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Development of interphase engineering techniques for the ductility improvement in CF/EP composites - comparison of NDT methods for delamination localization

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Abstract

In our ongoing research we are developing interphase engineering methods to improve ductility in endless fiber reinforced composites. In the present paper we locally weakened interfacial adhesion between the fibers and the polymer matrix by the application of a designed interlaminar pattern created by 3D printing from PCL (polycaprolactone), which can be solved in the epoxy resin matrix, leaving a low adhesion pattern, but not creating a third phase in the composite. We produced composites with three different weakened adhesion zones and investigated the compressive properties and the capability of acoustic emission (AE), full field digital image correlation (DIC) and infrared thermography (IR) to localize these zones.

Keywords: Interphase engineering; Ductility improvement; 3D printing; DIC;

1. Introduction

Although endless fiber reinforced composites provide exceptional mechanical performance, high strength to weight ratio, there is still a drawback compared to their metal competitors for structural applications: their limited ductility. Carbon fiber reinforced high performance composites have a tendency to fail instantly upon reaching their maximum load bearing capacity without any warning signs, or ductile plateau, plastic deformation, which could provide some extra safety in their application, additionally to the intricate structure of the laminate [1]. Ductility improvement in composites is mainly reached by the modification of the fibers or the matrix.

From the side of the reinforcing fibers, pseudo-ductility can be achieved by fiber hybridization [2-4], fiber misalignment [5,6] or the application of shortened, discontinuous fibers [7]. Promising results were achieved in this field, presenting the emergence of the pseudo-ductile plateau in the stress-strain graphs, but some problems are also introduced: different coefficients of thermal expansion of the different fibers can cause heat induced distortions, short fibers make material handling cumbersome, placement of misaligned layers in controlled conditions requires exceptional care or special equipment, failure can lead to asymmetry and unwanted anisotropy.

Introduction of pseudo-ductility from the matrix side can be even more problematic in case of fiber-dominated high performance composites. By applying rubber-like ductile matrices [8], the structure can withstand higher strains, but in most cases there are drawbacks in heat resistance and structural stiffness. The application of special fillers can also lead to ductility improvement, for example rubber particles [9,10] to pin the propagating cracks, but here the adhesion between the matrix and the particles has to be high enough, for the crack to enter the particle, and the filling content and dispersion has to be also sufficient.

In our ongoing research we provide a different approach, interphase engineering, to improve ductility. In our research we locally weaken interfacial adhesion between the fibers and the polymer matrix by the application of a designed interlaminar pattern created by 3D printing from PCL (polycaprolactone), which can be solved in the epoxy resin matrix, leaving a low adhesion pattern, but not creating a third phase in the composite. These local weakened zones can help to convert the main failure modes to more energy consuming delamination, creating a ductile plateau in the stress-strain curves.

The feasibility of our research has been already demonstrated [11]. In our present research we try to investigate the fundamental processes leading to the ductility improvement. Simple patterns are created in the tested composite

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coupons, and the local, designed formation of the delamination is investigated by acoustic emission (AE), full field digital image correlation (DIC) [12] and thermography. Based on the results we could compare the capabilities of AE and DIC for delamination detection and we could evaluate the quality of the low adhesion patterns.

2. Materials

In our present research we used IPOX ER 1010 (IPOX Chemicals Kft., Budapest, Hungary) DGEBA-based epoxy resin as a matrix material with IPOX MH 3111 high temperature hardener. The mixing weight ratio was 100:75 according to the producer's recommendation.

As reinforcement Zoltek PX35FBUD0300 (Zoltek Zrt., Nyergesújfalu, Hungary) unidirectional carbon weave was used (309 g/m2 surface weight), consisting of Panex35 50k rovings.

As the interface modification material we chose eMorph175N05 (Shenzhen Esun Industrial Co. Ltd., Shenzhen, China) PCL filament. We chose PCL because it is soluble in the matrix, it is easier to process than other thermoplastic additives due to its lower melting temperature, and because it is a biomaterial so it is not harmful to the environment, unlike many high-performance additives. The diameter of the filament was 1.75 mm (melting temperature, Tm = 60 °C, print temperature: >80 °C).

3. Composite production

For the composite specimens [0/45/-45/0] layup sequence was selected based on our preliminary FEA tests, because in case of this structure, the delamination formation and propagation is well visible in the DIC strain fields. After cutting the reinforcing layers for the laminate, the interfacial patterns were printed on the surface of the first 3 layers by a CraftBot Plus 3D (Craftunique Kft., Budapest, Hungary) printer. To investigate the formation of the delamination, a very simple pattern, just a central rectangular strip was selected. After the layup a strip with the same size in the same position was present in every interlayer of the composite. To investigate the effect of the strip width we produced composites with 3, 5 and 10 mm wide interlaminar patterns (Figure 1), where the completely filled printed rectangles had a length of 3, 5 and 10 mm along the longitudinal axis of the specimens, and after cutting the specimens the pattern was present in the whole width of the specimen..

During the composite production firstly we composed the whole reinforcement package (3 patterned layers and one layer without printed pattern) and pressed it in a heated platen press (Teach-Line Platen Press 200E, Dr. Collin GmbH, München, Germany) at 80°C temperature and 4.5 MPa surface pressure for 1 minutes. This step ensured that the pattern was in full contact to both adjacent reinforcing layers. After the pressing the composite plates were created using vacuum infusion. The infusion was done at room temperature with 0.5 bar vacuum. After the infusion the curing of the specimens took place in a Heraeus UT20 (Heraeus Holding GmbH, Hanau, Germany) drying oven at 90°C for 3 hours. Ater curing the specimens were cut using a Diadisc 5200 (MUTRONIC Präzisionsgerätebau GmbH, Rieden, Germany) diamond disc saw to the proper dimensions.

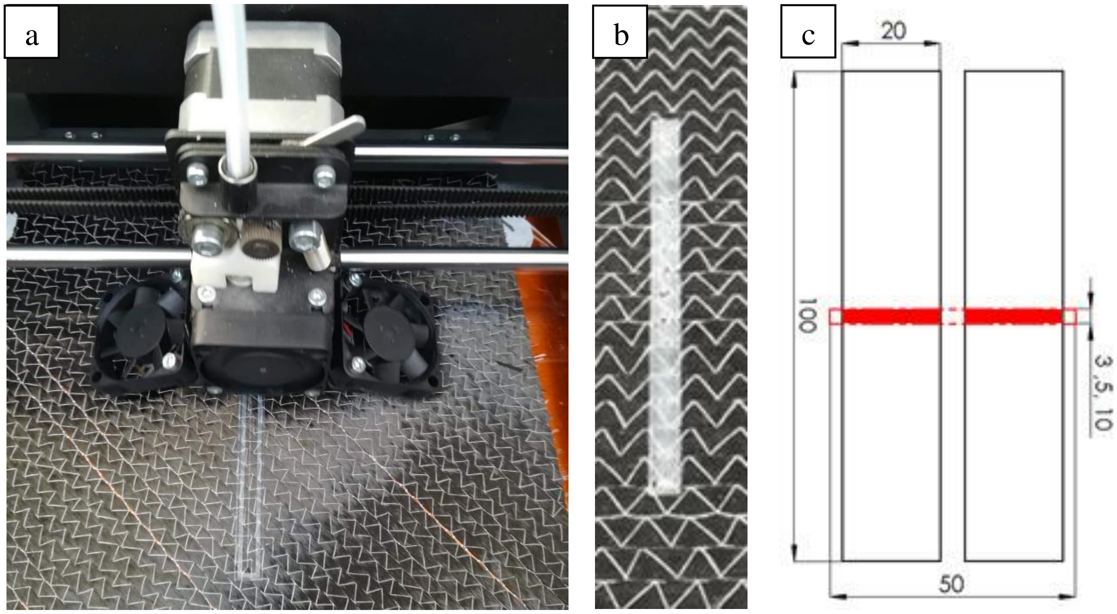

Fig. 1. (a) printing of the interfacial pattern; (b) printed pattern; (c) specimen layout (red area represents the shape and location of the printed pattern).

4. Test methods

Infrared thermography (IRT) was performed using a Flir A320 (FLIR Systems AB, Wilsonville, USA) infrared camera, the data was collected in the Flir ResearchIR software.

Compression tests were performed using a Zwick Z050 (Zwick/Roell GmbH GmbH, Ulm, Germany) computer controlled tensile tester at room temperature according to EN ISO 604. The specimen dimensions are presented in Figure 2. The initial grip to grip separation was 80 mm and the test speed was 2 mm/min. We chose compression tests because in our previous research we have investigated the effect of printed patterns in tensile setup [11], but for the onset of delaminations and the testing of their detection, compression tests are more suitable.

For fine strain measurement and the full strain field investigation a Mercury Monet 3D DIC (Sobriety Sro., Kurim, Czech Republic) system was used with two 5 Mpix resolution cameras at 10 Hz data acquisition rate.

The acoustic signals emerging during the test were acquired by a Sensophone AEPC-40/4 (Gereb es Tarsa Ltd., Budapest, Hungary) device with two Micros30s (Physical Acoustic Corporation, Princeton Junction, USA) microphones placed on the surface on the specimens symmetrically 30-30 mm from the centerline of the specimen in its main longitudinal axis.

Fig. 2. test setup of the compression tests.

5. Results and discussion

As a first reference measurement we used infrared thermography to check the location of the patterns in the specimens and check their consistency. For the measurements firstly the specimens were placed in a drying oven for 1 hour at 80°C, and after that the room temperature cooling process was investigated. PCL has nearly twice the specific heat capacity of epoxy resin, but their thermal conductivities are roughly the same. So based on this, the delaminated zone stores more heat energy, which should appear in the thermography images during the cooling process as a warmer place than the intact parts of specimens. One characteristic IRT shot is presented in Figure 3. From the image the exact location of the formed weakened adhesion zones can not be easily pinpointed. With the increasing pattern width, the inhomogeneity in temperature during cooling starts to form, but even at the largest damage zone, it is not suitable for localization. This may be due to slow cooling in the room temperature and the bigger thermal conductivity of carbon fiber reinforcement, which may have allowed the specimens to cool evenly. In the future, it might be worth trying other types of thermography techniques for example pulsed or lock-in thermography. The advantage of using IR thermography is its robustness and ease of use, but in our case we could also see the disadvantages: high thermal conductivity reinforcing materials can mask the effect of small imperfections, and what is not present in our static case, but also hinders the use of IR themography during operation of composite parts, is the time needed for the heat to propagate, which makes dynamic measurements almost impossible.

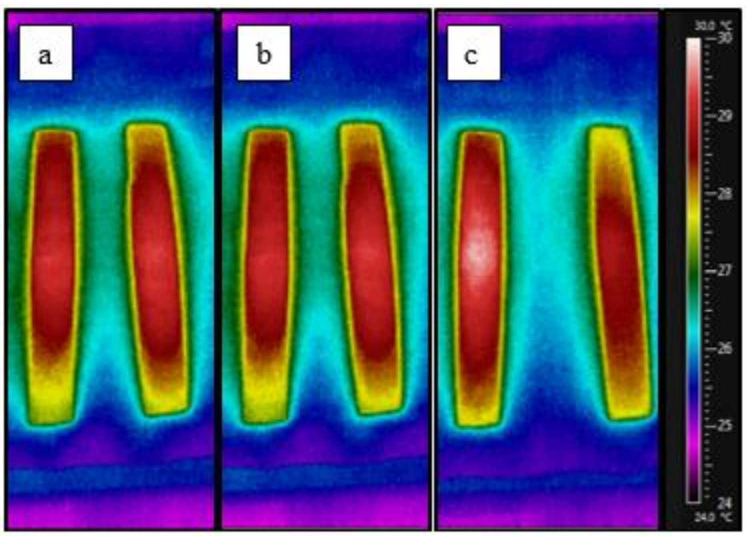

Fig. 3. characteristic IRT images (a) 3 mm pattern width, (b) 5 mm pattern width, (c) 10 mm pattern width.

To investigate the formation of the delamination and to compare the capabilities of AE and DIC for the localization of the delaminated zone, compression tests were performed on 2-2 specimens from each type up to 0.15% deformation to remain in the non-destructive testing range. The stress-deformation curves recorded during the compression tests are presented in Figure 4.

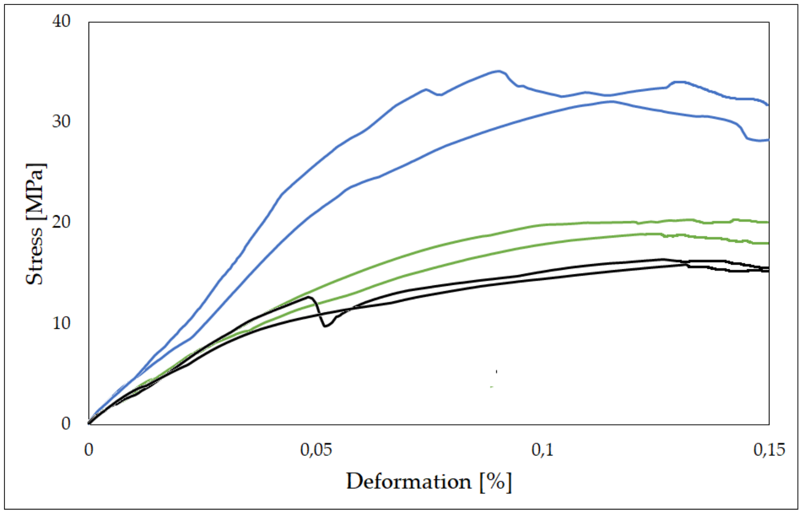

Fig.4 Stress-deformation curves of the compression tested specimens (blue – 3 mm pattern width, green – 5 mm pattern width, black – 10 mm pattern width)

The compressive curves of each specimen type are significantly different. While in case of the specimens with the thinnest pattern (3 mm) the curve reaches higher stress levels, with increased pattern width, the stress levels decrease, which indicates that the weaker adhesion zones significantly lower the total stiffness of the specimens.

Characteristic histograms of the AE tests are presented in Figure 5.

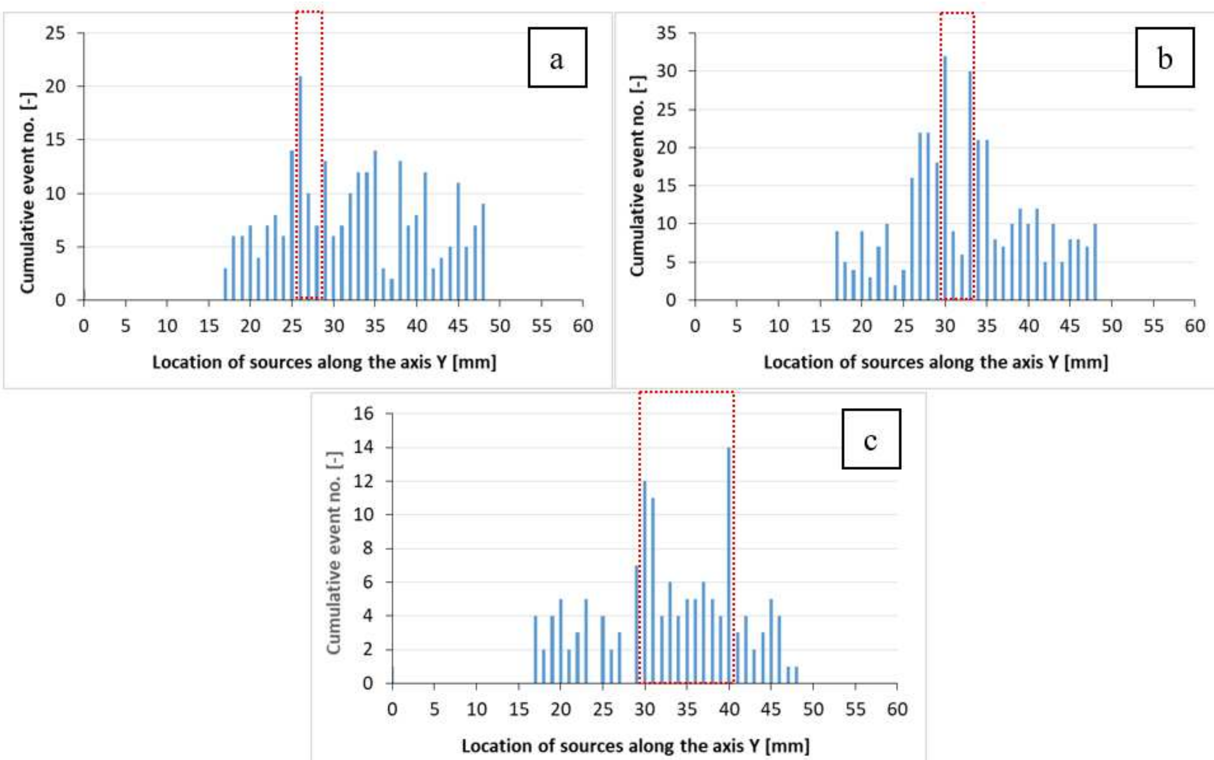

Fig. 5. characteristic AE histograms (a) 3 mm pattern width, (b) 5 mm pattern width, (c) 10 mm pattern width), edges of the pattern marked with red dashed rectangle.

From the histograms it can be seen that with the AE localization technique we were able to localize the delaminated zones caused by the patterns in case of 5 and 10 mm pattern width. It can be assumed, that with a weakened adhesion zone, the highest number of AE signals emerges from the edge of the delamination, where it starts to propagate. Inside the delaminated zone, and in the intact areas less signals are detectable. This assumption is justified by the results, we can see in the histograms corresponding to the specimens with wider patterns, that the most significant peaks with the highest ecent numbers are around the locations at the delamination borders. In case of the 3 mm wide interlaminar pattern, where edges of the delamination were probably too close to each other, so the two zones overlapped, which hindered the proper localization. As we can see AE was capable to localize the location of the delaminated zone, but there can be some disadvantages for its use during operation: noises can hinder the proper localization, and we have to consider the location of the delamination as a probability factor, also technically the delamination front has to at least slightly propagate to get results from the actual edge of the specimen, and finally the contour of the delamination can only be obtained by using numerous sensors during the localization.

Characteristic strain fields obtained from the DIC measurements measured at 0.1% deformation are presented in Figure 6.

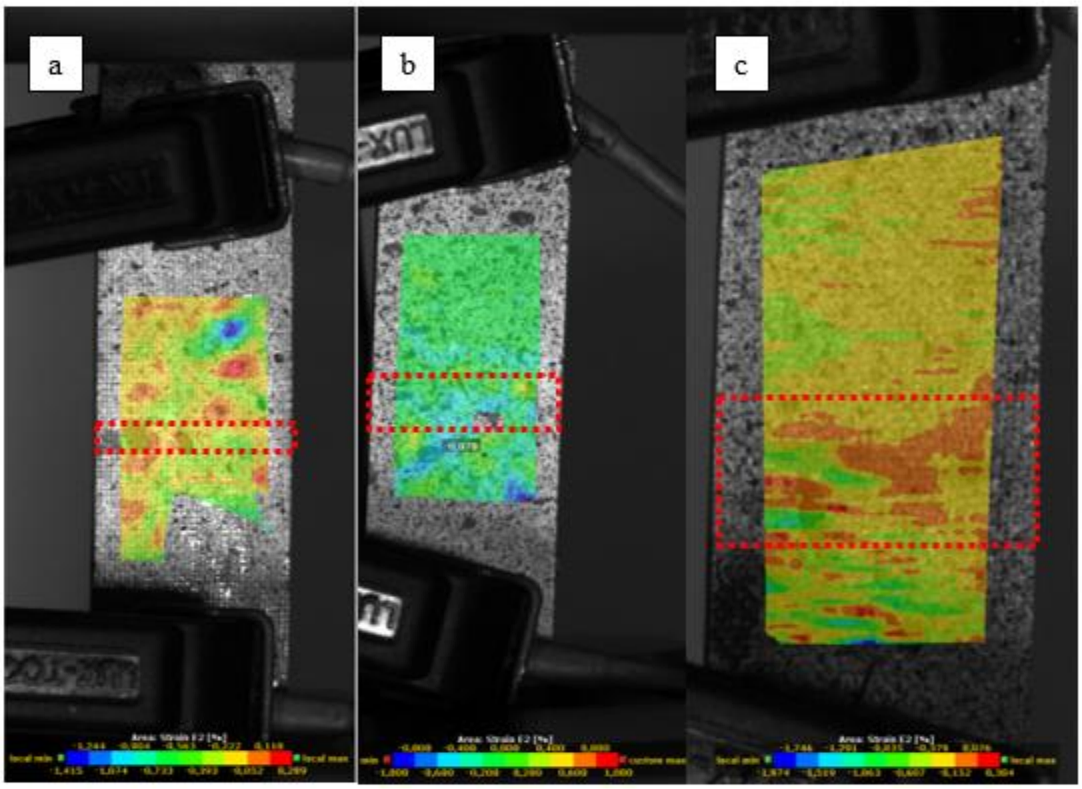

Fig. 6. characteristic DIC strain fields (a) 3 mm pattern width, (b) 5 mm pattern width, (c) 10 mm pattern width), edges of the pattern marked with red dashed rectangle

In the strain fields (E2 principal strain) a similar tendency can be observed as in case of the AE results. The 3 mm wide delamination did not introduce enough inhomogeneity in the strain field for localization. In case of the 5 mm wide printed pattern, the distorted strain field can be already observed in the inhomogeneous area. In case of the 10 mm wide printed pattern the DIC outperforms the AE technique. Not only the location but also the contour of the delamination can be seen. The different deformations observed in the delaminated zone are probably caused by the different adhesion levels between the matrix and the reinforcement. This can help to pinpoint the formation and progression of the delaminated zones. These are the most important advantages of using DIC for delamination/imperfection localization.

6. Conclusions

In our study we have created carbon fiber / epoxy composites with different interlaminar patterns introduced by 3D printing from PCL. We have compared the compressive characteristics of the samples with delaminations of different sizes. During the compressive tests we have performed AE and DIC data acquisition so the two techniques can be compared based on their performance to locate the artificially formed weakened adhesion zones. According to our results both AE and DIC were able to localize the delaminations. While AE localization could only detect the delamination edges, from the points with the highest cumulative event numbers, DIC gave us a better in-depth view of the status of the composite, with information about the contour and the internal adhesion levels.

In our further studies we will focus on using full-field DIC for damage localization in artificially and naturally damaged composite structures and check the feasibility of using DIC to pinpoint the onset of delamination progression in composites.

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