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# MICROMECHANICAL ANALYSES OF THE CRACK IN GLASS WOWEN-EPOXY COMPOSITES SUBJECTED TO IN-PLANE COMPRESSION LOAD

*Marina R. Stamenović, Slaviša S. Putić, Branislav B. Bajčeta and Dragana D. Vitković*

*The subject of this paper is micromechanical analyses of the crack on glass woven-epoxy composites subjected to in-plane compression load. Identification and understanding of the first damages as micro-cracks and micro-mechanisms and their further spreading during compression loading of construction of composite material are of great importance for every special usage and life prediction. Five different structures of glass-epoxy composites were tested with orientation of reinforcement 0º/90º and ±45º. The specific weights of reinforcement were 125, 170, 210, 550 and 880 g/m². The analysis of compression test results was performed on the basis of the structure. Micromechanical analyses of surface cracks of the samples was carried out by scanning electron microscopy, and the established models and mechanisms of initiation and propagation of cracks are very valuable in predicting the behavior of the real constructions subjected to compression load.*

KEYWORDS: Glass woven-epoxy composite, compression test, micromechanical analysis

## INTRODUCTION

Intensive development of polymer engineering, as well as capabilities of polymers in combination with other materials to form new synthetic structures of improved mechanical properties, have led to a real expansion in the usage of composite materials, followed by continuous improvement of technology of their making and usage. Composite materials have a wide range of application thanks to their good properties on loading, specific mechanisms of cracks, capabilities for accumulation of energy, and represent the greatest competition to the classical construction materials. The advantages are in relatively small mass, good balance strength/mass and stiffness/mass, good static and dyna-

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mic properties, good rejection of corrosion, simplified fabrication, time of assembly, etc. (1-3).

Testing of pressure loading is one of the most commonly used mechanical testing. These tests determine the influence of reinforcement on pressure strength and modulus of elasticity, but also provide the knowledge about the mechanisms of cracks which are unavoidable factor when we talk about the duration of testing composite structure (4-6). A dominant role in the initiation and propagation of crack when testing fibers reinforcement by composite materials has the reinforcement component, which will be confirmed by results and analysis shown in this paper.

## EKSPERIMENTAL

Composite material was fabricated under laboratory conditions at the Faculty of Technology and Metalurgy in Belgrade, Serbia. Selected fiber reinforcement was woven roving E-glass (Tables 1 and 2), produced by Tehnotex-Sombor, based on a silicate glass containing alkali up to 1%. The selected glass fibers had good mechanical, hydro-thermal and dielectrical properties. Five specific weights of glass woven reinforcement were used (A=125 g/m², B=170 g/m², C=210 g/m², D=550 g/m², E=880 g/m²) and two orientations (0º/90º and ±45º). The glass woven was made by classical procedures of spinning on different kinds of looms (Fig. 1).

**Table 1.** Structural components of "E" – glass

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

**Table 2.** Physical properties of "E"-glass fiber

| Properties | | |
|---|---|---|
| Specific weight | - | 2.54 |
| Tension strength | MPa | 2400 |
| Modulus of elasticity | GPa | 73 |
| Extension | % | 3.3 |
| Thermal extension | $10^{-6}$ $K^{-1}$ | 5 |
| Thermal installing | W/mK | 1 |
| Dielectrical constant | $\xi$ | 6.7 |
| Specific electrical resistance | Ωcm | $10^{14}$ |
| Moisture absorption at 20 °C | % | 0.1 |

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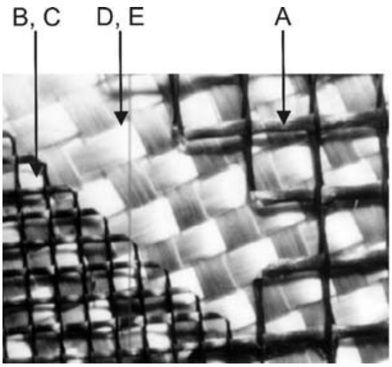
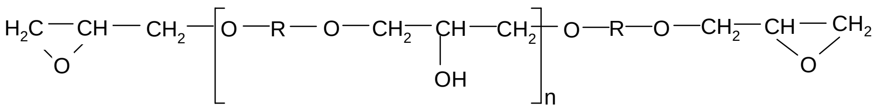
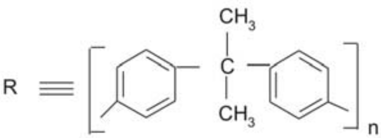



**Figure 1.** A view of the types of woven glass used

The matrix material was a polycondensation product of 2,2-bis-(4-hydroxyphenyl) propane (bisphenol A) and epichlorhydrin (Epidijan 6 made by Zaklady Chemiczne “Organika-Sarzyna” S.A):





3-aminomethanol-3,5,5-trimethanolcyclohexylamine, a modified cycloaliphatic amine of the same producer, was used as curing agent for fixing. The properties of resin are given in the producers catalog, Table 3.

**Table 3.** Catalog properties of epoxy resin

| **Properties** | | Specification | Analysis results |
|---|---|---|---|
| Appearance | | Viscous yellow liquid | Viscous yellow liquid |
| Epoxy number | val/100g | 0.51 – 0.54 | 0.520 |
| Epoxy equivalent | | 196 – 185 | 192 |
| Density | g/cm$^3$ | – | 1,26 |
| Viscosity at 25 ºC | mPas | 10000 – 15000 | 13700 |
| Color according to Gardner | | 3 | less than 3 |
| Contents of unevaporable components | % min | 99 | 99.5 |
| Contents of organic chlorine | % max | 0.3 | 0.17 |

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Original scientific paper

The epoxy-amine mixtures were prepared by heating the resin in an oil bath to 70 °C and adding the curing agent with continuous stirring until a clear homogeneous solution was obtained. Each laminate was fabricated by hand in a wet lay-up. Alternate layers of fibre reinforcement plies and liquid resin were placed inside a dam on a flat mould plate. The mould plate consisting of two metal boards upper and bottom whose dimensions were 292 x 230 x 13 mm, was tightened up with four screws to obtain the necessary pressure force of 67 N. The materials were cured during 48 h at room temperature, followed by 5 h at 90 °C, and final slow cooling. The structures of fabricated composites are given in Table 4.

**Table 4.** The structure of fabricated composite materials

| Sample | Number of reinforcement layers | Specific mass of reinforcement (g/m²) | Orientation of reinforcement | Mass fraction of reinforcement (%) |
|---|---|---|---|---|
| COMP-A-1 | 24 | 125 | 0°/90° | 42.7 |
| COMP-B-1 | 18 | 170 | | 33.7 |
| COMP-C-1 | 15 | 210 | | 34.2 |
| COMP-D-1 | 12 | 550 | | 56.7 |
| COMP-E-1 | 9 | 880 | | 66.3 |
| COMP-A-2 | 24 | 125 | ±45° | 41.1 |
| COMP-B-2 | 18 | 170 | | 37.1 |
| COMP-C-2 | 15 | 210 | | 35.4 |
| COMP-D-2 | 12 | 550 | | 58.4 |
| COMP-E-2 | 9 | 880 | | 68.2 |

Compression test was carried out on servo-hydraulic testing machine SCHENCK TREBEL RM 100, on the cut specimens (cuting was in two directions, with orientation 0°/90° and ±45°) of fabricated composite materials. Specimens of dimensions 120 x 10 x 5 (±10%) mm were machined from the 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. The testing procedure was defined by standard ASTM D 3410 (7, 8). Loading was registered by measuring cell of the capacity of 100 kN. Displacements were measured by double extensometer HOTTINGER DD1. During the test the diagrams stress-deformation ($\sigma$-$\varepsilon$) were plotted.

## RESULTS AND DISCUSSION

Compression strength, $R_{1,comp}$, was calculated from the following equation:

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

where:

$R_{1,comp}$ - compression strength, MPa,

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Original scientific paper

$F_{comp,max}$ - maximum compression force, N
$B$ - width of the specimen, mm
$D$ - thickness of the specimen, mm

Pressure modulus of elasticity, $E_{comp1}$, was calculated from equation [2] and the relationship $\Delta F/\Delta\varepsilon_1$ was determined by the method of linear regression from the rectilinear parts of curves pressure-deformation.

$$E_{1,comp} = \frac{\Delta\sigma}{\Delta\varepsilon} = \frac{\Delta F}{\Delta\varepsilon_1}\cdot\frac{1}{b\cdot d} \qquad [2]$$

The examples of the diagram force-shortening ($F$-$\Delta l$) obtained directly from the device for testing are shown on Fig. 2 (sample COMP-C-1-2) and Fig. 3 (sample COMP-C-2-1). From them, maximal force of breakage and modulus of elasticity were determined.

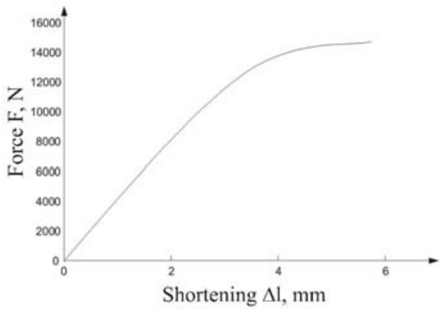


**Figure 2.** Diagram force (F) - shortening (Δl) got by testing of test tube COMP-C-1-2

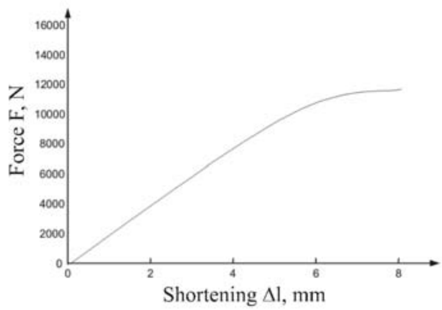


**Figure 3.** Diagram force (F) - shortening (Δl) got by testing of test tube COMP-C-2-1

Three samples of every structure were tested (Table 4). The calculated average values of compression strength and modulus of elasticity are shown in Table 5 and Fig. 4 and Fig. 5.

APTEFF, 39, 1-212 (2008) UDC: 677.52+678.686:678.017
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Original scientific paper

**Table 5.** Average compression properties

| Orientation | 0°/90° | | | | |
|---|---|---|---|---|---|
| Sample | COMP-A-1 | COMP-B-1 | COMP-C-1 | COMP-D-1 | COMP-E-1 |
| Compression strength $R_{1,comp,av}$ (MPa) | 212.9 | 218.6 | 224.0 | 378.4 | 473.8 |
| Compression modulus of elasticity $E_{1,comp,av}$(GPa) | 1.40 | 1.83 | 1.94 | 1.96 | 2.47 |
| | | | | | |
| Orientation | ±45° | | | | |
| Sample | COMP-A-2 | COMP-B-2 | COMP-C-2 | COMP-D-2 | COMP-E-2 |
| Compression strength $R_{1,comp,av}$ (MPa) | 150.6 | 191.5 | 196.9 | 264.5 | 277.1 |
| Compression modulus of elasticity $E_{1,comp,av}$(GPa) | 1.07 | 1.41 | 1.42 | 1.38 | 1.46 |

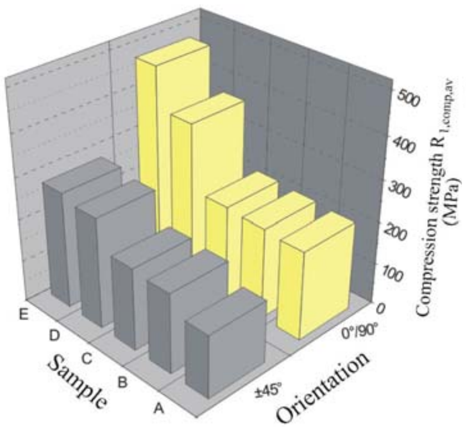


**Figure 4.** The comparative view of average values of compression strength

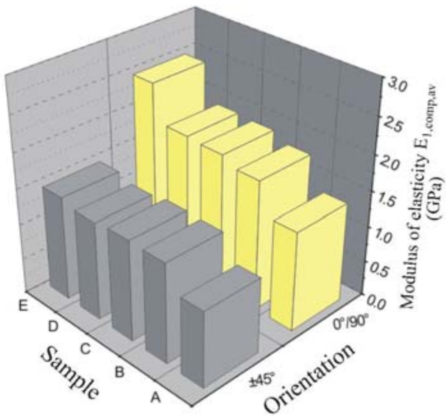


**Figure 5.** The comparative view of average values of modulus of elasticity

*Analysis of the results*

The results obtained from the compression test can be compared in respect of density of reinforcement (glass woven) and its orientation. With tested materials, no matter what the orientation of reinforcement is, the same dependance is seen between the compression strength and modulus of elasticity and the density of glass fibers. The best compression strength have materials COMP-E-1 and COMP-E-2 with the highest density of reinforcement (880 g/m$^2$). The cause for this is type of glass woven combined with the small separation between the braid of fabric, so it is not possible for empty space to appear that would be filled with air bubbles, which would cause a decrease of compression properties.

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Original scientific paper

The results are better in the case of orientation of layers 0º/90º (samples COMP-A-1, COMP-B-1, COMP-C-1, COMP-D-1 and COMP-E-1) than in the case of orientation ±45º (samples COMP-A-2, COMP-B-2, COMP-D-2 and COMP-E-2). This can be explained by the mechanisms of break of composites with these orientations of reinforcement.

*Micromechanical analisys of break with samples tested on compression load*

Scanning electron microscopy (SEM-JEOL JSM 5300) was performed on the fracture of surfaces of the mechanically failed specimens to study the mechanism of crack. The fracture of surfaces were vapour coated with a thin layer of gold to enhance the image.

The analysis was done in respect of the break of reinforcement because it is known that the reinforcement gives material strength and stiffness, and, on the other hand, it leads to different models of initiation of break and its spreading thanks to its specific properties. Fibers are not loaded in the same way because of interweaving of the fibers in cloths and different spreading of tension along the axe of fibers. The result of that is a different time of fibers crack, i. e. some fibers crack under smaller, and some under higher loading. The cracked fibers cause disturbance in the zone of crack, i. e. there are local tensions along the tore fiber, and further mechanism of crack was fully characteristic for this kind of test load.

The characteristic of samples with the orientation 0°/90° is a non-linear diagram of force-shortening, which is different from any other kind of composites. All cracks of samples are originated in elastic area and transversal to the direction of loading.

All characteristics of cracks can be seen with the composition of 0°/90° even at small magnification of the cracked samples:

- The layer with the orientation 0° has the axial cracks and pulled out fibers which indicate that cracks are due to loosening of the fiber-matrix connection and pulling out of fibers;
- The existence of axial cracks near to the point where the layers meet on the side of the layer of orientation 0°;
- Transversal cracks of the layers of orientation of 90° occur mainly through the middle surface fiber-resin but without changes that could be seen in the resin. As a rule, the axial cracks are missing in the layer of the orientation of 90°;
- Brittle cracks of fibers which show dominant axial component of tension; and
- Delamination.

The samples with the orientation of ±45° (samples-2) are characterized by the curves forces-shortening $F$-$\Delta l$ similar of test samples with the composition 0°/90° (samples-1). Consequently, it can be concluded that the character of the cracks of test samples is similar. In this case the removed components of compression are dominant, which leads to delamination and breaking of fiber-matrix connection after which the macroscopically seen cracks appear.

A Phenomenon which appears exclusively in the tests on pressure is twisting of the fibers. It can be said that on testing on pressure of composite materials the cracks appear only at the moment when twisted fibers crack, and a lot of them at the same time which weakens the sample.

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Original scientific paper

Even at small loads there were crackings of samples that definitely proved the appearance of the first cracks. They appeared on the sites where cracks in matrix already existed (Fig. 6) and on the sites where the connection fiber-matrix was separated caused by the micro twisting of fibers (Fig. 7).

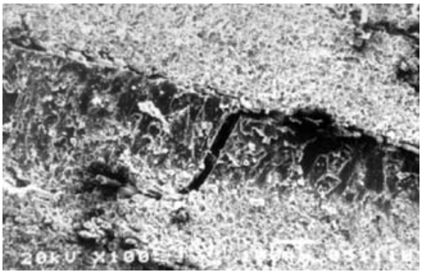


**Figure 6.** The crack in the matrix with test tube P-D-1-2 (magnification x 100)

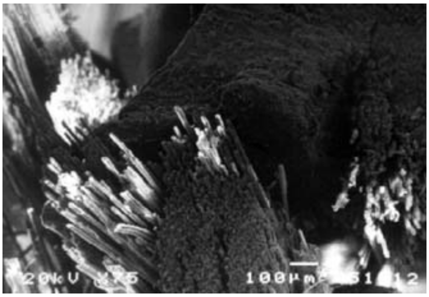


**Figure 7.** Characteristic appearance of cracked surface of sample P-E-1-1 (magnification x 75)

There are higher twisting and cracking of fibers in the cases when the fibers are not supported (Fig. 8).

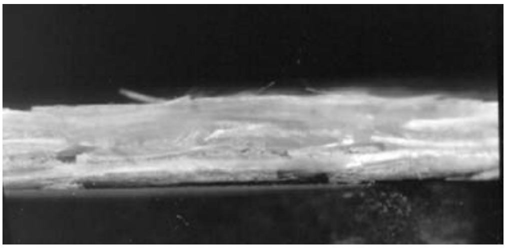

**Figure 8.** Twisting of fibers with test tube P-D-1-3

APTEFF, 39, 1-212 (2008) UDC: 677.52+678.686:678.017
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Original scientific paper

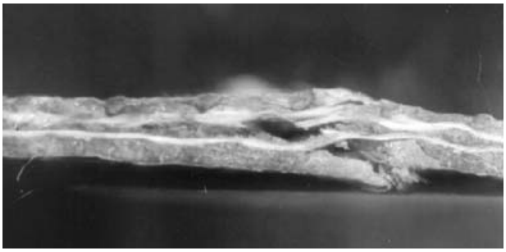

**Figure 9.** Crack and breaking into the layers of outer surface of the pressurized sample P-D-1-2

The first cracks appeared on outer surface, which confirms that a dominating mechanism of crack is twisting of the fibers. This is followed by cracking of fibers of outer surface under some angle but with progressive breaking into the close layers (Fig. 9). Such delaminating into layers can be seen by naked eye.

## CONCLUSION

The aim of this paper was micromechanical analysis of the appearance of damage on glass-epoxy composite samples broken under in-plane compression load.

Tests were done according to the standard method of testing on pressure to determine the influence of glass woven reinforcement on the existence and growth of damage, and also the compression characteristics, that is pressure strength and modulus of elasticity of this composite.

Five different structures were tested with orientation of reinforcement 0º/90º and ±45º. The specific weights of reinforcement were 125, 170, 210, 550 and 880 g/m². It can be concluded that materials with reinforcement of higher specific mass (type of glass woven E, $\rho$=880 g/m²) have shown better compression properties. Better results were obtained for the orientation of reinforcement 0º/90º than for ±45º because of the dominant shear stresses in this case.

A micromechanical analysis was performed in order to find out mechanisms and models of compression crack and better view of the quality of fabricated composite materials. The SEM micrograph of the fracture surface showed the models and the mechanisms of the compression crack known in the literature for similar structures and materials, like fiber crack, matrix crack, fiber debonding and delamination.

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## МИКРОМЕХАНИЧКА АНАЛИЗА ЛОМА КОМПОЗИТНОГ МАТЕРИЈАЛА СТАКЛО-ЕПОКСИД ИЗЛОЖЕНОГ ПРИТИСНОМ ОПТЕРЕЋЕЊУ

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У овом раду је приказана микромеханичка анализа лома композитног материјала стаклена тканина-епоксид услед притискујућег оптерећења у равни. Откривање и разумевање настанка првих оштећења у виду микропрслина и микромеханизама њиховог даљег ширења приликом притисног оптерећења конструкције од композитног материјала је од великог значаја, јер за сваку посебну примену одређује њен век трајања. Испитивано је пет различитих структура композитног материјала (површинске масе стакленог ојачања 125, 170, 210, 550 и 880 г/м$^2$ и оријентације ојачања 0°/90° и ±45°). Анализа добијених притисних својстава је изведена на основу ојачавајуће компоненте. Микромеханичка анализа је изведена на преломним површинама коришћењем СЕМ, где су уочени модели и механизми настанка лома услед притискујућег оптерећења. Ово сазнање представља важан показатељ у процени века трајања конструкције изведене од испитиваног материјала.

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