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ACOUSTIC EMISSION DETECTION OF MICROCRACK INITIATION IN CFRP UNDER SHEAR STRESS

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Summary: *Acoustic Emission was applied for detection of microcrack initiation in carbon fibre reinforced epoxy composites. Materials were prepared from PES bonded 1D NCF and 2D plain-weave carbon fibre fabrics, using the RTM technology. Small rectangular composite samples were cut from plates with* $[0]_n$, $[90]_n$, $[0/90]_n$ *and* $[\pm 45]_n$ *fibre layout. Fibre volume content of composite plates varied from small (34 for 2D/38% for 1D), through medium (51%) to high (68%). Side surfaces of selected samples were polished for microscopic observations. Short beam strength tests were performed on small samples (l/h=4) subjected to quasi-static 3-point bending tests, with two AE sensors attached to their surfaces for monitoring of damage initiation. Continuous control of selected AE parameters allowed to interrupt the loading sequence before the final failure. The Historic Index appeared to be the most efficient AE parameter in this regard. Details of microcracks developing on polished composite side-surfaces were observed under the SEM. Direct microscopic evidence confirms the fibre debonding to be the principal mechanism of crack initiation in these materials before any further damage.*

1 INTRODUCTION

One of the main fields of damage studies in composite materials where acoustic emission (AE) has found successful applications is the Felicity ratio analysis, most popular in pressure vessel testing [1-3]. A principal parameter of this methodology is so called Historic Index, used to establish the onset of significant emission based on signal severity [3,4]. This parameter is sensitive to change of the signal energy and it is particularly valuable for determining the onset of new damage mechanisms, independently of specimen size. The onset of significant acoustic emission is defined as the stress level where the value of historic index (HI) becomes equal or greater than 1,4 [4].

Surprisingly, this parameter has not been widely used in AE parametric analysis of composite materials. One of rare attempts of applying it to a pattern recognition analysis did not turn out promising [5]. The main problem appears in that, while ascribing the HI to the size of damage observed in a given study, the HI results of the range of dozens (30 even to 90) correspond to large, visually observed cracks and defects [6]. Enhancing the observation resolution (i.e. decreasing the size of detected defects) usually conforms with significant decrease of the value of HI. This correlation seems to be such, that the lower the HI at time of observation, the closer the damage to initiation stage.

The goal of this study was to observe directly the onset of microcrack initiation in CFRP subjected to shear stresses during the short beam strength (SBS) test, based on continuous monitoring of damage with use of AE, particularly exploiting the sensitivity of Historic Index (HI) to the onset of damage mechanisms.

2 MATERIALS AND METHODS

2.1 Sample preparation

Carbon fibre/epoxy matrix composite plates of the size 500 x 500 mm were manufactured by LZS ILK TU Dresden[1] with use of RTM technology, at resin transfer temperature 60^0C, injection pressure 6 bar and processing time 8 hrs. The epoxy resin L was used together with EPH 294 hardener[2].

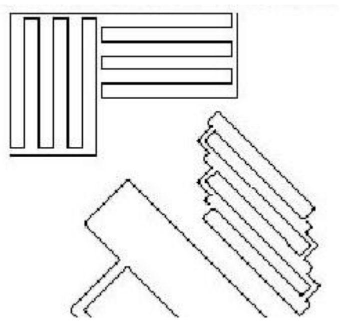
The 1D composite plates were made of PES bonded Toho Tenax E HTS40 non-crimp fabric (NCF) with carbon fibre strands of 12K filaments and with linear density 800 tex. Single Toho Tenax HTS40 carbon fibres used in this work have average tensile strength of 4300 MPa and tensile modulus 240 GPa. The 2D composite plates were formed from plain-weave fabric made of the same Toho 800 tex fibres, with average fabric weight of 600 g/m^2.

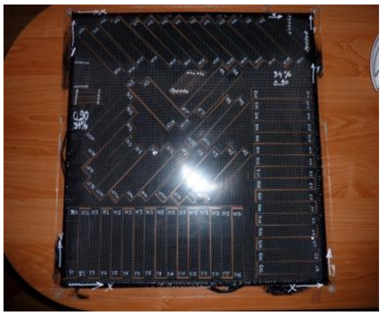
The number of fabric layers (5,7,10) and the amount of resin injected during plate moulding were varied to give final products with different volume content of fibres (small, medium, large): 38vol.%, 51% and 68%, respectively. The volume content of fibres in 2D plates was also purposely varied in the same way and has been determined by manufacturer as 34%, 51% and 68%, respectively.

Small rectangular composite samples of dimensions 45x4x2mm were water-jet cut from each of large 1D plates with varying fibre volume content in both principal fibre directions [0] and [90]. The same small size samples were cut from each of 2D plates with specific fibre volume content, to give cross-ply $[0/90]_n$ and angle-ply $[+45]_n$ fabric layout, in two

[1] Leichtbau-Zentrum Sachsen GmbH, Institut fur Leichtbau und Kunstofftechnik, Technische Universitaet Dresden, Germany
[2] both made by R&G Faserverbundwerkstoffe GmbH of Waldenbuch, Germany

orthogonal directions (denominated X and Y) with respect to principal RTM plate axes (Figure 1b).





a b

Figure 1: a) sample cutting scheme, b) RTM formed CFRP composite plate.

2.2 Mechanical properties: short beam strength tests in 3-point bending

In short beam strength (SBS) tests the interlaminar shear is a dominant mode of failure although usually internal stresses can be complex and variety of failure modes may occur, like compression and/or tension in flexure or even inelastic deformation [7-9].

The short-beam strength tests of the examined composites were carried out in 3-point bending of described above small samples, with loading span of 8mm (l/h=4). Tests were performed using mechanical testing machine Zwick Z100, equipped with testXpert v.3.1 software (by Zwick). Loading data were transferred through parametric output of Zwick testing machine to input module of the acoustic emission AMSY-6 system, allowing for simultanous registration of force and displacement and correlation with acoustic emission signals obtained from loaded samples.

Crosshead speed applied in most cases was 1mm/min, and in specific cases of very weak [90] samples it was reduced to 0,25 mm/min. The same speed of 0,25 mm/min was applied for samples with pre-polished side-surfaces (used later for microscopy) while a few very fragile microscopic [90] samples were loaded at 0,1 mm/min.

2.3 Acoustic Emission

The acoustic emission (AE) signals were registered with use of AMSY6[3] equipped with Vallen software for parametric and transient data acquisition and analysis. Two sensors small enough in size (d=4mm), Fuji AE144A, were attached to lower surface of each end of the sample with rubber band and vacuum grease as a coupling agent, followed by Vallen AEP4 preamplifiers set to 34 dB. The spectrum of Fuji AE144A broad-band sensor covers frequencies from 50 to 700 kHz and additionally the 75-660 kHz filter was applied.

The acoustic emission coupling was calibrated before each measurement using the Hsu-Nielsen source in form of 2H hardness graphite pencil 3±0,5 mm long [10]. The following parameters of acoustic emission data acquisition during the SBS bending tests were used: system gain 34 dB, threshold 25,3-46 dB (depending on type of sample), duration discrimination time 200 μs, TR sample rate 5 MHz, 2048 sample per TR set.

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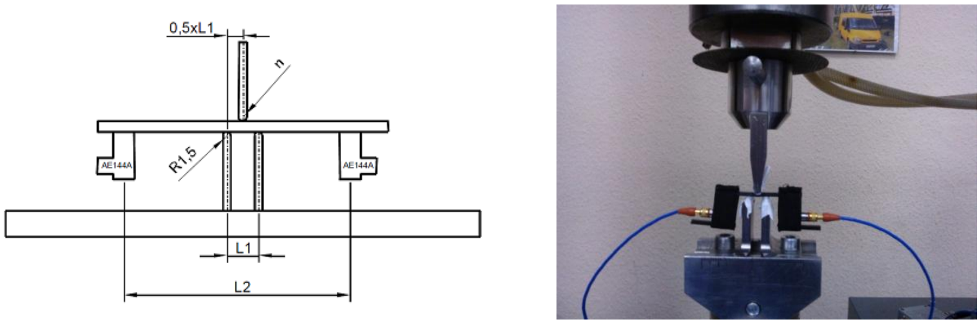
[3] made by Vallen Systeme GmbH, Germany



a b

Figure 2: a) experimental set-up for SBS 3-p bending tests with positioning of AE sensors
b) short-beam CFRP composite sample with AE sensors attached, in Zwick Z100 testing machine. N.B. Picture taken after sample's rupture registered by both AE and mechanical system, without any macroscopic damage visually detectable.

2.4 SEM observations

Observations of pre-polished side surfaces of selected samples before and after fracture were carried out using the high resolution scanning electron microscope. Uncoated samples were observed in secondary electron mode at relatively low voltage.

3 RESULTS and DISCUSSION

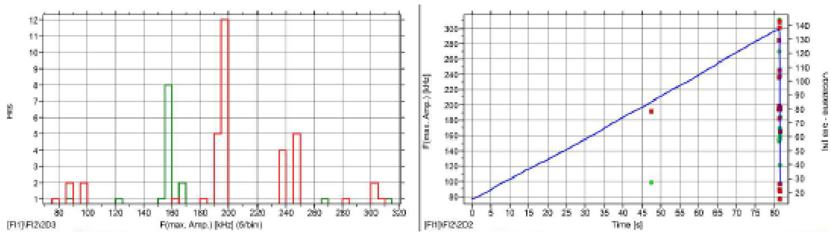
3.1 Parametric and transient acoustic emission

The acoustic emission (AE) parametric analysis did not reveal any characteristic pattern of amplitude, duration, event number, rise time or energy, neither in relation to fibre lay-out and/or volume fraction, nor to damage mechanisms observed in the examined composites [11]. It is worthwhile noting that examples can be found in the bibliography, reporting contradictory values of identical parameters obtained by different authors on similar materials and testing conditions [12]. One of the promising parameters resulting from the transient acoustic emission analysis is the signal frequency [13-18]. With all limitations related to complexity of propagation of various types of acoustic waves in heterogenous materials [1,13,16], 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 are calculated in this work using the Fast Fourier Transform (FFT) software provided by Vallen GmbH [19].

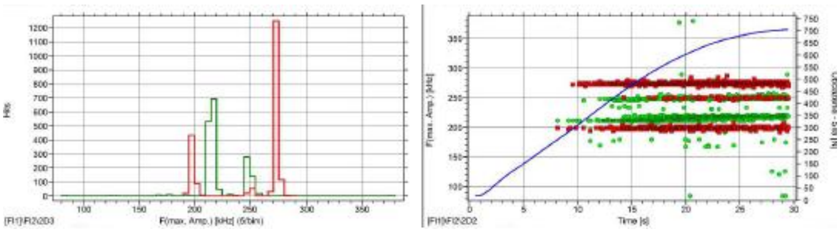
The AE frequencies (for maximum amplitude) of typical materials tested in this study are shown in Figure 3. the CFRP [90] samples (examined across fibres - direction X) show practically no signals till final fracture (Figure 3a), while CFRP [0] samples (examined along fibres – direction Y) show signals appearing early in the test (Figure 3b), mostly in frequency ranges (180-280kHz) indicated by other authors as typical for fibre/matrix debonding and/or pull-out [12,15-18].

Higher frequencies (>300kHz) of AE signals appear in cross-ply composites (Figure 3c,d) with number of signals increasing with increasing fibre volume. Such frequency ranges are often ascribed in the bibliography to fibre breakages [12,15-18]. The AE signals of average frequencies (~200kHz) appear in angle-ply composites [+45] later in the loading process and their number and range also seem to increase with increasing fibre volume (Figure 3e,f), which may involve phenomena at the fibre/matrix interface, i.e. fibre debonding and/or pull-out, and also later some fibre breakages [12,15-18].

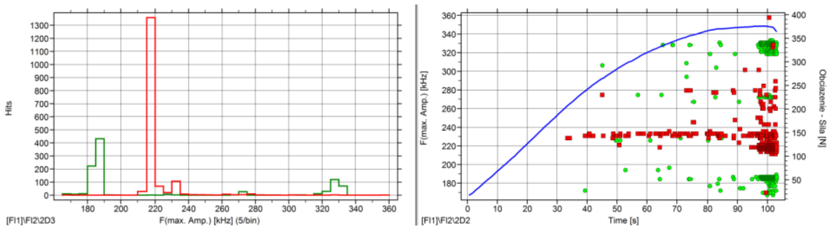
The physical decohesive phenomena described by these AE signals are usually of micrometer size and due to complexity of AE wave propagation in as heterogenous materials as composites, the accurate location of such defects is practically impossible [20,21]. For these reasons sufficiently reliable experimental evidence of described defects is still almost non-existent in available bibliography [22,23] and requires more dedicated research.



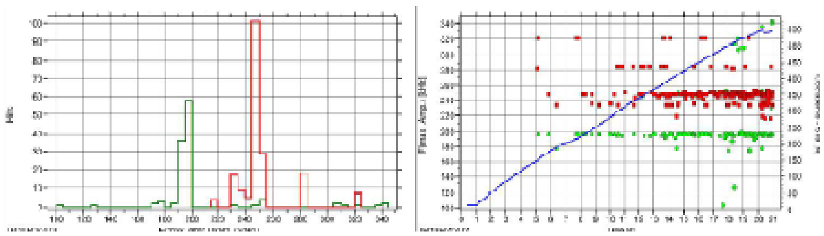
a) 1D 38 X



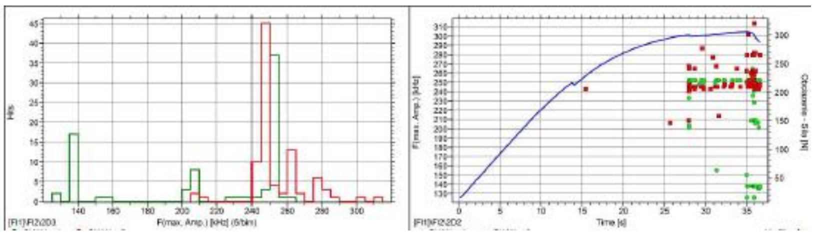
b) 1D 51 Y



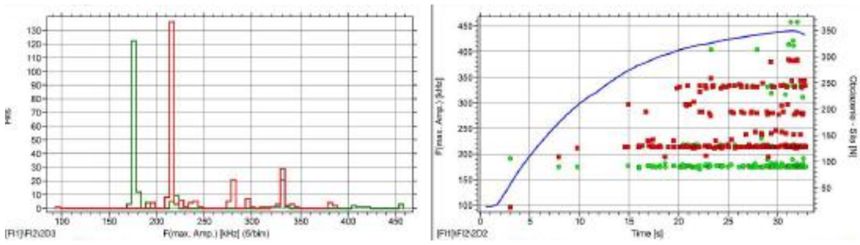
c) 0_90 34 X



d) 0_90 51 Y



e) 45 34 P

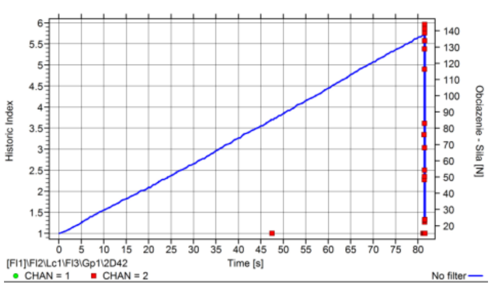


f) 45 68 L

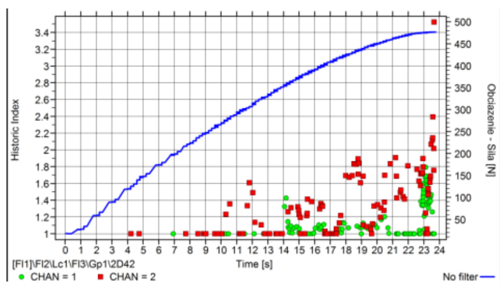
Figure 3: AE frequencies in CFRP with various fibre lay-out and volume content: a) $[90]_5$ PES bonded 1D NCF laminate, b) $[0]_5$ PES bonded 1D NCF laminate, c) plain-weave cross-ply $[0,90]_5$ laminate, d) cross-ply $[0,90]_5$, e) angle-ply $[-45]_5$, laminate, f) angle-ply $[+45]_{10}$.

3.2 Historic Index –AE indicator of damage initiation

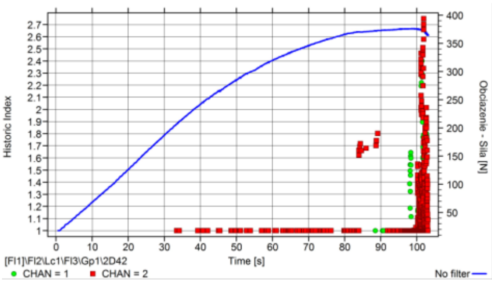
From all the AE parameters considered in this study, the most valuable from the point of view of controling the damage initiation stage, appeared to be the Historic Index. Its values in function of time of test for different fibre lay-outs and volume fractions are presented in Figure 4. These plots prove that damage initiation occurs in all examined materials (except 1D $[90\ ]_n$ – Figure 4a) well before the catastrophic failure, with different intensity and at different stress levels, depending on carbon fibre lay-out and volume (Figure 4b,c,d).



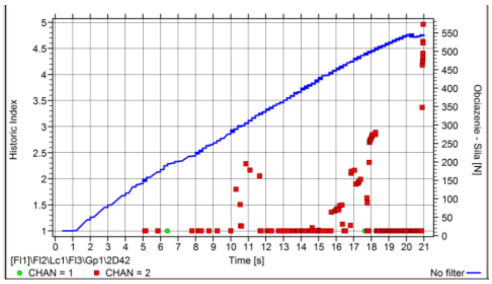
a) 1D 38 X



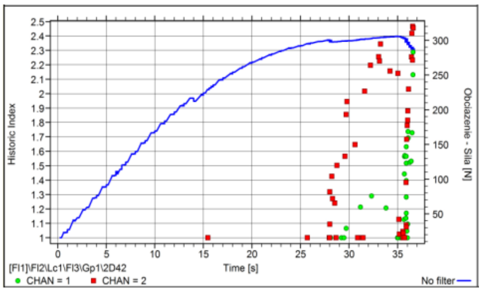
b) 1D 51 Y



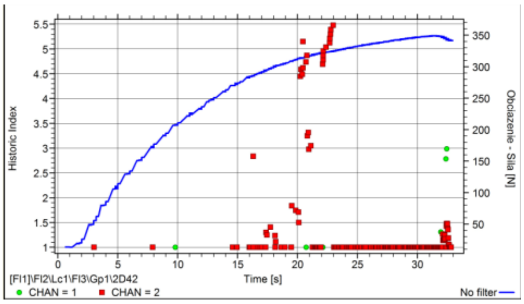
c) [0,90] 34 X



d) [0,90] 51 Y



e) [-45] 34 (P)



f) [+45] 68 (L)

Figure 4: AE Historic Index indicating the onset of damage for CFRP with various fibre lay-out and volume content:
a) $[90]_5$ PES bonded 1D NCF laminate, b) $[0]_7$ PES bonded 1D NCF laminate,
c) plain weave cross-ply $[0,90]_5$ laminate, d) cross-ply $[0,90]_7$, e) angle-ply $[-45]_5$,
f) angle-ply $[+45]_{10}$ laminate

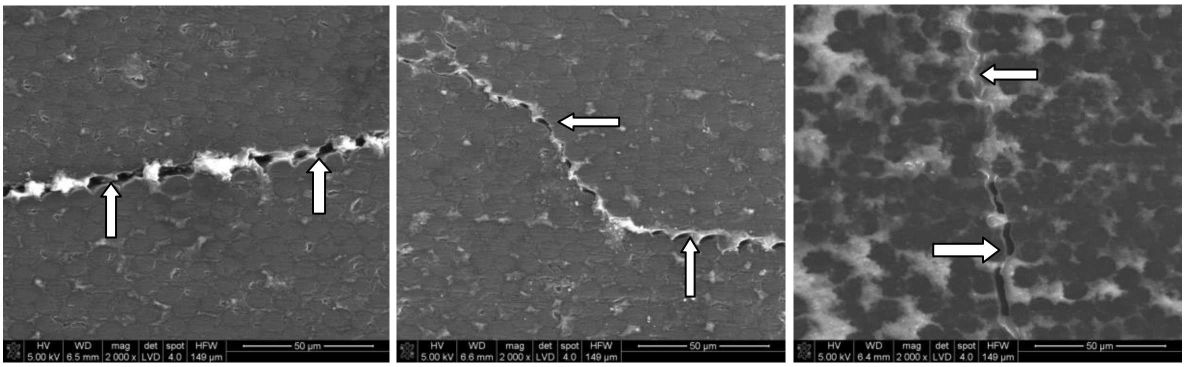
3.3 Microscopy (SEM)

After loading of unpolished samples, accompanied by continuous monitoring of AE parameters and gaining some experience in controling the evolution of Historic Index, the SBS loading tests of samples with polished side-surfaces were interrupted after the Historic Index reached the value near 1,4 and their side surfaces were carefully observed under the SEM[4] for microcrack development. The SEM observations of 3pb SBS samples' side-surfaces for crack initiation were performed at stress levels lower than critical (Figure 5a,b) and also after the final fracture of samples (Figure 5c).

Interestingly, presented here results very closely correspond to crack initiation model proposed by Lomov et al. [23] on the basis of experiments carried out on similar materials but on samples subjected to uniform tensile stresses. In their case, despite this specific experimental set-up, micro-cracks developed in direction paralel to the acting force, which may confirm the dominant role of shear stresses in crack initiation in these materials.

Figure 5c shows the crack passage from neutral (shear) axis of SBS sample to its tensile surface, mosty through fibre/matrix boundaries, very similar in nature to initiating cracks shown in Figures 5a and b. At this point the final fracture of the sample losing its load bearing capacity was registered by both mechanical and AE systems (compare Figure 2b).

[4] Nova NanoSEM 200 (FEI Company)



a) b) c)

Figure 5: a and b) microcracks initiating by fibre debonding inside fibre strand in angle-ply [$\pm$45]$_n$ CFRP samples loaded below critical stress in the SBS test
c) crack approaching the tensile surface in fractured CFRP sample after the SBS 3pb

In view of these observations, it seems that macro-defects often described in FRP as „delaminations" are in most cases the result of dramatic and unnecessary overload of the material and its catastrophic destruction within the inappropriate (unsafe) working stress range [4].

4 CONCLUSIONS

- Shear damage in bending is often ecountered as the mechanism of microcrack initiation in composite materials subjected to real working stresses and its detailed study is of particular interest.
- Applied methodology allowed to observe directly the very moments of microcrack initiation in samples the examined CFRP with different fibre structure and volume.
- Acoustic emission Historic Index is an excellent indicator of damage initiation in CFRP, irrespective of fibre lay-out and volume content.
- Damage initiation in the examined composites is dominated by fibre/matrix debonding.

5 ACKNOWLEDGEMENTS

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