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Manufacturing of composites with designed failure

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Abstract: The failure of polymer composites is a very complex process, which mainly occurs as a result of interactions between different types of damage in the material, often at random locations and without any particular signs. To increase the reliability of composites and enable their wider use in safety-critical components, it is crucial to make their failure processes more controllable and predictable, which could be achieved by the local modification of the interfacial adhesion. The aim of our research is to develop and investigate a method for designing the failure process of polymer composites in terms of the location and the mode of failure. We produced interfacially engineered composites containing a weakened adhesion zone by adding polycaprolactone (PCL) thermoplastic interlayer material, which was applied directly on the surface of the reinforcing material by fused filament fabrication (FFF). Then, material tests were carried out, and the failure mode and position were evaluated as a function of the geometry of the interlayer material.

1. Introduction

Nowadays, polymer composites are becoming more and more common, mainly due to their excellent specific mechanical properties – especially strength and stiffness. Besides mass products, their applications include structural components as well. However, their further spread may be limited by their failure process, which can be considered unfavourable. Their heterogeneous microstructure, the significant differences in the properties of the reinforcement material and matrix, the presence of interfaces, and the anisotropic properties of composites all contribute to the complexity of their damage and failure mechanisms [1, 2].

Polymer composites contain different types of damage in a large number. Defects can be divided into several categories. Based on the source of the damage, material defects, manufacturing-induced flaws, and in-service damage can be distinguished. Damage can also vary in size, varying from the nano to the macro scale. The four basic types of damage are fiber failure – breakage or micro-buckling – matrix cracking, delamination, and fiber-matrix debonding [2-4].

Different forms of damage can occur simultaneously in the material under load. Their evolution and interactions can lead to the failure of the structure, which can take place in random positions or in several locations at the same time, often catastrophically [2-4].

Avoiding catastrophic failure by increasing the toughness of composites and achieving pseudo-ductile behaviour is a hot topic. There are several solutions for this purpose. Interface modification provides a great opportunity for it, for example, by applying nanofiber mats or polymer films [5, 6]. The application of polycaprolactone (PCL) interlayer material is also a promising interface engineering technique, which enables the local modification of adhesion. It is important that PCL is soluble in the epoxy matrix and does not create a new phase. The application of PCL interlayer material seems successful in modifying the failure behaviour and increasing the ductility of the material [7].

A further disadvantage of polymer composites is that their damages are challenging to detect and localise, but non-destructive testing (NDT) methods can offer a solution. Alongside widespread NDT methods such as acoustic emission testing and ultrasonic testing, the digital image correlation (DIC) technique is gaining popularity in the composite industry. DIC is a non-contact, optical method that has been used for a relatively long time in the materials testing of composites to determine the displacements and strain fields under load, which is able to detect the presence and propagation of meso- and macro-scale damage in the composite, based on high local strain values and changes in the strain-field [4, 8].

In our research, we manufactured composite samples containing PCL interlayer material as a locally weakened adhesion zone. The aim was to predetermine the failure in terms of location and mode. The failure process was investigated under different loading conditions.

2. Experimental

2.1. Materials

IPOX MR 3010 (IPOX Chemicals Kft., Budapest, Hungary) epoxy resin (EP) with IPOX MH 3124 curing agent was applied as matrix material. The components were mixed in a weight ratio of 100:35. We used PX35FBUD0300 (Zoltek Zrt., Nyergesújfalu, Hungary) stitch-bonded unidirectional carbon fabric (309 g/m2 surface weight), consisting of Panex35 50k roving, as reinforcement material. We applied eMorph175N05 (Shenzhen Esun Industrial Co. Ltd., Shenzhen, China) poly (ϵ-caprolactone) (PCL) filament (1.75 mm diameter, 60°C melting temperature, 180°C printing temperature) as interlayer material.

2.2. Test methods

Firstly, the fiber volume fractions of the composite plates were estimated from density measurements carried out with a Sartorius Quintix 125D (Sartorius Lab Instruments GmbH, Göttingen, Germany) weighing scale, based on the rule of mixtures.

Then, Charpy impact tests were carried out according to MSZ EN ISO 179-2 with a Ceast Fractovis 9350 impact tester equipped with a DAS 8000 data acquisition unit on composite specimens with 80 mm length and 10 mm width. The test was performed with a flatwise impact with 15 J impact energy and an impact velocity of 3.7 m/s with a support distance of 62 mm.

The tensile tests were carried out according to MSZ EN ISO 527-4 with a Zwick Z250 (Zwick GmbH, Ulm, Germany) universal testing machine equipped with a 250 kN load cell. Specimens with bonded end-tabs, a free length of 150 mm and a width of 15 mm were used for the tensile tests. The applied test speed was 5 mm/min. The displacements were measured with a Mercury Monet (Sobriety, Kurim, Czech Republic) optical strain measuring system (DIC) with a 5 MPixel resolution IDS U3-3080CP-P-GL (Imaging Development Systems Inc., Obersulm, Germany) camera and two LED lights. The results were recorded and processed with the Mercury RT-v2.9 software. For the data acquisition, a frame rate of 10 Hz was used. Before the test, the optical strain measurement system was calibrated to avoid lens distortion and to correlate the real size of the specimen with its size in the recorded images. For the global strain measurement, a gauge length of 75 mm was used with a line probe in the software. Two additional line probes were used, one for the further measurement of global strain with a length of 50 mm and one for local strain measurement in the region of the PCL interlayer material with a length of 15 mm. A strain field map was also generated by DIC, where the calculated points were 20 pixels from each other, and the facet window size was 40 pixels × 40 pixels.

2.3. Sample production

Reference composites without PCL and composite plates containing PCL interlayer material were manufactured with $[0°]_6$ layup sequence. For the latter, the PCL filament was printed directly on the surface of the carbon fabric by fused filament fabrication (FFF) method using a CraftBot Plus 3D printer. The samples manufactured for the impact tests contained interlayer material inclined at an angle of 45° to the fiber direction, so that the effect of the weakened adhesion zone on the failure location could be better observed. The interlayer material in the tensile test specimens was applied perpendicular to the fiber direction (Figure 1 and Figure 2). The interlayer material, thus the weakened adhesion zone, differed in geometry: the zone had the same width for each plate, only its thickness varied. Composite plates were produced by vacuum-assisted resin transfer moulding (VARTM). Then, specimens were cut from the composite plates with a Mutronic Diadisc (Mutronic, Rieden am Forggensee, Germany) diamond disc cutter.

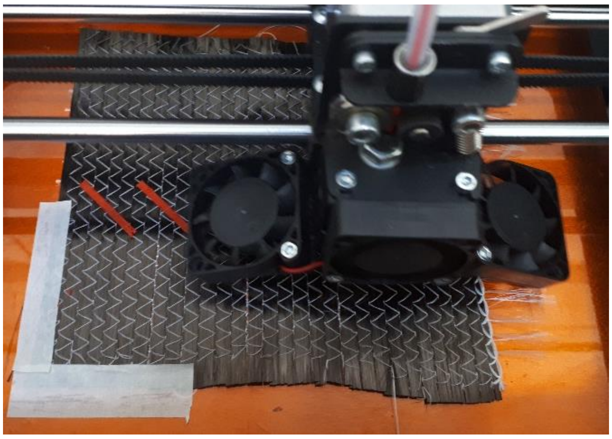

Figure 1. Printing of PCL on the UD carbon fabric by FFF method for the impact test specimen.

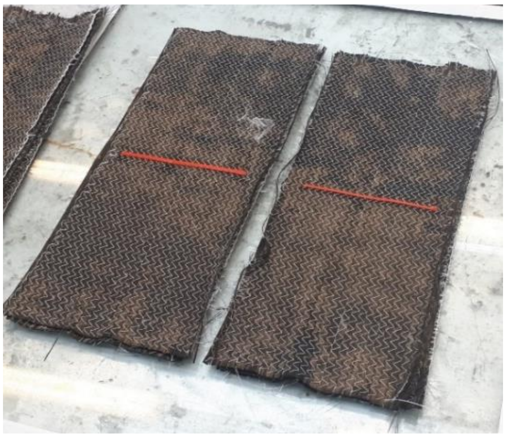

Figure 2. UD carbon fabric containing the PCL interlayer material during the manufacturing of the tensile test specimen.

3. Results

The average fiber volume fraction of the composite plates was 41.6±1.7 V/V%. These differences do not significantly affect the mechanical properties and the material behaviour.

3.1. Results of the impact tests

During the impact tests, the following samples were investigated:

- Reference samples containing no PCL,
- Specimens containing PCL interlayer material with a width of 2 mm and a thickness of 0.2 mm,
- Specimens containing PCL interlayer material with a width of 2 mm and a thickness of 0.8 mm.

First, the dynamic mechanical properties of the different composites were determined from the measured data (Table 1).

Table 1. The dynamic mechanical properties of the different composites.

	Impact strength [kJ/m^2]	Ductility index [-]
Reference	124.5±13.6	5.1±1.6
PCL with 2 mm width, 0.2 mm thickness	93.8±8.7	5.4±1.6
PCL with 2 mm width, 0.8 mm thickness	88.0±6.8	48.9±9.0

Table 1 shows that the ductility index is only significantly affected by the application of the 0.8 mm thick, 2 mm wide interlayer material, which results in a significant increase compared to the reference samples. In addition, it is important to note that the impact strength shows a decrement in case of composites containing interlayer material compared to the reference specimens, which can be explained by the presence of PCL.

Then, the failure mode and position were evaluated. The results show that the reference composites failed in a brittle manner along the line of the impact, as expected (Figure 3). For the composites containing PCL interlayer material, Figure 4 and Figure 5 suggest that the smaller thickness does not significantly affect either the fracture path or the mode of failure. However, the 0.8 mm thick interlayer material had a significant effect on both failure mode and position: two specimens failed by delamination, while for three specimens, the fracture path was increased, with failure occurring along the line of the interlayer material.

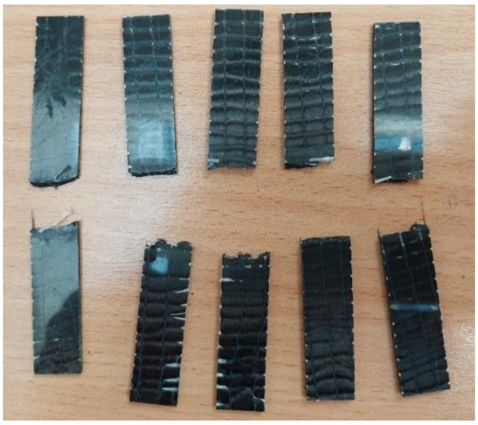

Figure 3. Reference specimens after the impact test.

Figure 4. The specimens containing PCL interlayer material with a width of 2 mm and a thickness of 0.2 mm after the impact test.

Figure 5. The specimens containing PCL interlayer material with a width of 2 mm and a thickness of 0.8 mm after the impact test.

Impact tests suggest that it is possible to influence the mode and position of failure by using PCL interlayer material, thus creating zones of weakened adhesion.

3.2. Results of the tensile tests

In the tensile tests, we focused more on the effect of the geometry of the PCL interlayer material, so we investigated the following composites:

- Reference samples containing no PCL,
- Specimens containing PCL interlayer material with a width of 2 mm and a thickness of 0.2 mm,
- Specimens containing PCL interlayer material with a width of 2 mm and a thickness of 0.4 mm,

- Specimens containing PCL interlayer material with a width of 2 mm and a thickness of 0.8 mm.

First, the mechanical properties – tensile strength, tensile modulus and strain at break – of the different composite plates were determined (Table 2 and Figures 6-8).

Table 2. The tensile properties of the different composites.

	Tensile strength [MPa]	Tensile modulus [GPa]	Strain at break [%]
Reference	993.7±78.3	98.7±2.5	1.15±0.12
PCL with 2 mm width, 0.2 mm thickness	1039.0±67.0	73.7±3.3	1.26±0.19
PCL with 2 mm width, 0.4 mm thickness	968.5±60.6	71.3±12.2	1.37±0.15
PCL with 2 mm width, 0.8 mm thickness	994.6±71.9	73.1±2.8	1.16±0.09

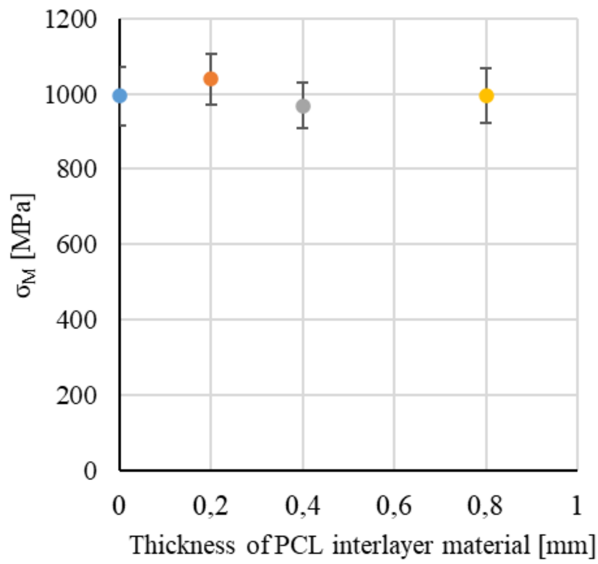

Figure 6. The effect of the thickness of PCL interlayer material on the tensile strength

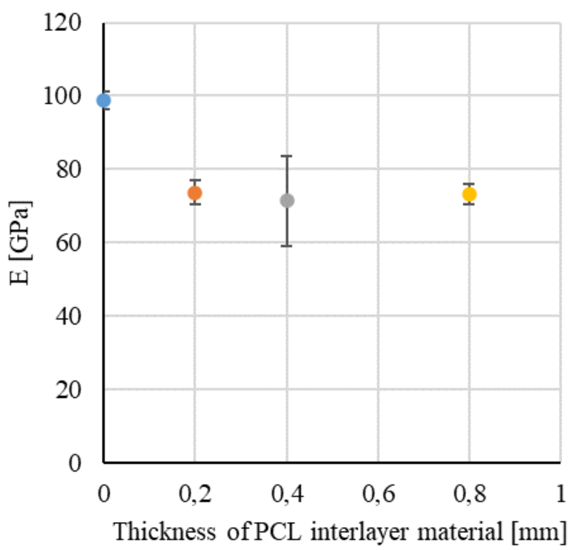

Figure 7. The effect of the thickness of PCL interlayer material on the tensile modulus

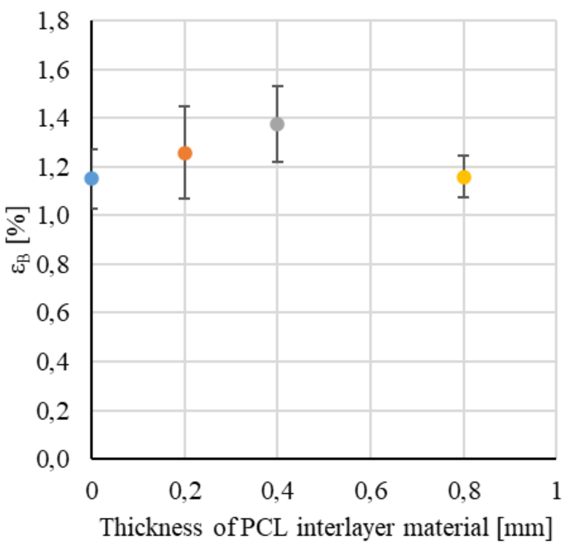

Figure 8. The effect of the thickness of PCL interlayer material on the strain at break

The results suggest that using interlayer material only leads to a significant change in the tensile modulus. The value of the tensile modulus decreases with the application of the interlayer material. However, the measured data show that for the same width, the thickness of the interlayer material does not have a significant effect on the value of the modulus.

Next, data from Line Probe (LP) and full-field measurements, recorded by DIC during the tensile tests were evaluated, which allowed the detection of possible damage, especially delamination. During the line probe test, we determined the elongation along three lines with different lengths, in the arrangement shown in Figure 9, so that line probes A2 and A3 provided information on the global strain, while A1 provided information on the local strain in the region of the interlayer material. The test was also performed for the reference specimens. The results of the line probe tests can be seen in Figures 10-13 for one representative specimen per specimen type.

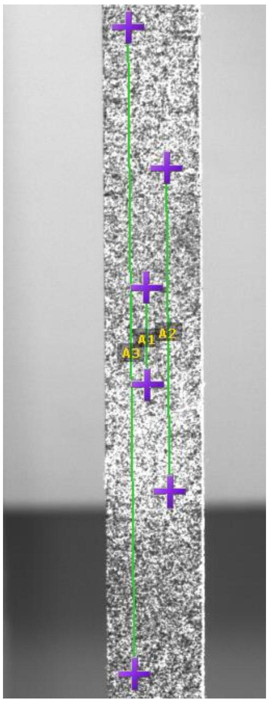

Figure 9. Arrangement of the line probe test, A1= 15 mm for local strain measurement, A2=50 mm and A3=75 mm for global strain measurement

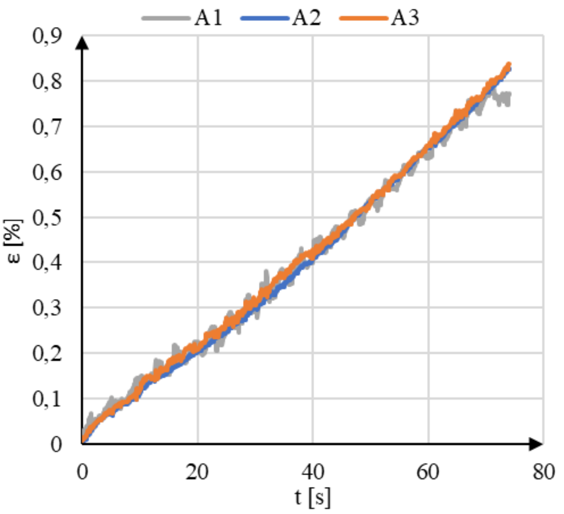

Figure 10. Typical result of a line probe test of a reference specimen

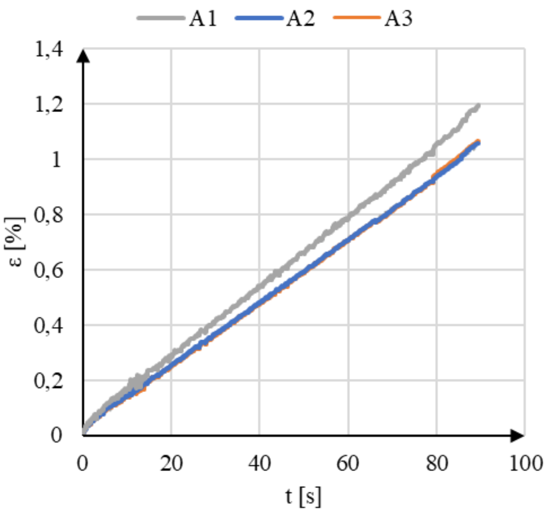

Figure 11. Typical result of a line probe test of a specimen with 2 mm wide, 0.2 mm thick PCL

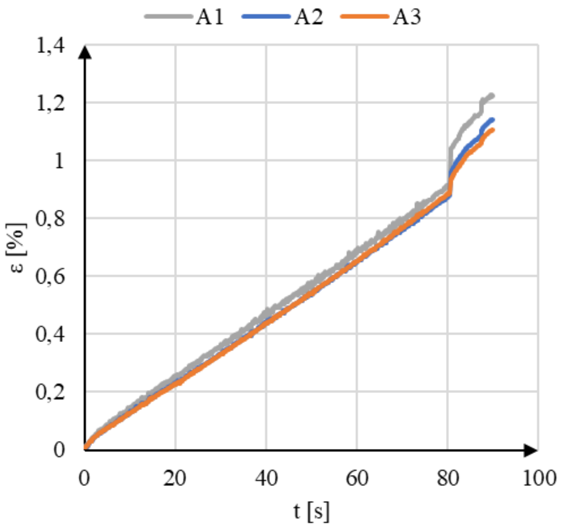

Figure 12. Typical result of a line probe test of a specimen with 2 mm wide, 0.4 mm thick PCL

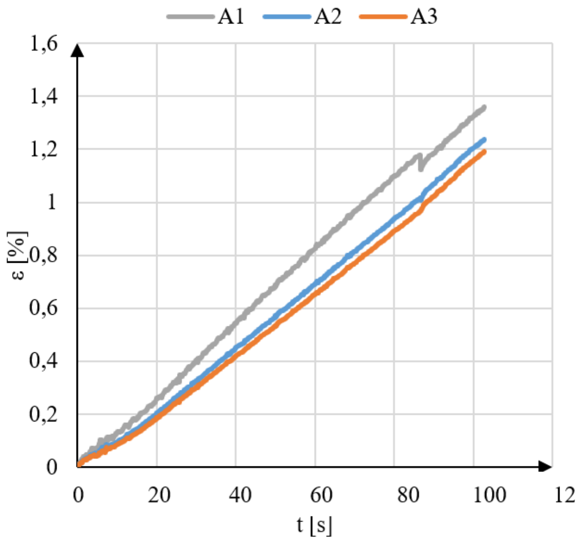

Figure 13. Typical result of a line probe test of a specimen with 2 mm wide, 0.8 mm thick PCL

The results of the line probe tests show that the local strain exceeds the global strain for specimens containing interlayer material, indicating the failure location and the presence of possible local delamination, likely as a result of the weakened adhesion zone. In contrast, there is no significant difference in the strains measured by the line probes for the reference sample.

We determined the strain-fields (50 mm x 15 mm) at the same global strain value for each specimen type (Figures 14-17).

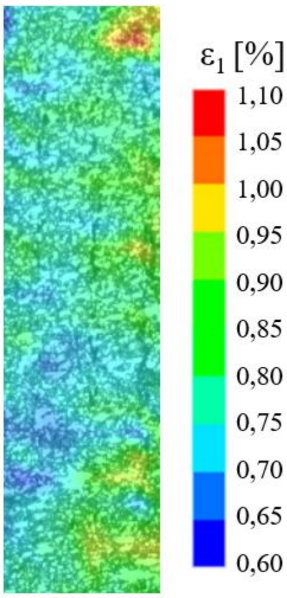

Figure 14. Typical strain field of a reference specimen

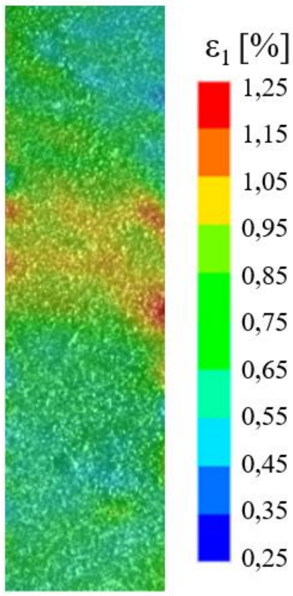

Figure 15. Typical strain field of a specimen with 2 mm wide, 0.2 mm thick PCL

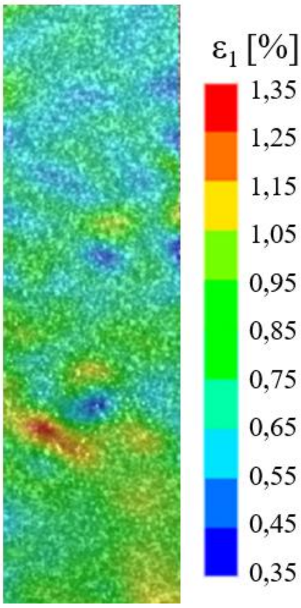

Figure 16. Typical strain field of a specimen with 2 mm wide, 0.4 mm thick PCL

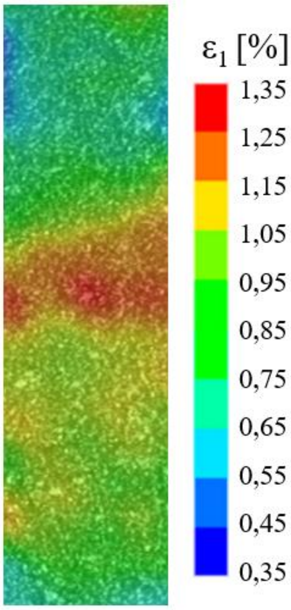

Figure 17. Typical strain field of a specimen with 2 mm wide, 0.8 mm thick PCL

The results of the full-field strain measurements confirmed the conclusions of the line probe test. The strain field of the reference sample is quite homogenous. It only contains a random failure, while the weakened adhesion zones formed by the PCL interlayer material cause a local increase in strain, predicting the location of the failure and indicating the formation of a delamination in the region. The interlayer material with 0.8 mm thickness has the most significant effect, which, in addition to modifying the adhesion, might create higher local strain and possible delamination through the formation of local fiber waviness and resin-rich areas.

4. Conclusion

We manufactured composite plates containing PCL interlayer material for the local modification of interlaminar adhesion. We investigated the effect of the application of PCL and the geometry of the weakened adhesion zone on the failure process and the mechanical properties of composite plates. We found that the use of PCL interlayer material enables the design of the failure mode and position, and the effectiveness of the method depends on the geometry of the weakened adhesion zone. The tensile tests suggest that the mechanical properties do not decrease significantly, while the impact test indicates a decrement in the impact strength but an increment in the ductility index.

5. References

[1] Talreja R and Singh C V 2012 *Damage and Failure of Composite Materials* (Cambridge: Cambridge University Press)
[2] Heslehurst R B 2014 *Defects and Damage in Composite Materials and Structures* (Boca Raton: CRC Press)
[3] Wang B, Zhong S C, Lee T L, Fancey K S and Mi J W 2020 Non-destructive testing and evaluation of composite materials/structures: A state-of-the-art review *Advances in Mechanical Engineering*. **12** 1-28
[4] Duchene P, Chaki S, Ayadi A and Krawczak P 2018 A review of non-destructive techniques used for mechanical damage assessment in polymer composites *Journal of Materials Science*. **53** 7915-7938

IOP Conf. Series: Materials Science and Engineering **1313** (2024) 012014 doi:10.1088/1757-899X/1313/1/012014

[5] Molnar K, Kostakova E and Meszaros L The effect of needleless electrospun nanofibrous interleaves on mechanical properties of carbon fabrics/epoxy laminates *Express Polymer Letters.* **8** 62-72
[6] Marino S G and Czél G 2021 Improving the performance of pseudo-ductile hybrid composites by film-interleaving *Composites Part A.* **142** 106233/1-106233/16
[7] Magyar B, Czigány T and Szebényi G 2021 Metal-alike polymer composites: The effect of inter-layer content on the pseudo-ductile behaviour of carbon fibre/epoxy resin materials *Composites Science and Technology*. **215** 109002/1-109002/8
[8] Hliva V and Szebényi G 2023 Non-Destructive Evaluation and Damage Determination of Fiber-Reinforced Composites by Digital Image Correlation *Journal of Nondestructive Evaluation.* **42** 43/1-43/15

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