# ACOUSTIC EMISSION DETECTION OF DAMAGE INITIATION IN PLAIN-WEAVE CFRP LOADED IN BENDING

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**Abstract**
Acoustic Emission (AE) was applied to detect micro-crack initiation in carbon fiber reinforced composites subjected to 4-point bending. Materials were made by RTM from plain-weave carbon fiber fabrics and epoxy resin. Fiber volume content varied from small (34%) through medium (51%) to high (68%). Samples of the size 120x20x2mm were cut from 2D composite plates in two orthogonal fabric directions [pseudo 0/90] and in two bias directions [pseudo +/-45] in relation to fiber lay-up. Fracture stress of composites with varying fiber contents were determined in 4-point bending on rectangular samples with two AE sensors attached to their surfaces for damage monitoring. Same samples were subjected to step-loading in 4-point bending, while Acoustic Emission sensors attached to samples' surfaces monitored damage initiation. Selected samples had their outer surfaces (between inner supports) polished for microscopic (SEM) observations. Continuous control of AE parameters allowed to interrupt the loading sequence of cycling tests before failure. Historic Index was the most reliable AE parameter indicating damage initiation. Micro-cracks were observed under the SEM on polished tensile surfaces of composites. Fiber debonding and breakage occurred simultaneously and were the first defects to initiate in the examined materials.

## 1. Introduction

Plain-weave composites structurally constitute a convenient experimental and analytical case, in comparison with other-than-unidirectional FRP composites, many of them having very complex 3D structures, and using various fabrics or braided fiber architectures [1, 2]. Due to sensitivity of plain-weave materials to interlayer delamination, these composites do not gain much commercial attention, however they are very suitable research materials, particularly with respect to micro- and/or meso-scale damage phenomena [3] and analytical modeling of their properties is very advanced [4, 5].

One of the main fields of damage studies in composite materials where acoustic emission (AE) has been successfully applied is the Felicity Ratio (FR) analysis [6-8], mostly used in pressure vessel (COPV) testing as well as in other structural applications [9]. A principal parameter in this methodology is so called Historic Index (HI), which is used to establish the onset of significant emission based on signal severity [10, 11]. This parameter is sensitive to variation of signal energy and its changes can be measured in function of time, therefore, it is particularly valuable in determining the

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onset of new damage mechanisms, independently of specimen size and shape. The onset of significant acoustic emission is defined as the stress level where the value of HI is equal or greater than 1,4 [11].

This parameter has not been widely used outside the FR studies of AE in composite materials. One of rare attempts of applying it to a pattern recognition analysis was not considered quite successful [12]. The main problem may appear in correlating the HI indications with the size of damage observed in a given study. The HI indications in the range of dozens (20 even up to 90) relate to severity of cracks observed visually or using NDE methods less sensitive than AE, like C-scan or X-ray imaging [13]. It is reasonable to believe that enhancing the observation resolution (i.e. decreasing the size of observed defects) should conform to significantly lower HI value. The lower the HI at time of observation, the closer the damage to initiation stage. Therefore, detecting the HI values between 1,4 and 2 or 3, should allow for observation of initiating defects in micrometric scale. It is worthwhile mentioning also, that good control of HI requires highly skilled equipment operators, reacting momentarily to sample's behavior and adjusting promptly the parameters.

In many practical applications composite materials are subjected to bending forces and to comply with these conditions, damage initiation studies are also carried out in 3-point bending [14-20] or 4-point bending [21-26], despite the fact that theoretical basis for applying mechanical solutions in composite bending set-ups is being questioned, due to large fracture strain of these materials [27]. However, significant deformation in flexion may also become advantageous for purposes of defect localization, as the region of highest risk of damage is limited to the maximum stress area (e.g. tensile in center of outer surface in bending or compression [25] in inner surface).

The goal of this work was to observe directly the onset of micro-crack initiation in plain weave CFRP multilayer laminates during the 4-point bending tests of rectangular composite beams (at outer surfaces subjected to tensile stresses), based on continuous monitoring of damage evolution with time of test using the AE, particularly exploiting the sensitivity of AE Historic Index (HI) to the onset of new damage mechanisms.

## 2. Materials and Methods

### 2.1. Sample preparation

Composite plates of the size 500 x 500 mm were made of carbon fiber fabric and epoxy resin matrix in Technical University Dresden[1] using the RTM technology. The resin transfer temperature was $60^0$C, injection pressure 6 bars and processing time 8 hrs. The epoxy resin L used together with EPH 294 hardener[2] constituted the matrix material.

Toho Tenax E HTS40 carbon fiber textiles with 12K filament tows and with linear density 800 tex were used for composite processing in form of plain-weave fabric of average weight of 600 g/m$^2$. The number of fabric layers and the amount of resin injected during plate moulding were varied to give final products with different volume content of fibers (small, medium, large): 34, 51and 68 vol. %, respectively, as determined by the manufacturer.

Rectangular composite samples of dimensions 120x20x2mm were water-jet cut from each RTM plate with specific fiber volume content. Samples were cut in two orthogonal directions (Fig. 1) following the principal RTM plate axes (denominated X and Y). This gave the fiber lay-up in the samples corresponding to cross-ply $[0/90]_n$ unidirectional composites. They will be named "pseudo cross-ply" further in the text. Another set of samples was water-jet cut in two bias directions (marked L and P) with respect to principal fabric axes, giving fiber lay-up in bending samples corresponding to angle-

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ply $[+45/-45]_n$ in unidirectional composite materials. They will be named shortly “pseudo angle-ply”. Selected samples had their tensile surfaces polished in the area between the inner supports of 4-point bending set-up, to allow for SEM observations after interruption of step-loading tests. Similar procedure has been proven successful by de Carvalho et al. [25] in examining compressed faces of composite samples in 4-point bending.

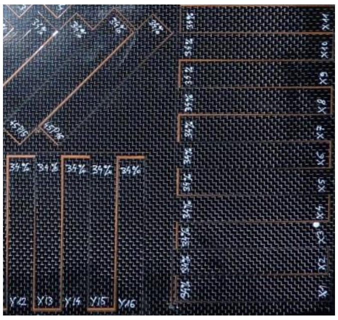


**Figure 1.** CFRP rectangular samples cut for 4-point bending tests from RTM plate.

## 2.2 Mechanical properties testing

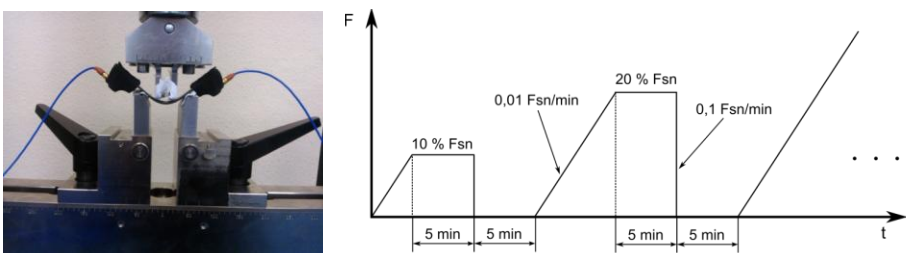
### 2.2.1 Flexural strength in 4-point bending

Prior to cyclic step-loading experiments, the strength limits of the examined CFRP composite materials were determined in monotonic 4-point bending tests [21, 26], accompanied by monitoring of their acoustic emission (AE) response [28, 29]. Electro-mechanical testing machine Zwick Z100 was used in these experiments, driven by the testXpert v.3.1 software (also by Zwick).

The crosshead speed applied in most cases was 1mm/min, and for samples with pre-polished tensile surfaces (used later for SEM microscopy) it was reduced to 0,25 mm/min. The experimental set-up in 4-point bending tests together with positioning of AE sensors on CFRP sample is shown in Fig.2a.

### 2.2.2 Flexural step-load tests

The increasing load (step-load) tests were performed in 4-point bending [11, 30-33], using the servo-hydraulic MTS 810 machine equipped with FlexTest software, version 3.5B 1787.




a b

**Figure 2.** a) experimental set-up in 4-point bending tests, with positioning of AE sensors
b) loading schedule in step-load tests

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Load was applied following the scheme shown in Fig. 2b, with 10% increasing steps (in relation to rupture stress), followed by 5 min hold. The load was applied with the average rate of 0,01 of fracture stress per minute and recorded using force sensor MTS 661 19F-01. Unloading rate was on average 0,1 of fracture stress per minute, down to the level of ~1% of fracture stress, to avoid testing machine's instability. Unloading was followed by another hold period of five minutes. The described sequence was repeated till final failure of the sample or until interruption of the test.

### 2.3. Acoustic Emission

The acoustic emission AMSY-6 system[3] equipped with Vallen software for parametric and transient data acquisition and analysis was used for damage initiation monitoring during the monotonic 4-point bending tests of the examined CFRP composite samples. Loading data were transferred through parametric output of Zwick testing machine to the input module of acoustic emission AMSY-6 system, allowing for simultaneous registration of force applied and cross-head displacement, and thus allowing for correlation with acoustic emission signals obtained from loaded samples. The AE signals were registered using two broad-band sensors Fuji AE144A (50-700 kHz) attached to sample's surface (Fig. 2a) with rubber band and vacuum grease as a coupling agent. The Vallen AEP4 preamplifiers had the gain set to 34dB. System threshold setting varied from 34 to 46 dB (depending on type of sample), and transient files registration TR used 2048 samples per set. Before actual measurements, standard signal calibration procedure was performed using the Hsu-Nielsen source [34].

The AMS-3 acoustic emission system (also by Vallen Systeme GmbH) equipped with Vallen software for parametric and transient data acquisition and analysis was used for damage monitoring during the increasing load (step-load) tests in servo-hydraulic MTS 810 testing machine equipped with force sensor MTS model 661 19F-01. Signals were collected by two Vallen resonant sensors V150-M followed by Vallen AEP3 preamplifiers. Sensors were attached to samples' end surfaces with rubber bands and vacuum grease was used as coupling agent. Preamplifiers gain was set to 34dB and system threshold in most cases was set to 40,7 dB, with 2048 samples per TR set. Signal calibration procedure was applied [34] prior to measurements.

### 2.4 Microscopic observations

Selected samples for each carbon fiber fabric lay-up and volume content had part of their central outer surface (between lower supports - see Fig. 2a) pre-polished for microscopic studies of damage initiation. The SEM observations were carried out on these surfaces using the high resolution scanning electron microscope Nova NanoSEM 200[4]. Uncoated samples were observed in secondary electron mode (SE) in low vacuum conditions (60 Pa) and at relatively low voltage (5kV).

## 3. Results and Discussion

The acoustic emission (AE) parametric analysis of signals obtained during monotonic and cyclic step-loading did not reveal any specific pattern of amplitude, duration, hit number, rise time or energy, neither in relation to fiber architecture and/or volume fraction, nor to damage mechanisms observed in the examined composites [35, 36]. It is not quite disappointing, as the examples can be found in the bibliography, reporting contradictory values of identical parameters obtained by different authors on similar materials and in similar testing conditions [13, 37-41].

[3] manufactured by Vallen Systeme GmbH, 82057 Icking, Germany. http://www.vallen.de/
[4] FEI Company, Europe NanoPort, Achtseweg Noord 5, 5651 GG Eindhoven, The Netherlands

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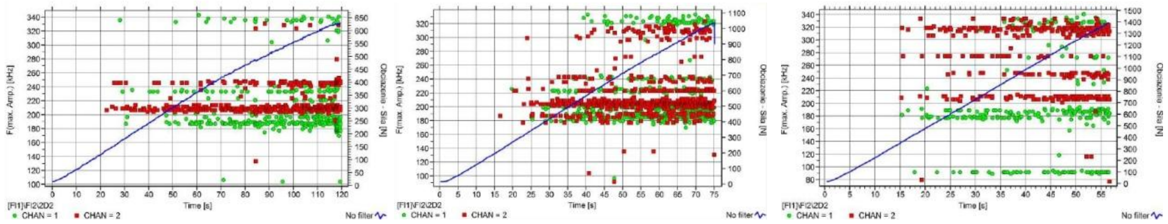
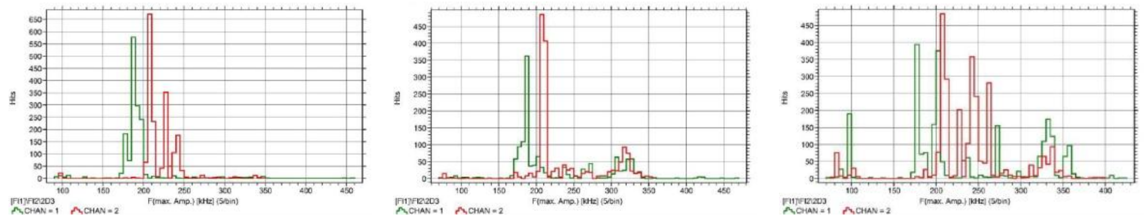
Signal frequency appeared to be in this study a useful parameter resulting from transient acoustic emission analysis, quite consistent with other published results [37-39]. Even considering all limitations related to complexity of propagation of various types of acoustic waves in heterogeneous composites [42-44], this parameter may be indicative of the type of damage developing in the material, although without quantification and accurate definition of time of occurrence. The frequency spectra of acoustic emission signals were obtained in this work using the Visual TR software provided by Vallen Systeme GmbH.

The AE frequencies (for maximum amplitude) of typical materials tested in this work in monotonic 4-point bending are shown in Fig. 3. The plain weave pseudo cross-ply CFRP show most of AE signals in frequency ranges 180-240 kHz (up to 280 kHz at highest fiber content). Similar values are indicated by other authors as typical of fiber/matrix interface phenomena [37, 39, 40]. Higher frequencies (>300 kHz) of AE signals also appear in pseudo cross-ply composites (Fig. 3 b,c), with number of signals increasing with the increasing fiber volume content. Such increase seems rational, considering statistical nature of fiber strength [35]. These higher AE frequencies (>300 kHz) are often assigned in the bibliography to fibre breakages [17, 39, 40, 45] and microscopic observations shown here confirm these results.




a) 34 v/o b) 51 v/o c) 68 v/o

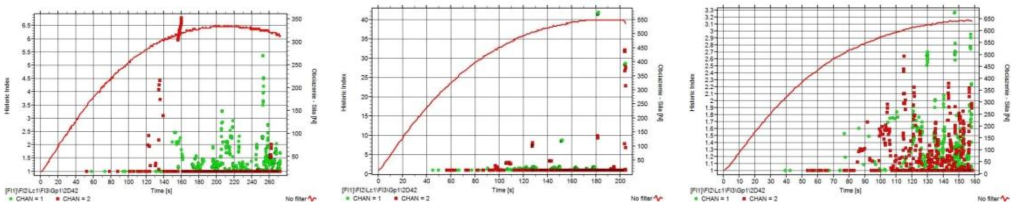



d) 34 v/o e) 51 v/o f) 68 v/o

**Figure 3.** AE frequency in 4pb tests of pseudo cross-ply CFRP with varying fiber content (a,b,c) frequency histograms vs #AE hits (d,e,f) for pseudo angle-ply CFRP

In pseudo angle-ply composites the frequencies of most of AE signals are centered around values near 200 kHz. Following the results of other works [37, 38], the initiation of damage may involve here the phenomena in fiber/matrix interface. Fiber debonding is confirmed in this work by SEM observations. Similarly to pseudo cross-ply CFRP materials examined, the frequency histograms in Fig. 3d,e,f show increasing number of hits related to fiber breaks (300-350 kHz), following the increase of fiber volume content in the samples.

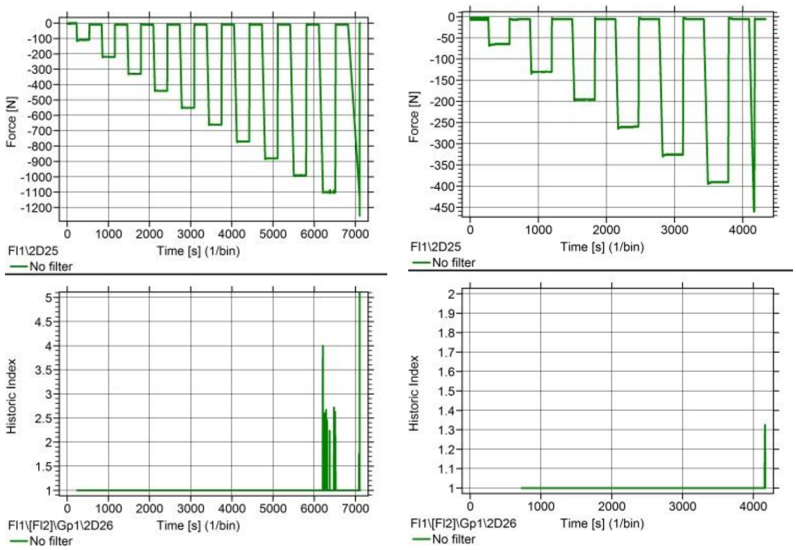
The most valuable AE parameter in terms of indication of accurate timing of damage initiation in the examined composites during both monotonic and cyclic step-load tests was the Historic Index. Fig. 4 shows the readings of AE Historic Index during monotonic 4-point flexure loading of pseudo angle-ply CFRP composites with varying fiber volume content. Values of HI exceeding 1,4 are believed to be indicative of the appearance of new damage mechanism [11], which was directly confirmed in this work by SEM observations shown later in the text.



a b c

**Figure 4.** AE Historic Index in monotonic 4-p bending tests of pseudo angle-ply CFRP samples (a-34, b-51, c-68 v/o of fibers).

Fig.5a (bottom) shows the indications of AE Historic Index appearing in full 4-point flexure cyclic step-load test performed till sample's rupture. Fig. 5b shows the indication of AE Historic Index at point of interruption of step-load test, after which sample's surface was observed under the SEM.




a b

**Figure 5.** Step-loading tests (top) of CFRP with corresponding HI plots (bottom)
a) full cycle test (till rupture) for pseudo cross-ply sample
b) interrupted loading for pseudo angle-ply sample

The ability of AE Historic Index to indicate accurately the timing of damage initiation may become very useful in more advanced AE analyses, like pattern recognition and/or wavelet transform [18], since the AE waveform characteristic of particular type of damage can be defined at the exact point of time of occurrence. In most works to date specific defects (matrix cracks, fiber breaks, debonding, pull-out, finally - delamination) have their time of occurrence attributed analytically, based on assumption of particular sequence of damage events, without support of direct experimental evidence [20, 46, 47].

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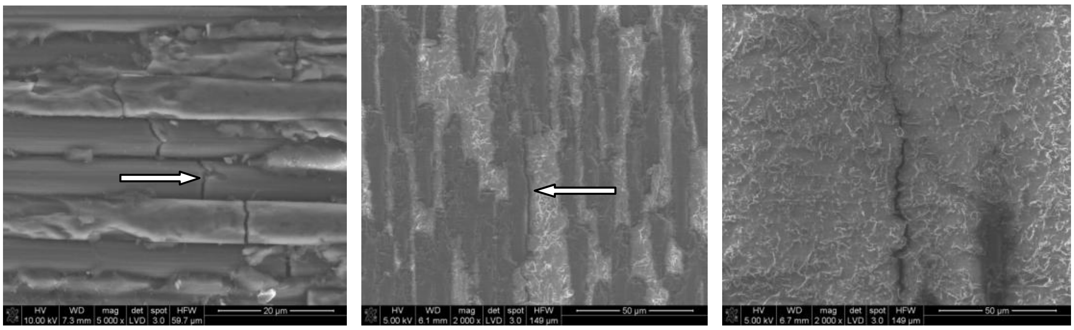
Following the indications of AE Historic Index and after interruption of step-loading cycle (but enough before final failure), the examined CFRP samples were subjected to careful microscopic (SEM) observations of their polished outer surface, subjected to tensile stress during 4-p bending loading. Typical features noted in pseudo cross-ply materials, irrespective of volume fraction, were the fiber-breaks in tows parallel to tensile surface (Fig. 6a) and fiber-debondings in tows vertical to tensile stress (Fig. 6 b,c). The features shown in SEM micrographs below were observed simultaneously, and no sequence [3, 17, 48] could be specified as to priority of one damage mechanism over the other.



a b c

**Figure 6.** Damage initiation in pseudo cross-ply CFRP (51v/o fibers) after step-load test interruption. a) fiber breaks in tows parallel to tensile stress, b) debonding in tows vertical to tensile stress, c) “apparent” matrix crack, resulting from fiber debonding underneath

Similarly to angle-ply unidirectional composites [49, 50], the samples of plain weave CFRP with pseudo angle-ply fiber fabric lay-up examined in this work in two bias directions, have their flexural strength almost two times lower than their pseudo cross-ply counterparts. Their fracture strain is twice that of pseudo cross-ply materials. However, the SEM observations of tensile surfaces of pseudo angle-ply samples with step-loading interrupted, show similar damage initiation features as in plain-weave pseudo cross-ply materials, i.e. fiber-breaks and fiber-debonding (Fig.7). These micrographs confirm that damage initiation observations in stressed surfaces based on determination of accurate timing may serve as better direct experimental evidence than micrographs of fracture surfaces of composite materials. The latter, as suggested by Lomov et al. [3], can be named “post mortem” analysis, and it can not reveal any information neither on damage initiation, nor on the sequence of damage events.

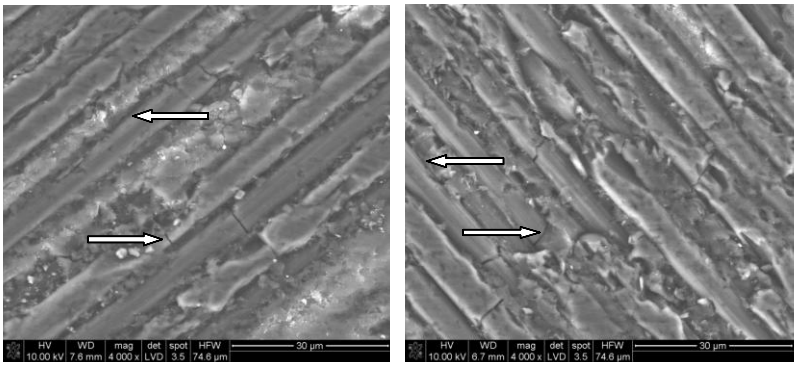


**Figure 7.** Fiber breaks and debondings in tows oriented at 45 deg. angles to tensile stress, after interruption of step-load tests of pseudo angle-ply CFRP

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## 4. Conclusions

- Acoustic Emission technique was efficient in monitoring damage initiation during the 4-point monotonic and cyclic step-load bending tests of plain weave CFRP with different fiber lay-up and volume content
- Acoustic Emission frequency was confirmed to be indicative of type of damage initiating in plain weave CFRP examined
- The Acoustic Emission Historic Index parameter appeared to be an excellent indicator of the time of occurrence of damage initiation during the loading process of CFRP samples
- Microscopic (SEM) evidence correlated well with AE monitoring and showed that fiber-matrix debonding and fiber breakage were the dominant mechanisms of damage initiation in the examined CFRP plain weave fabric laminates

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