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Mechanical analysis of multilayer composite materials with duroplastic matrix after exposure to low temperatures

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ABSTRACT

Purpose: Cryogenic engineering is gaining more and more interest in various industry sectors, which leads to an intensive search for effective solutions. The article presents the findings of mechanical testing conducted on glass-epoxy laminates at room temperature and after long-term contact with liquid nitrogen.

Design/methodology/approach: To compare the impact properties and flexural strength, the samples were tested under cryogenic and room conditions, and then the fracture locations were identified using the Leica DVM6 microscope. The study brings value to the emerging field of cryogenic engineering by providing valuable information on the mechanical properties of glass-epoxy composites under cryogenic conditions.

Findings: It has been found out that immersing the glass-epoxy composites into the Dewar had minimal influence on impact and flexural strength properties. The most noticeable changes were observed in the case of the EP_4_2 composite. The material consists of a solution of brominated epoxy resin in an organic solvent. It is used to produce laminates in electrical engineering and printed circuits in electronics, where it should exhibit excellent impact properties.

Research limitations/implications: One of the prospective research directions is a thorough analysis of the mechanical properties of the developed composite materials during cryogenic cycles.

Originality/value: The study aims to determine the effect of different compositions of glass fabric-reinforced resin with a weight of 205 g/m^2 on the mechanical properties of the developed composite materials at both room temperature and after long-term exposure to liquid nitrogen. Those investigations serve as surveillance for developing of new material solutions directed towards cryogenic applications and are essential for subsequent stages of research.

Keywords: Composite, Cryogenic, Polymer, Mechanical testing, Epoxy-glass laminate

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PROPERTIES

1. Introduction

Low production costs, market availability and excellent mechanical properties make composites with a duroplastic matrix considered advanced engineering materials. Composites applied to cryogenic conditions are used as structural elements, fuel tanks, electrical insulation and load-bearing elements [1,2] effectively develop high-performance epoxy-glass laminates for cryogenic conditions, it is necessary to be aware of and understand the mechanical characteristics of the developed composite materials operating at cryogenic temperatures. Equally important is to examine the effect of elements such as hardener content and matrix type on cryogenic properties [3]. The choice of hardener and matrix type can significantly affect the cryogenic properties of epoxy glass laminates. Various hardeners and matrices may have different coefficients of thermal expansion, glass transition temperatures, and chemical properties. Those parameters can affect the behaviour of the composite material at cryogenic temperatures..

Impact toughness is a physical property that describes the resistance of a material to impact or shock loads. Low temperature affects the impact toughness of composite materials. In such conditions, materials may become more brittle and susceptible to mechanical damage, reducing impact strength [4,5]. Liquid nitrogen causes changes in the structure of composite materials, such as deformations and fractures, ultimately leading to alterations in their mechanical properties. Therefore, it is important to consider the influence of low temperatures on the mechanical properties of materials during their design and application.

The studies on materials used at cryogenic temperatures described in the literature undoubtedly require ordering and standardisation to enable their rational comparison. Most of the literature focuses on modifying the resin as the matrix material to increase its ductility and impact toughness under extreme temperature conditions [3,6,7]. Other studies focus on introducing a well-defined reinforcing phase into the system, which appears in the form of particles or fibres, to minimise the effects of low temperatures on the entire composite (reducing the occurrence and propagation of microcracks). Zsombor Sápi [3] analysed literature published since 1994 on the behaviour of composite materials at cryogenic temperatures. The review confirmed that low temperature, especially short-term contact, positively affects the properties of the tested composites – their strength, Young's modulus, fatigue strength and thermal properties increase. Zsombor Sapi [3] emphasised that "there is no distinct temperature point that defines the field of cryogenics,but it is typically considered to be below -150°C, where the boiling temperature of oxygen, nitrogen, hydrogen and helium occurs. The upper limit of so-called "high temperature" cryogenics is -50°C, which is also selected for the upper temperature." In the article, it refers to the range -273°C (0 K) to -150°C (123 K) as cryogenic temperature (CT), the range -150°C (123 K) to -50°C (223 K) as low temperature (LT) and around 23°C as room temperature (RT).

M. Surendra Kumar [8] investigated the mechanical behaviour of glass/epoxy composites at cryogenic temperatures. It was found out that they are sensitive to loading rate. Studies by Hei-lam Ma [5] indicate that GFRP (Glass Fiber Reinforced Polymer) composites in cryogenic conditions showed (1) less visible damage, (2) higher stiffness compared to other composites, and (3) relatively weak energy absorption. The research also shows that post-curing can reduce visible damage and increase the energy absorption of GFRP composites. In impact tests of GFRP laminates at temperatures (-50°C to 120°C), Salehi-Khojin et al. [9] discovered that the laminates become more rigid at low temperatures.

The study aims to determine the influence of the varied composition of a resin reinforced with a 205 g/m^2 plain-woven glass fabric on the mechanical properties of the developed composite materials at room temperature and after long-term exposure to liquid nitrogen. Those investigations serve as a reconnaissance for developing new material solutions for cryogenic applications. This research project was carried out in collaboration with "IZO-ERG S.A." in Gliwice.

2. Experimental studies

The developed material is in the form of a laminated glass-epoxy sheet. It was produced by layering successive plies of glass fabric saturated with an epoxy resin composition. In the first stage, the following epoxy resins were selected: EPIDIAN 11M80 (Sarzyna Chemical), YD-128 and YDPN 638A80 (Kukdo), which were then modified with different hardeners (DICY, DDS and Nowolak P). The E-type glass fabric with a gram weight of 205 g/m^2 and plain weave structure was impregnated with these prepared compositions provided by Joint Company.

Table 1 presents the composition and designation of the developed epoxy resin compositions, while Table 2 contains the technical data of the glass fabric.

The designations of the composite material in the table should be interpreted as follows:

EP_X_Y_RT/1D/7D

Legend: EP – epoxy resin, X – type of epoxy resin, Y – type of hardener, RT,1D,7D – exposure time in liquid nitrogen.

Table 1.
Designation of selected composite materials

No	Symbol	Composition
1.	EP_1_1	YDPN 638 A 80 + Nowolak
2.	EP_2_2	YD -128 + DICY
3.	EP_2_1	YD -128 + Nowolak
4.	EP_1_3	YDPN 638 A 80 + DDS
5.	EP_4_2	EPIDIAN 11M80 + DICY

Table 2.
Technical Parameters of Joint Stock Company 7628 Type E Glass Fabric

No	Parameter	Parameter
1.	Grammage	205 g/m^2
2.	Weave type	Plain
3.	Glass type	„E”
4.	Thickness	0.1697 mm
5.	Width	110.3 cm

Two different forms of resin were used in the experiments: an unmodified resin based on bisphenol A, and a solution of epoxy resin enriched with bromine diluted in an organic solvent - ethyl methyl ketone. Those resin forms are widely available and used in production processes at “IZO-ERG S.A.” in Gliwice.

Preparation of the material

Composite sheets were manufactured at IZO-ERG S.A. in Gliwice. The process consisted of:
1. Preparing the resin composition.
2. Impregnating the substrate with the resin composition.
3. Using the pressing process to form the product in the form of sheets.

To prepare the matrix, specific quantities of selected composition components, such as resin and hardener, were dissolved in the solvent – acetone and then subjected to mixing for several minutes until complete dissolution of DICY.

Table 3.
Parameters of the prepared material

Symbol	Fluidity, %	Resin content, %
EP_1_1	24.6	38
EP_2_2	13	33.6
EP_2_1	17.9	35.48
EP_1_3	13.1	34.6
EP_4_2	21	34

Table 3 presents the parameters of the prepared material (matrix). The finished composites’ final properties also depend on the manufacturing process.

The previously prepared EP_X_Y resin compositions were used to coat glass fabric at 160°C using a Hoesch laboratory coater. The selected carrier was impregnated with the appropriate amount of resin, and the carrier-resin system was partially cured using the coater mentioned above. The next step involved dividing the prepared semi-finished product into sheets of 30x50 cm. The sheets were pressed at 165°C for 120 minutes using a Hoesch laboratory press. The presented parameters of the pressing process allowed for the crosslinking effect to be fixed and for a full range of strength properties to be obtained. The produced laminate contains eight sheets of epoxy-glass prepreg, arranged in parallel configuration with an orientation of [0°/90°].

3. Research methodology

According to the PN-EN ISO 179-2 [10] and PN-EN ISO 178 [11] standards, samples were precisely cut out from the obtained composite sheets. Long-term exposure of samples to liquid nitrogen was performed in Dewar YDS-5-200 by the “Cryogen” company. The prepared samples were placed in a dipper and immersed in liquid nitrogen. The samples were exposed for 24 hours and seven days, with the liquid nitrogen level being monitored daily. After exposure, the samples were carefully removed, protected, and directed for further mechanical testing.

3.1. Charpy impact testing

The Charpy impact tests were conducted using the PSW-40 Leipzig pendulum hammer as part of the study. Those tests followed the standard PN-EN ISO 179-2 “Plastics. Charpy impact strength determination. Instrumental impact test” [10]. A hammer with a breaking energy of 40 kJ was used for this purpose. It is a standard hammer used for impact testing of production laminates. Measurements were performed for each developed composite material under room temperature conditions and after 1 and 7 days of exposure to liquid nitrogen. Six measurements were made for each series of samples. The impact strength of the developed composite materials was calculated based on the obtained data using formula (1) [10]:

$$R_e = \frac{E_c}{h*b} * 10^3 \left[\frac{\mathrm{kj}}{\mathrm{m}^2}\right] \quad (1)$$

where:
R_e – Impact strength, kJ/m^2,

E_c – Energy absorbed during fracture, J,
h – Thickness of the test specimen, mm,
b – Width of the test specimen, mm.

According to the current standard, Table 4 contains designations for fracture characteristics.

Table 4.
Designation of fracture characteristics according to standard PN-EN ISO 179-2 [10]

Fracture character	Symbol
Complete fracture	C
Hinged fracture	H
Partial fracture	P
No fracture	N

3.2. Flexural property testing

The three-point bending test was performed according to the standard PN-EN ISO 178, "Plastics - Determination of flexural properties" [11]. The test was conducted using a universal testing machine INSTRON 500 N-50 kN. The distance between the supports was 32 mm, and the bending speed was 2 mm/min. Six tests were performed for each sample series. Before starting the test, the width and thickness of the beams were measured using a calliper. Based on the obtained values, the modulus of elasticity in flexure and the flexural strength were determined.

Due to economic reasons, the composite materials' thickness is less than 3 mm.

4. Discussion of results in varied temperature conditions

4.1. Results of Charpy impact testing

The results of the impact tests at room temperature and after exposure to liquid nitrogen are presented in Table 5 and Figure 1.

Studies have shown that the exposure of multilayer composites to liquid nitrogen for 1 and 7 days does not cause significant changes in their impact properties. Materials EP_1_1, EP_2_1, EP_2_2 and EP_3_3 exhibit stability in the properties above, with differences in impact strength after exposure being less than 5%.

The most noticeable changes were observed for EP_4_2, which showed the best impact performance at room temperature and after exposure. At room temperature, the impact strength of this material was 157 kJ/m^2, and after 1-day exposure, it was 152 kJ/m^2. After 7-day exposure, it decreased to 131 kJ/m^2, representing a decrease of approximately 32.3% in this property compared to the other materials. The lack of significant impact on the impact properties of liquid nitrogen can be attributed to the samples returning to their original state after exposure to liquid nitrogen.

Table 5.
Average impact test results for five sample configurations at room temperature (RT)j, after 1(1D) and 7(7D) day exposure to liquid nitrogen

	Symbol	Impact strength, kJ/m^2	Type of fracture
RT	EP_1_1	101	P
	EP_2_2	104	C
	EP_2_1	97	P
	EP_1_3	98	C
	EP_4_2	157	C
1D	EP_1_1	106	P
	EP_2_2	103	C
	EP_2_1	98	P
	EP_1_3	102	C
	EP_4_2	152	C
7D	EP_1_1	104	P
	EP_2_2	100	C
	EP_2_1	97	P
	EP_1_3	98	C
	EP_4_2	131	C

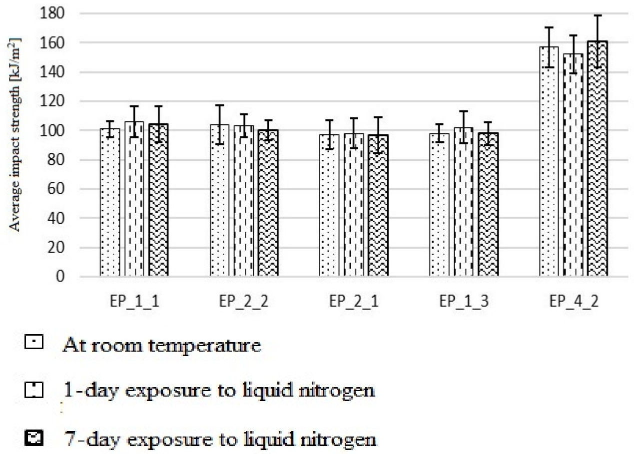

Fig. 1. Evaluation of the influence of room temperature, 1-day and 7-day exposure to liquid nitrogen on the impact properties of different laminate configurations.

Dr M. Elamin [12] conducted research to determine the effect of temperature (23°C and -70°C) on the damage initiation force, maximum damage force, lower damage force and impact load decrease. At -70°C, the matrix damage was noticeably greater than at room temperature. Dian-sen Li et al. [13] performed Charpy impact tests on 3D Multi-axial warp-knitted composites at room temperature (20°C) and liquid nitrogen (-196°C). The results showed that the impact resistance of the composites significantly improved at the temperature of liquid nitrogen compared to room temperature. Furthermore, the impact properties significantly decrease with the increase of the angle of inclination of the fibre layer in both temperature conditions. Further impact testing on glass-epoxy laminates, conducted using the Charpy method at room temperature and after contact with liquid nitrogen, showed that these laminates exhibited better impact properties after short-term contact with liquid nitrogen [14].

For comparative purposes regarding the obtained fractures, microscopic observations were conducted. Figure 2 presents exemplary images of the tested laminate surfaces captured at 40x magnification. The photos were taken using the Leica DVM6 digital microscope from Leica Microsystems, allowing precise 2D and 3D image analysis. No circular indentations or traces of air bubbles, or other inclusions/discontinuities were identified during the observations. The absence of defects indicates proper impregnation of the reinforcing phase with resin and a well-executed manufacturing process with appropriately selected process parameters. In most materials, a brittle behaviour was observed upon impact fracture. Comparing the images at both room temperature and different durations of exposure to liquid nitrogen, it can be concluded that the cryogenic temperature did not significantly affect the material's structure or the nature of the fracture.

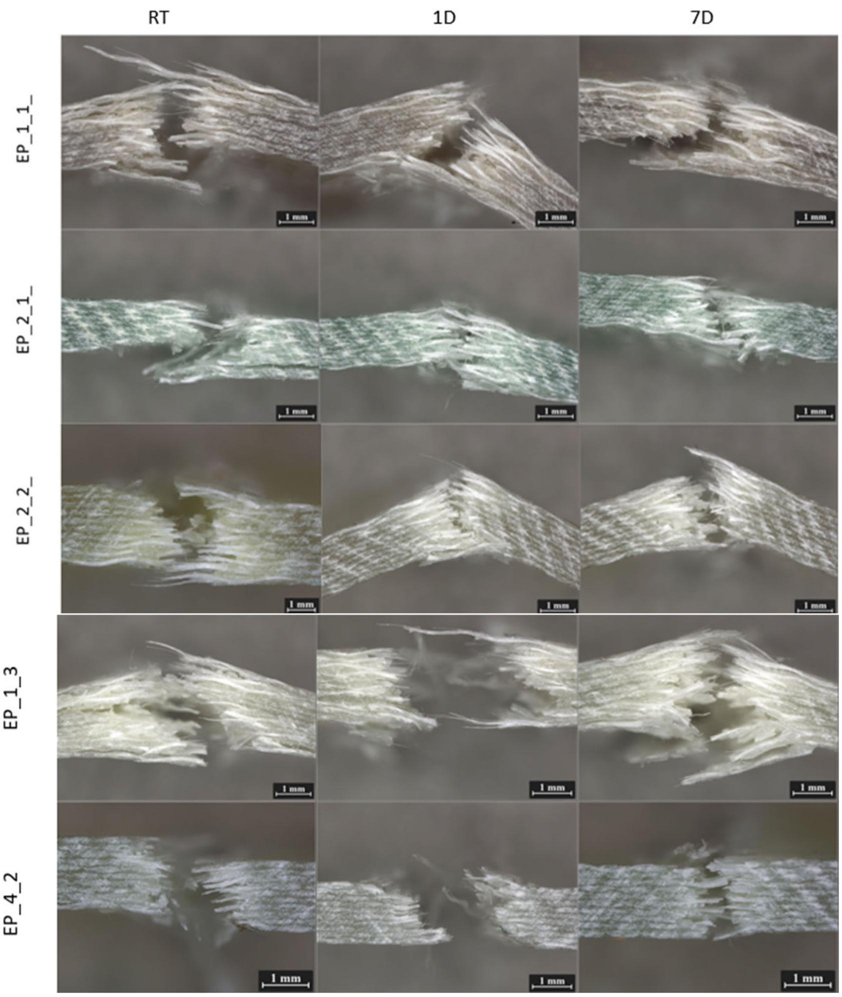

Fig. 2. Comparison of laminate images at 40x magnification

4.2. Results of flexural property testing

Table 6 and Figures 3,4 present the results of Young's modulus test from the three-point bending and bending strength tests. According to the IEC-893 standard – "Part 2: Methods of Test, Specification for Industrial" [15], epoxy-glass laminates' flexural strength should be less than 350 MPa. All tested laminates meet the requirements of the IEC-893 standard.

Table 6.
Average results of flexural strength tests and Young's Modulus values of samples at room temperature after 1 and 7 days of exposure to liquid nitrogen

Symbol		Flexural strength, MPa	Young's modulus, GPa
RT	EP_1_1	571.72	22.27
	EP_2_2	617.3	20.93
	EP_2_1	583.52	24.62
	EP_1_3	535.2	21.64
	EP_4_2	775.8	29.59
1D	EP_1_1	551.52	21.31
	EP_2_2	599.2	20.85
	EP_2_1	585.44	25.63
	EP_1_3	520.52	21.86
	EP_4_2	750.36	22.44
7D	EP_1_1	546.86	22.14
	EP_2_2	584.1	21.38
	EP_2_1	587.28	26.28
	EP_1_3	500.7	19.64
	EP_4_2	762.92	23.29

The results of the conducted tests indicate that the EP_4_2 composite exhibited the highest flexural strength among all composite materials. At room temperature, its value was 776.51 MPa, and after a one-day exposure to liquid nitrogen, this value remained unchanged at 776 MPa. After seven days of exposure, the value slightly decreased to 763 MPa. On the other hand, the lowest flexural strength results were recorded for the EP_1_3 composite, which had a value of 535.44 MPa at room temperature and 535 MPa after one-day exposure to liquid nitrogen. After seven days of exposure, this value significantly decreased to 501 MPa, representing a 35.4% decrease compared to the best composite. Notably, the modulus of elasticity for both tested composites, EP_4_2 and EP_1_3, decreased with the duration of exposure to liquid nitrogen, indicating a negative impact on their stiffness.

The EP_2_1 composite showed no changes in flexural strength compared to other samples after exposure to low temperatures. It means that EP_2_1 is a stable composite material. Additionally, prolonged exposure to liquid nitrogen contributed to an increase in the modulus of elasticity of EP_2_1, which means that its elasticity decreased. Similar trends were observed for EP_1_1 and EP_2_2. Furthermore, laminates with epoxy resins having a higher molecular weight equivalent have better mechanical properties. Only EP_4_2 contains the epoxy resin EPIDIAN 11M80 with an average equivalent molecular weight of 208 g/eq among the tested composite materials. It is reflected in better mechanical properties.

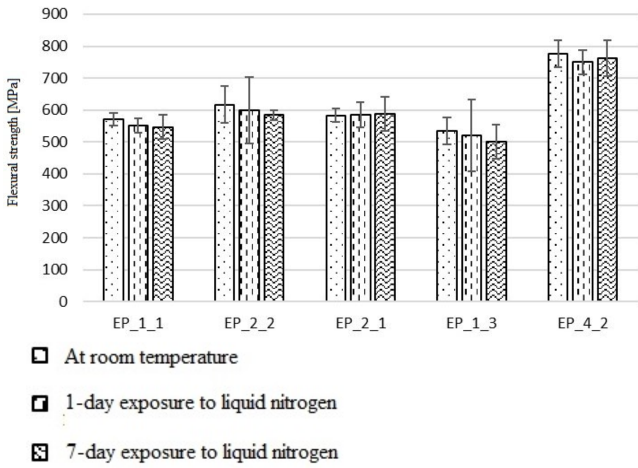

Fig. 3. Comparison of flexural strength of tested series of samples

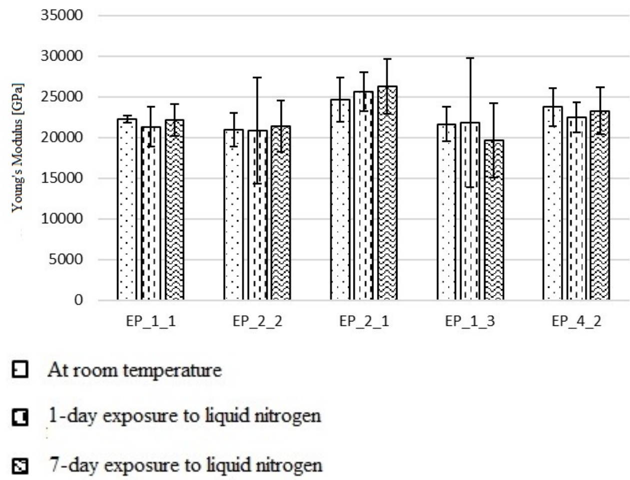

Fig. 4 Comparison of Young's modulus values from the three-point bending test

5. Conclusions

The main objective of the research was to analyse the mechanical properties of developed glass fibre-reinforced composite materials in cryogenic conditions. The behaviour of the composites was examined under room temperature conditions and after 1 and 7 days of exposure to liquid nitrogen. The obtained results are a starting point for further research on new material solutions adapted to cryogenic applications. Five different material variants, varying in epoxy resin composition and curing agent, were used in the study.

The main conclusions from the conducted research are as follows:

(1) 1 and 7 days exposure to liquid nitrogen hardly affects the impact properties of the tested materials.

(2) The EP_4_2 composite has exceptionally high impact properties that remain stable at room and low temperatures.

(3) Young's modulus of configuration EP_2_1 showed an increasing trend after seven days of exposure to liquid nitrogen.

Changes in the impact strength and flexural strength of glass-epoxy laminates under the influence of liquid nitrogen may be related to the weakening of chemical bonds between the glass fabric and epoxy resin. The process can lead to material degradation, decreasing impact and flexural strength. However, it should be noted that the extent of property changes in the tested laminates after exposure to liquid nitrogen may depend on factors such as material composition, direction duration, temperature, and pressure.

Table 7 presents the flexural strength and Young's modulus results at room temperature based on a brief review of the scientific literature.

Table 7.
Brief review of the scientific literature

Flexural strength, MPa	Young's modulus, GPa	Reference
103.78	4.5	[16]
420	18	[17]
449	28	[18]
A comparison of the results obtained in the article		
571.72	22.27	EP_1_1
535.2	21.64	EP_1_3
583.52	24.62	EP_2_1
617.3	20.93	EP_2_2
775.8	29.59	EP_4_2

Azhary [16] investigated the mechanical properties of laminated composites (glass-epoxy) using tensile, bending and impact tests. The resin used in the trials was an epoxy resin based on bisphenol A, and the reinforcing phase was E-glass mats (so-called chopped strand mat). The flexural strength was 103.78 MPa, while Young's modulus remained at 4.5 GPa.

Reed [17] conducted shear and flexural strength tests at 77 K and 296 K on glass-epoxy laminates of various weaves – satin and linen. The resin consisted of an anhydride bisphenol type F resin system. The type 7500 plain weave laminate had the following mechanical properties: flexural strength – 420 MPa (295 K) and 740 MPa (77 K) and Young's modulus – 18 GPa (295 K) and 20 (77 K).

Wang [18] investigated the mechanical properties and the structure of cracks in epoxy composites reinforced with E-glass fabric depending on the used silicon-organic coupling agent. The matrix was an epoxy resin based on bisphenol A, the reinforcing phase was E-glass fabric and δ-Aminobutyl-triethoxysilane (ABS), the flexural strength was 449 MPa, and Young's modulus was 28 GPa.

A comparison of the obtained results with the literature data shows that the EP_X_Y composite materials exhibit excellent flexural properties and Young's modulus. For comparison, the EP_4_2 composite at room temperature, after 1 and 7 days of immersion in liquid nitrogen, had an average flexural strength of 763.03 MPa. This represents a 7.3-fold, 1.8-fold, and 1.69-fold increase compared to the results obtained by Azhary [16], Reed [17], and Wang [18], respectively.

Table 8 presents the averaged impact test results at room temperature based on a brief review of scientific literature.

Table 8.
Brief review of the scientific literature.

Impact strength, kJ/m^2	Reference
200	[16]
83.08	[14]
284.6	[19]
A comparison of the results obtained in the article	
101	EP_1_1
98	EP_1_3
97	EP_2_1
104	EP_2_2
157	EP_4_2

Azhary [16] also studied the impact properties of laminated composites. The average impact strength of the glass-epoxy laminate was 200 kJ/m^2.

In a previous article [14], glass-epoxy composites' impact properties were investigated at room temperature and after brief contact with liquid nitrogen. The average impact strength of the EP_4_2 composite at room temperature was

83.08 kJ/m^2. The conducted research indicates that short-term exposure to liquid nitrogen increased the impact strength of the EP_4_2 composite, reaching approximately 132.29 kJ/m^2.

Devendra [19] studied the mechanical properties of epoxy composites reinforced with E-glass fibre filled with various filler materials. The average impact strength of the glass-epoxy composite without filler was 284.6 kJ/m^2.

Comparing the impact tests results of composite materials in RT and after being immersed in liquid nitrogen for 1 and 7 days, with the literature data, it can be concluded that there is a need to improve the impact properties. A significant improvement in EP_4_2 can be observed compared to the results obtained in the article [14]. It corresponds to a 43.35% increase in the average impact strength of EP_4_2, which can be attributed to the introduced modifications and better manufacturing quality.

However, the conducted study differs in terms of the dimensions of the test samples, their conditioning and test equipment from the cited literature data, which may explain some differences in the results obtained.

Various composite configurations have been selected based on the analyses described in this publication. Those materials were produced in a laboratory environment significantly impacting the mechanical test results and the final quality of the laminates. However, it should be noted that the presented study has some limitations that may affect the overall interpretation of the results. The described experimental sample size needed to be bigger, which may lead to some underestimation of observed phenomena.

The results presented in the study open up several new research directions worth exploring in detail in the future. One of them is to investigate the effect of cryogenic cycles on the mechanical properties of composite materials. The next stage of the research will be to compare the results of cyclic load-unloading tests, starting from 0.25% until the sample is damaged (cyclic loading-unloading tests) both at room temperature and reduced -50°C. Also, conducting research in a climatic chamber to extend the exposure of the tested materials to lower temperatures is being considered.

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