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Effect of matrix modification of a sandwich composite on selected strength properties

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ABSTRACT

Improvements in materials and the availability of an apparatus enabling mechanical, strength, thermal testing of sandwich structures, including fatigue tests, allow for a dynamic development of research work confirming the effectiveness of the use of structural composites as energy-intensive structures. This paper presents the results of selected strength tests of a layered epoxy-glass composite with a polyurethane-modified matrix and a porous core. The conducted experimental studies were proposed as a basis for the creation of a test procedure for comprehensive strength and mechanical characterisation of the composite. Obtaining a layered material with a modified matrix is a formative factor for impact energy absorption properties. The quantitative and qualitative effects of material selection and matrix modification on impact properties were characterised, and an analysis of composite damage and component interaction under loads occurring during the proposed experimental tests was carried out.

Keywords: composites, mechanical engineering, modification of composites, mechanical properties.

INTRODUCTION

Composites are most commonly used for the design of energy-intensive structures due to their very good properties, low density and the highest ratio of strength and stiffness to specific weight among materials [1, 2, 3] and when compared with metallic materials, they exhibit much higher values of relative absorption energy.

The sandwich composite design generates many more damage possibilities, the potential occurrence of which should be particularly considered at the design stage. An important problem of sandwich composites is the strategy of introducing external loads [4]. The effect of low-energy impacts is often subsurface delamination, which is difficult to identify visually or manifests itself as a very slight deformation of the composite surface. This type of damage, which causes progressive degradation of structural properties, is referred to as barely visible impact damage (BVID) [5]. The fluctuating stresses lead to a loss of stiffness and ultimately to uncontrolled structural failure, hence the importance of monitoring highly stressed composite structures, reacting in a timely manner and performing the necessary structural maintenance.

An important aspect is also the choice of materials. Using epoxy resin as matrix, a favourable price/quality ratio is obtained, but its weaknesses are brittleness and low resistance to cracking [6]. In order to eliminate the disadvantages of the epoxy matrix and to improve the mechanical and processing properties obtained, various types of modifications are used. These modifications may involve the addition of appropriate modifiers or fillers, e.g. based on aromatic polyethers and polysiloxane elastomers [7], on thermoset rubbery [8] and liquid natural rubber [9], dispersion of clay platelets [10], hyperbranched polyester [11], low concentrations of comb-shaped fluorinated [12], polyurethane [13, 16], carboxylated nitrile rubber

[14], other different types of elastomer [15], or modifications in the composite structure in the case of multilayer structural composites [17].

Easy availability and very good properties mean that porous components combined with other materials allow attaining the strength parameters not achievable with a single component. The trend towards lighter structures has allowed porous materials to qualify as a new generation material. In standing structures, weight is a secondary requirement, while in vehicles, appliances, machinery, flying, portable and protective structures it is of paramount importance [18].

Fibre-reinforced polymer (FRP) composites meet the requirements of high material strength with a low specific weight of the composite. The fibres take the largest part in load transfer, while the matrix binds the fibres together and protects them from external influences [2]. The operational demands placed on fibres are very similar to those placed on the matrix itself, i.e. high strength parameters and the maintenance of relatively stable properties during operation at different temperatures and under the influence of various external factors (atmospheric, chemical) [19]. In a sandwich composite, the core is responsible for counteracting the deformations caused by stresses perpendicular to the surface of the cladding layers. Its properties vary according to the arrangement in such a way as to optimise the selected properties of the overall composite as much as possible [20].

The type of damage resulting in strength reduction and the ability to absorb impact energy are key aspects that determine the impact characteristics of structural composites, on which the impact resistance and effectiveness of the protective properties of the structure largely depend.

Energy-absorbing composites with layered structures are often subjected to impact loading perpendicular to the surface. Visible or invisible deformations occur as a result of such loading. The deformations that are difficult to identify generate an uncontrolled reduction in the strength and stiffness of the composite, initiated within the material by fibre or matrix cracking or delamination [19]. Various methods are used to characterise composites under impact loading [21], among others, high [22] and low [24] impact velocity tests and specific ballistic impacts [23]. The impact resistance of a composite material is usually determined by key properties: damage tolerance and puncture resistance. Damage tolerance is directly related to impact damage mechanisms. It signifies the ability of the material to carry the load despite its occurrence. The magnitude of the load and the damage as well as its nature should reflect the operating conditions and especially the damage should be able to be located and assessed by a specific method based on unambiguous parameters [22].

The puncture resistance of structural composites usually consists of two stages:

- exposure to transverse impact loads (impact);
- testing of a selected strength or mechanical property which, in relation to the to the impacted specimen to determine the so-called residual strength.

Many studies also focus attention on compression after impact (CAI) tests; however, it is much more common for sandwich composites to be required to withstand bending loads, hence they should be subjected to bending after impact (BAI) tests [25, 26]. Dynamic mechanical analysis (DMA), on the other hand, is a method that characterises the mechanical properties of materials simultaneously as a function of time, frequency and temperature [27] These tests are also worth comparing with other tests such as bending or compression strength to be able to fully reflect service conditions. It also appears to be important that there is little literature data on the use of chemical modification of the matrix of sandwich composites as a factor in improving material properties. Finding this research gap allowed the work to be carried out and the objective to be formulated by appropriately selecting a modifier in combination with an epoxy matrix, glass fabric reinforcement and a lightweight flexible core, a composite with very good mechanical and strength properties was obtained at a low production cost, and the proposed experimental testing procedure was designed to determine the effect of modification, composition and correlation between composite layers on selected strength characteristics and failure mechanisms.

MATERIALS AND METHODS

The epoxy resin was modified with polyurethane to create interpenetrating polymer networks (*IPN*), which significantly enhance its strength properties [28]. Without this modification, the resin is often deemed unsuitable for structural applications due to its high brittleness and lower strength compared to other plastics. The amount of polyurethane added was determined based on previous studies on epoxy resin modification [29]. Table 1 outlines the components necessary for producing the structural composite.

The initial composite layer was created by thoroughly saturating the glass fabric with the modified resin and evenly distributing it across the entire reinforcement surface. An extruded polystyrene (*XPS*) layer was then placed on top of this prepared layer. Subsequently, another layer of resin-impregnated fabric was applied over the core. This process was repeated until a total of three core layers and four reinforced cladding layers were formed. The specimens were then cut using a diamond blade cutter in accordance with the experimental test standards.

Five composite specimens of both types were prepared for each test.

Dynamic mechanical analysis of the cladding layers was carried out using a DMA Q800 (TA Instruments, USA). The specimens measuring 40 × 15 × 1 mm were tested on cantilevers at 1 Hz in the temperature range from -120 to 140 °C (heating rate: 4 °C/min). The conservative and loss modulus and tangent δ as a function of temperature were recorded.

Impact tests of composite specimens with a rectangular cross-section (without notches) were tested using the Charpy method according to ISO 179 for layered materials [30]. The dimensions of the specimens were 100 × 15 and the thickness dependent on the number of core layers. The specimens were struck with a pendulum hammer with an energy of 7.5 J using an Impact 25 apparatus (Galdabini, Cardano al Campo (VA), Italy) with a support spacing of 75 mm so that the hammer struck exactly in the centre of the specimen. The composites were impacted in two directions: perpendicular (plane impact) and parallel (edge impact) bv.

The drop-weight impact test was performed using an Instron CEAST 9340 testing machine. The specimens were placed freely on a cylindrical base with holes. A hemispherical impactor attached with a 2.65 kg weight was dropped from a height of 385 mm at a speed of 2.74 m/s to generate an impact of 10 J.

The residual strength of the composites was determined using the bending after impact (*BAI*) method. It is directly related to the falling weight impact test and the bending strength.

The composite specimen was first impacted with an appropriate energy. Then, the material strength changes were determined according to the three-point bending scheme on the Zwick Roell Z-100 universal testing machine. The tests were carried out in accordance with the EN ISO 178 standard with distance of supports equal to 55 mm. The reference values were the specimens subjected to the bending test only (Bending Only).

RESULTS

On the basis of previous experimental studies using PU in the range of 0–20% and an analysis of the literature [16], a modification of the matrix in the amount of wt. 0–10% PU, striking a balance between improving the mechanical properties of the composite and minimising the material losses resulting from achieving only a slight improvement in strength parameters. The liquid polymer modification used in this study led to the formation of interpenetrating polymer networks (Figure 1a) between the reactive groups – OCN in the structural chain of the polyurethane and the resin. It was noteworthy that polyurethane could also form chemical bonds with the hydroxyl groups present on the glass surface (Figure 1b).

Due to the novelty of using chemical modification of the matrix, the composite layer (matrix + reinforcement) was subjected to dynamic mechanical analysis (DMA), which is an effective form of characterising sandwich composites under varying conditions of temperature, frequency and strain. It allows the composite manufacturing process to be evaluated and the properties of the entire composite to be visualised, as it is largely

Table 1. Components to produce the structural composite

Core	Reinforcement	Matrix	Modifier	Hardener
XPS foam (3 mm)	Glass fabric (250 g/m^2 plain) (324 g/m^2 twill)	Epoxy resin (Epidian 5)	Liqud polyurethane (Desmocap 12)	Triethylenetetramine (*Z1*)

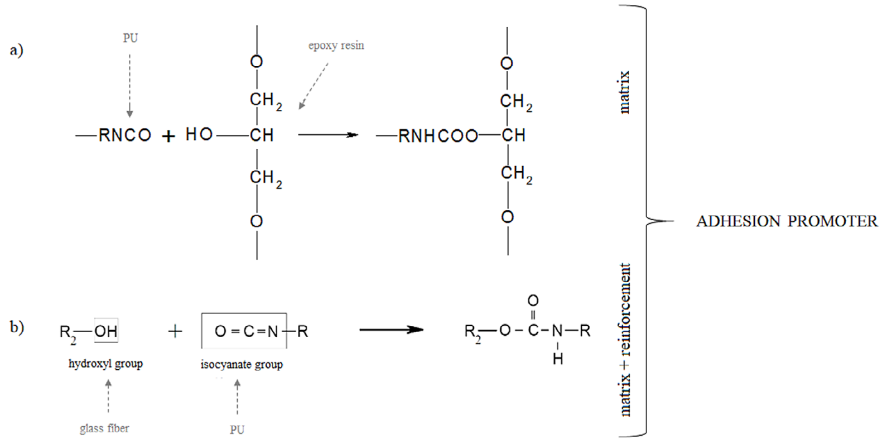


Figure 1. The formation of interpenetrating polymer networks (a) between the reactive groups – OCN, (b) with hydroxyl groups present on the glass surface

the cladding in the transmission of loads (including impact loads). Figures 2–3 present a summary of the properties measured during the dynamic mechanical analysis of the cladding (storage modulus, loss modulus and tangens *δ*).

The DMA test made it possible to characterise the composite layer and confirm the mechanism of reinforcement depending on the modifier content in the negative and positive temperature range and under constant, repeated loading. The addition of 5 wt% modifier in the epoxy matrix with GF250 reinforcement in the positive temperature range resulted in an increase in the loss modulus E (169.80 MPa). The ratio of material stiffness to viscoelastic properties (tgδ) was lowest for the EP0250GF composite (0.18) and highest for the 10PU250GF composite (0.37). The conservative modulus of the composites, which determines the stiffness of the material, showed that the modified composites exhibited a sharp decrease in stiffness at temperatures around 60–70 °C. The curves as a function of negative temperatures showed characteristic peaks in the loss modulus, indicative of a low-temperature secondary transition of the matrix (secondary transition) inherent in the reinforcement properties.

The analysis of the mechanical properties of the composites started with Charpy impact tests in two impact directions. The results are shown in Table 2.

The maximum impact strength in the perpendicular impact test was noted for specimen 10PU250GF3XPS – 11.82 kJ/m^2, an increase of 10% compared to the composite without the modified matrix. In the impact test in the parallel direction, the maximum value was recorded for specimen 5PU324GF – 30 kJ/m^2. Plain fabric has a higher stiffness compared to twill fabric, but considering the failure mechanisms, composites reinforced with twill fabric show better behaviour from the point of view of shielding structural applications. Examples of failure modes for unmodified and PU-modified composites with 5% PU content are shown in Figure 4.

Microscopic observations were also made, examples of which are shown below (Figure 5). The addition of the modifier effectively inhibited crack propagation in the cladding layer, also counteracting fibre cracking.

Observing the slope, the shape of the graphs (Figure 6) and the area under the curve, the information about the stiffness of the material and the amount of impact energy absorbed is obtained. For the 5PU250XPS composites, the lowest displacement (22 mm) and the highest value of load absorbed by the material (1399.42 N) were observed. The reduction in average displacement was therefore about 5% compared to the reference composite and about 10% compared to the composite containing 10% modifier by weight. The reduction in displacement also indicates an increase in the resistance of the composite to delamination, as confirmed by visual identification after testing. Analysis of the behaviour of the plain fabric reinforced materials therefore suggests that the low PU content effectively reduces perforation displacement while increasing puncture resistance.

From the force-displacement diagram and the results of individual specimens generated by the test programme, it is also possible to calculate the absorption energy of the composite, i.e.

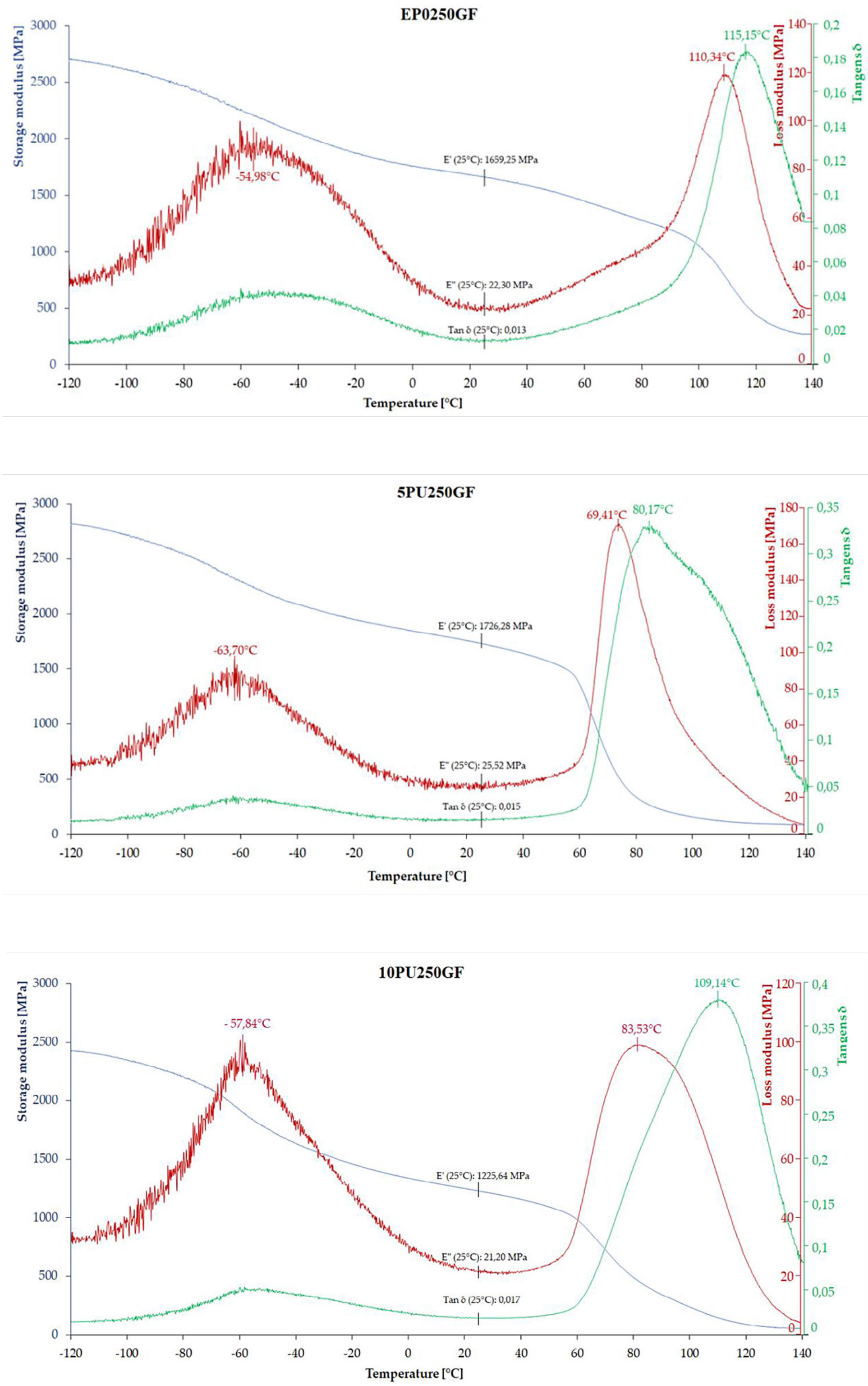


Figure 2. Dynamic mechanical analysis of EP/PU/250GF composites

the actual value of the energy absorbed by the composite as a result of the impact. This parameter is one of the most important characteristics of materials used as protective structures in vehicles and machinery. The absorption energy (EA) was calculated using equation (1), and the values of the energy absorbed by the composite are summarised in Table 3.

$$EA = \int_0^l P \cdot dl \qquad (1)$$

where: P – the value of the destructive force and dl is the displacement.

Visual identification of damage in the composites also confirms the improved damage tolerance by modifying the matrix. No visible damage was observed in the lower layers of the polyurethane-modified matrix composites, indicating that the outer (Figure 7) and middle layers of the composite effectively inhibit damage propagation. This confirms the potential of using polyurethane-modified composites as components or equipment shields, as the lower part of the composite (closest to the component to be protected) does not lose the continuity of its structure and effectively protects the component inside the shield. The composites with an unmodified matrix cracked in both the top layer and the bottom cladding layer

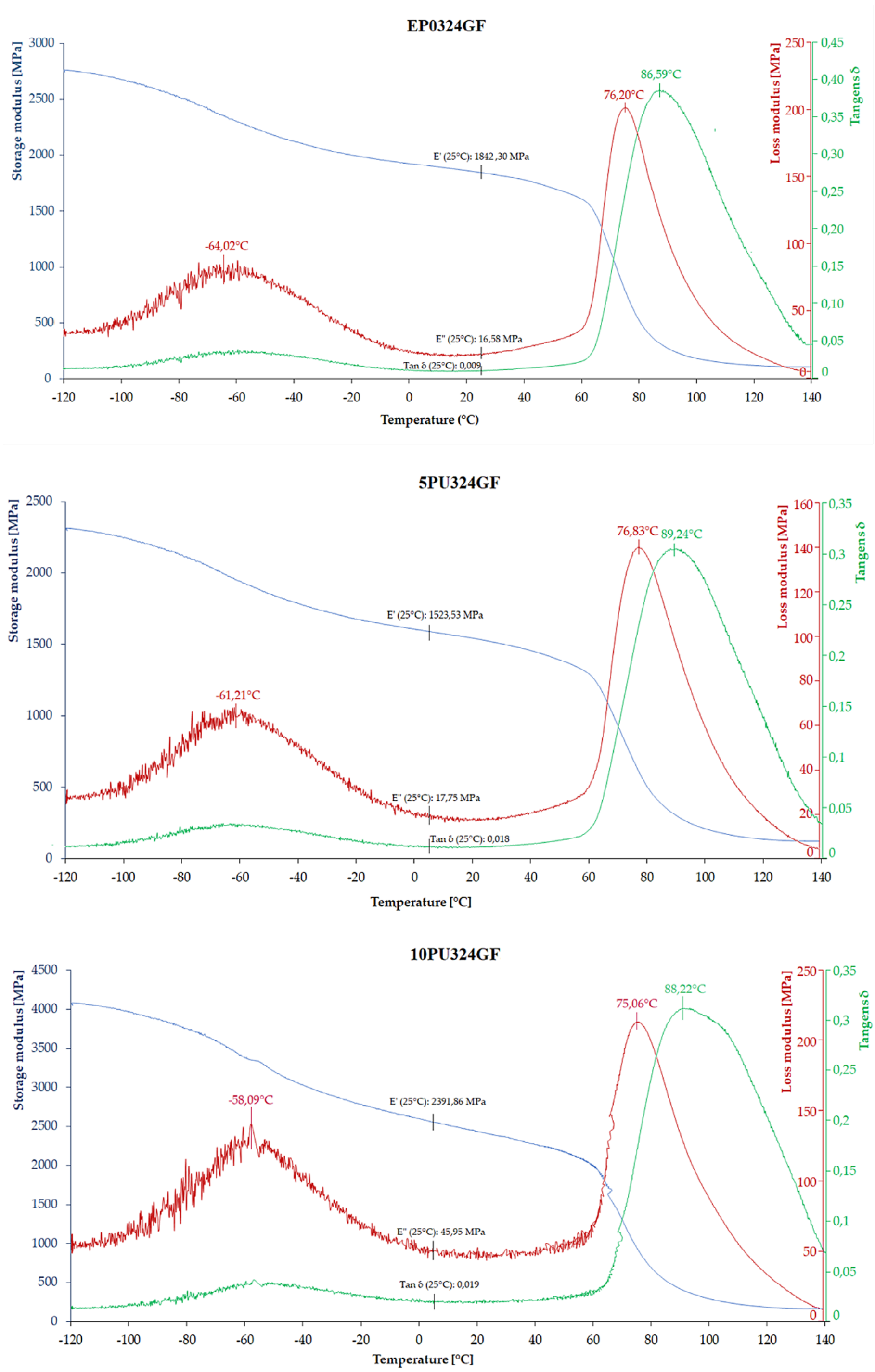


Figure 3. Dynamic mechanical analysis of EP/PU/324GF composites

Table 2. Impact test results

No.	Type of composite	Impact test [kJ/m^2]	
		Perpendicular	Parallel
1	EP0250GF	10.84	14.08
2	EP0324GF	8.76	23.66
3	5PU250GF	11.28	17.67
4	5PU342GF	8.56	30.00
5	10PU250GF	11.82	18.58
6	10PU324GF	8.86	22.80

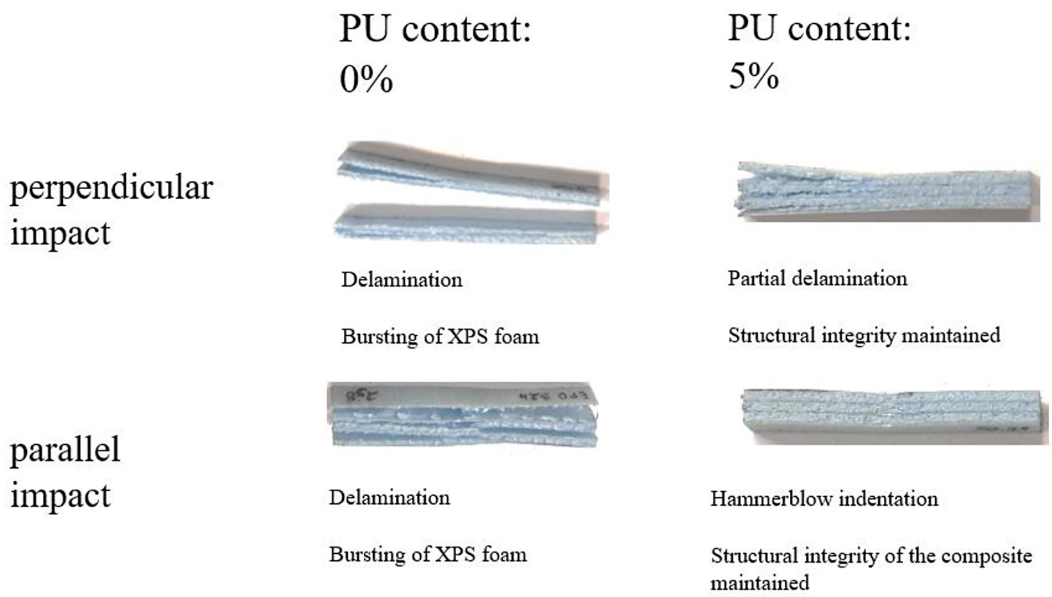


Figure 4. failure modes for unmodified and PU-modified composites with 5% PU content

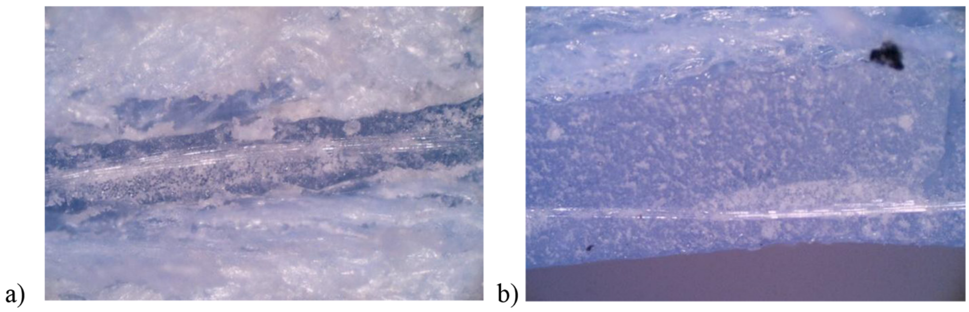


Figure 5. (a) 10 PUR 250 GF after impact test, impact area and middle layer
(b) 10 PUR 250 GF – after impact test, impact area and surface layer

(Figure 8), implying a very low ability to absorb impact energy (low damage tolerance).

The puncture resistance of composites with three core layers was determined by three-point bending after impact (BAI). The method allows an assessment of the degree of reduction in the values of the determined properties after impact and bending compared to the original values (bendable composites only). Table 4 summarises the results obtained from the bending test of composites before and after impact. The non-impacted specimens are denoted as BO and the specimens subjected to the falling weight test before the bending test are denoted as BAI. Puncture resistance is one of the parameters that is crucial in load-bearing applications subjected to impact loads.

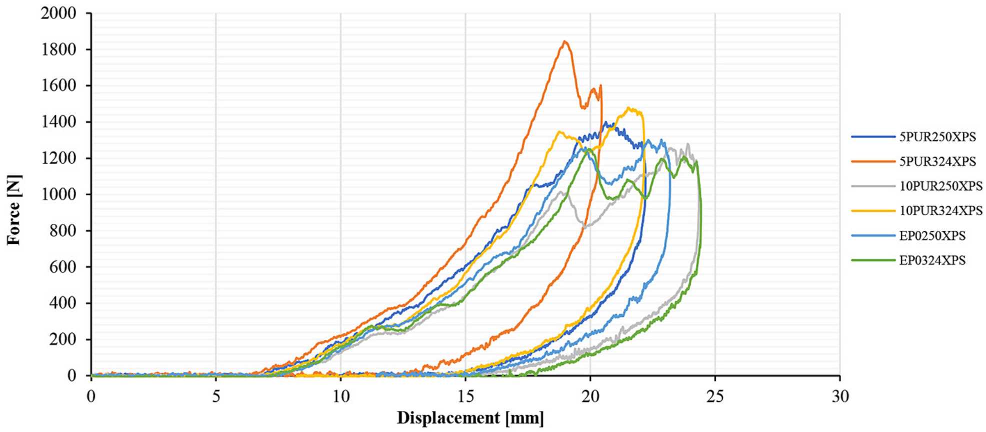
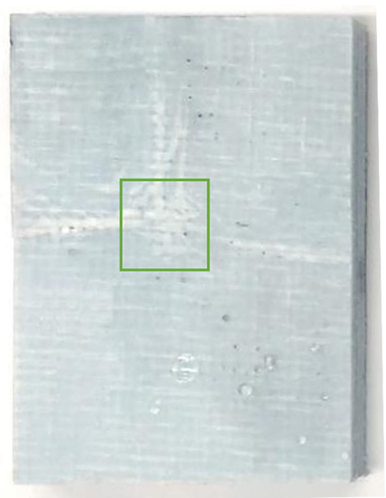


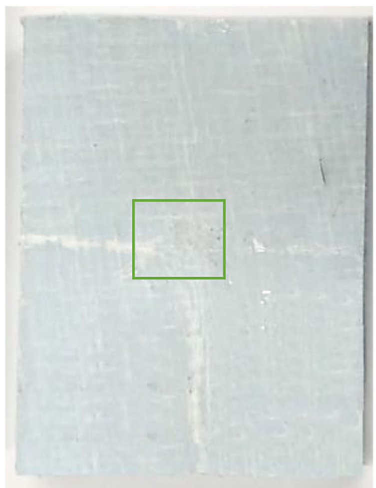
Figure 6. Force-displacement curve in the damage tolerance test

Table 3. Energy absorbed by the composite

No.	Type of composite	Energy absorbed by the composites	
1	EP0250GF	7.89 J	± 0.40 J
2	5PU250GF	8.17 J	± 0.41 J
3	10PU250GF	7.92 J	± 0.40 J
4	EP0324GF	7.08 J	± 0.35 J
5	5PU342GF	7.76 J	± 0.39 J
6	10PU324GF	8.90 J	± 0.45 J



5PU250GF3XPS



5PU324GF3XPS

Figure 7. Damage to the upper cladding of *5PU* composites

Analysis of the results shows that the composites with 5% *PU* have the highest residual strength. For the 5PU324GF3XPS composite, both its initial E_z value (9.11 kJ/m^2) and its value after impact and bending (8.11 kJ/m^2) were the highest among the materials tested. This means that the composite retained 89% of its initial strength, considering the amount of work required to destroy the specimen. The 10PU324GF3XPS composite had the lowest failure energy, confirming the conclusions of other studies that the 10% polyurethane proportion is too high and contributes to the deterioration of properties.

When considering the results determining the flexural modulus of the composites tested, the composites with 5% modifier content also

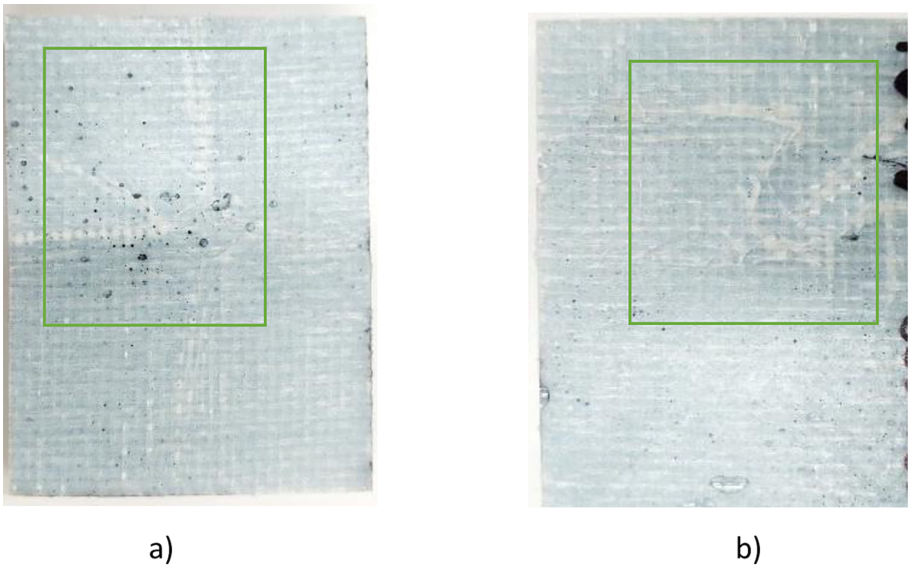


Figure 8. Example of damage to composites with unmodified resin after a 10 J impact; (a) top cladding, (b) bottom cladding

Table 4. Results of the bending test of composites before and after impact

No.	Type of composite	E_z [kJ/m^2]			Flexural modulus [MPa]		
		BO	BAI	Residual strength	BO	BAI	Residual strength
1.	EP0250GF3XPS	6.38	5.03	79%	41.95	26.40	62%
2.	EP0324GF3XPS	7..21	5.69	78%	50.20	26.95	53%
3.	5PU250GF3XPS	6..55	5.32	81%	35.57	27.10	76%
4.	5PU324GF3XPS	9.11	8.11	89%	42.50	32.00	75%
5.	10PU250GF3XPS	6.47	4.97	76%	40.75	24.90	61%
6.	10PU324GF3XPS	8.55	5.61	65%	42.20	23.15	55%

showed a lower decrease in stiffness when compared to the other materials. Although the bending modulus in the BO test for these composites was initially the lowest (35.57 MPa), after the BAI test their modulus value was the highest of all composites, retaining 75–76% of the primary strength (27.10 MPa). The EP0324GF3XPS composite had the lowest puncture resistance, with its flexural modulus almost halving from its primary value (BO – 50.20 MPa, BAI – 26.95 MPa).

CONCLUSIONS

The applied liquid polymer modification caused the reactive -OCN groups in the structural chain of the polyurethane to form IPN polymer networks with the resin, which interpenetrate each other. Reinforcement at the physical level – the polyurethane acts as a plasticising polymer filler – and at the chemical level – the modifier acts as an adhesion promoter.

The materials used in the preparation of the composite, the introduction of internal composite layers into the composite structure and the grading of the core have a beneficial effect on the durability, properties of the composite:

The addition of 5 wt% modifier in the epoxy matrix with GF250 reinforcement in the positive temperature range resulted in an increase in the loss modulus E (169.80 MPa). The ratio of material stiffness to viscoelastic properties (tgδ) was highest for the 10PU250GF composite (0.37) an increase of 10% compared to the composite without the modified matrix in the perpendicular impact test was noted for specimen 10PU250GF3XPS – 11.82 kJ/m^2.

The composites with 5% *PU* have the highest residual strength – the 5PU324GF3XPS composite, both its initial E_z value (9.11 kJ/m^2) and its value after impact and bending (8.11 kJ/m^2).

The flexural modulus of the composites tested, the composites with 5% modifier content also showed a lower decrease, after the BAI test their modulus value was the highest of all composites, retaining 75–76% of the primary strength (27.10 MPa).

Visual identification of surface damage showed very similar behaviour of the composites – impactor point impact trace and linear cracks radiating in two directions. The unmodified composites cracked in the matrix layer, also damaging the fibres in the reinforcement layer.

Modified composites maintained structural continuity in the composite layer due to the elasticising effect of the modifier.

Visual identification of damage in composites with modified matrix showed a significantly better load response during bending (BO) and post-bending impact (BAI) tests. The inhibition of crack propagation was the result of good matrix adhesion and reinforcement caused by the action of the polyurethane modifier.

In summary, the epoxy-glass composites with 5% polyurethane and XPS foam core have an effective ability to absorb shock energy or impact load and very good response parameters to bending and compressive loads. This proves the validity of using chemical modification that has a significant impact on the properties of the composite confirmed by experimental studies. in addition, The very low cost of manufacturing EP/PU/GF/XPS composites with a favourable price-to-property ratio of the layered material is a noteworthy aspect considered at the design stage. This response of the material allows it to be used effectively in protective applications.

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