Analysis of behavior of composite material during loading tests

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Abstract. Composites are widely used as a material suitable for construction applications, but the operating conditions can lead to reduction of materials properties, damage initiation and collapse of the structure, so it is necessary to monitor condition of components and thus prevent its catastrophic failure. The unidirectional glass fiber reinforced polymer matrix composite (GFRP) was inspected with usage of non-destructive (NDT) acoustic emission during the static loading tests. The 0° test specimens were manufactured and tested under tensile and three-point flexural load for which the custom-made apparatus was used. Failure mechanisms were inspected by detailed analysis using scanning electron microscopy (SEM) to deduce the damage sequence. Tensile stress-initiated matrix cracking which induces formation of scars, ribbons and riverlines followed by delamination and fiber failure with formation of radials. Flexural load showed cracks in matrix, delamination and typical compression regions in form of fiber microbuckling. Based on the speed of wave propagation and linear localization, damage processes were detected in form of lateral cracking, delamination and integrity loss during the tensile and flexural tests. The data obtained from mechanical testing were correlated with selected acoustic emission parameters (counts, events, amplitude and duration). Behavior of GFRP material during the tensile test can be described very well with usage of counts while the flexural test provides much less information. The evaluation of damage processes using event parameter based on the amplitude and duration proved to be problematic due to the noise interference. The Short-time Fourier transform (ST-FFT) and peak frequency was used for identification of failure modes. In case of tensile and flexural load the matrix cracking has lower frequencies while delamination and fiber failure have higher values.

1. Introduction

Nowadays the application of fiber reinforced polymer composite materials (FRP) is widespread in various industry as suitable material for construction. Due to good mechanical properties (strength, stiffness), low density and corrosion resistance, its usage is mainly in aerospace, aviation, automotive and military [1, 2]. Due to complex nature of composite material, it is difficult to detect damage modes inside the material. The resolution power of many common NDT technique used for monitoring of damage progress is limited, because they can only detect the defect of a certain dimension [3]. The acoustic emission method does not have this limitation and thus is more suitable for damage analysis than other techniques.
1.1. Acoustic emission

The acoustic emission (AE) is NDT method for materials testing which have great impact in the area of real-time in-situ health structure monitoring. This technique measures and analyzes elastic stress waves generated by formation or propagation of defect. Ability to detect and localize defect inside the material helps to secure the safety of construction parts during operation and extends their lifetime, since many defects are irreversible [4]. Most of the defects are of outer or inner character (on the surface or inside the material) or they might be present at difficult-to-access places so it is often difficult or impossible to use other common NDT methods like ultrasound or X-ray [5]. The common approach for evaluation of damage processes inside the polymer composite material is usage of acoustic emission parameters, such as events, counts or energy [6–8]. As many authors have stated, the increase of cumulative values or change in their activity is tied to damage mechanisms initiated in material due to the applied stress. For example, at the beginning of a load, during the matrix cracking, the overall increase of signal activity is small because less energy is needed for crack growth and propagation. The amplitude is usually low as well while fiber related damage processes have higher amplitude and signal activity, since a larger amount of energy is needed [9]. Popular approach is waveform analysis [10] where during the matrix cracking the amplitude is lower and duration time is also shorter, while for fiber damage the amplitude and duration time is higher [4]. The peak frequency of an AE signal is another popular technique for signal classification. It defines specific range of frequencies for each of damage modes. [7,11,12] The efficiency of acoustic emission is based on sensitivity to numerical methods used for evaluation of measured data. Many of transition AE signals that exists inside the materials structure are not tied to real defects but to other types of processes i.e. background noise. It is necessary to determine the characteristics which will differ the sought phenomenon from undesirable one. For example, crack is indicated by medium or higher value of amplitude with higher duration (based on type of material) while burst signal with lower than three counts and low duration is undesirable noise. [9,13]

The aim of this paper is to perform fast AE damage analysis without using the advanced techniques with correlation of events based on the amplitude distribution and duration, which is supported for comparison by standard evaluation techniques.

2. Methods

2.1. Material

The unidirectional (UD) reinforced epoxy composite was used for tensile and three-point flexural tests. Unsaturated polyester resin was reinforced with E-glass fibers with diameter range of 12-20 µm. The test specimens were cut from 1.5x1.5 m composite plate with variable thickness of 6 mm. The plate was manufactured by hand lay-up method. The vacuum was applied to spread the resin throughout the fiber. The curing process of resin was done at ambient temperature. The quality control and determination of volume fiber fraction was done according to standard ČSN 64 4002 and then compared with image analysis made by the analytic software NIS Elements AR and light microscope Carl Zeiss Neophot 32. The volume fraction of fibers was determined to be 45 ±3% and matrix porosity in form of voids and bubbles is 3.42 ±0.5%. During the image analysis (Figure 1), the irregular distribution of fibers in matrix was found in form of resin-rich areas which may potentially act as a site for damage initiation.

![GFRP microstructure (etched by hydrofluoric acid)](image-url)
2.2. Mechanical testing

The unidirectional reinforced test specimens with 0° fiber (fibers are parallel to the tensile load) orientation were manufactured in accordance with ASTM D3039 [14] and ASTM 7264 [15] for each type of test with dimension of tensile test specimens 200 × 20 × 6.5 mm and 85 × 20 × 6 mm for flexural testing. Tabbing material was not applied on the tensile specimen ends because it was not possible to ensure the efficient adhesive and the tabs tended to debond during the tensile test. The thickness of the specimen could not be changed, so the specimens remained without the tabs. Tensile test was carried out on 200 kN static and 100 kN dynamic universal testing machine Instron 1273 with crosshead displacement rate set to 0.5 mm·min⁻¹ at temperature 19.2 °C and 30% R.H. For three-point flexural test, the custom-made apparatus was used (see Figure 2), where movement of transducer ABM U9B is realized by rotation of M5 thread. M5 is set to motion with pair of meshing gears where the pinion is mounted on the shafts output. By stepper engine rotation, the gear and pinion also rotate and simultaneously sets in motion the M5 thread with transducer ABM U9B which acts as a loading element and simultaneously as a measuring load force. Deflection is measured by LVDT E-300 transducer mounted opposite to ABM transducer. To control the apparatus the Data Acquisition/Control Unit HP 3852A was used together with set of digits integrating voltmeter HP 44701A, analogue output arbitrary waveform DAC HP 44726A and HP Agilent 44709A. In the development environment LabWindows™/CVI, the software was written for data acquisition and apparatus control. The displacement rate was set to 0.7 mm·min⁻¹ (equal to 1 rpm) and span to 63 mm. The test was performed at temperature 22.7 °C and 36% R.H. (relative humidity).

Figure 2. The apparatus scheme for three-point flexural test together with control detail.

2.3. Acoustic Emission

The acoustic signal was being monitored during the mechanical tests by PCI-2 based AE system from Physical Acoustic Corporation (PAC) with pair of differential wideband sensors (100-900 Hz). Each sensor was connected to EPA 1220A preamplifiers with 40 dB gain and mounted on the surface of testing specimens (see Figure 3) with M Bond 200 adhesive kit in the distance of 60 mm for tensile specimen and 40 mm for flexural specimen. For a noise removal the signal passed 20 kHz low-pass filter and 1000 kHz high-pass filter. The threshold type was fixed with value of 30 dB and sample rate was set to 4 MSPS. The AE signal was analyzed by using the following parameters: cumulative counts, amplitude, events and duration time. Analyzing the waveform, the frequency spectra was obtained.
Figure 3. The arrangement of AE sensors: (a) Tensile test, (b) Flexural test.

For the linear localization of AE signal, the velocity of waves spreading through the testing specimens was measured by Krautkamer USM 25 Ultrasonic Flaw Detector equipped with dual 2 MHz SEB-2 KF5 probe. Results were compared to the Hsu-Nielsen pencil lead break test which was performed by breaking the 2H Ø0.5 mm lead on the surface of testing specimens to detect acoustic emission signal. Based on the difference in time of arrival, the wave velocity was calculated. The elastic wave propagates in GFRP material with velocity of $4500 \text{ m/s}$ for both types of testing specimens. The data correlation with tensile test was used to identify the failure sources.

2.4. Fractography

The failure mode after tensile and flexural test was evaluated according to ASTM standards and described as three-part failure code. The fracture surfaces were sputtered by chromium evaporation using vacuum evaporator JEOL JEE-4X with current set to 50 A. Scanning electron microscope (SEM) JEOL JSM-7600F has been used for damage process evaluation. Ultra-low voltage gentle beam (GB-low) method with acceleration voltage set to 1 kV and secondary electrons (SEI) were used for fracture surface observation.

3. Results and discussion

3.1. Mechanical testing

Two main failure mechanisms can be observed by naked eye after tensile test. The first are lateral cracks on the surface layer (gelcoat) accompanied by smaller capillary lateral cracks (see Figure 4a). Cracks are under approx. 45° which corresponds to shear stress component, where the highest stress value is under 45°. Lateral cracks are followed by the delamination which initiates in the area of AE sensor and propagates through the grip area (see Figure 4b) towards the specimen edge. The final code was determined as M(ALG)MV where multiple failure (M) type was found in form of angled (A), lateral (L) and grip (G). Failure area was also evaluated as multiple (M) with various (V) location.

For three-point flexural test the failure occurred under the loading pin. Figure 4b shows that lateral cracks appeared on the surface layer before the main failure initiation. In this case, the code is M(TB)AV, where the failure mode is multi-mode (M) with tension (T) and compression (C) with failure area at loading nose (A) and various failure location (V). The ultimate tensile strength is 390 MPa, while flexural strength is 224 MPa.
3.2. Fractographic analysis

Both fiber and matrix dominant fractographic features were observed on fracture surfaces and some of them are similar for both tensile (Figure 5c-f) and flexural load (Figure 5a-b), e.g. fiber fracture or matrix microflow. The axial tension led to initiation of intralaminar failure mechanisms in form of matrix cracking in the resin-rich areas. The interaction of multiple cracks leads to microflow of matrix and formation of fractographic features, such as scarp, ribbon and riverlines.

Figure 5. SEM fractograph of flexural and tensile test failure: (a) Microbuckling due to shear stress, (b) Fiber failure, (c) Matrix microflow, (d) Brittle fracture and fiber related failure, (e) Matrix failure mechanism at fiber imprint area, (f) Matrix failure mechanism at resin rich area.
Scarp formation (Figure 5c) not only shows that matrix has less stiffness, but their interconnection creates riverlines (Figure 5f) that can be used for determination of crack propagation. Scarps were also detected next to fiber imprints (Figure 5e) which indicates that crack reached the matrix/fiber interface followed by initiation of shear stress at the interface, this is confirmed by presence of riverlines. The result is either separation of fiber from matrix/debonding (Figure 5b), fiber pull-out (Figure 5d) or brittle fracture and formation of radials (Figure 5d). The fiber pull-outs with fiber fracture were observed infrequently, while the formation of radials is dominant morphological character, which corresponds to failure of 0° fibers. The presence of high amount of fiber imprint and scarps was also observed (Figure 5e) in the case of delamination failure.

Both tension a compression regions can be observed on the fiber fracture surface in case of flexural load. It is a result of fiber buckling due to induced compression stress where the side support of matrix is depleted, and fiber failed by microbuckling (Figure 5a-b). Since the matrix has low resistance in compression, the cracks initiate first then followed by the fiber dominant failure in a form of fiber fracture and microbuckling. Matrix has brittle behavior in flexural load as well which is confirmed by presence of riverlines (Figure 5f).

3.3. Acoustic emission
3.3.1. Linear localization. Figure 6 illustrates the results of linear localization during mechanical tests. This technique usually only determines the 2D coordinates of acoustic emission signal source, but certain patterns can be deduced. During the tensile test, the lateral cracks under 45° are induced in the gelcoat layer. These cracks are evenly distributed on the surface and similar distribution can be seen in the graph (Figure 6a). The event rate is also increasing towards the edge, where the sensor is mounted with high peak of event rate around 50 mm. This probably indicates initiation and propagation of delamination which ends with integrity loss and failure of testing specimen. Similar approach can be used in case of flexural load (Figure 6b). Large Gaussian event rate is in the middle of monitored area (around 20 mm) with occasional peaks around. The specimen failure occurred in the middle of testing specimen which corresponds with high rate of events between the sensors.

![Figure 6](image)

**Figure 6.** The linear localization of AE source: (a) Tensile test, (b) Flexural test.

3.3.2. Counts. Figure 7 illustrates the activity of AE signal during the mechanical tests which in case of tensile test creates three separated areas (Figure 7a). The high activity of counts can be seen at the early stage of loading with corresponding sharp increase of cumulative values. The lateral cracks and smaller lateral capillary cracks in surface layer appeared almost at the beginning of test and this type of damage process and can be assigned to the first area up to 7 kN in similar way as stated in [7]. This is followed by decrease of count rate and low signal activity with occasional peak increase and slight increase can be seen in case of cumulative counts and there is no change in profile till reaching the value of 11 kN. This corresponds to matrix cracks, since fractographic analysis showed that matrix cracks are induced at the beginning and need less energy to propagate. Final area begins with large increase in count rate together with sharp increase in cumulative values. Distribution of count rate is similar as it
was with events at linear localization. The fibers begin to break or are pulled-out. The slight load decrease at 44 kN indicates initiation of delamination and gradual loss of integrity, which corresponds with high count rate. It is similar to delamination initiation and parameter distribution in [9]. The activity drops together with load at 49 kN where fracture occurred.

![Figure 7](image.png)

**Figure 7.** The correlation of load and count parameter: (a) Tensile test, (b) Flexural test.

Completely different behavior can be seen in case of flexural test (Figure 7b) where cumulative counts have almost exponential type of profile. There are still two distinguishable areas. Low activity and slow increase in cumulative counts is sign of failure associated with matrix, since it occurred as first, while the change at 0.7 kN in cumulative counts and almost linear increase in signal activity indicates fiber failure. No decrease in load during the test was observed so it is difficult to distinguish other mechanism types.

### 3.3.3. Amplitude

Figure 8 shows correlation of acoustic emission events with amplitude and their classification according to the event duration. Based on the claim that shorter duration is associated with matrix damage and higher with fiber dominant, some amplitude bands can be created (see Figure 8a) for a tensile test. The damage associated with fiber can be found between 70-87 dB since the cluster is clearly distinguishable. Matrix damage however might either be in band of 60-70 dB and 87-100 dB (red color) or in 67-75 dB and 84-100 dB (orange). Second variant is more probable since the first one has too low duration and could be exchanged for noise. The amplitude distributions corresponds to Ref. [12]. The evaluation of flexural load is more difficult because there are no separated areas and distribution is completely different (Figure 8b). Red color can be again considered as noise and therefore the matrix failure (orange) is in band of 45-85 dB. It seems that in case of flexure, the damage associated with fiber initiate together with matrix at lower amplitude bands (40-50 dB) and can be also found at higher values (85 dB) which corresponds to amplitude distribution in Ref. [9].

![Figure 8](image.png)

**Figure 8.** The event rate based on Amplitude and event duration: (a) Tensile test, (b) Flexural test.
3.3.4. Peak frequencies. Decomposing waveforms by fast Fourier transform (FFT) and converting the time to frequency domain (Figure 9) is more effective method to identify the failure sources. The dominant frequencies at 50-150 kHz were observed at lower stresses for tensile test (Figure 9a). Therefore, low frequency events can be attributed to matrix-related mechanisms. Higher frequency belongs to interfacial debonding in form of delamination (200-300 kHz) while fibre damage is at 450 kHz. These frequencies assigned to damage mechanisms are in well accordance with Refs. [3,12,16].

![Figure 9. Peak frequencies: (a) Tensile test, (b) Flexural test.](image)

Similar approach can be used for flexural stress (Figure 9b). Highest frequency has matrix cracking at 50-100 kHz while fiber damage is at 450 kHz. No other damage type could be evaluated, since the rest of frequencies was below the threshold (lower than 5 %).

4. Conclusion
The UD testing specimens with 0° fiber orientation were tested under tensile and flexural load. Good results were obtained from flexural test with usage of custom-made apparatus so it can be further used and modified for acoustic emission analysis. Fiber and matrix damage processes were identified by SEM. From the observed fractographic features it was possible to deduce the sequence of damage in the composite microstructure during the loading tests. Linear localization provided not only coordinates for identification of acoustic emission sources, but it was also possible to assign lateral cracks in surface layer to event rate activity for both type of stresses. Based on the events distribution, the delamination at tensile failure could be also identified. Good data correlation was observed for damage mechanisms in case of tensile test. Matrix cracking, delamination and fiber related failure can be detected with counts and evaluated based on the rate and cumulative values. However less information was obtained from the flexural load where only areas related to matrix and fiber damage process were identified and thus no detailed information about specific damage processes could be determined. Amplitude and peak frequency distributions also provided another possibility to identify the failure sources. The evaluation of amplitude bands proved to be problematic. The assignment of matrix cracking to events based on the duration can be exchanged for background noise while fiber dominant failure is easier to identify since event duration is much higher. The distribution is completely different for tensile and flexural stress and it is difficult to determine correct amplitude distribution, so this approach seems to be less reliable. FFT analysis supported the identification of the failure mechanisms with easier distinguishing of damage processes. Matrix cracking has low frequency, while delamination and fiber related damage have higher values. The investigation showed that acoustic emission is suitable method for detection and identification of damage and failure mechanisms in UD GFRP composite, but it is necessary to choose appropriate parameters for data correlation. Higher attention to background noise should be paid since it can be easily confused with event related to damage process.
To improve damage detection by AE the following research will focus on usage of other signal parameters, like absolute energy and RMS (Root Mean Square). In addition to the correlation with load, the parameters will also be correlated with deformation. Further attention will be given to the waveform analysis using Continuous Wavelet Transform (CWT), Fourier Transform (FT) or Discrete Wavelet Transform (DWT).

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5. References
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