initiation thresholds in textile composites and fatigue life limits, *Proceedings TexComp-10, 10th International Conference on Textile Composites*, 2010.
- [7] DIN EN ISO 572-2, Plastics - Determination of tensile properties - Part 2: Test conditions for moulding and extrusion plastics, Deutsche Norm; 83.080.01, sixth ed., Beuth Verlag (2012).
- [8] DIN EN ISO 294-5, Plastics - Injection moulding of test specimens of thermoplastic materials - Part 5: Preparation of standard specimens for investigating anisotropy, Deutsche Norm, fourth ed., Beuth Verlag (2013).
- [9] ASTM D 5766, Standard Test Method for Open-Hole Tensile Strength of Polymer Matrix Composite Laminates, eleventh ed., ASTM International.
- [10] DIN EN ISO 572-1, Plastics - Determination of tensile properties - Part 1: General Principles, Deutsche Norm; 83.080.01, sixth ed., Beuth Verlag (2012).
- [11] ISO 13003:2003, Fibre-reinforced plastics - Determination of fatigue properties under cyclic loading conditions; 83.080.20, first ed. (2003).