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The influence of the matrix properties on mechanical properties of a glass fiber reinforced polymer made by the infusion method

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ABSTRACT

At every stage of the development of modern aerospace structures, engineers are keen to obtain the lightest possible product while maintaining high strength. In order to meet such requirements, composite materials are now widely used in the aerospace industry, with manufacturing methods constantly evolving. This thesis aims to investigate and analyze the influence of the matrix on the strength of a layered composite made using the infusion method. This method is currently among the most popular and effective ones. The technique makes it possible to produce a robust laminate using optimum labour and favourable costs. However, a number of rules must be followed during the production process to produce a satisfactory product. In relation to the objective of the thesis, three different composites were produced and tested, differing in the properties of the used resin composition. The matrixes were different in terms of the use of processes such as degassing and the ratio of epoxy resin to hardener. During the research, tensile strength, impact strength and bending tests were carried out. This work shows that on the basis of the results obtained from the strength tests and observations of the internal and external structure of the tested materials, it can be concluded that the ratio of resin and hardener has little effect on the strength properties of the material. The results also show that the specific conditions of composite production by the infusion method greatly affect the spontaneous degassing of the resin mixture.

Keywords: multilayered composites, composite manufacturing processes, infusion method, GFRP.

INTRODUCTION

The aerospace industry continues to grow and, consequently, the demands on the materials used in this industry are increasing [17]. In the early days of aviation, traditional materials such as wood and fabric were used, followed by metals and their alloys. Currently, solutions are being sought that allow for further weight reduction while still meeting high strength requirements [3, 5].

Composites are an example of plastics that meet these expectations. The use of layered composites in the construction of aircraft components such as coverings, enclosures, or shields allows for a reduction in fuel consumption costs by lowering the overall weight, thereby enabling carrying heavier loads.

In line with the ongoing trend to continuously modernize composite materials, innovations in their manufacturing are being sought, also pertaining to well-established production methods for these materials [13]. In the case of composite materials, original composite manufacturing methods are in-situ metallic based composites [29] fabrication of ceramic rich composite coatings (ceremets) via high velocity oxy-fuel (HVOF) spraying [30] or even reinforcing metallic structures with carbon fibre reinforced polymer (CFRP) composites [31]. The composite material consists of two main phases, namely reinforcement and matrix. In the case of reinforcement, it is responsible for the transfer of any loads to which the composite element is subjected. It must be added that it is definitely

a higher strength component. The matrix, on the other hand, is designed to bind and protect the reinforcement from environmental factors. Particles and fibres, such as glass fibre, can be used as reinforcement [32]. It is widely used in aviation due to its high tensile strength and non-flammability. The selection of the proportions of the composite components depends on the expected properties and its application.

Among the manufacturing methods, it is possible to distinguish the infusion method [1], which is currently one of the most popular and effective ways of producing composites. An important advantage of the infusion method is its low harmfulness and the ability to automate the process, which enables speeding it up, reducing labour input and faster production of manufactured components.

Glass fibres are an interesting material primarily due to their attractive property-to-price ratio [9, 10, 11]. They now form a very large part of the reinforcements used in the fibre-reinforced polymer composite industry, including aviation. Another material widely used in aviation is carbon fibre, which is characterized by high lightness and strength. It is used to manufacture components such as fuselages, wings, and control surfaces. It is also possible to use them to produce blades in rotorcraft, providing high resistance to external loads [33]. Papers [33, 34] contain examples of the use of this type of material for the fuselage of an unmanned aerial vehicle. The manufactured successive fuselage versions were tested for strength on specially designed test stands.

In the case of glass fibres, the characteristic feature is their high tensile strength and low Young's modulus, as well as their high shear modulus [4, 7]. With this type of fibre, their strength is diameter-dependent. As the diameter of the section decreases, its strength increases. It should also be noted that the smaller the cross-sectional diameter, the higher the elastic modulus and tensile strength, although at the same time the elongation at break decreases. Glass fibres achieve high strength due to their internal structure. However, many scratches, cracks and other irregular changes can be seen on their surface, which in turn reduces their mechanical properties. Fibre glass deforms elastically because the elastic limit and the breaking point are almost identical.

A typical E-glass fibre, with a diameter of approximately 10 μm, has a tensile strength of 1,000–1,400 MPa, with an elongation at break of 1.5–3.5%, and an elastic modulus of up to 77 GPa.

Another crucial property of glass fibre is its non-combustibility. It can operate at temperatures up to 300 °C without any change in strength. In the case of negative temperatures, no changes in strength properties are observed at -50 °C [26].

For the development of the article, three different composites were produced and tested, differing in the type of the used resin composition. The composites differed as a result of the use or non-use of liquid matrix degassing and as a result of the different proportions of epoxy resin to hardener. The scope of the study included the following activities: infusion manufacture of three composites, each differing in matrix properties, microscopic examination of the internal and external structure, and examination of the mechanical properties of samples prepared from the manufactured composites. Author's of the article wanted to check how different proportions of epoxy resin to hardener influence the results of strength tests (tensile strength, impact strength and bending tests) in the infusion method manufacture of composites. During research the thesis was also verified – that the infusion method production of layered composites largely affects spontaneous degassing of the resin mixture used in this method.

Preparation of materials for testing

In order to carry out the research, a composite panel was manufactured from nine layers of 390 g/m^2 glass fibre fabric with 370 g/m^2 saturation and "twill" weave [2, 7, 8]. A delamination layer was also applied to facilitate the separation of the other layers from the composite. In addition, a special mesh was used to facilitate the smooth and even distribution of the resin with a hardener in the reinforcement fabric.

After the reinforcing fabric sheets were cut, a mould was prepared - it was a flat pane of glass with a working area delineated with a butyl tape. The tape was bonded taking into account the dimensions of the composite panel as intended (300×600 mm). The size of the mould was 100 mm larger than the cut materials on each side in order to provide auxiliary space. The working area of the mould was waxed with TR Industries 104 wax in accordance with the manufacturer's recommended technology. This procedure was intended to facilitate the separation of the

fabricated composite from the mould. Nine layers of glass fabric were then laid in the mould, maintaining the appropriate distances from the edges of the mould. A delamination fabric was placed on the surface of the fibreglass layers, which was additionally taped to prevent displacement during the infusion process. The delamination fabric layer was correspondingly larger than the reinforcement layers preceding it. A mesh was laid on the surface of the delamination layer to distribute the matrix and then also secured with tape to avoid repositioning during the infusion process. The mesh such as delamination fabric was correspondingly larger than the delamination layer that preceded it. Spiral tubing was placed on the surface of the infusion mesh and connected to the solid tubing. Valves were screwed into the solid pipes. To prevent movement during the process, the tubing was secured with a tape and additionally with a butyl tape to the mould to keep it tight and stable. For the preparation of the matrix, MGS L285 epoxy resin and H287 hardener were used. Depending on the composite being prepared, different proportions were used. For the first laminate, a resin/hardener weight ratio of 100:40 [23] was used, i.e. in accordance with the manufacturer's recommendations. In this case, no matrix degassing process was used. For the second composite, the same proportions were adopted as for the first composite, however the degassing process was carried out before the mixture was delivered to the composite. In the third composite, a matrix was prepared with a resin/hardener weight ratio of 100:30. It was thus decided to test the effect of hardener deficiency on the strength properties of the composite material. Degassing was not used. Once all the components were properly aligned, a plastic film was applied to the mould that contained the composite. The sealing was done with a double-sided butyl tape. Following a positive leak test, the infusion process was carried out. Through one tube, the epoxy resin/hardener mixture was supplied, while the other tube was connected to a vacuum pump (Fig. 1) [12, 15].

Both valves were opened to start the process. This allowed the matrix to start being sucked out of the tank due to the vacuum pump creating a vacuum in the mould [16, 24]. The supersaturation of the composite layers began (Fig. 2).

Once the supersaturation of the reinforcement layers was complete, the valves of the piping supplying the resin mixture and the piping connected to the vacuum generation pump were closed. It was noticeable that the matrix moved in a much faster manner in places where there was no reinforcement. In order to cure the composite, it was left under negative pressure inside the mould for 24 hours. After this time, the tubing was disconnected and the composite was safely separated from the glass surface and the delamination fabric. The use of wax on a glass mould definitely made the process easier.

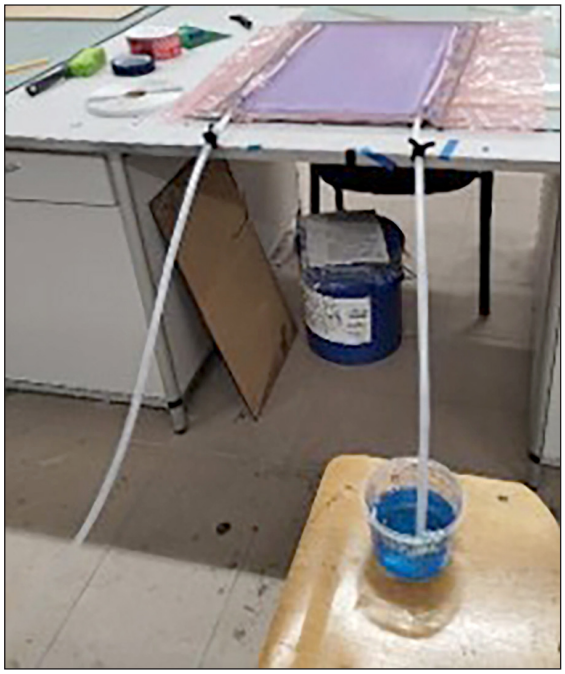

Figure 1. Composite before the supersaturation process

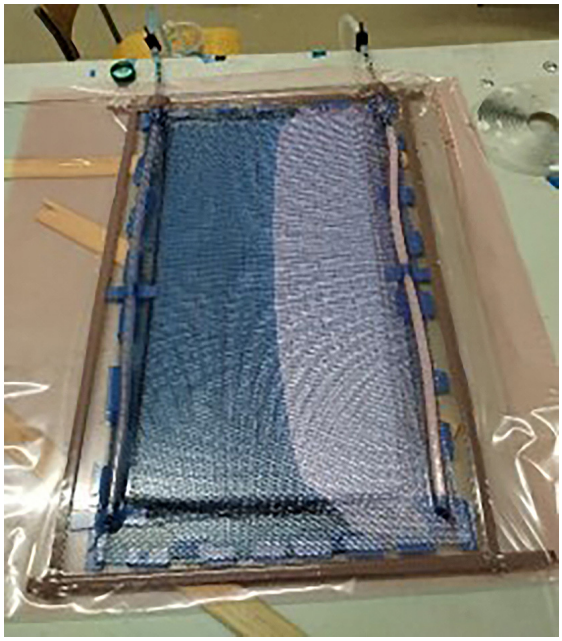

Figure 2. Course of supersaturation of composite layers with matrix

In this way, three composites were made with different matrix properties. For comparison, the propagation time of the matrix was measured as a function of its parameters [19, 22]:

- composite with matrix at a ratio of 100:40, without degassing – 14 minutes;
- composite with matrix at a ratio of 100:40, with degassing – 14 minutes 30 seconds;
- composite with matrix at a ratio of 100:30, without degassing – 15 minutes 50 seconds.

The obtained composites were visually inspected for adequate curing, the presence of discontinuities, inclusions and also other defects. No abnormalities were found during these tests (Fig. 3). Specimens were cut from the manufactured composites using a water jet cutting machine for testing the mechanical properties.

RESULTS OF EXAMINATION

A visual inspection of the fabricated laminates and the used resin/hardener mixtures was conducted prior to the strength tests. In the case of the resin composition, a visual inspection was carried out to determine the air bubble content in relation to its previous degassing. For this assessment, a pre-prepared sample batch was used, consisting of inlet and outlet pipes with the mixture solidified and the resin composition cast into a mould. The examination was performed with a microscope, using different magnification values. In the photographs taken of the mould samples (Fig. 4–6), the effect of degassing of the liquid matrix is noticeable, as revealed by fewer air bubbles. There are the fewest air bubbles in the case of the resin composition that was degassed after mixing.

During the visual examination of the prepared composite materials (Fig. 7–9), no significant differences in their structures were observed [14, 18, 21, 35, 36], which, combined with the analysis of the matrix with/without degassing, leads to a conclusion that the infusion process significantly affects the automatic degassing of the resin mixture during the propagation of the resin composition through the reinforcement layers in the infusion process.

For further research, the mass of the fabricated laminates was determined, their thickness

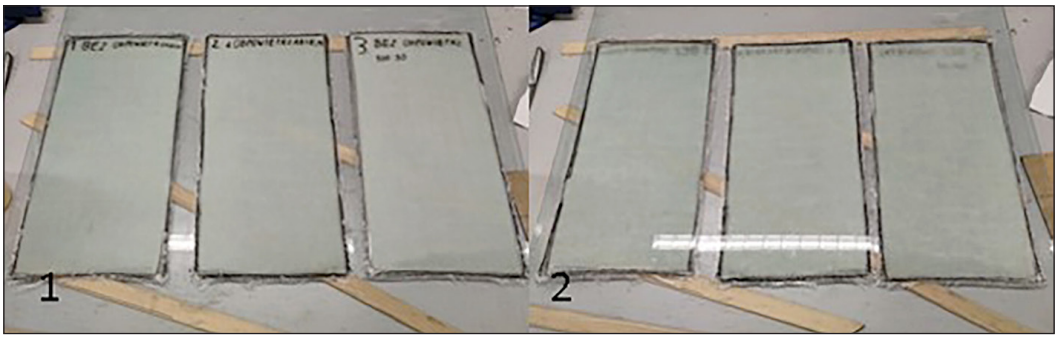

Figure 3. Manufactured composites: 1 – rough surface, 2 – smooth surface

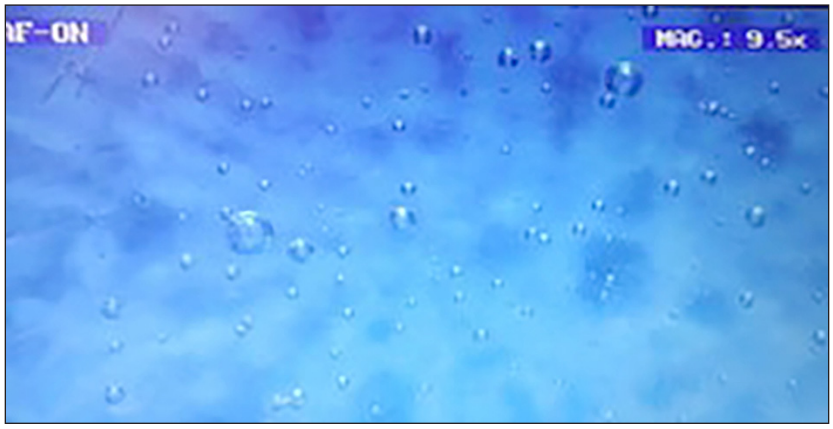

Figure 4. Resin composite (100:40) non-degassed – magnification 9.5×

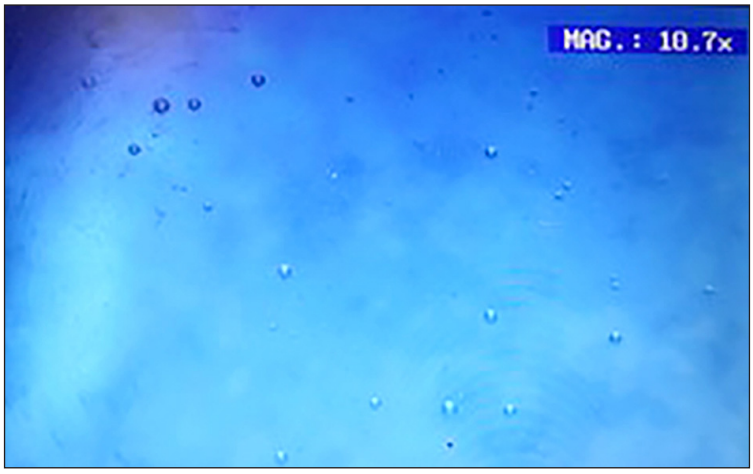

Figure 5. Resin composite (100:40) ventilated – magnification 10.7×

Figure 6. Resin composite (100:30) non-gasses – magnification 9.5×

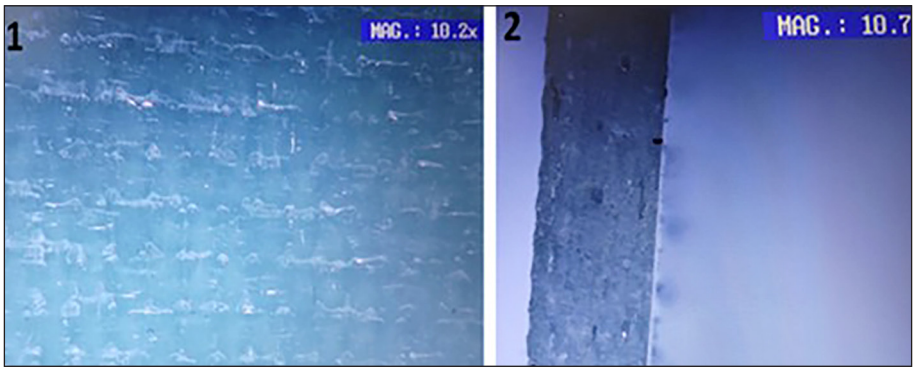

Figure 7. A laminate made using a resin mixture with a resin/hardener ratio of 100: 40, without degassing, 1 – external structure, 2 – internal structure

measured and the surface mass and percentage of reinforcement in their structure calculated. The results have been listed in Table 1.

An SW-5 impact hammer was used to determine the impact strength of the fabricated composite materials. The test involved testing the impact resistance of the laminate under plane and edge loading. In the course of the tests, the authors used 63 samples sized 11×80 mm. Twenty-one specimens were used from each composite

Figure 8. A laminate made using a resin mixture with a resin/hardener ratio of 100: 40 with degassing, 1 – external structure, 2 – internal structure

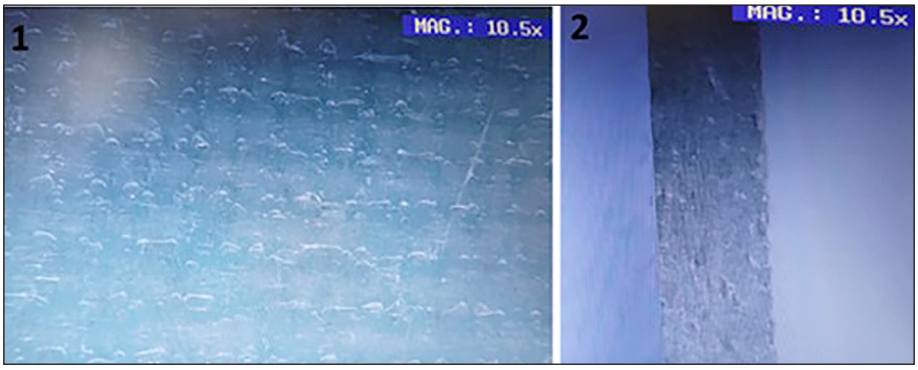

Figure 9. Laminate made using a resin mixture with a resin/hardener ratio of 100:30 without degassing, 1 – external structure, 2 – internal structure

Table 1. Parameters of composites prepared for testing

No.	Type of composite	Number of samples (pcs)	Average thickness (mm)	Surface weight (g/m^2)	Share of strengthening (%)
1	Matrix with degassing, ratio 100:40	19	2.95	5,067.90	69
2	Matrix without degassing, ratio 100:40	19	2.95	5,116.23	69
3	Matrix without degassing, ratio 100:30	19	2.92	5,062.50	70

for testing, with 10 specimens used for surface testing and 11 specimens used for edge testing.

The composite samples failed in a similar manner for each material (Fig. 10–11). When analyzing the results, it was noticed that the lower impact strength under surface loading was observed in the non-degassed matrix composite with a resin/hardener ratio of 100:30. It is noticeable that the average impact strength of the specimens was higher for the edge tests.

Flexural strength testing was carried out on undamaged specimens and specimens damaged by prior impact loading with different energy values. In the course of the tests, the authors used 57 samples sized 60×80 mm. This allowed for the comparison of flexural strength values between samples subjected to puncture testing with a specified energy and those not subjected to impact loading. An Instron Ceast 9340 drop hammer was used for the impact load. The energies used were 5 J, 10 J and 29 J. For the bending test, for specimens not subjected to impact loading and impacted at 5 J and 10 J, 5 specimens were prepared from each composite, while for specimens loaded at 29 J, the batch for each composite consisted of 4 specimens. Depending on the impact energy

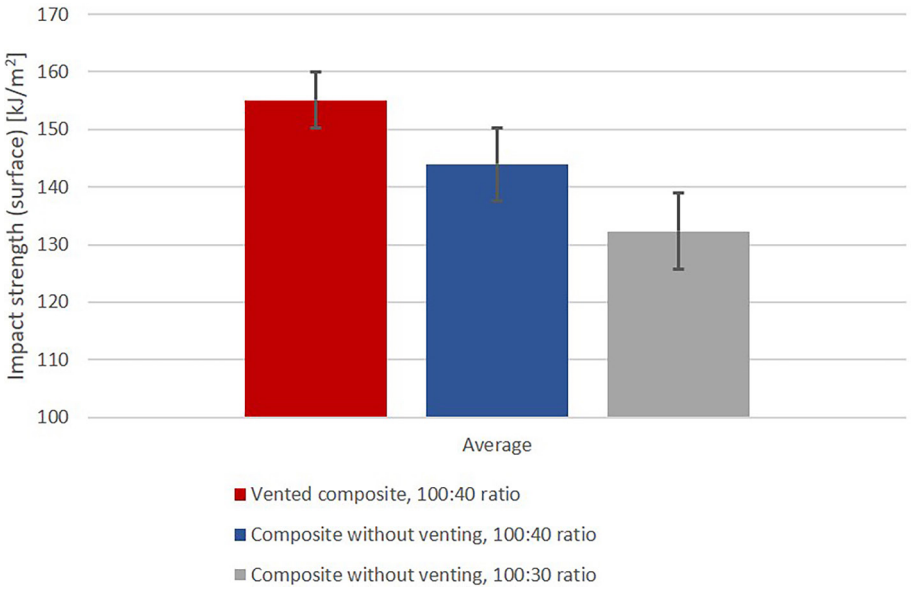

Figure 10. Surface impact strength of composites

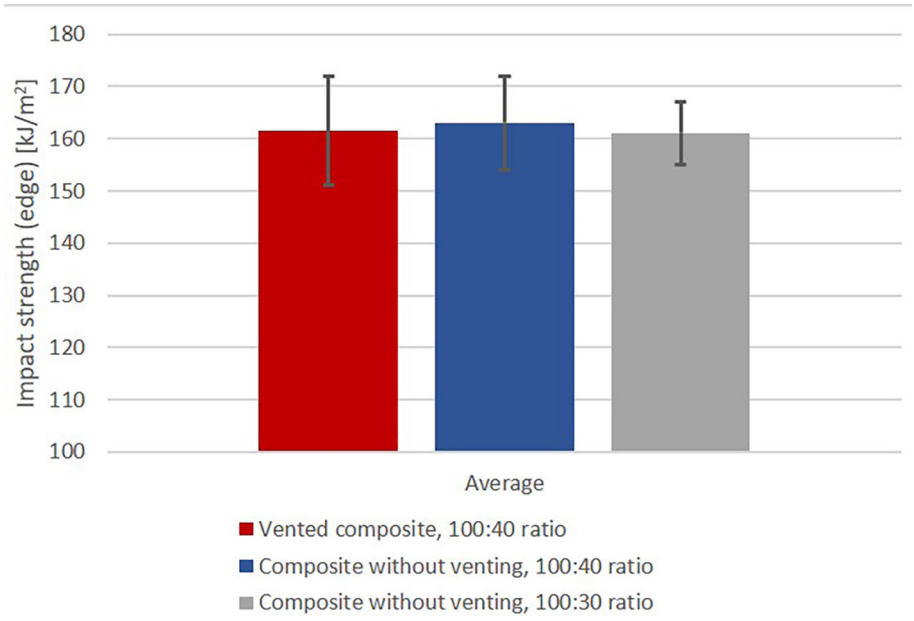

Figure 11. Edge impact strength of composites

used, the specimens were exposed to varying degrees of damage. As the applied energy increased, the diameter of the defect increased. For energies of 5 J and 10 J, the traces had a more regular shape. In contrast, the damage on the composite sample subjected to an impact energy of 29 J was characterized by a high degree of irregularity and far-reaching cracks and nicks. The greatest deformation was caused by an impact with an energy of 29 J, though the load did not completely destroy the specimen. For all samples, the damage was visible on both sides.

Bending strength testing of the composite was carried out on a Zwick/ Roel 5kN testing machine. The guidelines included in EN ISO 178 were applied during the experiment. During the tests, the bending strength was determined and the bending modulus was determined. Fifty-seven samples were subjected to strength tests. A batch of 19 pieces was prepared for each of the three composites: 5 undamaged specimens, 5 specimens impacted with 5 J, 5 specimens impacted with 10 J and 4 specimens impacted with 29 J. In the tests, the initial force was set at 0.1 MPa, the crosshead

speed during the bending modulus determination was 2 mm/min, while the testing speed in the remaining range was 10 mm/min. The results are shown in the graphs (Fig. 12– 17).

In order to facilitate comparison, a general bending strength graph was produced for each laminate (Fig. 18).

The highest bending strength among the non-impacted composites was obtained for the non-impacted matrix composite with a resin/hardener ratio of 100:30 and its average value of 601 MPa. The lowest strength in this group of 552 MPa was the degassed matrix composite, with a resin/hardener ratio of 100:40. Its bending strength was 8% lower than that of the laminate with an non-degassed matrix and a resin-to-hardener ratio of 100:30.

In the group of composites impacted with an energy of 5 J, the highest bending strength was obtained for a non-degassed matrix composite with a resin/hardener ratio of 100:30 and an average bending strength of 565 MPa. The lowest average bending strength was a degassed matrix laminate with a resin/hardener ratio of 100:40 characterized by an average strength of 539 MPa. The material with the highest strength in this group (with a non-degassed matrix with a resin/hardener ratio of 100:30) had a 5% higher bending strength than the material with the lowest strength.

The highest bending strength in the group of laminates impacted with an energy of 10 J was obtained for a non-degassed matrix composite with a resin ratio of 100:30 and an average

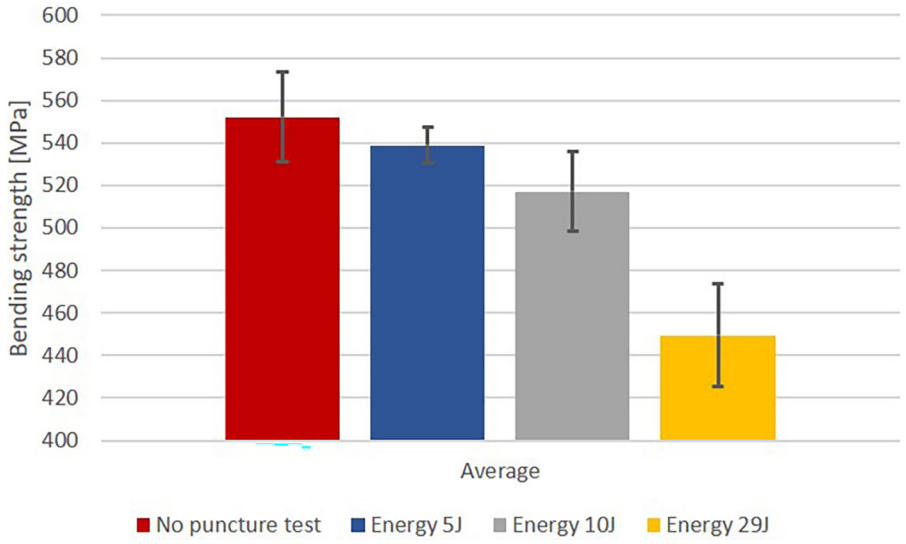

Figure 12. Bending strength of a degassed matrix composite with a resin/hardener ratio of 100:40

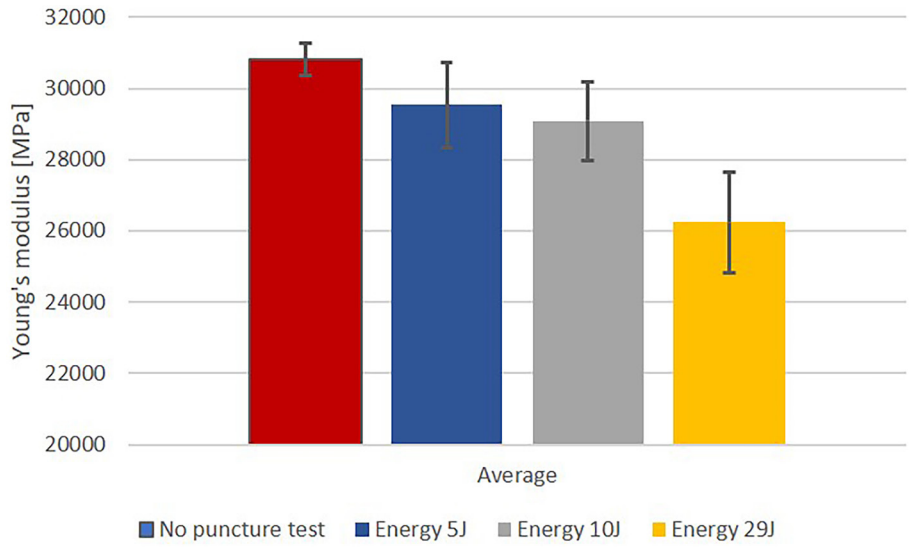

Figure 13. Bending modulus of a degassed matrix composite with a resin/hardener ratio of 100:40

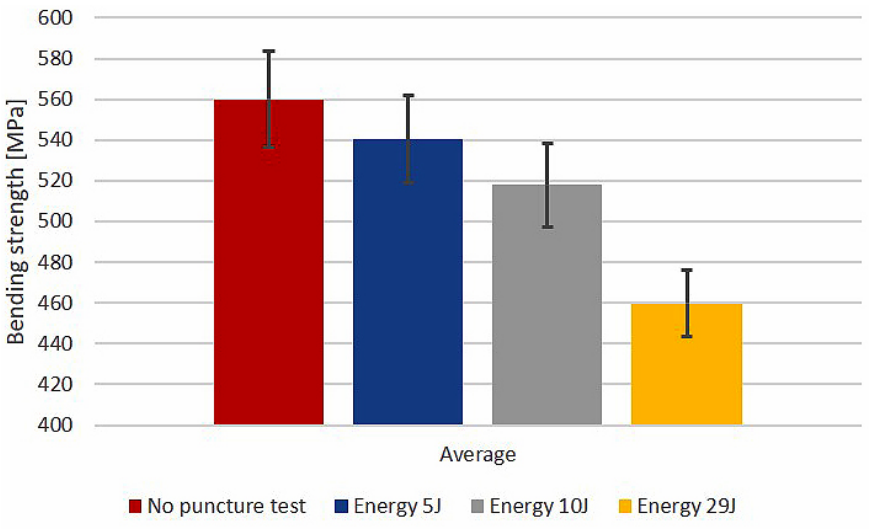

Figure 14. The bending strength of a composite with an non-degassed matrix and a resin-to-hardener ratio of 100:40

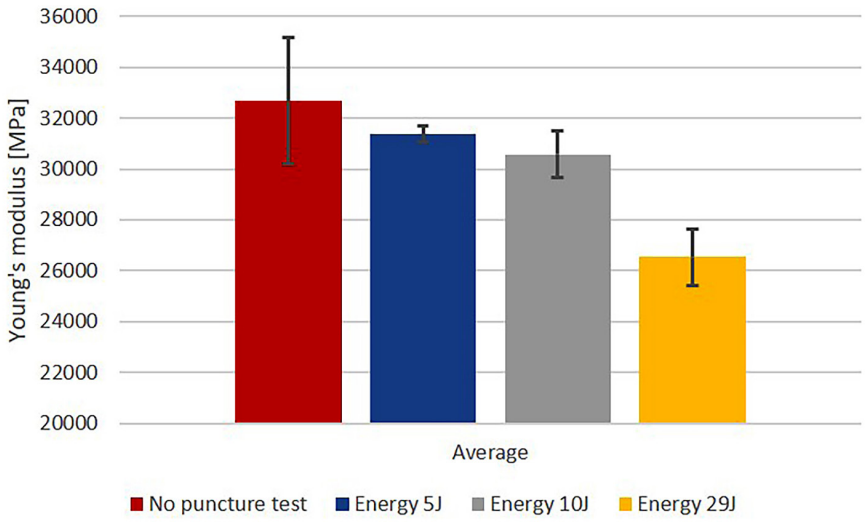

Figure 15. Bending strength of a non-gassed matrix composite with a resin/hardener ratio of 100:40

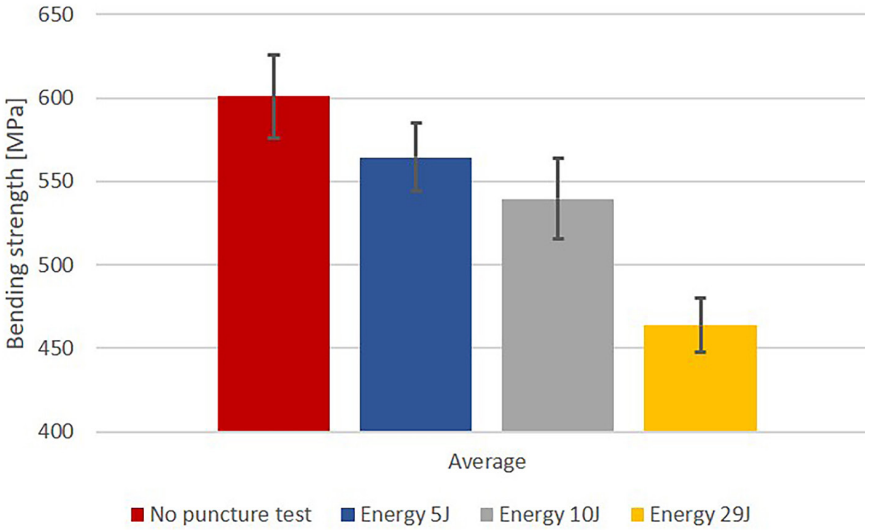

Figure 16. Bending strength of a non-gassed matrix composite with a resin/hardener ratio of 100:30

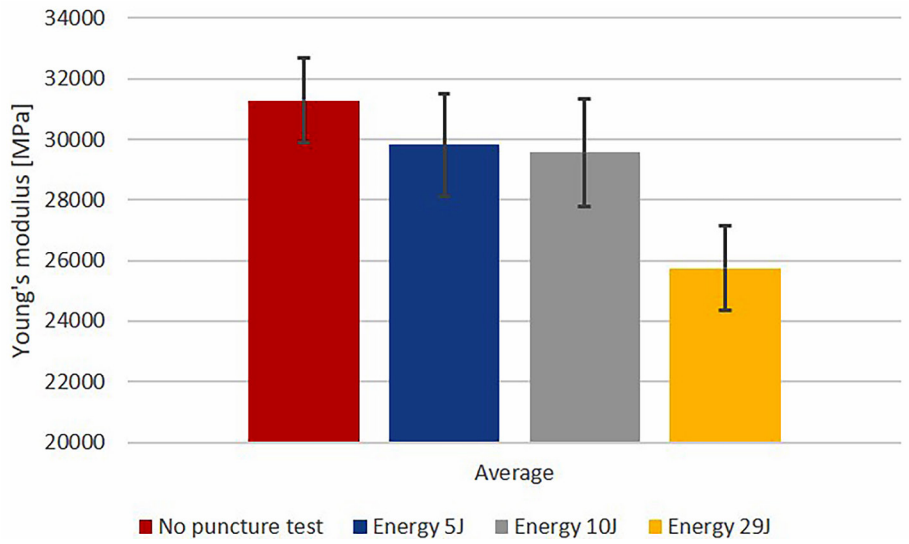

Figure 17. Bending strength of a non-gassed matrix composite with a resin/hardener ratio of 100:30

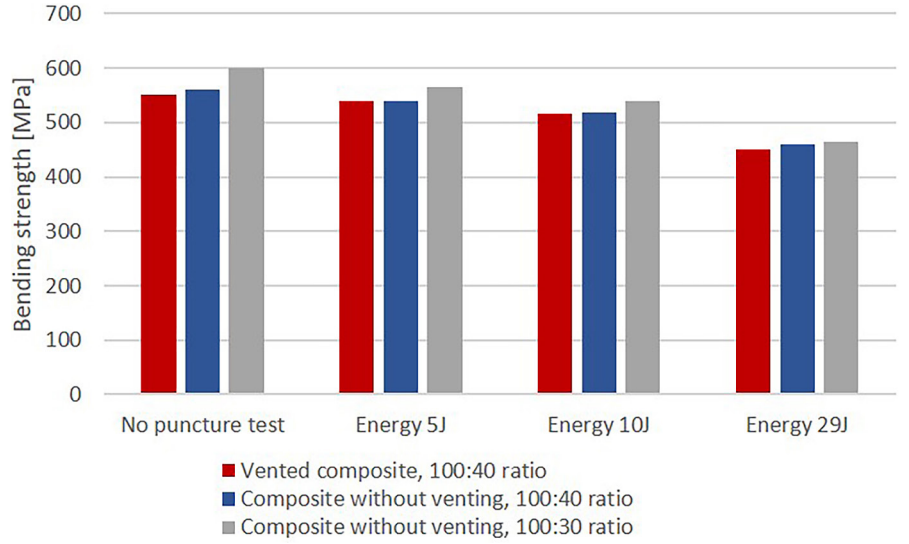

Figure 18. Summary of average bending strength values

strength of 549 MPa. In contrast, the lowest average bending strength value was that of the degassed matrix composite made with a resin/hardener ratio of 100:40. It was equal to 517 MPa. Its bending strength was 6% lower than that of the a composite material with an non-degassed matrix and a resin-to-hardener ratio of 100:30.

In the last group tested, where the composites were impacted with an energy of 29 J, the highest bending strength was also obtained for a non-degassed matrix laminate made with a resin/hardener ratio of 100:30 and its average value of 464 MPa. The lowest strength in this group of 450 MPa was the degassed matrix composite, with a resin/hardener ratio of 100:40. The material with the highest strength in this group (with a non-degassed matrix with a resin/hardener ratio of 100:30 had a 3% higher bending strength than the material with the lowest strength.

During the tensile strength testing, the longitudinal modulus of elasticity was determined, and the tensile strength of samples made from the produced composite materials was measured. The test was performed on an Instron 5982 static testing machine. The elongation was carried out at a rate of 2 mm/min. It was performed in accordance with ISO 527-5. "T-bone" shaped samples were prepared for this test. For each of the three composite materials, the batch size was 5 samples. They were subjected to axial elongation. A programme report was generated after each batch (Fig. 19).

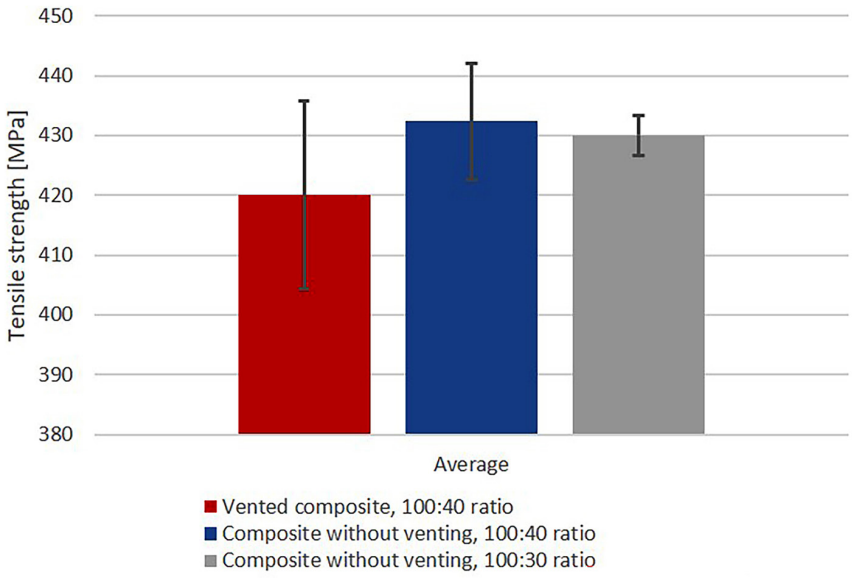

Figure 19. Tensile strength of the tested laminates

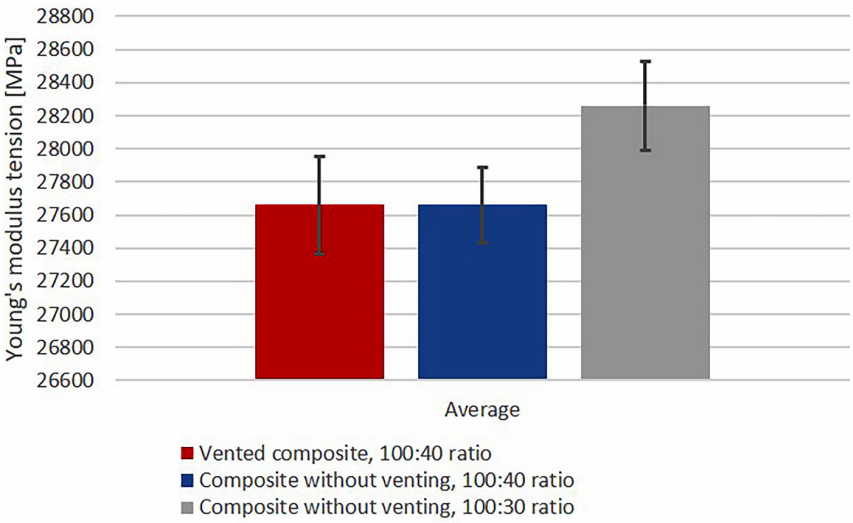

Figure 20. The longitudinal modulus of elasticity of the tested laminates

The highest tensile strength was obtained for the composite with a non-degassed matrix, with a resin-to-hardener ratio of 100:40, its average value was 432.38 MPa. The lowest strength of 419.97 MPa was the degassed matrix composite, with a resin/hardener ratio of 100:40. Its bending strength was 3% lower than that of the laminate with an non-degassed matrix and a resin-to-hardener ratio of 100:30 (Fig. 20).

CONCLUSIONS

Laminate samples were cut with water, which ensured high dimensional repeatability and, most importantly, no interference with the structure. After measuring and determining the mass of the individual samples, the surface mass and the percentage of reinforcement were calculated. The calculations showed that each of the laminates had a high proportion of reinforcement, around 70%. This confirms the correctness of the preparation of each composite material. After the composites had been measured, matrix laminates with a resin/hardener ratio of 100:40 showed an average thickness of 2.95 mm, both with and without degassing. In contrast, in the case of the non-degassed composite, where the resin/hardener ratio was 100:30, the average thickness had a value of 2.92 mm.

As part of the visual examination, both the resin mix and the resulting composite were visually inspected. In the case of the matrix not subjected to the infusion process (cast after mixing before being connected to the mould), the effect of degassing was noticeable. Under the microscope, it was possible to observe a significantly lower number of air bubbles when the degassing process was carried out. In contrast, during microscopic inspection of the individual composite materials, no differences in the internal and external structures were observed.

Strength tests began by determining the impact strength of the individual laminates. The specimens were surface and edge loaded using a pendulum hammer. Regardless of the composite from which they were made, they deteriorated in a similar manner. The test results indicated no significant differences between the properties of the materials. It was noticeable that the value of the calculated average impact strength was higher for the edge tests.

As part of the flexural strength testing of the prepared laminates, undamaged specimens and specimens previously struck with a dropping hammer were prepared. Before checking the bending strength, the specimens were impact-loaded using different impact energies. They had the following values: 5 J, 10 J and 29 J. None of these energies destroyed or punctured the laminates completely, while the greatest differences were seen after an impact energy of 29 J. During these tests, greater damage was seen not at the point of impact, but on the opposite side of the composite. This is one of the drawbacks of laminates, because in such cases, when damage occurs on the surface of an aircraft's covering and there is no access to its internal side, there is a risk of the damage going unnoticed or being improperly assessed. Depending on the material, the results obtained in bending do not differ to any great extent. Differences can be seen by considering the energy previously used. Both bending strength and bending modulus decreased as the energy of the prior impact increased.

The final test was the tensile strength test. In its course, the coefficient of longitudinal elasticity was also determined. During the test, each specimen was destroyed in a correct manner, i.e. not directly in the handles of the testing machine. Slow and gradual axial stretching yielded accurate results. From the obtained tensile test data, it can be concluded that there are no significant differences between the produced laminates in tensile strength, while the samples with the wrong resin/hardener ratio showed the highest Young's modulus value.

The results of each of the strength tests for the multilayer composites were very similar to each other. This may be due to the fact that, for each laminate, the reinforcement content was around 70 per cent and the reinforcement was the main component in the structural design. This mainly explains the fact that the strength properties of the composite obtained in a 100:30 resin/hardener ratio were very similar. From the analysis of the results of the outer and inner structure of the composite, it can also be concluded that the infusion process largely influences the automatic degassing of the matrix during propagation through the reinforcement material. Although differences between them exist, they can be considered negligibly small, as the reinforcement phase is the same in all the tested composites, and it is primarily this reinforcement that is responsible for the examined strength properties.

On the basis of the results obtained from the strength tests and observations of the internal and external structure of the tested materials, it can be concluded that the ratio of resin and hardener has little effect on the strength properties of the material (in the considered range of mass ratios and with the use of MGS L285 resin and H287 hardener) and that the infusion method of composite manufacture largely affects spontaneous degassing of the resin mixture used in this method.

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