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# LOW-TEMPERATURE TENSION PROPERTIES OF GLASS-EPOXY COMPOSITE MATERIALS

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*The aim of this paper was to present the determination of tensile strength* $R_m$ *and modulus of elasticity* $E_1$ *of glass-epoxy composites at two different temperatures (at room temperature* $t=20°C$*, and at* $t=-50°C$*). Standard mechanical testing was carried out on glass woven-epoxy composite material with different structures (two specific weights of reinforcement, 210 g/m*$^2$ *and 550 g/m*$^2$*) and orientations (0°/90° and ±45°). Micromechanical analysis of failure was performed on a stereo microscope and SEM in order to determine real models and mechanisms of crack.*

KEYWORDS: Glass woven-epoxy composite material; tension test; low temperature test; micromechanical analysis

## INTRODUCTION

A very common way of composite materials fabrication is glass fiber as reinforcement and epoxy resin as matrix, which are characterised 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 composite and specific mechanism of crack, characteristic for this type of material. However, in reality, during exploatation, a lot of construction parts is subjected to low temperatures. In these cases, the toughness of material changes, which can cause crack under the stress is different from the one got in static testing.

The research itself surveyed the most important papers about composite materials based on glass-woven reinforcement and expoxy resin as matrix. Also, micro-mecha-

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nics analysis and understanding are necessary to predict the tensile strength of composites at room and low temperatures. For the past few decades many researchers have studied mechanical properties of the composite materials itself, as well as the changes of materials properties with the changes of temperature (1-5), which represents the basis of the research presented in this paper.

In the theoretical part, the greatest care is paid to the structural analysis of fiber reinforced laminate composite materials, the application and the possibilities for the further development, mechanical properties, as well to the methods for studying of mechanical properties of materials.

In experimental part of the work a method of fabrication of composite material is given. The mechanical static test of glass woven-epoxy composite materials with different specific weight and orientation of reinforcement were performed for finding out the influence of glass reinforcement on the mechanical properties of fabricated composite materials. In order to determine the properties of composites of different structures in extreme conditions, the tension tests were performed at +20ºC and –50ºC. By fractography analysis of crack surfaces a complete picture has been formed of the fabricated glass woven-epoxy composite materials, as well as of the models and mechanisms of damages and the crack initiation and propagation at different load conditions.

## EXPERIMENTAL

In order to classify a composite material it is necessary to perform first its specification. This means that we need to have the knowledge, not only about the properties of material but also about the conditions under which the final product was shaped. During the processing of composite material it is necessary to maintain some of the parametars (pressure and temperature) within the preset limits. Nevertheless, in the real process of fabrication a disturbance of the mentioned parameters may appear, which may cause many irregularities, like the appearance of bubbles, higher moisture, higher pressure in the mold, etc. All these irregularities lead to the deviation of real mechanical properties of the composite material from those predicted on the basis of the theoretical analysis and calculations. Hence, subsequent checking of these characteristics by some chosen standard methods of examination are needed in order to get real characteristics for the classification and later for the statistical processing. These methods are standard and used worldwide. Practically, today it is not possible to imagine any firm which produces composite materials without its own laboratory with all kinds of equipment for various examinations.

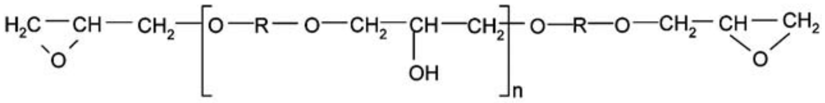
### *Material*

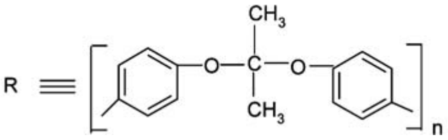
Materials have been fabricated in the lab conditions at the Faculty of Technology and Metallurgy in Belgrade. The basic structural components were: glass woven in two orientations (0º/90º and ±45º) as a reinforcement and epoxy resin as matrix. Materials have been fabricated manually, the reinforcement was done at room temperature, and after that the specimens for mechanical tests were cut.

**Reinforcement properties**. Glass woven on the basis of silicate glass which contains alkali up to 1% were used for reinforcement. They were also made of “E”-glass fibers which have good mechanical, hydro-thermal and electrical properties. Two specific masses of glass woven reinforcement were used: C=210 g/m² and D=550 g/m². Glass woven was made by classical procedures of spinning on different kinds of looms (Fig. 1).

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

**Epoxy resin** used as matrix material, was a polycondensation product of 2,2-bis-(4-hydroxyphenyl) propane (bisphenol A) and epichlorhydrin:





while 3-aminomethanol-3,5,5-trimethanolcyclohexylamine, a modified cycloaliphate amine of the same producer, was used for fixing.

*Fabrication process and design of the study*

The materials were fabricated manually in the mold (5, 6) consisting of two metal boards upper and bottom whose dimensions were 292 x 230 x 13 mm, tighten with four screws to obtain the necessary pressure.

The static tension mechanical tests have been performed on the cut specimens of fabricated composite materials. All combinations of the specimens with the standard dimensions (250 x 25 x 2.5 mm) are shown in Table 1. 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 delaminations could be readily discerned.

**Table 1.** The structure of fabricated composite materials

| Sample | Number of reinforcement layers | Specific mass of reinforcement (g/m²) | Orientation of reinforcement | Mass fraction of reinforcement (%) |
|---|---|---|---|---|
| T-C-1 | 5 | 210 | 0°/90° | 33.3 |
| T-D-1 | 4 | 550 | | 55.2 |
| T-C-2 | 5 | 210 | ±45° | 37.2 |
| T-D-2 | 4 | 550 | | 56.3 |

## RESULTS AND DISCUSSION

### *Tension test at t= 20°C*

The tension properties were studied first at the temperature $t$=+20°C. The aim of was to determine the influence of glass reinforcement on tension properties (tension strength and modulus of elasticity) and the basic properties of tested composite materials, as well as to compare the obtained values of tension strentgh with those measured at low temperature.

In accordance with ASTM D3039-76 (7), the tension test was carried out on a MTS hydraulic axial loading test machine with the usage of hydraulic jaws. During the test the strains in the longitudinal direction ($\varepsilon_l$) were registered continually.
The ultimate tension strength was calculated from thequation:

$$R_{m1,z} = \frac{F_{\max}}{b \cdot d} \qquad [1]$$

where:

$R_{m1,z}$ the ultimate tension strength, MPa
$F_{max}$ the maximal force, N
$b$ the width of the specimen, mm
$d$ the thickness of the specimen, mm
Modulus of elasticity was calculated from the equation:

$$\mathrm{E}_{1,z} = \frac{\Delta\sigma}{\Delta\varepsilon} = \frac{\Delta \mathrm{F}}{\Delta\varepsilon_1} \cdot \frac{1}{\mathrm{b} \cdot \mathrm{d}} \qquad [2]$$

in which the relation $\Delta F \Delta\varepsilon_l$ was calculated by linear regression method using the linear part of the registered diagram force-elongation. The examples of force-elongation diagram ($F$-$\Delta l$) obtained directly from the test machine are shown in Fig. 2. According to them the maximal force and modulus of elasticity were determined. The results of calculated tension strength and modulus of elasticity of all studied samples are presented graphically in Fig. 3 and Fig. 4.

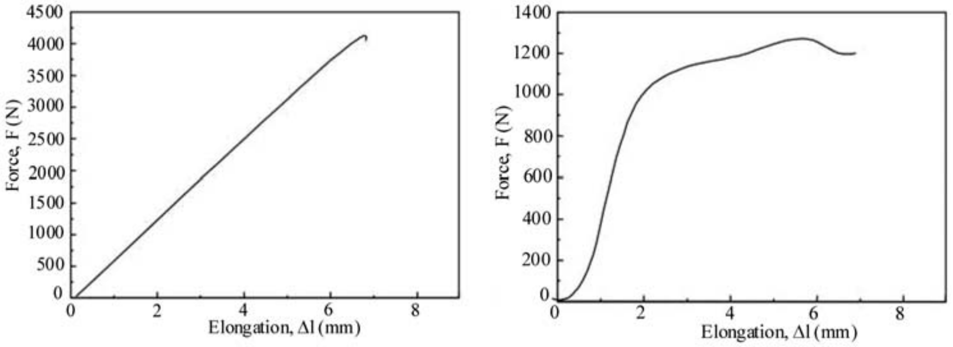


**Fig. 2.** Force–elongation diagram; left for T-C-1-2 specimen; right for T-C-2-1 specimen

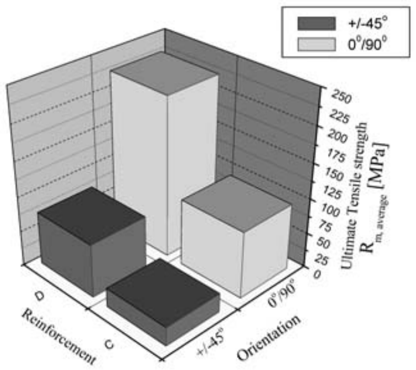


**Fig. 3.** Ultimate tensile strength; average values

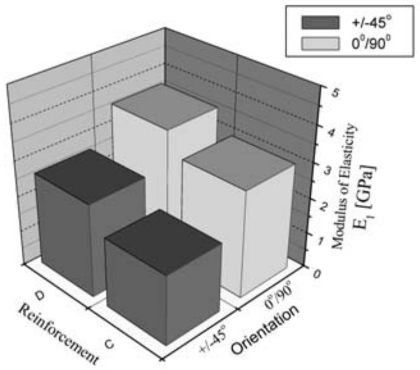


**Fig. 4.** Modulus of elasticity; average values

*Tension test at t=–50°C*

Studies of tension at –50°C were performed to see in what way the change of temperature influences mechanical properties of materials (5, 6). Three test specimens were prepared of each fabricated structure. The low temperature was obtained by using liquid nitrogen pumped into the cell. The time in which the specimen experienced the cryogenic temperature was varied from test to test because of the limitation of the pump controller. However, once the temperature remained constant, the test was conducted under that temperature until failure. This cell also contained the dummy thermal strain gage.

The result of the study was presented in a force-elongation diagram (Fig. 5), from which the value of maximal force was read directly. The values of ultimate tension strength and modulus of elasticity were calculated in the same way as described in the procedure of tension test at room temperature (Equations [1] and [2]). The tension test results at –50°C are presented in the form of diagrams in Figs. 6 and 7.

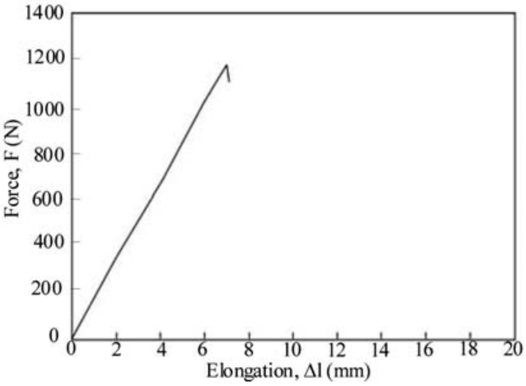


**Fig. 5.** Force–elongation diagram; specimen T-D-1-4 (*t*=-50°C)

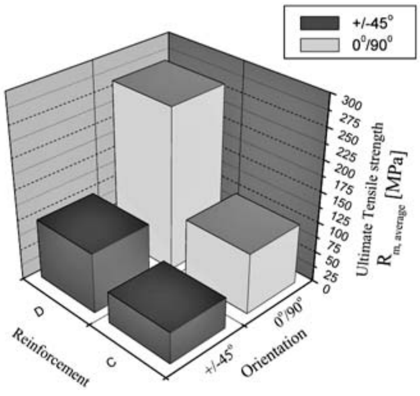


**Fig. 6.** Ultimate tensile strength; average values at *t*=–50°C

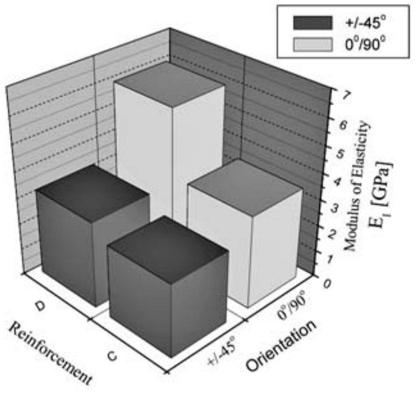


**Fig. 7.** Modulus of elasticity; average values at *t*=–50°C

To compare the tension test results obtained at +20°C and –50°C, average calculated values of ultimate tension strength and modulus of elasticity obtained at the two experimental temperatures were presented in Table 2, and in Figs. 8 and 9.

Generally, based on the experimental results it can be concluded that mechanical properties increase with increase of specific weight of glass woven reinforcement in the composite materials (type “D”). It is the same for both types of the reinforcement orientation, whereby the results are better in the case of the orientation 0°/90° (specimens C-1 and D-1) than in the case of orientation ±45° (specimens C-2 and D-2). Also, these series of specimens have greater mass part of reinforcement (Table 1) which confirms the assumption that greater mass part of reinforcement gives better tension strength of composite materials.

The presented results can be explained in terms of micromechanisms of crack. The composite materials unlike the conventional construction materials have the specific models of damage and crack mechanisms. The detailed knowledge of these mechanisms certainly allows one to get a complete picture of the studied material. Since this work repre-

**Table 2.** Ultimate tensile strength and modulus of elasticity for all test temperatures

| **Sample** | **Test temperature** | | | |
|---|---|---|---|---|
| | +20°C | | -50°C | |
| | Average value | | | |
| | $R_{m,\ average}$ | $E_{1,\ average}$ | $R_{m,average}$ | $E_{1,\ average}$ |
| T-C-1 | 95.0 | 3.01 | 99.4 | 3.51 |
| T-C-2 | 26.7 | 1.95 | 51.2 | 2.76 |
| T-D-1 | 227.3 | 3.62 | 266.1 | 6.01 |
| T-D-2 | 75.9 | 2.62 | 98.3 | 3.26 |

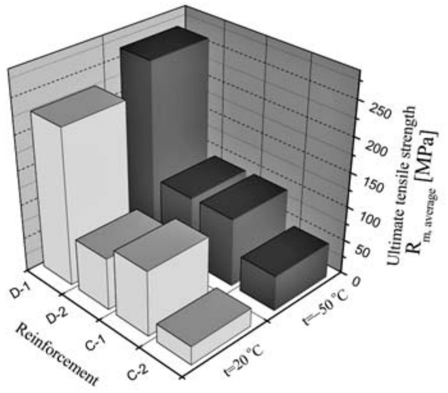


**Fig. 8.** Ultimate tensile strength;
average values for both test temperatures

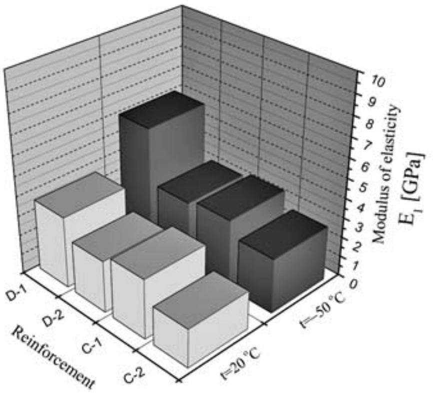


**Fig. 9.** Modulus of elasticity;
average values for both test temperatures

sents a characterization of a new fabricated material, a micromechanic analysis has been done in order to check all the mechanisms of crack known from the literature. The analysis was done above all according to crack of reinforcement layer because it was known that reinforcement was the structural element that gave the material strength and rigidity, on one side, and on the other, due to its specific qualities, brought to different models of crack initiation and its further propagation. What is common, is that because of interwining of fibers in woven and different distribution of stress along the longitudinal direction of the fiber, the fibers are not loaded in the same way. The result of that is also a different time of crack in fibers, during the study, some fibers have broken at a lower and some at higher load. The fibers that broken in a shorter time caused a disorder in the crack zone, i.e. the local stresses have appeared by the broken fiber. A further mechanism of crack depends on the type of study, which will be explained in more detail.

For all tested materials with 0°/90° reinforcement, the major initial damage mechanisms appeared in the form of transverse microcracking and delaminations at the fiber undulation areas. These delamination regions are known as “meta delaminations” (Fig. 10).

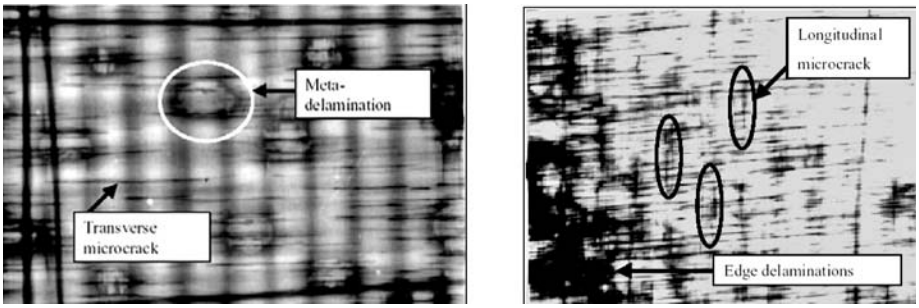


**Fig. 10.** Radiograph of specimens with longitudinal and transversal microcracks subjected to tesion load at t=20°C

This initial damage was followed up by the growth of transverse cracks, which were extended through one transverse tow at maximum length, and the introduction of longitudinal microcracking, which did not grow significantly around the fiber undulation areas. Finally, a fiber-matrix debonding and edge and interply delaminations typical of laminates were preceded by catastrophic fiber fracture. This behavior is not unusual – it is well documented that the transverse and longitudinal cracks intersect with each other, fibers, and adjacent plies lead to delamination. The delamination inhibits the ability of the composite to distribute load, so the fibers fail as the stress increases to a critical level.

As far as the samples with reinforcement of orientation ±45° are concerned (samples 2), the character of the curve force-elongation $F$-$\Delta l$ (Fig. 2) is different from the curve for the specimens with the orientation 0°/90° (samples 1) in which the non-linear part is even more pronounced in all studied cases. It can be concluded that the character of crack of test specimensis is not similar. In this case, the shear stress components are dominant, which leads to the appearance of delamination and breaking the fiber-matrix interface (debonding), after which appears a macroscopically visible crack. It is characteristic to notice that there was cracking of fibers in one direction (conditionally under the angle of +45°) and delamination in the other direction (conditionally -45°), Fig. 11.

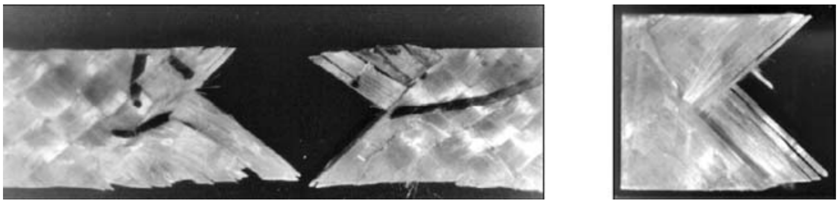


**Fig. 11.** Cracked surface; test specimens T-C-2

For these samples the burst of fibers is characteristic also in the plaits of glass woven as a consequence of the low mass part of resin in matrix. The fibers did not crack in a definitely orientied layer but randomly in all directions. However, the crack of fibers at some angle (±45°) can be absorbed, which confirms the existence of dominant shear stresses.

In Fig. 12 is shown the specific appearance of the crack surface of test specimens for both studied temperatures.

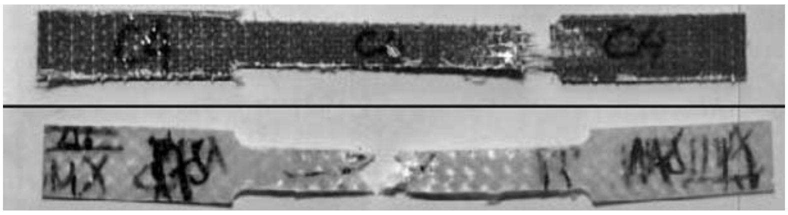


**Fig. 12.** A view of cracked test specimens studied
t=–50°C (T-C-1); bottom - at t=+20°C (T-D-2)

Two types of deformations are characteristic for the materials with polymer matrix: viscous flow (a permanent deformation that remains after the effect of tension which caused it) and all elastic deformations (which are lost after the effect of tension). The relation between these material deformations directly influences the final properties of polymer composite material, and mostly depends on the temperature. With the decrease of temperature the crack of test specimens is brittle (Fig. 13). With the increase of viscidity the elastic component of deformation decreases, the slope of the curve is decreased too, and the values of ultimate tension strength ($R_m$) and modulus of elasticity ($E_1$) are the lowest at +20°C and highest at –50 °C.

The appearance of viscidity is directly connected with epoxy resin as matrix which, depending on temperature, behaves as an elastic solid body, on one side and a viscous liquid, on the other.

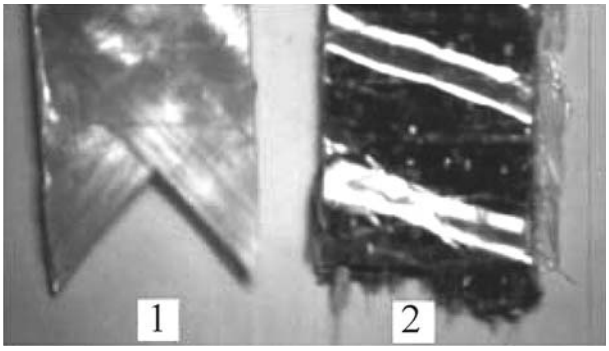


**Fig. 13.** The view of cracking surface of testing specimens (brittle crack)
subjected to tensile load at $t$=–50°C ((1) T-D-2 and (2) T-C-1)

The cracks such as shown in Fig. 14, verify that the specimens at low temperature are delaminated more than the specimens subjected to the tension at room temperature.

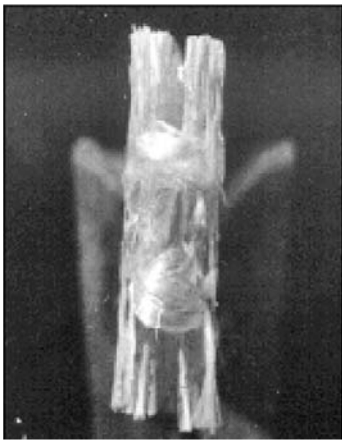


**Fig. 14.** Delamination; test specimen T-C-1

The decrease of tension strength with increase of temperature can be further explained by the deformations that appeared in the material due to the difference in thermal expansion coefficients of reinforcement (glass) and matrix (resin). These deformations cause the appearance of cracks mostly on the fiber-matrix interfaces, which are the areas of high stress condition rousing debonding and cracks in the fibers. Also, with the increase of temperature there appear uneven stress distribution over the cross area of test samples: outer layers are more deformed than the inner one, the delamination appears, and the final break is thus made faster.

By analyzing the effect of temperature it can be concluded that the dominant influence on the tension properties has the reinforcement, which means that the samples with higher specific weight and mass fraction of reinforcement have better properties.

## CONCLUSION

The advances in composite processing and fiber design have resulted in new applications for lightweight and high strength composite material systems. Recently, the fiber-reinforced systems have been found to be uniquely suited for the smart material applications. General overviews of the current state of manufacturing and application of composite systems are available. As limits are reached in the specific strength and moduli of fibers and matrix materials, there is a need to understand how the fabric architectures affect the behavior of composites, i. e. optimal arrangements can be designed and utilized for specific applications. Many important advantages are accomplished for the construction itself (in the area of technology of development and exploitation itself) compared to the application of some other constructional materials. It especially favors their application in the machine constructions of grater responsibilities, such as pressure vessels, small turbine or pump shovels, parts of planes and other means of transport, but also constructions that do not demand a great reliability, such as small boats, kiosks, water tanks, different sport stuff, etc. For the obvious wide spectrum of usage, the composite materials have to thank first of all to their structure and specific mechanisms of static failure that give them the good mechanical properties, followed by a relatively small weight of parts, which was the subject of study in this paper. The performed studies represented a very complex problem that demanded complete theoretical understanding, development of the fabrication process and experimental studies. By combining all the aspects a real picture of studied composite materials can be achieved.

The glass woven-epoxy composite materials of different structures (mass fraction and orientation of reinforcement), fabricated in laboratory conditions were studied. In the experimental part of the work static mechanical properties of fabricated glass woven/epoxy composite material subjected to tension load based on standard methods but adjusted to the studied materials and the conditions of work were determined. Besides, since the epoxy resin is a highly elastic material that shows important changes with the change of temperature that directly influences properties of composites, the tension strength of composites at –50ºC was determined to define the behavior of these materials under real conditions.

It can be concluded that the materials with reinforcement of higher specific weight (type of glass woven D → $\rho$=550 g/m$^2$) have shown better properties. Also, it is confirmed that materials with a higher mass ratio of reinforcement have better mechanical properties. The tension strength at low temperatures are greater than at room temperature.

The static mechanic properties of test specimens from series C are generally much lower than those of series D, which could be explained by lower specific weight and smaller mass ratio of reinforcement. Also, it has to be said that their values for series C are much lower than the expected values for similar structures of materials known in the literature, so they are not recommended for usage in some more responsible constructions.

In order to get a more complete picture of the quality of fabricated composite materials, a micromechanical analysis was performed. On cracked surfaces of test specimens all models known in the literature were observed, and the mechanisms of crack for the similar structures and materials. However, the most obvious mechanisms (in some cases visible even with the naked eye) is maybe the lack of matrix that practically represents the existence of cracks in the material. It is known that cracks in composite materials provoke a big stress concentration and greatest danger to their integrity, and that they may be one of the most important reasons for lower values of mechanical properties compared to the expected and familiar ones from the literature. This certainly means that this method of fabrication should be used with a greater care in order to obtain the material better properties, without air bubbles. First of all, the pressure in mold should be raised, weight ratio of fixers should be increased, and the final product should be fixed at elevated temperature, which certainly can be a subject of further work.

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# ОДРЕЂИВАЊЕ ЗАТЕЗНИХ СВОЈСТАВА СТАКЛО-ЕПОКСИ КОМПОЗИТНИХ МАТЕРИЈАЛА НА НИСКИМ ТЕМПЕРАТУРАМА

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Предмет и циљ испитивања приказаних у овом раду представља одређивање затезне чврстоће ($R_m$) и модула еластичности ($E_t$) стакло-епокси композитних материјала испитивањем на затезање на две различите температуре ($t$=20°C и $t$=–50°C). Испитивања су изведена према стандарду на стакло-епокси композитном материјалу различитих структура (две специфичне тежине ојачања 210 g/m$^2$ и 550 g/m$^2$) и оријентација ојачања (0°/90° и ±45°). Допринос представља и микромеханичка анализа на преломним површинама изведена на стерео и електронском микроскопу којом се дошло до стварних модела настанка и развоја оштећења при изведеним испитивањима.

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