

Indication of the Destruction Stage of Epoxy PES-Filled Composite Using the Connected Parameter of Acoustic Emission Rate and the Spectral Characteristics of AE Signals

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Abstract – The methods of non-destructive testing have an important role in the problem of maintenance of transport and transport infrastructure. They make possible the early detection of internal defects in engineering structures and elements of vehicle design. The samples of composite materials used in the construction of transport infrastructure elements and transport vehicles have been tested. This paper shows the features of AE, which may allow more efficient and accurate analysis of the state of composite objects. The relation of the intensity and frequency characteristics of signals has been revealed. This work discusses the possibility of unambiguous identification of the stage of composite object destruction on the basis of these parameters.

Keywords – Acoustic emission method, non-destructive testing, multiparameter acoustic emission analysis, polyethersulfone composite.

I. INTRODUCTION

Acoustic Emission (AE) method belongs to the family of non-destructive testing methods and is used to detect defects at the early stages of their development. The method is used to detect and locate defects in loaded structures and components. AE carries information about the origin of dislocations in the material and makes it possible to monitor the development of internal and external cracks. Comparing the possibility of AE and other NDT methods, the following differences can be identified. AE can monitor changes in material behavior over a long time and without moving any of its components, i.e. sensors. This makes the technique quite unique along with the ability to detect crack propagation occurring not only on the surface but also deep inside the material [1]. Before applying the AE method in practice, it is necessary to select some specific criteria for the control of the physical processes, which are planned to be assessed by using AE.

The existing parameters and methods of acoustic emission assessment include signal waveform processing. Being digitally recorded, a single AE-event can provide valuable information about the material being tested. All AE features can be derived from signal amplitude, frequency and time properties. The following amplitude properties of an AE event bring information about physical processes in materials: attack, decay, sustain and release [2]. The frequency features of an AE signal also carry information about the process of destruction. Increase in the amplitude of resonant frequencies can be used to determine the severity of the defect [3]. Changes in frequency over distance show such material properties as the presence of inhomogeneities, cavities, grains, as well as elastic features.

The rate of AE flow over time is another criterion of structure assessment. AE-event flow in time domain representation is more informative. Some earlier studies conducted in 1970–1990s showed the relation between the number of acoustic emission intensity and structural damage under cyclic loading [4]–[6] This criterion was regarded as a useful scalar measure of damage.

In the same years, a diagnostic parameter proposed by the researchers in Riga was introduced [7] and successfully proved [8]–[10] as an informative technical assessment indicator. At the moment of crack initiation in the material, the speed of AE signal accumulation begins to increase sharply. This leads to a jumplike increase in the tangent to the graph depending on the total count of acoustic emission in time. Previous researchers call this angle α -criterion of crack appearance (Fig. 1).

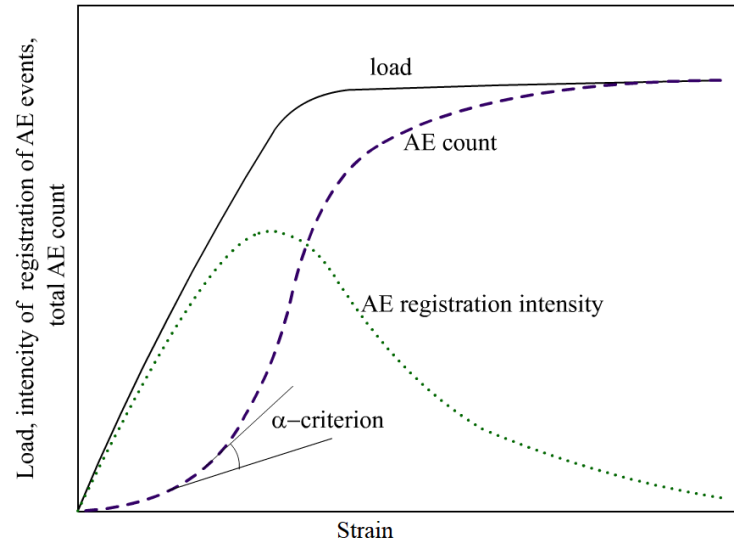


Fig. 1. Criterion for crack initiation in the diagram of cumulative AE.

It is difficult to define this criterion, because its appearance is not usually preceded by the horizontal sections of total AE. Therefore, it is more convenient to determine the time of α -criterion occurrence through the second time derivative of the total registered AE. Also, the complexity of determining α -criterion is a consequence of the discrete nature of the flow of AE events. It is impossible to accurately determine the exact instant angle of the tangent to the graph of the accumulated amount of AE. Another difficulty in using the α -test is the absence of strictly expressed linear segments on the graph of cumulative AE (Fig. 1). Because of this it is hard to find the time of occurrence of a fatigue crack. Despite the constraint, α -test is an informative parameter for the assessment of the status of materials and components, which has been successfully used in laboratory tests.

Recent AE systems are powerful enough to perform AE signal waveform recording and analysis in real-time. Analysis in frequency domain can reveal the characteristics of signals and systems which are useful in dealing with systems [11]. The frequency analysis is mostly used in composite material fracture studies: failure mode of composite assessment [12], glass/polypropylene composites [13], fracture monitoring of lightweight composite-concrete beams [14] and so on. There are known some successful attempts to use AE spectral analysis for the monitoring of machinery parts, ground and underwater structures, for example, the state of rolling bearings [15], classification of leakages in water pipes [16], recognition of fracture signature of underwater synthetic mooring [17]. The listed works consider AE frequency feature as a unique, yet static fingerprint of fracturing process.

Research Objectives:

- to determine the behavior of dynamic (α) – AE criterion in composite material for different epoxy composite PES-filler concentrations;
- to determine the spectral characteristics of AE signals in composites being destroyed.

II. MATERIALS AND METHODS

This research shows the possibility of processing different AE parameters to define composite material destruction phase under a loading program. The term “composite” in terms of composite

materials mean two or more materials combined at a macroscopic level in the form of the third material with new and useful properties. Generally, not all of these properties can be simultaneously improved in a composite. Some of the properties may conflict with each other, for example, insulating properties and thermal conductivity properties. An example of such deterioration in the material properties is contained in the study of damage to the 7-layer fiber composite conducted by the author of this paper [18]. The airplane RRJ elevator experienced artificial lightning bolt damage. The replacement of this unit with a multilayer fiber-reinforced composite increased the quality of reliability and reduced the weight of the product, but at the same time, the destruction left by lightning was significant.

In land transport, there exist examples of composite parts that have a direct impact on the safety of trains. These are fairings of diesel multiple units (DMU unit), axle boxes, transmission box covers for diesel and electric locomotives. Among the circumstances preventing the spread of the use of composites in the construction of road infrastructure, the researchers mention the lack of simple and reliable methods for the inspection of composite objects and procedures for their repair (RGS Europa, 2014). These arguments motivated the choice of material for tensile testing and method development.

A. Material Characteristics

The study used the following materials: epoxy filled composition (epoxy resin Araldite® LY 1564 / Aradur® 3486) with different filler content. Filling: polyethersulphone brand Ultrason® E 2020 P SR micro (PES). The investigated epoxy resin composition has a 12.5 % content of PES weighing 0.0 %, 5.0 %, 7.5 %, 10.0 %, and is obtained by mixing in a conventional mixer at room temperature for 30 minutes and cured in accordance with the rules for 4 hours at a temperature of 100 °C

B. Samples and Setup

To determine the AE characteristics of composite structures, it is important to determine these for the source material. During the testing of epoxy composite PES-filling samples, the method of stretching the sample to break was used. The tests involved the use of 30 composite samples sized 2 mm × 10 mm × 150 mm with varying concentrations of PES (0.0 %, 5.0 %, 7.5 %, 10 % and 12.5 %). The control of the samples was carried out on a tensile testing machine Zwick / Roell 2.5 until the destruction of a sample. An AE sensor was installed on each of the test specimens with the help of tightening clamps (Fig. 2). The following parameters were measured: stress, strain, time to failure and total acoustic emission.

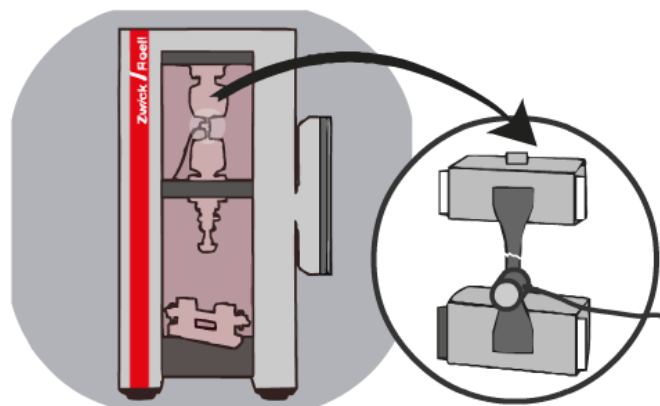


Fig. 2. Schematic attachment of AE sensors to “dogbone” epoxy samples in the testing machine.

The monitoring was carried out for the samples in an amount from 5 to 7 at each concentration of filler. Preparations for the AE control: before the actual registration of AE signals, AE tract

calibration was performed in order to maximize the sensitivity of the equipment and reduce extraneous noise. The calibration was performed using test AE signals from the breaking of pencil lead with hardness T , diameter 0.5 mm – SU-Nielsen method [19].

C. Testing Procedure

The tensile tests were performed on the test machine, Zwick / Roell 2.5, using a maximum load of 2.5 kN. The tensile test was chosen due to the uniformity of stress distribution across the sample section. The testing machine crosshead speed was 2 mm/min. Before the test, an AE sensor (model R6-ALPHA) by Mistras company was attached to the sample. MICRO-II AE device by Mistras company was used for measuring the acoustic signals. Before testing, the samples were conditioned at a temperature of $+22\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ and a relative humidity of 50 %. During the tests, the values of the applied load, specimen deformation and AE signals were recorded.

In all cases, the duration of the experiment was different. Besides, the elongation and load at the moment of sample rupture was different as well. This made it difficult to compare the dynamics of AE pulse emergence between both the groups of samples with different concentrations of PES and the samples of the same group. To solve this problem it was proposed to shift from the coordinate system with absolute time to the relative coordinate system (normalization) in the time domain where 0.0 is the beginning of the experiment, and 1.0 is the time of sample fracture. Also, load and elongation data from the computer of ZWIG / Roel 2.5 testing machine were subject to normalization.

D. Data Normalization

Each experiment in each composite sample group had a unique time of occurrence of sample rupture, as well as a unique score of AE at break. Conducting a comparative analysis of such experiments without reduction to a single scale is quite difficult. In order to solve this problem, there was proposed a normalization method by which all time and AE count data were re-calculated from absolute values to relative ones. On the time axis and total AE axis, the beginning of the experiment was marked with 0 and the end of the experiment (sample break) was marked with 1. The result for the 12.5 % PES sample is shown in Fig. 3.

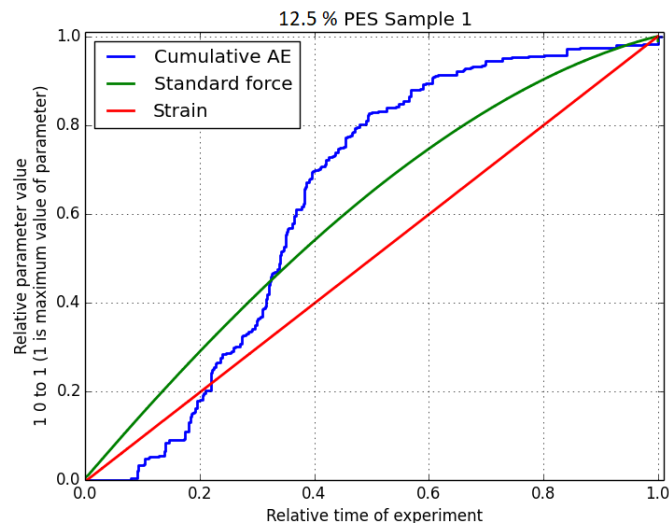


Fig. 3. Normalized representation of experimental data for sample No. 1 at 12.5 % PES Sample 1 concentration. Time moment 1.0 corresponds to sample breakdown.

The normalization is carried out as follows: an array of data with the readout time of AE signal registration are represented by vector t_x ,

$$t_x = \langle t_{x,0}, t_{x,1}, \dots, t_{x,n-2}, t_{x,n-1} \rangle, \quad (1)$$

where

- x serial number of experiment in a group;
- n number of registered AE count changes.

Each value of t_x vector corresponds to the value of c_x vector (cumulative count) which determines the cumulative AE count at the moment in time.

$$c_x = \langle c_{x,0}, c_{x,1}, \dots, c_{x,n-2}, c_{x,n-1} \rangle, \quad (2)$$

where

- n number of registered AE count changes;
- x serial number of experiment in group;
- $c_{x,i}$ cumulative AE count value at the time moment $t_{x,i}$.

The normalized values of time are determined by vector t_x^{norm}

$$t_x^{\text{norm}} = \frac{t_x}{t_{x \max}}, \quad (3)$$

where

- x serial number of experiment in a group;
- t_x vector of time values before normalization.

The normalized cumulative AE count values are determined by vector c_x^{norm}

$$c_x^{\text{norm}} = \frac{c_x}{c_{x \max}}, \quad (4)$$

where

- x serial number of experiment in a group;
- c_x cumulative AE count value before normalization.

Thus, after performing the normalization, the time and average AE count values for all experiments were represented in relative coordinates (Fig. 3).

E. Determination of α_1 and α_2 by Using the Smoothing Function

The fracture criterion (α -test) is determined by the sum of positive increments of AE curve, which has a bend at some point. The increment represents the change in the dynamics of summed AE signals. Due to the lack of expressed linear areas on the graph of total AE, it is difficult to use the α -test for the detection of the moment of occurrence of a fatigue crack. In this connection, the second derivative of the total of registered AE becomes a convenient indicator of the occurrence of a fatigue crack.

When calculating the dependence of the second derivative on the operating time by using direct differentiation in certain time intervals, the high sensitivity of this criterion to the change of total AE dynamics provides a large number of sign change areas at the stage of fatigue damage accumulation.

To solve this problem it was proposed to introduce the element of inertia in the graph of total AE by the smoothing method. A smoothed normalized vector of total AE values c'_x is generally calculated as follows:

$$c'_{x,j} = \frac{\sum_{i=j}^{k+j} c_{x,i}^{\text{norm}}}{k},$$

$$c'_x = \langle c'_{x,j} \rangle; j = \langle 0, 1, \dots, n - k - 1 \rangle, \quad (5)$$

where

- k number of elements of smoothing window;
- n number of registered AE count changes;
- x serial number of experiment;
- $c_{x,i}^{\text{norm}}$ value of cumulative AE in x^{th} experiment at the time moment $t_{x,i}$.

After the smoothing of input vector of acoustic emission count, resulting vector c'_x with a length of n elements is sent to the input of digital differentiation function helping to calculate a vector with a length of $n - 2$, which is a derivative of the second order along the time axis

$$\alpha_x = \frac{\partial^2 c'_x}{\partial t^2}, \quad (6)$$

where α_x resulting vector.

Resulting vector α_x is also sent to the input of the smoothing function described above, and based on the result a graphical representation of the second derivative, the total AE, elongation and stress are built in the system of relative coordinates.

Fig. 4 shows the result of data processing in the experiment for the composite sample containing PES 12.5 %. The dotted line indicates 4 expressed zones of rise and fall for the second derivative of the total AE. At the same time a smoothing function with a window of 6 % of the duration of the experiment was applied to both the cumulative AE and its second derivative. The beginning of the first zone rise corresponds to the beginning of AE accumulation. The subsequent recession and beginning of the second zone rise corresponds to α_1 . The joint between the second and the third zones indicates α_2 and finally, the last rise coincides with the achievement of the ultimate strength. Similarly, the data have been processed and the described 2nd derivative behavior was confirmed for all other experiments on the composite containing PES 5.0 %, 7.5 %, 10.0 %, 12.5 %. The control samples of pure epoxy with 0 % concentrations did not show the described behavior. Thus, the problem of the complexity of interpretation of the amount of AE increase rate was solved and this made the visualization of α -criteria a more informative and easier to read indicator of the developmental stages of PES-filled epoxy composite destruction.

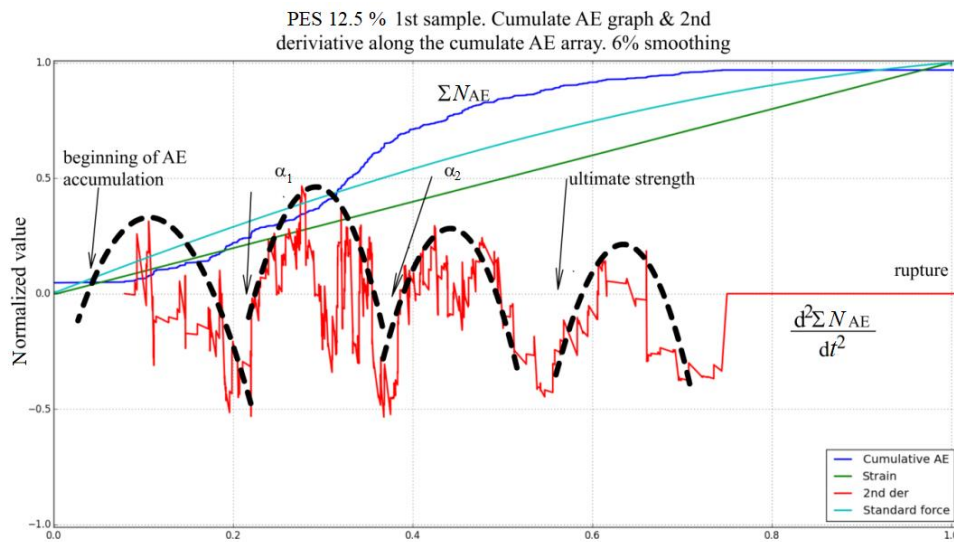


Fig. 4. The total AE and second derivative of smoothed total AE array obtained by direct differentiation.

For each group with the same concentration of PES, there were calculated the average values of AE and tensile load in the time domain. In each of the groups of samples, there were defined α_1 and α_2 points. In addition to the analysis of AE as a stream of discrete events, there was also carried out an analysis of the spectral characteristics of AE pulses emitted by the material being destroyed. During the experiment, the waveforms of AE pulses were recorded, and then analyzed with the help

of software developed within this work. There were investigated: the spectral characteristics of individual events (Fig. 5) and distribution of the peak energy of frequency spectrum in the time domain (Fig. 6).

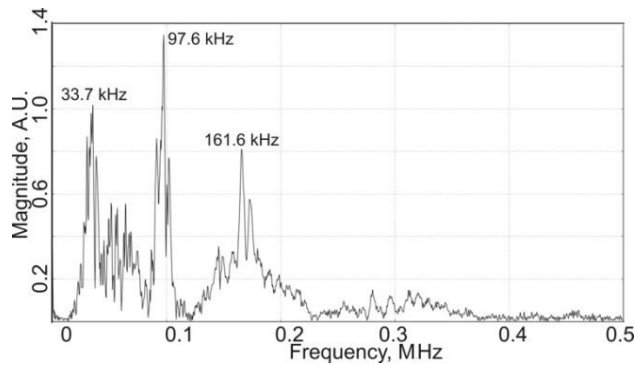


Fig. 5. Spectral density of a single AE-event in a crumbling PES-filled epoxy composite.

The frequency analysis of AE signals showed that for the samples with all tested concentrations of filler there are two characteristic AE broadband signals - the lower and the upper. The lower is lying in the frequency band from 37 kHz to 39 kHz, and the upper – from 97 kHz to 103 kHz. This suggests that in a matrix of this type of composite material the deterioration of adhesion presumably occurs for two characteristic geometric dimensions of drop cracks corresponding to the specified frequencies.

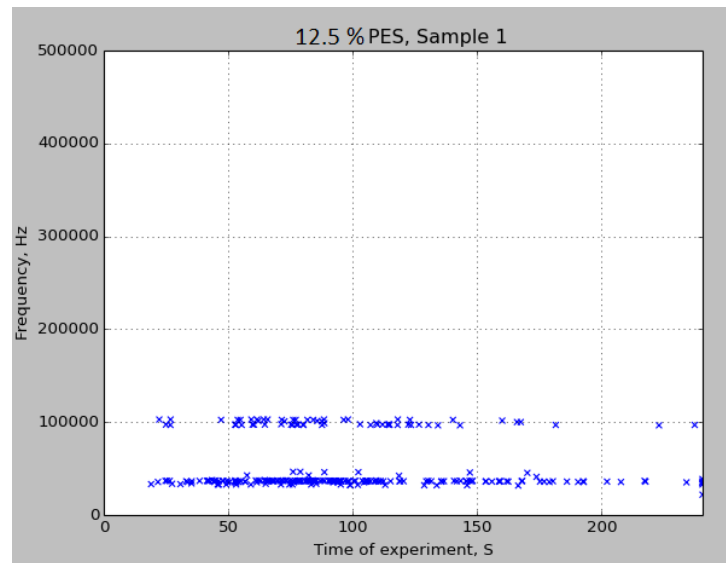


Fig. 6. Maximum energy distribution in the spectrum of frequencies of all events in the time coordinates.

F. Determining the Connection of α -Criterion With the Spectral Characteristics of AE Signals During the Destruction of Epoxy Composite

In each experiment on PES filled samples, there were observed two points – designated as α_1 and α_2 – characterized by an expressed increase in the rate of cumulative AE growth. The change in the peak frequency of AE was uneven. In order to determine the behavior of the maximum frequency a polynomial approximation function (fit-function) was used. The plot was drawn based on the time vector, showing the cumulative AE count during the time of the experiment (Fig. 7). Additionally, the data on stress, strain and elongation of composite samples were added to the plot.

Throughout the experiment, the frequency generally decreases for the full group of high and low frequencies, as well as separately for both groups. At the same time, after the occurrence of each of the two α -criteria, the behavior of frequency is changing. After the onset of α_1 , the fit-function changes from monotonically decreasing to monotonically increasing.

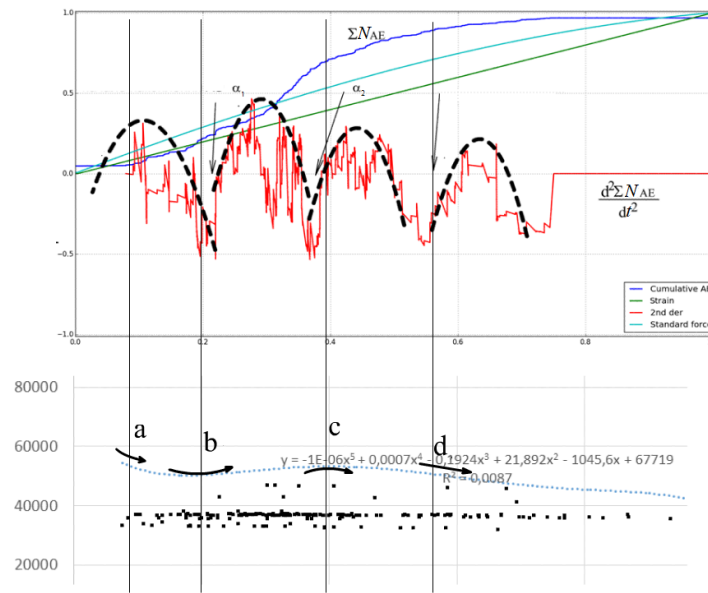


Fig. 7. Scheme of the compliance of 2nd derivative of total AE and the dynamics of the approximating function of the maximum frequency in the 1st experiment with the content of PES 12.5 %.

The values of the frequency of AE events reach their maximum values. Then, after the registration of a new package of active emission corresponding to α_2 , the fit-function again begins to decrease monotonically and reaches its minimum value just before the sample breaks. At the end of the experiment, it is already at σ yield point, when the sample is about to break, there is observed a short-term increase in frequency for the last time. Thus, four distinct phases of AE dynamics change look as shown in Fig. 7. Each stage is characterized by its own unique combination of two parameters: the dynamics of the 2nd derivative of the total AE and the dynamics of the maximum power spectral density. All observed phases are summarized in Table I.

TABLE I

RELATIONSHIP BETWEEN THE DYNAMICS OF AE SIGNAL PARAMETERS AND A DESTRUCTION STAGE

Stage characteristics	Behavior of the fit-functions of max. spectral density frequency	Behavior of the 2 nd derivative of the cumulative AE count	Stage marking
Start of AE accumulation	Decreases with flattening	Reduction changes to growth	a
α_1	The lower extreme. Decline changes to growth.	Reduction changes to growth	b
α_2	Growth changes to reduction	Reduction changes to growth	c
Ultimate strength achievement	Nearly uniform parameter reduction	Reduction changes to growth	d
Area in which 2 nd derivative reaches the local minimum, but the change of the form of cracking does not occur. Without the use of frequency setting section it may be mistakenly identified as an event in groups 1–4	Growth	Reduction changes to growth	Interval between b and c
All others intervals	Change of value	Function dynamics does not show the change of decrease to growth	All others intermediate intervals

In addition to the four stages of composite sample destruction associated with an obvious change of the second AE derivative from decrease to increase, most experiments on the samples have also revealed a phase which can be erroneously classified as a change of fracture type.

This step is in section b–c (Fig. 7). In order to eliminate the possibility of misclassification of this stage the frequency parameter is used.

III. RESULTS

The tensile tests conducted with the use of 30 composite samples with varying concentrations of PES (0.0 %, 5.0 %, 7.5 %, 10 % and 12.5 %).

The acoustic emission signals during tensile tests were recorded and analyzed using the software developed within the research. There were investigated: the spectral characteristics of individual events and distribution of the peak energy of frequency spectrum in the time domain

The peak frequencies of AE signals were determined within two basebands - the lower and the upper. The lower is lying in the frequency band from 37 kHz to 39 kHz, and the upper – from 97 kHz to 103 kHz.

In each experiment on PES filled samples, there were observed two distinctive α_1 and α_2 points with expressed increase in the rate of cumulative AE growth.

The dynamics of the 2nd derivative of the total AE and the dynamics of the maximum power spectral density were analyzed. The behavior of the AE intensity 2nd derivative used as a parameter connected with spectral properties of AE signals.

IV. CONCLUSION

It was concluded that the cumulative AE-criterion (α -criterion) is a parameter related to the dynamics of frequency change. Its use suggests the following approach: starting from an arbitrary point in time, immediately after the release of acoustic emission with a typical 2nd derivative pattern change, it is necessary to record the change of AE event frequency. During the tests, it is possible to determine the stage of failure by the nature of changes in the approximation function of maximum AE signal frequency. The analysis of cumulative AE count indicates the existence of three different stages of damage accumulation in the composite. These stages cannot be identified by using only strain diagrams, but they are distinguishable on AE diagrams. The time of onset of these phases is determined by the moment of α_1 and α_2 criteria manifestation.

The characteristics of AE signal frequency spectrum are changing while progressing through the stages of material destruction and may be used as an additional parameter for the assessment of the state of destruction. It was found that the average frequency of maximum spectral power at the end of each experiment is lower than at the beginning. It is possible to define the parameters of the relationship between the dynamics of AE signal appearance and the dynamics of the frequency pattern of signals in the PES-filled epoxy composite samples. This requires further studies for other groups of materials: metals, alloys, concrete and other materials used for transport facilities. Simultaneous use of the criterion of the second derivative and the criterion of maximum event frequency change can help identify the phase of sample destruction, starting the recording from an arbitrary moment in the process of loading. At the same, the time identification interval is minor. It is sufficient to have several tens to hundreds of AE events to observe the features characterizing the stage of destruction.

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