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MICROSTRUCTURAL CHARACTERIZATION OF GLASS-EPOXY COMPOSITES SUBJECTED TO TENSILE TESTING

Jelena M. Petrović[a], Dragoljub. Ž. Bekrić[b], Ivica. T. Vujičić[a], Ivana D. Dimić[c] and Slaviša S. Putić[a]*

[a] University of Belgrade, Faculty of Technology and Metallurgy, Karnegijeva 4, 11120 Belgrade, Serbia
[b] University of Belgrade, Faculty of Mechanical Engineering, Kraljice Marije 16, 11000 Belgrade, Serbia
[c] University of Belgrade, Faculty of Technology and Metallurgy, Innovation Centre, Karnegijeva 4, 11120 Belgrade, Serbia

The main objective of the research presented in this paper was to carry out a statistical-mechanical analysis concerning the tensile test of glass-epoxy composite materials in order to calculate their relevant tension properties and micromechanical structure destruction process. The analysis was done at room temperature, and its results were derived based on the structure of the glass woven 280 g/m², "twill texture" with the width value of 100 cm, type - Interglass 92125, and epoxy resin type MGS L 135. Samples were shaped by hands with 35% of fabric volume part. The consumed portion of resin was 220 g/m², the thickness of the laminate was 0.308 mm, and the mass of the laminate was 500 g/m². There were a total of eight layers built in the panel. The micromechanical analysis was derived from the crack surfaces data collected on a scanning electronic microscope, and it showed the mechanisms of damage, and development of cracks until the occurrence of the final break under the tension load.

KEY WORDS: glass-epoxy composite materials, tension mechanical properties, micromechanical analysis

INTRODUCTION

The main objective of this study was the characterization of structural damages of the impellers made of a glass-epoxy composite material, used in the wind energy systems, and which usually occur during their operation. In the analysis, it was necessary to meet certain criteria for the strength and impeller structure of wind energy systems for the preplanned working settings and extreme stress conditions defined by the standards. Under the ideal conditions, the impellers of the wind energy systems should meet all of the operating criteria in their surroundings. The analysis of the behavior of glass-epoxy composite materials that are used for impeller of wind energy systems, through the definition of reliable and high-grade parameters, represents a fundamental scientific contribution.

Contrary to classic construction materials, the mechanical properties of composite materials (as heterogeneous materials) are formed during the process of their production.

*Corresponding author Jelena Petrović, University of Belgrade, Department of Technology and Metallurgy, Karnegijeva 4, 11000 Belgrade, Serbia, e-mail: jecca_zeleznik@yahoo.com

Since the properties of composite materials are not stable but dependent on the production process, the mechanical tests become even more significant. The need for a proper knowledge of the material properties as well as of the conditions under which the final product is made is clearly understandable. During the process of fabrication it is important to keep some of the parameters (for example: time, temperature, pressure) lower than the predetermined limits. However, during the fabrication process some changes in the conditions that were established in advance could occur, and that can cause appearance of many irregularities, such as formation of voids, humidity, increased pressure in the mould, etc. All those irregularities may bring about the deviations of real mechanical properties from those obtained theoretically and by calculations. Additional check of those properties is required, and this is done using standard static and dynamic methods for classifications, which are later used for the statistical analysis of the results (1-5). In the last few decades, many researchers have considered these points. In the work of Torabizadeh and Fereidoon (6), a model was developed to perform the progressive failure analysis of quasi-isotropic composite plates at low temperatures. Measurements of the temperature changes during the uniaxial tensile test in polymer composites were reported by Lindhagen and Berglund (7). The results of thermo-mechanical characterization of glass/epoxy composite specimens using Infrared Thermography technique was presented by Muneer et al. (8). The specimens used for the study were fabricated in-house with three different lay-up sequences and were tested on a servo-hydraulic machine under uniaxial loading. The results obtained in the parallel testing of the epoxy-glass composite specimens, using conventional mechanical methods and thermography, were presented by Kutin et al. (9). The tensile properties of composites were followed by thermography. The mechanical properties of G10 glass-epoxy composites were also determined in compression and tension tests in the work by Ravi-Chandar and Satapathy (10).

Tension tests are very widely applied. They provide the information about the values of the modulus of elasticity, the tension strength, and Poisson's coefficient. During the examination flat specimens that were cut from the shaped panel are used, and the magnitude of strain as well as the longitudinal and transversal strains are continuously recorded (11, 12). Hydraulic loading test machines with the use of hydraulic jaws are applied as the examination equipment. The hydraulic jaws can be separated by different speeds (1-500 mm/min) and that depends on the type and the structure of the examined material. The test machine has to continuously register the strain on the test tube that is under tension, until its final crack occurred. The device has to possess the appropriate instrument to be able to show the elongation of the specimen as a function of the load at any given moment of examination.

The objectives of this research could be grouped as general and specific. The general objectives are referred to the definition of the conditions under which the damage of the impeller occurred while the impeller was in the domain of critical stress values. Specific objectives referred to the examinations that included both, macro and micro analysis of the glass-epoxy composite material complex structure, the impeller being made of this material. The analysis was done before and after the exposure of the impeller to the stress conditions until the breakage occurred. The characterization of the damages could contribute to the quality estimate of the operating lifetime of the impellers, primarily from the practical use of the materials for their manufacturing.

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EXPERIMENTAL

Material

The structure of the analyzed material was the glass fabric 280 g/m², "twill texture" with the width value of 100 cm, type - Interglass 92125 produced by P-D Interglass Technologies GmbH and epoxy resin type MGS L 135 produced by Martin G. Scheufler Kunstharprodukte GmbH. Samples were hand-made with 35% of fabric volume part. It should be noted that this was a new type of material and that the study of the glass-epoxy composite material with 35% of fabric volume part is extremely important because of the increased tendency for the application of lighter composite materials with good mechanical properties. The portion of resin that was consumed was 220 g/m², the thickness of the laminate was 0.308 mm, and its mass was 500 g/m^2. There were eight layers built in the panel. The properties and chemical composition of "E"-glass fibers used to form the textures are shown in Table 1 and Table 2, respectively. The physical and mechanical properties of the resin are shown in Tables 3 and Table 4.

Table 1. Properties of the "E"-glass fibers

Property	Unit	Value
Diameter	μm	8-16
Specific weight	g/m^2	450 ± 5%
Tensile strength, R_m	[Mpa]	2500-3450
Modulus of elasticity, E_1	[Gpa]	72.5
Deformation, ε_l	[%]	3.3-3.5
Thermal elongation	[10^{-6} K^{-1}]	5
Thermal conductivity	[W/m·K]	1
Dielectric constant	[ξ]	6.7
Electrical resistivity	[Ωcm]	10^{14}

Table 2. Chemical composition of the "E"-glass fibers

Structural component	Fraction (wt%)
SiO_2	52 – 56
Al_2O_3	12 – 16
B_2O_3	5 – 10
Na_2O and K_2O	0 – 2
MgO	0 – 5
CaO	16 – 25
TiO_2	0 – 1.5
Fe_2O_3	0 – 0.8
Fe	0 – 1

Table 3. Physical properties of the uncured epoxy resin.

Property	Unit	Value
Appearance	-	Yellow viscous liquid
Epoxy number	n/100 g	0.53-0.59
Epoxy equivalent		170-189
Density	g/cm^3	1.14-1.18
Viscosity at 25°C	mPa·s	2300-2900
Color (Gardner color scale)		Less than 3
Non-volatile components content	% min	99.5
Organic chlorine content	% max	0.17

Table 4. Mechanical properties of the cured epoxy resin

Property	Unit	Value
Tensile strength, R_m	MPa	68-80
Deformation, ε	%	5-7
Bending strength, R_f	MPa	110-130
Modulus of elasticity, E_l	MPa	2.9-3.2
Pressure strength	MPa	110-130
Impact energy /Charpy/, a_n	KJ/m^2	30-50

The composite material was made with the aid of a handcrafted mold. The mold consisted of two metal plates screwed with screw bolts to ensure an appropriate pressure force. Once placed in a mold, the material was left for 24 hours at room temperature to cure and harden. After 24 hours, the mold was opened and the sample was hardened. The sample of the composite that was without any significant defects was taken out of the mold and left to cure completely in air for 7 days at room temperature. The standard specimens for mechanical testing were cut out from the prepared composite.

Tensile testing

The three specimens (T-GE) were prepared for mechanical testing. The specimens dimensions were 250x25x5 mm (the middle measurment part with the length l_l=100 mm and width b=15 mm). Before testing, the specimens thickness and width were precisely measured (±1%). Further machine processing of the specimens was performed with a diamond tool tip moving at a speed that reduced generation of heat in the specimens. Cutting was carried with a notched cutter, the thickness of 1 m/min on an ALG-100 machine.

The testing was carried out on the hydraulic loading test machine SCHENCK TREBEL RM 100 with the use of hydraulic jaws according to the standard test method ASTM D3039 (13). The incorporated load was registered with the aid of a measuring cell (capacity of 100 kN). The data were collected using an UPM 60 measuring device. During

the analysis, the increasing values of the force (ΔF) and elongation (Δl) were continuously recorded, and, based on this information, the values of the stresses (σ) and strains (ε_l) in the longitudinal direction were calculated. The elongation was measured by using a dual extensimeter Hottinger DD1. There were two parallel extensimeters, to measure the elongation on both sides of the specimen, and the parallel connection to extensimeter facilitated the averaging of the measured values. The measuring range of this extensimeter was ± 2.50 mm, and its work was based on the principle of tape measurement with the accuracy of 0.05.

Micromechanical analysis was done on a scanning electronic microscope TESCAN Mira3 XMU at 5 kV. Prior to the analysis, the specimens were coated with a layer of the alloy of Au-Pd type.

The cross-sectional dimension and the values of tensile strength and modulus of elasticity of the specimens were calculated by using the equations [1-3] (13).

The cross-sections of the specimens were calculated by the following equation:

$$A_0 = b \cdot d \qquad [1]$$

The tensile strength was calculated by equation [2], as follows:

$$R_{m,1} = \frac{P_{\max}}{b \cdot d} \qquad [2]$$

where:

$R_{m,1}$ - tensile strength in the longitudinal direction, Mpa

P_{max} - maximal force at the break, N

A_0 - cross-section of the specimen, mm^2

B - specimen width, mm

D - specimen thickness, mm

The modulus of elasticity (E_1) was calculated from equation [3], where the ratio $\Delta P/\Delta\varepsilon_l$ was determined by linear regression method from the straight part of the registered stress curve as a function of strain:

$$E_1 = \frac{\Delta\sigma}{\Delta\varepsilon} = \frac{\Delta P}{\Delta\varepsilon_1} \cdot \frac{1}{b \cdot d} \qquad [3]$$

RESULTS AND DISSCUSION

The tensile test in the longitudinal direction was performed on three specimens, and the values of the tensile strength and modulus of elasticity were obtained. It should be noted that the test was successful as for all tested specimens the fracture occurred in the middle part of the specimen (the measurement part). The calculated values of the tensile strength in the longitudinal direction and the corresponding modulus of elasticity are given in Table 5. The stress vs. strain diagrams ($\sigma - \varepsilon$), obtained directly from the test machine for all tested specimens, are shown in Fig. 1.

Table 5. Results of the tensile tests

Specimen	Specimen width b, mm	Specimen thickness d, mm	Cross section A_0, mm^2	Max force at break P_{max}, kN	Tensile strength R_{m1}, MPa	Modulus of elasticity E_1, GPa
T-GE-1	14.9	5.03	74.95	31.6	422	89.7
T-GE-2	15.1	4.95	74.75	32.1	429	90.1
T-GE-3	14.7	5.02	73.80	30.6	414	86.7

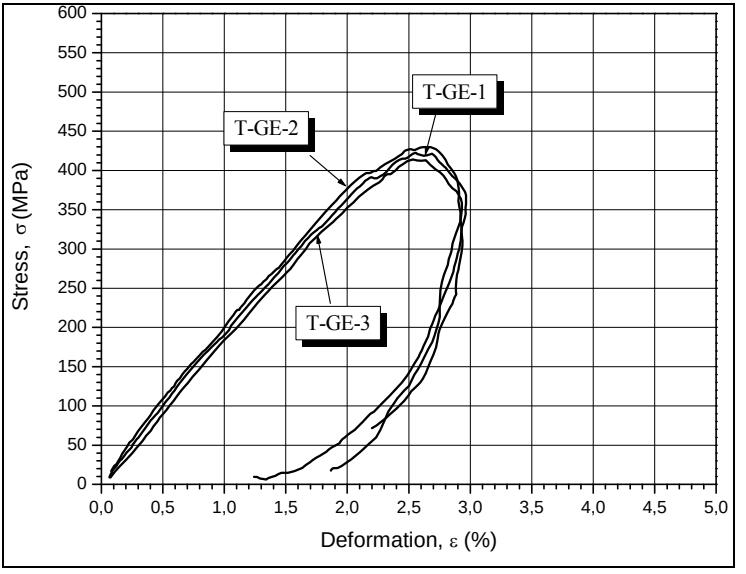

Figure 1. Comparison of the stress-strain (σ–ε) curves

It can be concluded that the values of the maximal forces P_{max} obtained were relatively equal, although there was a slightly lower value for the sample T-GE-3 (30,6 kN), which can be explained by the smaller dimension of its cross-section measuring part. The breakage of the sample T-GE-1 is shown in Fig. 2.

Figure 2. Cracked sample T-GE-1

Based on the results of the three analyzed samples, we calculated the average value of the tension strength, which was 421.7 MPa and the average value of the modulus of elasticity, which was 88.73 GPa. Therefore, it can be concluded that there was a relatively small deviation pf the measured values from the average calculated values for both the tension strength and modulus of elasticity.

A slightly higher deviation was observed for the values of the modulus of elasticity, which can be explained by the fact that is harder to precisely determine elastic module because of the unstable linear part in the diagram and the relatively small beginning of the screening of the tension-strain (σ–ε) curve. As far as the tension strength is concerned, it is well known that because of the entwisted fibers and different distribution of the stress through the center line of the fiber, all fibers do not experience the same load. The result of this is the different times of the breakage of the fibers – some of them break under the lower and some of them under the higher values of the load. The fibers that broke earlier created a disbalance in the breakage zone, therefore shearing and local shearing stresses occurred on the sides of the broken fiber. Such situations did not affect the same way all the samples, and this was one of the reasons why there was a difference in the values of the maximal forces under which the crack occurred.

During the analysis of the sample in the longitudinal direction, not all of the fibers experienced the same amount of demand because of the specific texture of the fibers, the way fibers were entwisted and different distribution of tension through the center line of the fiber. As a result, the times of the fiber break were different (Fig. 3).

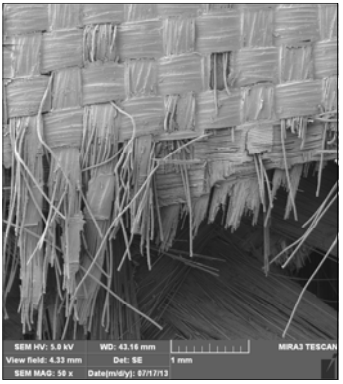

Figure 3. Different times of the fiber cracking

It was possible to observe a smaller dispersion of the results of the tension strenght, and even more of the modulus of elasticity that were obtained in the analysis. During the process of shearing, the increased demand showed "sliding" of the whole coiled layers of the fibers through the sample thickness, leading to the disbalanced stress conditions and strains in the layers. This is schematically shown in Fig. 4. At the beginning, the fibers were undestructed, but the microcracks that already existed at the host were growing progressively and were creating a macrocrack in them. In that way the host was becoming destructed and the fibers were "sliding" through those macrocracks on the thickness between the layers. The fibers were still undestructed, but their original places of coil were changed significantly and that created a noticable holes on the thickness. Because of this, the collections of fibers in these zones were forced to hold additional demand which resulted into extra separation of the fibers from the host, with formation of new cracks and first breakages of the fibers (14).

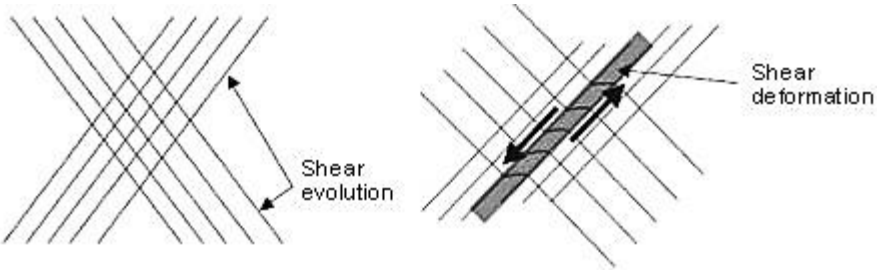

Figure 4. Schematic of the shear evolution;
Left: Normal structure; Right: Shear deformation

The examples of the occurrence of the shearing of the layers as a whole are shown in Fig. 5 and Fig. 6. It is obvious that out of the whole layer, there were only a few fibers left, whereas all of the other fibers slided to the adjoining layer under the demand. In this way, the layer that remained experienced an additional demand.

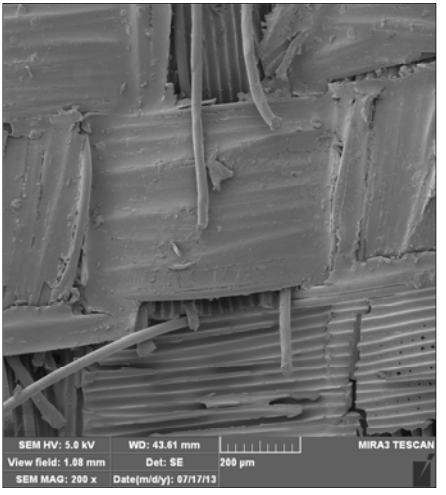

Figure 5. Layer shear evolution

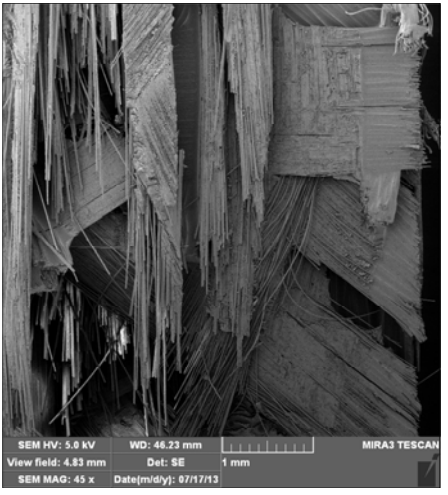

Figure 6. Fiber shear evolution

The shearing components of tension had very important role, and this is evident from the calculated stress-strain (σ–ε) graphs, which were not linear, in contrast to the graphs of the majority of composites. The nonlinearity was usually occurring at roughly 30÷35% of the maximal stress values. The increased value of stress led to the breakage of the fiber-resin bonds and crack that occurred after the breakage of fiber was growing through the adjoining fibers and caused a macrocrack, Fig. 7 and Fig. 8. As a result, there appeared the fiber crack and local differentiation, but the composite kept carrying external load. With a continuous increase of stress, the local damage kept spreading and the breakage of the whole group of fibers occurred, which led to a progressive differentiation and final crack. The final crack was followed by a strong acoustic effect, which was a consequence of the breakage of a huge amount of fibers at the same time.

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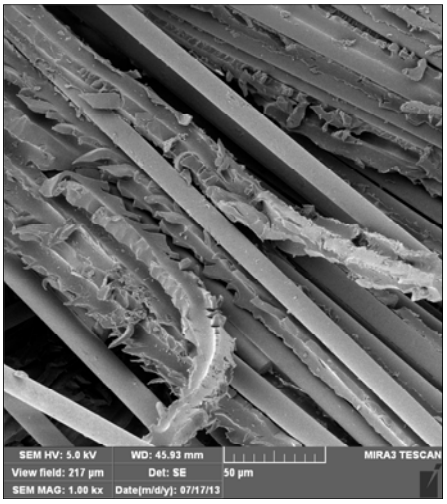

Figure 7. Macrocracks that have occurred during the breakage of the fiber-resin bond

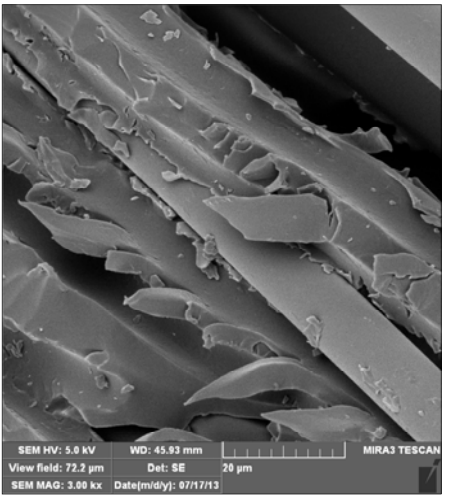

Figure 8. One macrocrack between the layers

It is obvious that delamination was the outcome of the process of destruction of the samples. The delaminated surface had the appearance that fitted the surface of interlaminar shearing, which can be seen in Fig. 9 and Fig. 10.

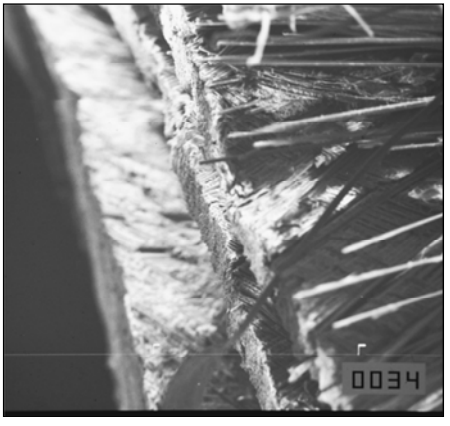

Figure 9. Delamination of the samples

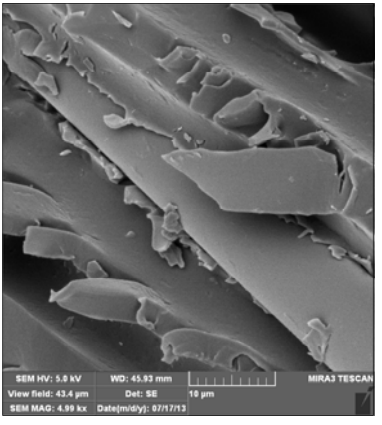

Figure 10. Sample delamination during the tension load under the higher enlargement

The appearance of the beginning of the cracks was very distinctive because of the absence of resin (Fig. 11 and Fig. 12), and a consequence of that was that the fibers breakage did not occur at the same time. The fibers that were not covered broke first, and

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after that breakage occurred in the others, as well as in those fibers that were not overwhelmed with the tension force (Fig. 13 and Fig. 14).

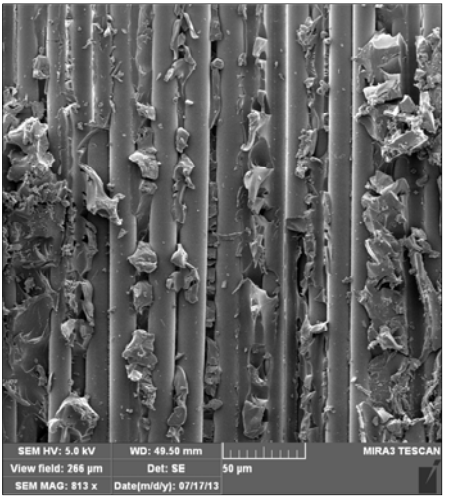

Figure 11. Deficiency of the resin

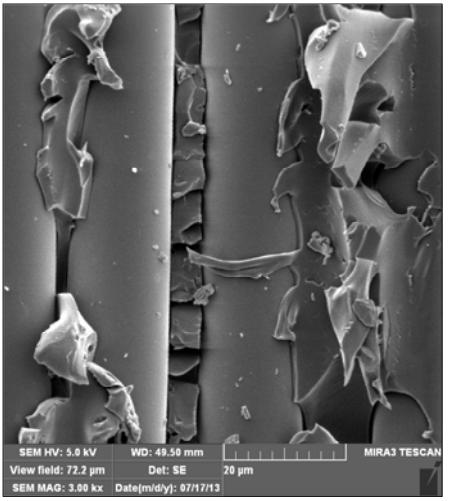

Figure 12. Deficiency of the resin under higher enlargement

Figure 13. Different times of fiber cracking in dependence of the resin deficiency

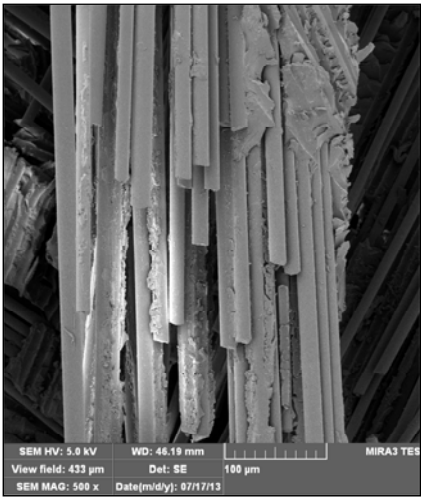

Figure 14. Different times of fiber cracking in dependence of the resin deficiency under higher enlargement

CONCLUSION

This paper presented the results of a mechanical analysis of the glass-epoxy composite materials in the tension test in the longitudinal direction. The values of the tensile strength and of the modulus of elasticity were calculated. The micromechanical analysis showed all the mechanisms that usually occur under the analysis. As a consequence of the specificity of the texture and orientation of fibers, as well as of the different distribution of tensions at the center line of the fibers, all of the fibers are not demanded the same way. A result of this is the different times of the breakage of the fibers, which means that some of the fibers broke under a lower demand and some under the pressure of higher demands. It can be concluded that the obtained results are in the range of the known literature data for this type of analysis and materials with a similar structure. A special contribution is that the micromechanical analysis led to valuable data about the time durability and mechanisms for breakage appearance of the material during the tension load.

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МИКРОМЕХАНИЧКА КАРАКТЕРИЗАЦИЈА СТАКЛО-ЕПОКСИ КОМПОЗИТА ИЗЛОЖЕНОГ ЗАТЕЗНОМ ОПТЕРЕЋЕЊУ

Јелена М. Петровић[а], Драгољуб Ж. Бекрић[б], Ивица Т. Вујичић[а], Ивана Д. Димић[в] и Славиша С. Путић[а]

[а]Универзитет у Београду, Технолошко-металуршки факултет, Карнегијева 4, 11000 Београд, Србија
[б]Универзитет у Београду, Машински факултет, Краљице Марије 16, 11000 Београд, Србија
[в]Универзитет у Београду, Технолошко-металуршког факултет, Иновациони центар, Карнегијева 4, 11120 Београд, Србија

Предмет и циљ истраживања приказаних у овом раду су статичко механичка испитивања на затезање стакло-епокси композитног материјала ради добијања његових релевантних затезних својстава и микромеханичке анализе оштећења структуре. Испитивања су изведена на собној температури. Анализирани су композити на основу епоксидне смоле типа МГС ЛР 135 и стакленог платна 280 g/m², "twill" ширине 100 cm, тип - Интерглас 92125. Узорци су обликовани ручном методом са 35% запреминског удела влакана. Утрошак смоле је био 220 g/m², дебљина ламината 0,308 mm а маса ламината 500 g/m^2. Укупно је уграђено 8 платана у панелу. Анализом преломних површина на скенинг електронском микроскопу изведена је микромеханичка анализа којом се дошло до сазнања о механизмима оштећења и развоју прслине до коначног лома при деловању затезног оптерећења.

Кључне речи: стакло-епокси композитни материјали, затезна механичка својства, микромеханичка анализа

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