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# The influence of high and low temperatures on the impact properties of glass–epoxy composites

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*Abstract*: The aim of this paper is to present the influence of high and low temperatures on the impact properties glass–epoxy composites. The impact strength $a_n$ is presented for four different glass–epoxy composite structures at three different temperatures, *i.e.*, at room temperature $t$ = 20 °C, at an elevated temperature $t$ = +50 °C and at a low temperature $t$ = –50 °C. Standard mechanical testing was carried out on the composite materials with specific masses of reinforcement of 210 g m$^{-2}$ and 550 g m$^{-2}$ and orientations 0°/90° and ±45°. Micromechanical analysis of the failure was performed in order to determine real models and mechanisms of crack and temperature influence on the impact properties.

*Keywords*: glass woven–epoxy composite material, impact test, high and low temperature impact tests, micromechanical analysis

## INTRODUCION

A very common way of fabrication of composite materials is using glass fibers for reinforcement and an epoxy resin as the matrix, which results in materials characterized by good physical, chemical, thermal, and mechanical properties. Some of the most important technical characteristics of these materials are their static and dynamic properties, which are due to the structure of the composite and the specific mechanism of crack formation. However, in reality, during exploittation, a lot of construction parts are subjected to high and low temperatures. In these cases, the toughness of the material changes, which can cause cracking under the stress, which is different from the one obtained in standard testing.

For the past few decades many researchers have studied the mechanical properties of the composite materials themselves, as well as the changes of the properties of the materials with changing temperature, which represent the basis of the research presented in this paper.

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Howard and Hollaway[1] studied the highly elastic properties of an epoxy resin and showed how they influence the properties of the composite material itself, as well as the changes of the properties of the materials with changing temperature.

Shindo, Ueda and Nishiok[2] in their joint work dealt with the thermal mechanical response of non-metalic woven composites with temperature-dependant properties. The composite material in generalized plane strain was assumed. First, a finite element method was used to study the influence of crack formation, residual thermal stresses and weave curvature on the mechanical performance of glass–epoxy laminates at low temperatures. Subsequently, they examined the stress state at the free edge of the woven composites. Finally, they calculated the mechanical properties of the woven composites. Numerical results on the distribution of the stresses and the mechanical properties at different temperatures and the warp angles were obtained and presented in graphical form.

Myung–Gon Kim *et al.*[3] researched the tensile properties of a glass/epoxy composite which had been cycled with thermo-mechanical loads at low temperatures using an environmental test chamber. Thermo-mechanical tensile cyclic loading (up to 10 cycles) was applied to laminates from room temperature (r. t.) to –50 °C, to –100 °C, and to –150 °C (c. t.). The results showed that the tensile stiffness significantly increased with decreasing temperature, while thermo-mechanical cycling had little influence on it. The tensile strength, however, decreased as the temperature was decreased down to c. t., while the rate of strength decrease was reduced after c. t. cycling. For the analysis of the test results, the coefficients of thermal expansion of the laminate composite specimen, both at r. t. and c. t., were measured, and the interface between fiber and matrix was observed using SEM images.

Abdel–Magid *et al.*[4] studied the properties of E–glass/epoxy composites at 65 °C. The values of the module of elasticity, stress and strain were examined. They were compared with the values obtained at room temperature; a decrease in value of the module of elasticity was noticed as well as of the break elongation. As a result, these authors concluded that longer exposure of the samples to higher temperatures caused ductile breaks on E–glass/epoxy composites.

In their work, Sefrani and Berthelot[5] presented an analysis of the temperature effect on both the stiffness and the damping of glass fibre composite materials. The experimental results showed that the mechanical properties were appreciably maintained up to the glass transition temperature, where the damping increased sharply in a small temperature interval.

Khalid[6] tested aramid/epoxy composite samples reinforced with glass fibers by the Sharpy impact method. The composite samples were made manually and had the dimensions 50 mm×10 mm×10 mm. The testing was performed in temperature interval + 40 to –40 °C with 10 °C steps. The volume fractions of fibers were 0.45, 0.55 and 0.65. The effects of the volume fraction of fiber and the temperature on the impact toughness of the composite samples were tested. Damage of the tested samples was observed using a microscope at a magnification of 100. The results showed a slight increase of the impact toughness of the composite samples with increasing temperature in the interval –40 °C to –10 °C. This was followed by a larger increase of the value of the impact toughness with increasing temperature in interval –10 °C to 40 °C. It is shown that the aramid/epoxy has a higher impact toughness than glass/epoxy at all the tested temperatures. On the other hand, breaking of the fibers was found in the composite sample and the appearance of damage as a coma in the sample itself. Increasing the volume fraction of fiber decreased the impact toughness of the glass and aramid/epoxy.

Ray[7] suggested the need for investigating and characterizing the freeze-thaw response of polymer composites under different loading rates and also at ambient and sub-ambient temperatures. The presented experiments were performed with three weight fractions (*i.e.*, 0.55, 0.6 and 0.65) of glass fibers reinforced polyester composites. The specimens were suddenly exposed to a temperature of –80 °C for 2 h and then either immediately tested at that temperature or after allowing the samples to thaw to ambient temperature for 1 h.

According to the previous review, it is obvious that many scientists analyzed the properties of polymer composite materials at different temperatures. However, only a few of them tested their properties on impact loading and made conclusions about the change of their impact properties with changing temperature. That was one of the main reasons for the experiments reported in this paper.

In order to determine the properties of composites of different structures under extreme conditions, the impact tests were performed at +20 °C, +50 °C and –50 °C. By fractographic analysis of the crack surfaces, a complete picture was formed of the fabricated glass woven–epoxy composite materials, as well as of models and mechanisms of damage and crack initiation and propagation under load conditions.

## EXPERIMENTAL

The composite materials were fabricated under laboratory conditions at the Faculty of Technology and Metallurgy in Belgrade, Serbia. The selected fiber reinforcement was woven roving E–glass (Tables I and II), produced by Tehnotex, Sombor, which is based on silicate glass containing up to 1 % alkali. The selected glass fibers have good mechanical, hydro-thermal and dielectrical properties. Two specific weights of glass woven reinforcement were used ($C$ = 210 g m$^{-2}$, $D$ = 550 g m$^{-2}$) and two orientations (0º/90º and ±45º). The glass woven was made by classical procedures of spinning on different kinds of looms (Fig. 1).

The matrix material was a polycondensation product of 2,2-bis-(4-hydroxyphenyl) propane (bisphenol A) and epichlorhydrin (Epidijan 6 made by Zaktady Chemiczne "Organika–Sarzyna" S.A) with the structure shown in Sheme 1.

3-Aminomethanol-3,5,5-trimethanolcyclohexylamine, a modified cycloaliphate amine of the same producer, was used for fixing. The properties of the employed resin were taken from the producer's catalog and are presented in Table III.

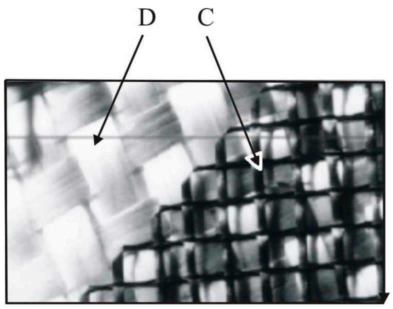
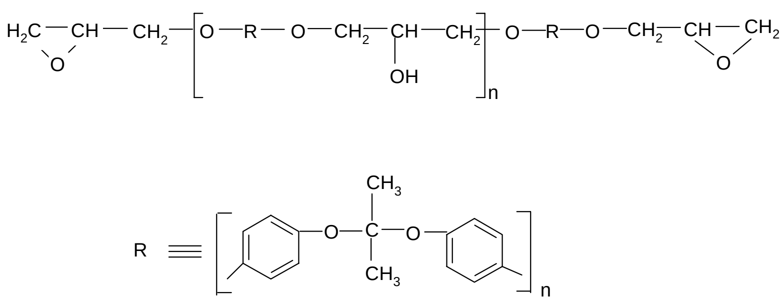



Fig. 1. A view of used types of woven glass.



Scheme 1. Polycondensation product of 2,2-bis-(4-hydroxyphenyl) propane (bisphenol A) and epichlorhydrin.

TABLE I. Structural components of E–glass

| Structural component | Content / % |
|---|---|
| Silicium(IV)-oxide | 52 – 56 |
| Aluminium(III)-oxide | 12 – 16 |
| Boron(III)-oxide | 5 – 10 |
| Sodium(I)-oxide, Potasium(I)-oxide | 0 – 2 |
| Magnezium(II)-oxide | 0 – 5 |
| Calcium(II)-oxide | 16 – 25 |
| Titanium(IV)-oxide | 0 – 1.5 |
| Iron(III)-oxide | 0 – 0.8 |
| Iron | 0 – 1 |

The epoxy–amine mixtures were prepared by heating the resin in an oil bath to 70 °C, adding the curing agent and continuously stirring until a clear homogeneous solution was obtained. Each laminate was fabricated by hand in a wet lay-up. Alternate layers of fiber reinforcement plies and liquid resin were placed inside a dam on a flat mould plate. The mould plate[8,9,10] consisting of an upper and bottom metal board of dimensions 292 mm×230 mm×13 mm was tightened with four screws to obtain the necessary pressure force of 67 N. The materials were cured for 48 h at room temperature, followed by 5 h at 90 °C and a final slow cooling. The structures of the fabricated composites are given in Table IV.

The impact test was performed on an impact test machine with a pendulum for Sharpy experiments in accordance with ASTM[11] on cut specimens (the cutting was in two directions, with orientation 0°/90° and ±45°) of fabricated composite materials. All combinations of the specimens with dimensions (55 mm×10 mm×2 mm) are given in Table IV. The specimens were machined from flat panels using a high speed diamond saw with liquid cooling. This machining operation resulted in very smooth, square cuts. One edge of each specimen was polished so that cracks and delamination could be readily discerned.

TABLE II. Physical properties of E–glass fiber

| | |
|---|---|
| Specific weight | 2.6 N $m^{-3}$ |
| Tension strength | 2400 MPa |
| Modulus of elasticity | 73 GPa |
| Extension | 3.3 % |
| Thermal extension | $5\times10^{-6}$ $K^{-1}$ |
| Thermal installing | 1 W $mK^{-1}$ |
| Dielectrical constant | 6.7 |
| Specific electrical resistance | $10^{14}$ Ω cm |
| Absorbing moisture, at 20 °C → 65 % | 0.1 |

TABLE III. Catalog properties of epoxy resin

| Properties | Specification | Analysis results |
|---|---|---|
| Appearance | Viscous yellow liquid | Viscous yellow liquid |
| Epoxy number/ val/100 g | 0.51– 0.54 | 0.520 |
| Epoxy equivalent | 196– 185 | 192 |
| Density/ g $cm^{-3}$ | – | 1.26 |
| Viscosity at 25 ºC/ Pa s | 10–15 | 13700 |
| Color according to *Gardner* | 3 | less than 3 |
| Contents of unelaborated components/ % min | 99 | 99.5 |
| Contents of organic chlorine/ % max | 0.3 | 0.17 |

TABLE IV. The structure of fabricated composite materials

| Sample | Number of rein-forcement layers | Specific mass of reinforcement/ g $m^2$ | Orientation of reinforcement | Mass fraction of reinforcement/ % |
|---|---|---|---|---|
| I–C–1 | 5 | 210 | 0°/90° | 34.2 |
| I–D–1 | 4 | 550 | | 56.7 |
| I–C–2 | 5 | 210 | ±45° | 35.4 |
| I–D–2 | 4 | 550 | | 58.4 |

Five test specimens for each of the studied materials and test temperatures were used for the experiments. The heating and cooling of the test specimens for study at the high and low temperature was conducted in small closed chambers for the temperature regulation and subsequently they were set on the test machine. The temperature was measured with a digital thermometer and at the moment when the expected temperature was reached, the test specimens were set in the testing device and tested.

Scanning with an electronic microscope (SEM–Jeol JSM 5300) was performed on the fracture surfaces of mechanically failed specimens to study the mechanism of crack formation. The fracture surfaces were vapor coated with a thin layer of gold to enhance the image.

## RESULTS AND DISCUSSION

*Impact test at* t=*20 °C*

The values of impact energy were directly read on the scale of the testing device. According to it and the dimensions of the test specimens, the impact strength was calculated from Eq. (1):

$$a_n = \frac{A_n}{bh} \qquad (1)$$

where: $a_n$, J $m^2$, is the impact strength of the test specimen; $A_n$, J, is the impact energy that the specimen absorbs; $b$, m, is the width of specimen and $h$, m, is the thickness of specimen.

The average values (five tested specimens) of the impact strength are given in Fig. 2.

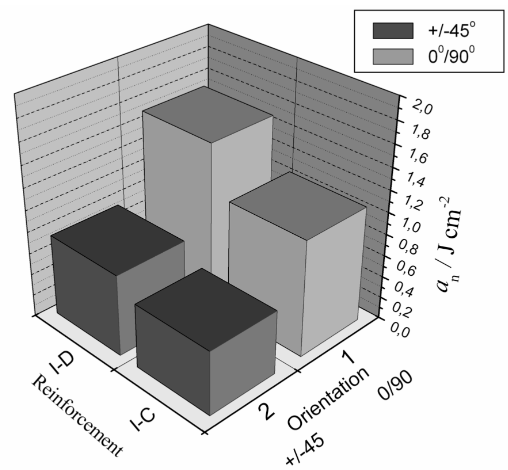



Fig. 2. Impact strength, average values at 20 °C.

Based on the experimental results presented in Fig. 2, it can be concluded that the impact properties increased for both types of orientation of the reinforcement with increasing specific mass of the glass woven reinforcement in the sample, although the results were better in case of orientation 0°/90° (samples C–1 and D–1) than in case of orientation ±45° (samples C–2 and D–2). In both cases (0°/90° and ±45°), a greater impact strength was observed for the samples with reinforcement of the type D, which confirms the assumption that a greater specific mass fraction of reinforcement imparts a better impact strength on composite materials.[8,9,10] Also, a second explanation of these results is the type of reinforcement cloth. The employed reinforcement cloth D (Fig. 1) has small spaces between the plaits of weaving. This means that spaces without resin (in other words with air bubbles) do not exist. In the other case, when the reinforcement was type C (with large spaces between the plaits of weaving, Fig. 1) the situation was different. In this case, the area between the plaits of weaving contained a lot of air bubbles, which caused the decrease of impact strength. This conclusion will be proved in the micromechanical analysis in this paper.

*The impact test at elevated (*t = *+50 °C) and low (*t = *–50 °C) temperatures*

In order to determine the influence of temperature on the value of the impact strength, studies were performed at +50 °C and –50 °C. The procedure of the study, the calculation (Eq. (1)) and the employed device were the same as for the procedure of the study at room temperature. For a better comparison of the results, Fig. 3 shows the average calculated values of the impact strength at the three test temperatures. Comparison of test results was conducted in relation to results of the study at room temperature.

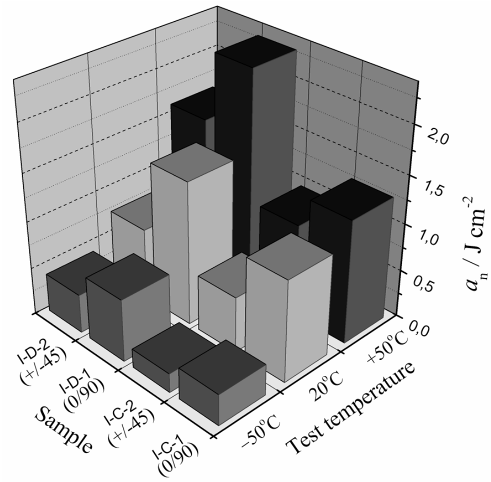



Fig. 3. Impact strength, average values for all test temperatures.

*Micromechanical analysis of failure*

An impact load crack was initiated in all the test specimens on the side of the outer layer which was subjected to tension (Fig. 4). The break occurred at the moment a critical state of stress in the material was reached, which caused the occurrence of a critical value of crack and its progressive growth. The location of the critical crack is related to fiber–matrix debonding (Fig. 5), after which the fibers cracked. It is obvious that on the spot where the first break appeared (outer layer) more broken fibers existed which had previously been debonded and pulled out from the matrix. Crack propagation leads through to the inner surface of test specimen in the transversal direction of the test specimen. Lateral on this crack, cracks and delamination in test specimens appeared (Fig. 6) as a result of shear stresses in the layers. The existence of shear stresses is characteristic for the impact test, especially in the case when the orientation of the glass woven reinforcement was ±45°. The inner layers which were loaded on impact were exposed to great axial stresses. The final crack was in the vertical plane of the test specimen.

It is very interesting that the broken test specimens tested at a temperature of –50 °C had a completely different appearance to those tested at +50 °C. This is totally understandable bearing in mind the two specific types of deformations characteristic for a polymer matrix subjected to different test temperatures: viscous flow and elastic deformation. These two types of deformations in the material directly influence the final properties of a polymer composite material. After the experiment, the specimens tested at the higher temperature showed characteristics of ductile fracture (Fig. 7), whereas those tested at the lower temperature had a brittle crack (Fig. 8).

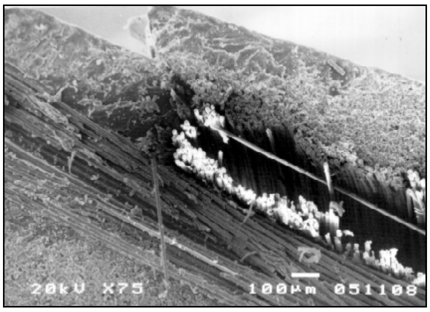



Fig. 4. SEM Micrograph of the cracking of outer layer of specimen subjected to impact load on enlargement ×75; test sample I–D–1.

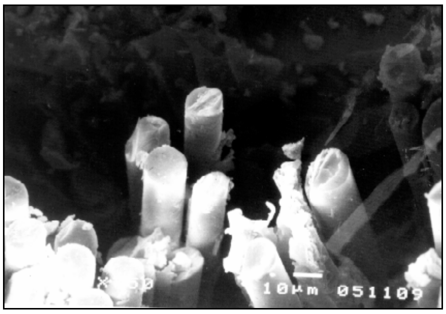



Fig. 5. SEM Micrograph of the fiber–matrix debonding on enlargement ×350; test sample I–D–1.

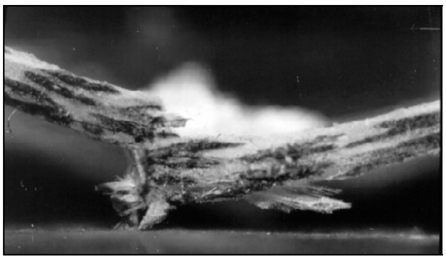


Fig. 6. Delaminations between layers in test sample I–C–2.

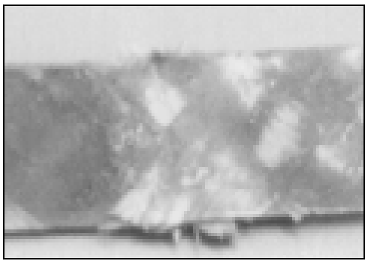


Fig. 7. A view of impact cracked test specimen at $t$ = +50 °C (the appearance of ductile fracture); test sample I–D–2.

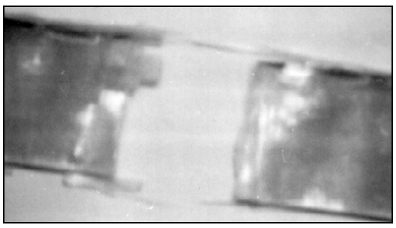


Fig. 8. A view of impact cracked test specimen at $t$ = –50 °C (the appearance of brittle crack); test sample I–D–1.

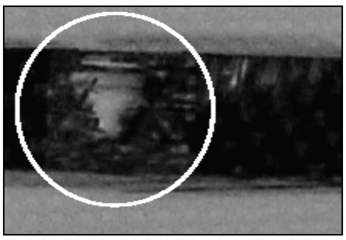


Fig. 9. Delamination and failure fracture surface on elevated temperature.

Cracks such as that shown in Fig. 9 verify that at the elevated temperature delamination of the specimens occurred to a greater extent than at room temperature. The edge replicas also indicate a greater amount of fiber/matrix debonding in the specimens tested at the elevated temperature, but the edge replicas should be considered with caution since they show only a small section of the specimens.

## CONCLUSIONS

In this study the impact strength of glass woven–epoxy composite materials at 20, –50 and +50 °C were determined. The test materials were glass woven/epoxy composite materials with two different specific masses of glass woven reinforcement and two different orientations.

It was shown that materials with reinforcement of higher specific mass (type D of woven glass → $\rho$ = 550 g $m^{-2}$) exhibited better impact properties. The impact strength obtained at the elevated temperature was greater than that at room temperature. On the other hands, the test results showed minimal values of impact strength at the low temperature, with characteristics brittle cracks.

In order to obtain a better picture of the quality of the fabricated composite materials, micromechanical analysis was performed. The SEM micrographs of the fracture surfaces confirmed the models and the mechanisms of impact crack known in the literature for similar structures and materials, such as fiber crack, matrix crack, fiber debonding and delamination.

## ИЗВОД

## УТИЦАЈ ВИСОКИХ И НИСКИХ ТЕМПЕРАТУРА НА УДАРНА СВОЈСТВА СТАКЛЕНО−ЕПОКСИДНИХ КОМПОЗИТА

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Циљ овог рада је да прикаже утицај високих и ниских температура на ударна својства стаклено-епоксидних композита. Ударна јачина $a_n$ приказана је за четири различите структуре стаклено−епоксидних композита на три различите температуре, тј. на собној $t$ = 20 °C, повишеној $t$ = +50 °C и ниској температури $t$ = –50 °C. Стандардно механичко тестирање обављено је за композите са масама ојачања 210 g $m^{-2}$ и 550 g $m^{-2}$ и орјентације 0°/90° и ±45°. Микромеханичка анализа оштећења изведена је са циљем да се одреде реални модели и механизми лома и утицаја температуре на ударна својства.

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