

# The Effect of Cryogenic Cycling on the Mechanical Properties of Epoxy-Glass Composites

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## ABSTRACT

Glass-epoxy laminates are characterized by exceptional properties such as high thermal insulation, resistance to mechanical damage, and stability at low temperatures. These crucial characteristics make them suitable for diverse applications, including cryogenics. Their application in cryogenics allows them to maintain low temperatures in research and industrial processes. This article analyzes the effect of cryogenic cycles on the functional properties of composite materials. The study investigated the influence of cryogenic cycles on the mechanical properties of glass-epoxy laminates. Three sets of cycles were employed, each consisting of 1, 5, and 10 cycles. After each set of cycles, the mechanical properties, including impact strength, flexural strength, and Young's modulus, were measured and compared. Additionally, after each series, scanning electron microscopy (SEM) was used to carefully observe the material's surface and detect possible changes in its appearance and structure, such as cracks or deformations. Conclusions from the conducted research provide essential information on the correlation between cryogenic cycles and the functional properties of composites obtained by coating. The research results can be used to design and improve these materials in various industrial applications. This work determines the effect of a different composition of resin reinforced with glass fabric weighing 205 g/m<sup>2</sup> on the mechanical properties of composite materials subjected to cryogenic cycles. This research aims to create innovative materials adapted to work in cryogenic environments.

**Keywords:** composite, cryogenic, cycle, mechanical testing, epoxy-glass laminate.

## INTRODUCTION

The influence of cryogenic cycles on the mechanical properties of laminates composed of an epoxy matrix reinforced with glass fabric is a crucial aspect of materials engineering and structural design. Research in this area holds significant importance, particularly for aerospace applications, as these structures experience extreme temperature variations. The frequent occurrence of thermal cycles causes thermal stresses in the structure, which can lead to the formation of microcracks and degradation of the material's mechanical properties. For such applications, research is crucial to determine the impact of cycles on the

strength and durability of laminates [1, 2]. Composite materials, especially those with a polymer matrix, are exposed to degradation of mechanical properties under the influence of thermal cycling, which can damage the material system. Various parameters can influence the mechanical properties of polymer matrix composites, making a comprehensive experimental analysis necessary to define the main parameters and understand their effects [3]. Research on the impact of thermal cycling on the mechanical properties of composites attracts the attention of research communities and the aerospace and aviation industries [4–7]. To properly define thermal cycles, Hancox [8], in his research paper, thoroughly

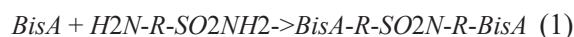
and extensively described the terminology of the thermal cycle/shock and reviewed the scientific literature on thermal cycles. Thermal cycling involves multiple exposures of a sample or material between two temperatures, with adequate exposure times to extreme temperatures to ensure thermal equilibrium. It is generally assumed that the cooling or heating rate is insufficient to cause thermal shock, a sudden temperature change. For example, immersing an unprotected sample at room temperature into liquid nitrogen would result in a thermal shock. Thermal cycling excludes the effects of external factors such as weathering (due to temperature change), exposure to sunlight or moisture, thermal degradation due to oxidative attacks on the matrix or fibers (which may result from prolonged exposure to high temperatures), and combinations of these factors. Ghasemi et al. [3] conducted an analysis of the scientific literature on the impact of cryogenic cycles on laminated composite materials, which contributed to the organization and expansion of knowledge about the impact of cryogenic cycles on the properties of polymer matrix composites. The research presented in the article provides valuable knowledge on the mechanisms and phenomena occurring in the material during cryogenic cycles, which may be important for the design and manufacture of polymer matrix composites.

Rinaldi et al. [9] tested the tensile strength of glass/epoxy samples cycled at temperatures from 0 °C to 110 °C up to 60 times. Test results showed that the polyaminophenol curing agent was better than the long chain amine curing agent. Heidari-Rarani et al. [10] researched the mechanical durability of optimized epoxy-polymer concrete in three freeze/thaw cycles. They demonstrated that the resistance to brittle cracking and tensile strength of the tested materials decreased with an increase in the average temperatures of thermal cycles. Grandidier et al. [11] presented an approach to modeling high-pressure thermo-oxidative conditioning of an epoxy resin material, which was applied in the design and optimization of accelerated thermo-oxidation tests. Gall et al. [12] studied the deformation of multi-layer materials in microelectromechanical systems (MEMS) under the influence of thermal cycling. The structures deformed non-linearly and warped at high temperature changes. Mohammad Abedi et al. [13] conducted research to investigate the effect of the cyclic process of freezing and thawing on the behavior of the corrugated glass/epoxy laminate. Mechanical tests were also carried out during the research,

including bending strength. The results showed that with an increasing number of cycles, various forms of damage leading to ultimate failure were concentrated at the notch tips. This work determines the effect of a different composition of resin reinforced with glass fabric weighing 205 g/m<sup>2</sup> on the mechanical properties of composite materials subjected to cryogenic cycles. In the previous article [14, 15], an attempt was made to analyze the mechanical properties of laminates by subjecting them to 1/7 – day and 3 – minute immersion in liquid nitrogen. This research aims to create innovative materials adapted to work in cryogenic environments.

## EXPERIMENTAL MATERIALS

The material used for the research is a glass-epoxy laminated plate made by layering successive layers of glass fabric saturated with epoxy resin. The matrices in the newly developed composite materials are made from the following epoxy resins: EPIDIAN 11M80 (Sarzyna Chemical), YD-128, YDPN 638A80 (Kukdo) modified with curing agents DICY, DDS, and novolac P. The E-type glass fabric required for the experiments was supplied by a Joint Company. For the purposes of testing, composite materials will be marked as follows: EP\_X\_Y\_RT/1D/7D, where: EP – epoxy resin, X – type epoxy resin, Y – hardener type, RT, 1C, 5C and 10C – number of cycles in liquid nitrogen. Technical parameters and designation of epoxy resins are presented in Table 1, while Table 2 contains technical data of the glass fabric. The research includes two commonly available and used forms of resin: (1) unmodified resin based on bisphenol A and (2) an epoxy resin solution enriched with bromine dissolved in an organic solvent. The structural scheme of the resins is shown in Figure 1. The formation of chemical bonds between bisphenol A and diaminodiphenylsulfone (DDS) molecules is the polymerization of these two compounds [16]. The presence of a catalyst is necessary to start the reaction forming a polymer network with permanent chemical bonds. The product of the described reaction is a rigid and durable material, i.e. epoxy resin. The reaction proceeds according to the scheme:



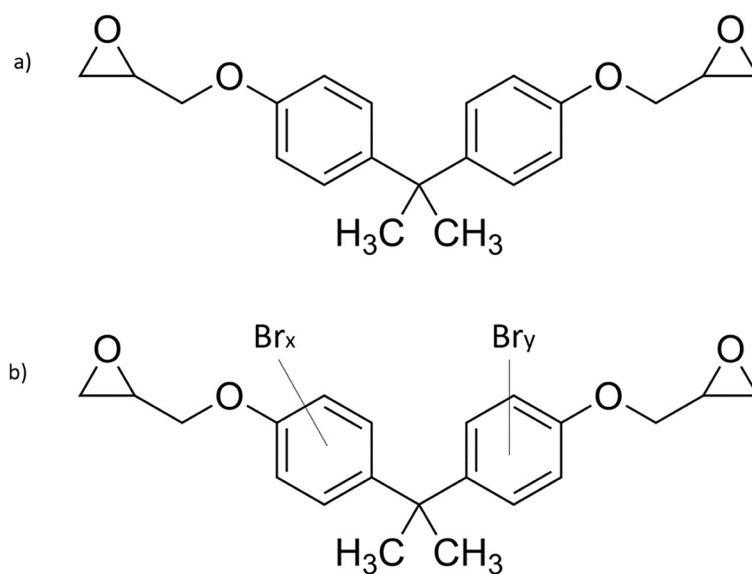
where: *BisA* – bisphenol A; *H<sub>2</sub>N-R-SO<sub>2</sub>NH<sub>2</sub>* – diaminodiphenylsulfone; *BisA-R-SO<sub>2</sub>N-R-BisA* – epoxy resin polymer.

**Table 1.** Designation of selected composite materials

Symbol	Type of epoxy resin	Hardener
EP_1_1	YDPN 638 A 80	Novolac
EP_2_2	YD -128	DICY
EP_2_1	YD -128	Novolac
EP_1_3	YDPN 638 A 80	DDS
EP_4_2	EPIDIAN 11M80	DICY

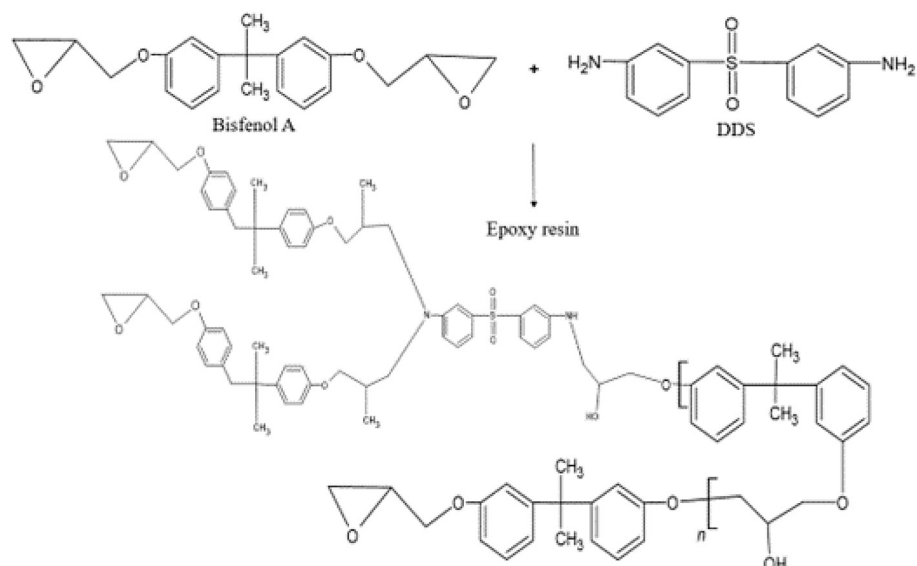
**Table 2.** Technical parameters of Joint Stock Company 7628 Type E Glass Fabric

Parameter	Value
Grammage	205 g/m <sup>2</sup>
Weave type	Plain
Glass type	„E”
Thickness	0.1697 mm
Width	110.3 cm

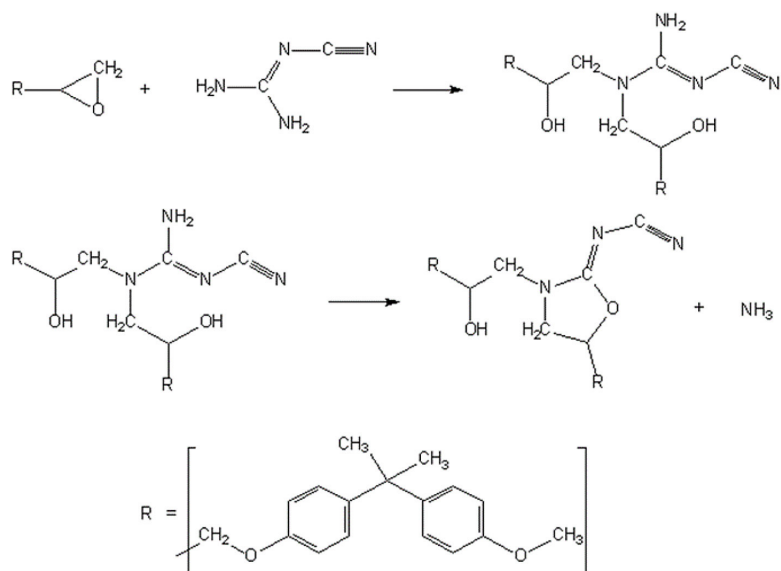
**Figure 1.** Structural formulas of selected epoxy resins (a) epoxy resin based on bisphenol A and (b) brominated epoxy resin.

Figures 2–3 present a simplified scheme of the polymerization reaction. As a result of the reaction of bisphenol A with dicyandiamide, an epoxide bond is formed due to the nucleophilic attack of the epoxide group of bisphenol A on the nitrile group in dicyandiamide, a dicyandiamide derivative is also formed. The reaction catalyst can be heavy metal ions (positive catalysts). The resulting polymer exhibits high chemical and mechanical durability. It is also a versatile material suitable for various applications. Copolymerization condensation is a

reaction resulting from the mechanism of bisphenol A (multifunctional monomer) with novolac P, which combines through formed polymer bonds. The functional groups involved in the reaction are epoxy and hydroxyl groups, bound to bisphenol A and novolac P. The outcome of the reaction is a copolymer characterized by improved environmental resistance and higher mechanical properties compared to monomers [16, 17]. The reaction mechanism of brominated epoxy resin with novolac P is a chemical process. It occurs between the epoxy functional groups on the resin and the



**Figure 2.** Simplified scheme of the epoxy resin reaction based on bisphenol A with DDS [11]



**Figure 3.** Reaction of epoxide functional groups with dicyandiamide, proposed by the Zahir [12, 13]

amino groups on the novolac, with hydrocarbon linkages between them. The new molecules resulting from this process have enhanced stability and mechanical properties. A detailed description of the reaction mechanism depends on the chemical composition of the compounds used and the reaction conditions – temperature, time, and molar ratio [18]. The complexity of the reaction process results in elaborate structural formulas of the products, which is why they have been omitted in this context. Composite sheets were fabricated at the Plastics Plant IZO-ERG S.A in Gliwice, the process consisted of the following stages:

1. Resin composition preparation.
2. Supersaturation of the carrier with the resin composition.
3. Forming the board/sheet product using a pressing process.

The selected components of the matrix composition - resin and hardener - were dissolved in the solvent in appropriate proportions. The ingredients were subjected to a mixing process until DICY was completely dissolved. Table 3 presents the parameters of the prepared material - matrix, the final properties of the composites depend on the manufacturing process. The preparation

**Table 3.** Parameters of composite materials

Symbol	Fluidity [%]	Resin content [%]
EP_1_1	24.60	38.00
EP_2_2	13.00	33.60
EP_2_1	17.90	35.48
EP_1_3	13.10	34.60
EP_4_2	21.00	34.00

process of the materials was also thoroughly described in the articles [14, 15].

A Hoesch lab coater coated glass cloth with EP\_X\_Y resin compositions at 160 °C. Then, the carrier was saturated with an adequately selected amount of matrix, and the prepared carrier-resin system was pre-hardened. The effect of the described activities was the creation of a semi-finished product, which was divided into sheets of 30×50 cm. In a Hoesch lab press, the sheets were pressed for 120 minutes at 165 °C. The pressing parameters were chosen to ensure the crosslinking effect and achieve a full range of strength properties. The obtained laminate consists of 8 sheets of epoxy-glass prepreg arranged in a parallel orientation [0°/90°].

### RESEARCH METHODOLOGY

The samples were cut from the prepared composite plates according to standardized sizes. Cryogenic cycles were carried out on a workstation for cooling samples in liquid nitrogen – Dewar YDS-5-200 from “Cryogen.” The samples were successively placed in a bucket and submerged. A complete cycle was defined as 2 minutes in liquid nitrogen (LN2) and 5 minutes at room temperature + 23 °C. After the cooling phase, the scoop with the samples was placed on the table at room temperature. One cycle lasted 7 minutes. Five different sample configurations were cycled 1, 5, and 10 times. Samples that have not been subjected to cooling cycles in liquid nitrogen. After the cooling process, the samples were carefully preserved to be used for mechanical analysis.

#### Charpy impact testing

The Charpy impact test was used to determine the impact strength using the PSW-40 Leipzig pendulum hammer. The tests were carried out

following the recommendations of the PN-EN ISO 179-2 standard “Plastics. Charpy impact strength determination. Instrumental impact test” [16]. The hammer used for the tests is a standard hammer used in impact tests of production laminates with a breaking energy of 40 kJ. For each developed composite material, measurements were made in room conditions and after exposure to liquid nitrogen, which was carried out as described previously. The impact strength was determined for each composite, and the average of the measurements was made for 6 samples.

The impact strength was calculated according to the Equation 2 [16]:

$$Re = \frac{Ec}{h \times b} \times 10^3 \left[ \frac{kJ}{m^2} \right] \quad (2)$$

where: *Re* – impact strength [kJ/m<sup>2</sup>]; *Ec* – breaking energy [J]; *h* – thickness of the test piece [mm]; *b* – width of the test piece [mm].

The thickness and width of the sample were measured with a caliper before starting the impact test. Table 4 contains the indications of the fracture character in accordance with the applicable standard.

#### Flexural properties testing

The PN-EN ISO 178 standard, “Plastics - Determination of bending properties” [17] was used to determine the conditions for performing a three-point bending test on the INSTRON 500N-50 kN testing machine. The distance between the supports of the machine has been fixed at 32 mm, and the bending speed was 2 mm/min. All series of samples were subjected to 6 bending tests. The

**Table 4.** Designation of fracture characteristics according to standard PN-EN ISO 179-2 [12]

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

width and thickness of the beam were measured with a caliper prior to the start of the test; it is a measurement necessary to construct the strain vs. stress plots. The obtained values made it possible to calculate the longitudinal modulus of elasticity and the bending strength. The thickness of the composite materials produced is less than 3 mm.

## DISCUSSION OF RESULTS IN VARIED TEMPERATURE CONDITIONS

### Results of Charpy impact testing

The results of impact strength tests after 1.5 and 10 cycles of exposure to liquid nitro-gen are presented in Table 5, and Figure 4. The tests have shown that cryogenic cycles of multilayer composites cause minimal changes in their impact properties. Materials EP\_1\_1, EP\_2\_2, EP\_2\_1, EP\_1\_3, and EP\_4\_2 are stable, and differences in impact strength after cycles are less than 5%. The most noticeable changes were noted for EP\_4\_2, which showed the best impact toughness results in both the 1st, 5th and 10th cycles.

### Microscopic observations

Microscopic structures of the external surface of the tested laminates (Fig. 5) taken with a Leica DVM6 digital microscope (from Leica Microsystems with the possibility of precise 2D and 3D image analysis) at 40x magnification. Impact test results on damaged specimens show no signs of structural delamination, round dents, or air bubbles. This proves proper saturation with resin and good technological quality, enabling adequate oversaturation for the carrier. During

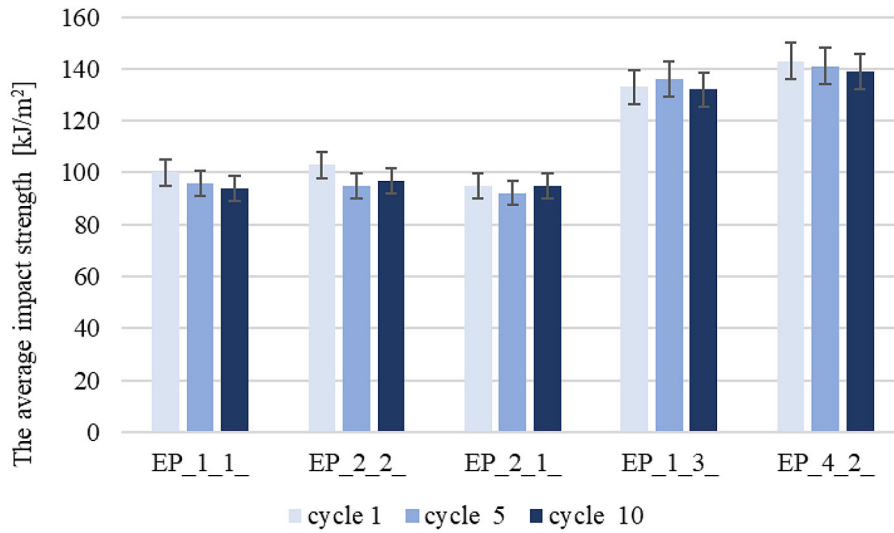
the observations, no defects were found, indicating successful manufacturing. Most materials exhibited brittle behavior upon impact. Cryogenic cycles had little effect on the material’s structure or fracture characteristics. Only in the case of EP4\_2\_ was a complete fracture observed.

### Results of flexural properties testing

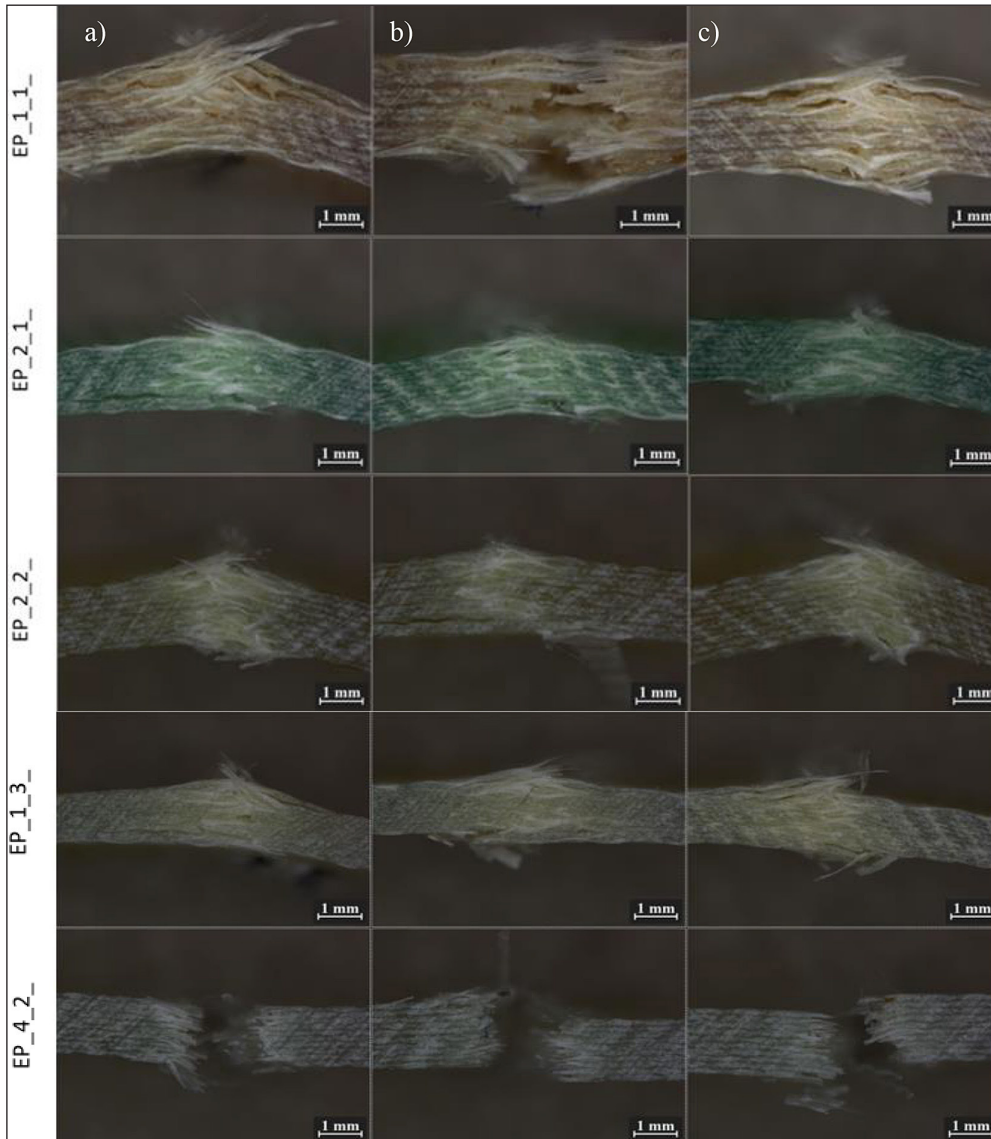
The results of the flexural strength and elastic modulus tests and the graphically obtained mechanical results of the tested composite materials are shown in Table 6 and Figures 6–7, respectively. According to the IEC-893 standard – “Part 2: methods of test, specification for industrial” [18], the bending strength of epoxy-glass laminates should not be less than 350 MPa. All tested laminates meet the requirements of the IEC-893 standard. The analysis of the obtained results revealed that cryogenic cycles in almost all cases resulted in the deterioration of the bending properties of the composite materials. The exception is EP\_2\_1, whose bending property increased with the number of cycles, and its stiffness modulus is the highest among the tested materials. The results of the tests indicate that the EP\_4\_2 composite had the highest flexural strength among all composite materials. After 1 cycle, its value was 817 MPa; after 5 cycles, 799 MPa; and after 10 cycles, 790 MPa. The worst flexural strength results were recorded for the EP\_1\_3 composite, which had a value of 526 MPa after 1 cycle and 521 MPa after 5 cycles. After 10 cycles, this value dropped significantly to 512 MPa, which is a 37.33% decrease compared to the best composition. It is worth noting that the modulus of elasticity for both tested composites, i.e., EP\_4\_2 and EP\_1\_3, decreased with the increased number of

**Table 5.** Average impact test results for five sample configurations after 1.5 and 10 cycles

	Symbol	Impact strength [kJ/m <sup>2</sup> ]	Type of fracture
1C	EP_1_1_	100	P
	EP_2_2_	103	P
	EP_2_1_	95	P
	EP_1_3_	133	P
	EP_4_2_	143	C
5C	EP_1_1_	96	P
	EP_2_2_	95	P
	EP_2_1_	92	P
	EP_1_3_	136	P
	EP_4_2_	141	C
10C	EP_1_1_	94	P
	EP_2_2_	97	P
	EP_2_1_	95	P
	EP_1_3_	132	P
	EP_4_2_	139	C



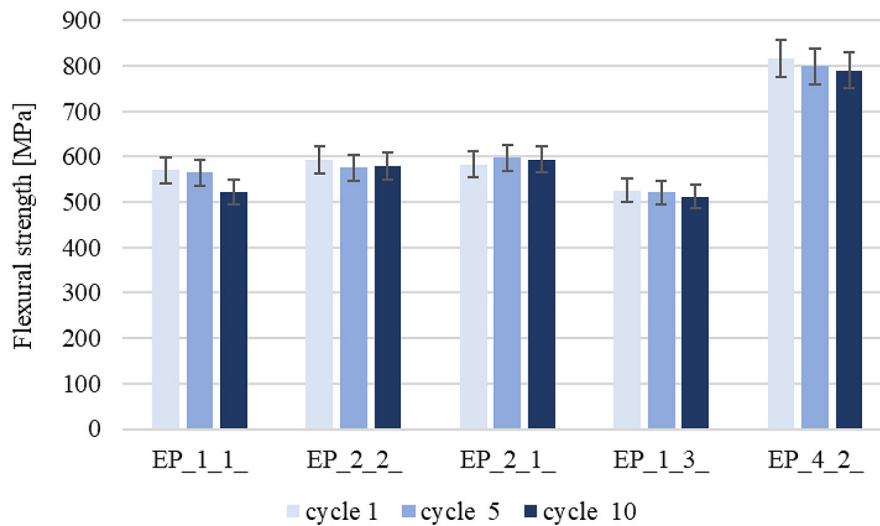
**Figure 4.** Evaluation of the influence of 1/5/10 cycles on the impact properties of different configurations of laminates



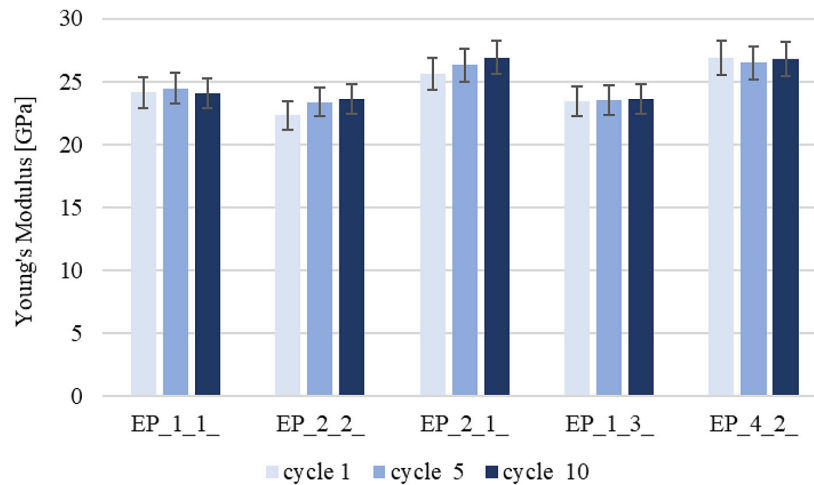
**Figure 5.** Comparison of photos of laminates in 40x magnification; (a) 1C, (b) 5C and (c) 10C

**Table 6.** Average results of flexural strength tests and Young’s Modulus values of samples after 1/5/10 cycles

	Symbol	Flexural strength [MPa]	Young’s modulus [GPa]
1C	EP_1_1_1C	570	24.10
	EP_2_2_1C	593	22.29
	EP_2_1_1C	583	25.59
	EP_1_3_1C	526	23.40
	EP_4_2_1C	817	26.89
5C	EP_1_1_5C	565	24.46
	EP_2_2_5C	576	23.37
	EP_2_1_5C	597	26.31
	EP_1_3_5C	521	23.54
	EP_4_2_5C	799	26.48
10C	EP_1_1_10C	522	24.06
	EP_2_2_10C	579	23.62
	EP_2_1_10C	594	26.91
	EP_1_3_10C	512	23.63
	EP_4_2_10C	790	26.77



**Figure 6.** Comparison of flexural strength of tested series of samples



**Figure 7.** Comparison of Young’s modulus values from the three-point bending test



cycles, so it had a negative effect on their stiffness. The composite EP\_2\_1 is remarkable as it showed an increase in bending strength properties after 1, 5, and 10 cycles compared to other samples. This is an inverse trend. Additionally, cryogenic cycles contributed to an increase in the elastic modulus of EP\_2\_1, meaning that the material became stiffer. Similar trends were observed for EP\_1\_1 and EP\_2\_2. The results of bending strength and Young's modulus based on a brief review of the scientific literature are presented in Table 7. Comparing the flexural strength results obtained in this study with the findings from the article [15], it can be observed that the difference between the obtained values is not more significant than 5%. This indicates that 1, 5, and 10 cycles do not significantly impact the results, and the materials remain stable under the specified test conditions. However, for EP\_4\_2, the difference was approximately 9%, suggesting that this material slightly improved its flexural properties after the cycles. Regarding Young's modulus, the cycles and exposure to liquid nitrogen were found to influence these values. After the cryogenic cycles, all materials showed an increase of more than 10% in their Young's modulus [15]. In the case of impact strength, neither condition significantly affected the values except for EP\_1\_3, which showed an increase of approximately 35% compared to the values obtained during immersion. It is worth conducting how different resins and catalysts affect impact properties in the future. Comparing the data with scientific literature, it can be confidently stated that promising results were obtained in terms of flexural strength, impact, and Young's modulus. Azhary [18] studied a material composed of bisphenol A-based epoxy resin, a polyamide hardener, and E-glass fabric. The flexural strength of the epoxy/glass composite was 103.78 MPa, while the tensile modulus was 4.8 GPa. Nagaraja's research group [19] analyzed the flexural and modulus properties of carbon-glass/epoxy hybrid composite laminates, which were 615.48 MPa and 15.40 GPa, respectively. On the other hand, Wang [20] conducted research

on E-glass fiber reinforced epoxy composites as a function of the silane coupling agent used, namely  $\gamma$ -Aminopropyltriethoxysilane (APS) and  $\delta$ -aminobutyltriethoxysilane (ABS). The results were 449 MPa for flexural strength and 28 GPa for tensile modulus. In the article [15], a detailed study was conducted on the impact of a low-temperature environment on the mechanical properties of glass fiber reinforced epoxy composite laminates (GFRP). However, the reduction in stiffness due to accumulated damage was more significant in laminates tested at low temperatures (~17% vs. ~11%). Since a relatively small decrease in stiffness (~2–3%) was observed in some of the tested laminates, it can also be stated that the composition of the matrix material plays an important role in delaying the initiation and formation of damage. However, in article [14], the impact properties and strength of composite materials were compared. The samples were tested at room temperature (RT) and after 1 and 7 days of immersion. It was found that immersing glass-epoxy composites in a Dewar vessel had minimal impact on their strength properties for impact and bending. 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 for the production of laminates in electrotechnics, where it should demonstrate excellent impact properties. Table 8 presents the results based on a brief review of scientific literature.

**Scanning electron microscope observations**

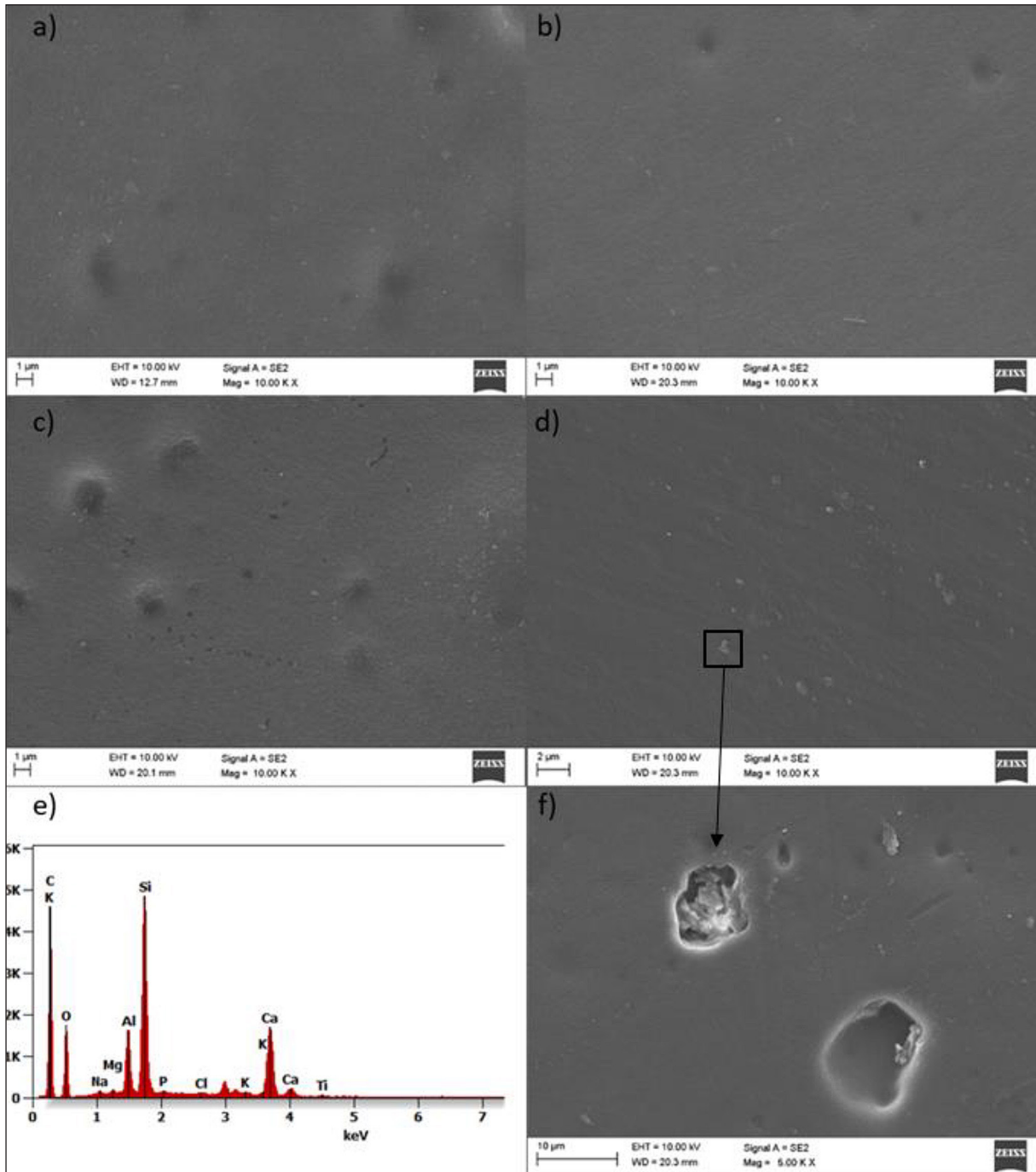
Figures 8–12 present microphotographs of the surface of composite materials at 10,000x magnification. These microphotographs were taken using a Zeiss Supra 35 high-resolution scanning electron microscope equipped with the GEMINI optical column (SE and BSE detectors were used). Sample preparation involved sputter coating the specimen with a conductive Ag material using a Bal-Tec SCD 050 device with the following parameters:

**Table 7.** Brief review of the scientific literature

Flexural strength [MPa]	Young's modulus [GPa]	Reference
103.78	4.80	[18]
615.48	15.40	[19]
449.00	28.00	[20]

**Table 8.** Brief review of the scientific literature

Material	Impact strength, kJ/m <sup>2</sup>	Flexural strength, MPa	Reference
RT_EP_4_2	157	775.8	[14]
1D_EP_4_2	152	750.36	
7D_EP_4_2	131	762.92	
Epoxy-glass composite	200	103.78	[18]
	–	449	[20]
	–	420	[21]



**Figure 8.** Surface characteristics of EP<sub>1\_1</sub> particles, magnification 10,000x, (a) cycle 0, (b) cycle 1, (c) cycle 5, (d) cycle 10 and (e), (f) analysis of the chemical composition of the unidentified area (EDS)

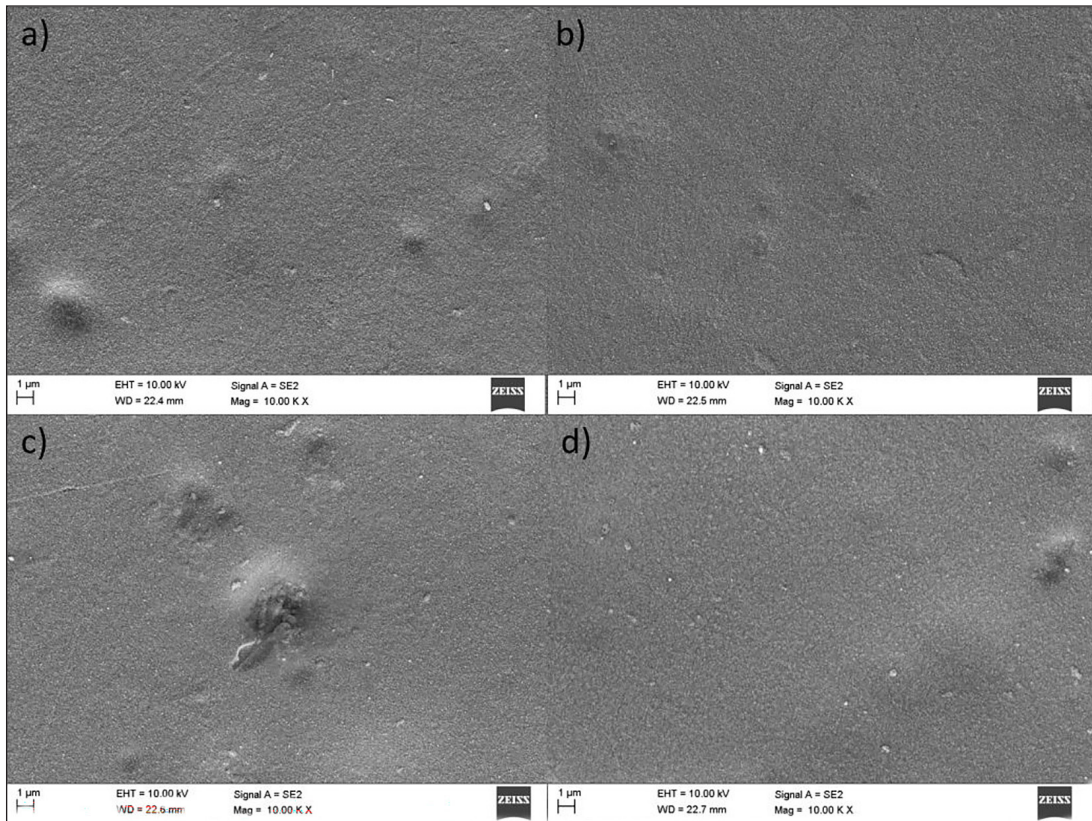


Figure 9. Surface characteristics of EP\_1\_3 particles, magnification 10,000x, (a) cycle 0, (b) cycle 1, (c) cycle 5, (d) cycle 10

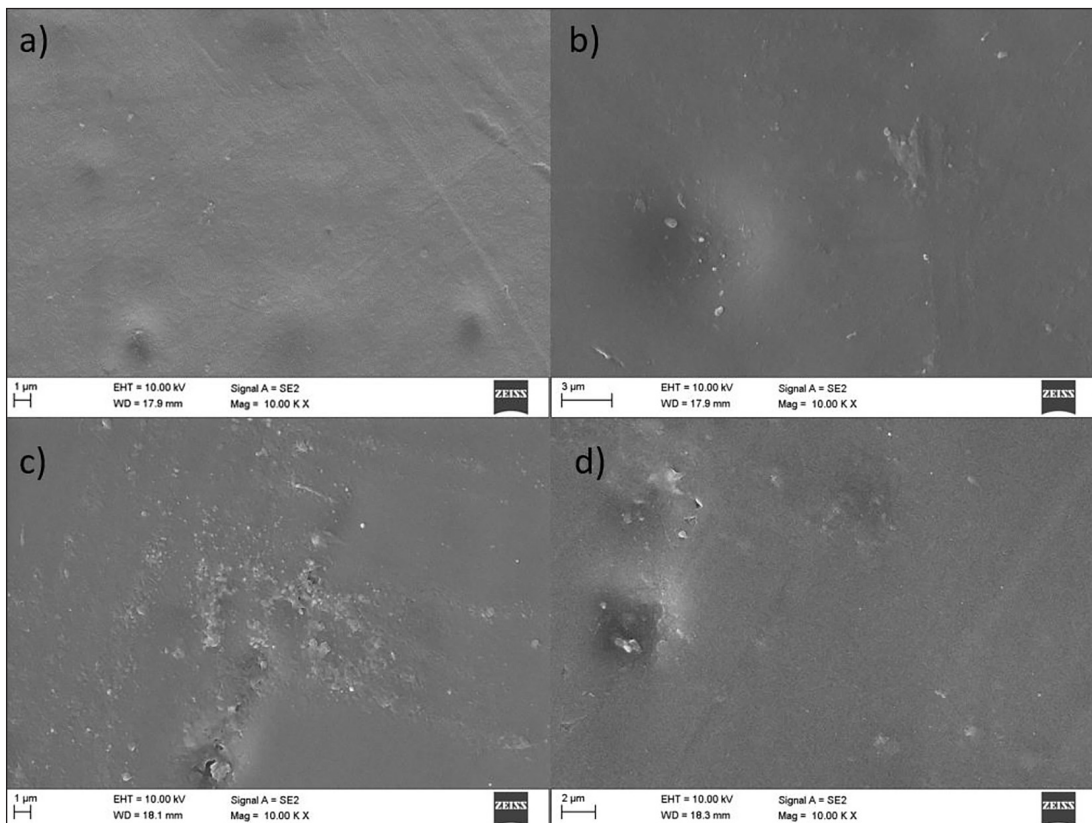
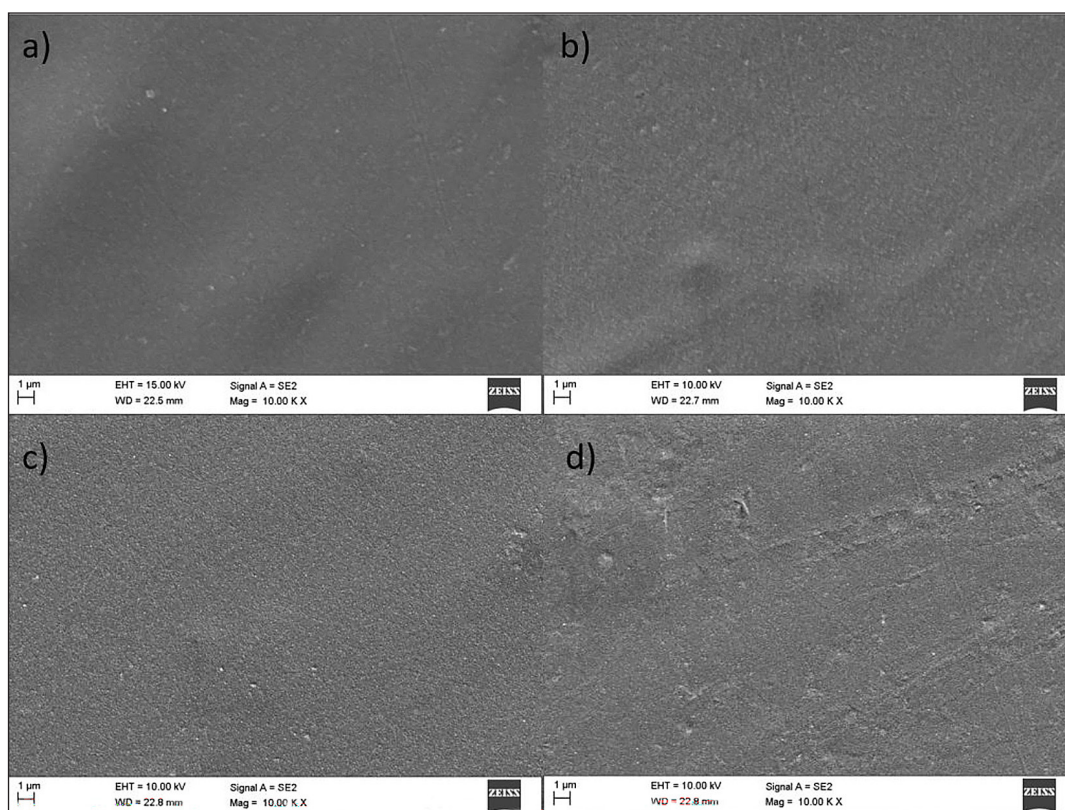
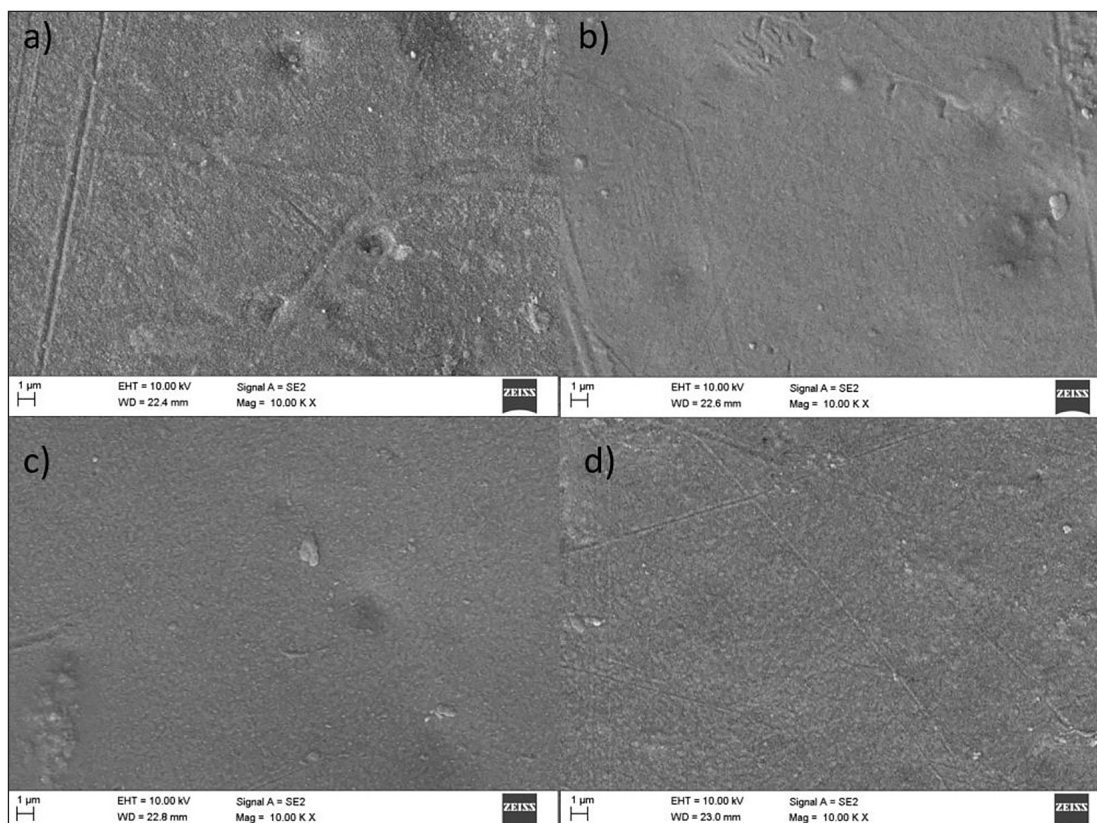


Figure 10. Surface characteristics of EP\_2\_1 particles, magnification 10,000x, (a) cycle 0, (b) cycle 1, (c) cycle 5, (d) cycle 10



**Figure 11.** Surface characteristics of EP\_2\_2 particles, magnification 10,000x, (a) cycle 0, (b) cycle 1, (c) cycle 5, (d) cycle 10



**Figure 12.** Surface characteristics of EP\_4\_2 particles, magnification 10,000x, (a) cycle 0, (b) cycle 1, (c) cycle 5, (d) cycle 10

- spraying time: 20 seconds,
- current intensity: 50 mA,
- voltage (resulting): 480 V,
- table temperature: 21 °C.

Microanalysis of the chemical composition of the investigated surfaces of the samples was performed using an EDX UltraDry detector from Thermo Scientific™. Themicrophotographs (8–12) showed many imperfections like indentations, undissolved epoxy resin, and some mechanical damage, probably from manufacturing or cut-ting the material. No microcracks due to cryogenic cycles were observed. EDS analysis re-vealed the presence of impurities like K, Na, Mg, Al, Si, P, Cl, Ca, and Ti. The Si indicates the presence of glass fabric particles, likely from the sample-cutting process.

## CONCLUSIONS

The research’s main goal was to analyze the mechanical properties of the developed composite materials reinforced with glass fabric. It aimed to determine how the compo-sites behave after 1, 5, and 10 cryogenic cycles. The obtained results will be a starting point for further research on new material solutions adapted for cryogenic applications. Five different material variants, with different types of epoxy resin and hardener, were used in the study. The main conclusions from the conducted research are as follows:

- the effect of 1, 5, and 10 cycles in liquid nitrogen influenced the impact properties of the tested materials;
- composite EP\_4\_2 stands out with exceptionally high impact properties, which remain stable both at normal and lower temperatures;
- all other composites have stable impact properties, regardless of temperature conditions;
- the EP\_4\_2 composite had the highest flexural strength among all composite materials;
- Young’s modulus of configuration EP\_2\_1 tended to increase after 1,5 and 10 cycles;
- all configurations meet the requirements of IEC-893 - “Part 2: Methods of Test, Specification for Industrial”;
- SEM did not detect any microcracking caused by cryogenic cycles.

The next step will also involve increasing the cryogenic cycles to 35, 50, and 65 to verify the mechanical properties and detect any microcracks induced by thermal shock.

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