Micromechanical analysis of fatigue and crack growth in carbon-fiber epoxy composites based on mechanical testing

Aleksandra N. Jelić[1], Milica M. Sekulić[2], Marina R. Stamenović[3], Vukašin Đ. Ugrinović[1] and Slaviša S. Putić[4]

[1]*Innovation Centre of the Faculty of Technology and Metallurgy, Belgrade, Belgrade, Serbia*
[2]*University of Belgrade, Vinča Institute of Nuclear Sciences, Belgrade, Serbia*
[3]*College of Vocational Studies – Belgrade Polytechnic, Belgrade, Serbia*
[4]*Faculty of Technology and Metallurgy, University of Belgrade, Belgrade, Serbia*

Abstract

The paper presents static and dynamic tests of carbon/epoxy composite materials with fiber orientation at 0°/90° and ±45°. The main tensile properties were determined as a basis for subsequent dynamic tests, in which permanent dynamic strength, crack growth, and crack growth rate in the material due to the action of fatigue load were assessed. Comparisons were made regarding the structure of the tested specimens. Samples were obtained from prepregs with a specific density of 1600 kg/m^3. The tests were performed at room temperature. Scanning electron microscopy (SEM) was used to analyze the damage in the material during these tests, the mechanisms of their further damage progression and, the impact on the growth and growth rate of the initial crack in the material. The analysis of numerical results and micromechanical analysis confirmed the dominant role of the reinforcing structural element in the material in all performed tests. The obtained results are of great importance in the application of composite materials of such structures under different operating conditions and load regimes.

Keywords: composite material; static tests; dynamic tests; crack growth; SEM.

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1. INTRODUCTION

As macroscopic combinations of two or more distinct materials with a discrete yet identifiable interface separating them, composite materials have been used in almost every aspect of our lives. The strength combined with stiffness and lightness is one of the main advantages of composite materials [1]. Specific matrix and reinforcement characteristics directly influence the properties of the composite materials and their potential use. One of characterization techniques of composite materials is tension testing with the aim to determine following two mechanical parameters: strength (tensile strength, R_m) and stiffness (modulus of elasticity in longitudinal direction, E_1). While composite materials are designated as insensitive to fatigue, they do fail under fatigue loads, especially when compared to metallic materials [2]. In recent years, fatigue behavior of composite materials has been in the focus of intensive studies, as fatigue is considered to be responsible for most failures of structural components. Damage to isotropic materials, such as metals and polymers, typically occurs monotonously leading to material failure; in composite materials, on the contrary, four main modes of damage have been observed under fatigue loading, namely matrix cracking, fiber-matrix debonding, delamination, and fiber fracture.

Currently, great attention has been paid to a group of advanced composite materials consisting of carbon fiber (CF) reinforced epoxy resin (EP) due to the combination of high specific strength, high modulus of elasticity, low density, thermal stability, and chemical resistance [3–8]. However, several studies were performed due to the concern with the fatigue behavior. Capela *et al.* [9] investigated the fatigue behavior of short carbon fiber reinforced epoxy composite

Corresponding author: Aleksandra N. Jelić, Innovation Centre of the Faculty of Technology and Metallurgy, Belgrade, Serbia; Tel.: +38169604150
E-mail: jelicalexandra@gmail.com
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materials where fatigue life was influenced by fiber dispersion and porosity. Hashim *et al.* [10] studied the effects of fiber orientation on fatigue behavior of intraply carbon-Kevlar reinforced epoxy hybrid composite materials. Fulco *et al.* [11] investigated the effects of aging caused by high temperatures and moisture in carbon fiber reinforced composite materials and its influence on fatigue behavior. Mandegarian *et al.* [12] determined a fatigue life at a reasonable accuracy after a certain number of fatigue testing of carbon epoxy composite materials. Bertorello *et al.* [13] studied the influence of the matrix type in carbon fiber reinforced composite materials on the fatigue delamination process under mode III fracture. These studies show the influence of matrix and fiber properties, fiber orientation, and processing on materials exposed to variable loads and their potential applications.

Even for nominally equivalent composite materials, the experimental findings cannot generalized due to the effects of the material properties, manufacturing conditions, and configuration on the resulting composite fatigue output. It is apprehended from the works published in the literature that various parametric studies were performed on composite materials under static loading. Bhatia *et al.* [14] investigated the behavior of repaired carbon/epoxy laminates under tensile fatigue loading at the frequency of 3Hz and the load ratio R=0.1. Llobet *et al.* [15] conducted tension-tension load-controlled fatigue tests at a frequency of 5Hz. Wu *et al.* performed fatigue tests with a load frequency set as 16Hz, and the minimum to maximum stress, of 0.1 [16]. Liu *et al.* [17] conducted fatigue tests, at a constant amplitude sinusoidal waveform cyclic loading at a frequency of 5Hz with temperature rises limited within 10°C. The potential for the temperature rises during composite fatigue is recognized in literature and it mainly depends on the degree and frequency of cyclic loading and could be related to a variety of different heat sources.

The aim of the present research is determination of the high-frequency fatigue behavior of a cross-ply and angleply carbon fiber reinforced epoxy by *S* – *N* testing and crack growth analysis. To minimize the impact of residual strains due to the temperature increase, which can disrupt measurements of fatigue, we suggest a method for determining a more accurate value of the fatigue strength defined as the actual fatigue strength in this study.

2. EXPERIMENTAL

2. 1. Materials

The investigated carbon-fiber epoxy composite material was collected from Wing d.o.o (Belgrade, Serbia). The specific density of the material was 1600 kg/m^3, and the fiber diameter was 7÷15µm. The composite material was processed using unidirectional carbon-epoxy prepregs (HexPly® 914, Hexcel, USA), with a thickness of approximately 0.15 mm for one single layer. The specimens were processed in an autoclave, at 175 °C and 7bar for 1 hour, followed by 190 °C and 7 bar for 4 hours, as prescribed by the manufacturer, and gradually cooled down to 40 °C. The fiber mass fraction for each specimen was around 65 %. Various specimen dimensions were used according to the method of investigation and the associated size.

The specimens were cut using WaterJet – water cutting machine (PTV, Czech Republic). Table 1 provides an outline of the cases with designations of the test sequence used.

Table 1. Program of investigations with specimen series and designations

Determined properties	Lay-up		Dimensions, mm	Number of layers	Number of specimens
	0°/90°	±45°			
Static tensile properties	Z-1		250×20×25	20	10
Static shear properties		Z-2	250×30×3	24	10
Fatigue strength	Z-1	Z-2	140×30×2	16	15; 18
Fatigue crack growth	Z-1	Z-2	55×30×8	64	3; 3

2. 2. Characterization

The goal of the tensile testing is to obtain the values of tensile strength, modulus of elasticity and Poisson's ratio. Tensile testing was carried out at a material testing laboratory according to the ASTM 3039 tensile testing protocol. The machine used for testing was Shimadzu AGS-X Series Universal Electromechanical Test Frame (Shimadzu, Japan) with hydraulic grips. The strain measurements were measured by an extensometer.

Fatigue strength investigations were conducted by a high frequency pulsator (Amsler, Germany) with a variable tensile load, which can produce sinusoidal variable loads in the range from -100 to +100 kN. Mean values of the load amplitude and load were reported with a precision of ±50 N. The stress ratio in fatigue was 0.1, and the frequency was in the range from 120 to 130 Hz. For residual strain measurements, strain gauges were fixed to the specimens and attached to a PC with a converter using a multi-channel dynamic recording system (Alpha 2000, Alpha Technologies, Canada). Dimensions of the specimens from the Z-1 and Z-2 series were: width of 20 mm and a radius of 34 mm.

Tests to evaluate the crack growth rate were performed in three-point bending on a high-frequency resonant pulsator (Cracktronic, Switzerland). The experiments were performed with force regulation at the same stress ratio of 0.1 and with the dynamic moment ranging from 12.7 to 4.4 Nm. The frequency ranged from 132 to 155 Hz. Subsize Charpy specimens were cut from small bars collected from panel samples using a hard-metal milling cutter. Before the study, series of three specimens from each lay-up were prepared mechanically and wrapped with a crack gage foil (Rumul RMF A-5, Rumul, Switzerland) glued like a typical strain gauge to measure the growth of a fatigue crack.

Fracture surfaces of mechanically fractured specimens were examined for the analysis of fatigue micromechanisms by scanning electron microscopy (SEM) (MIRA3 TESCAN, Tescan, Czech Republic) The surfaces of the fractures were vapor-coated with a thin film of gold to increase visual clarity.

3. RESULTS AND DISCUSSION

3. 1. Tension test results

Tension tests were performed in order to determine the values of tensile strength, strain and modulus of elasticity. Typical comparative stress – strain curves for both specimen types are shown in Figure 1 while the average values of the tensile strength, strain, and modulus of elasticity are presented in Table 2.

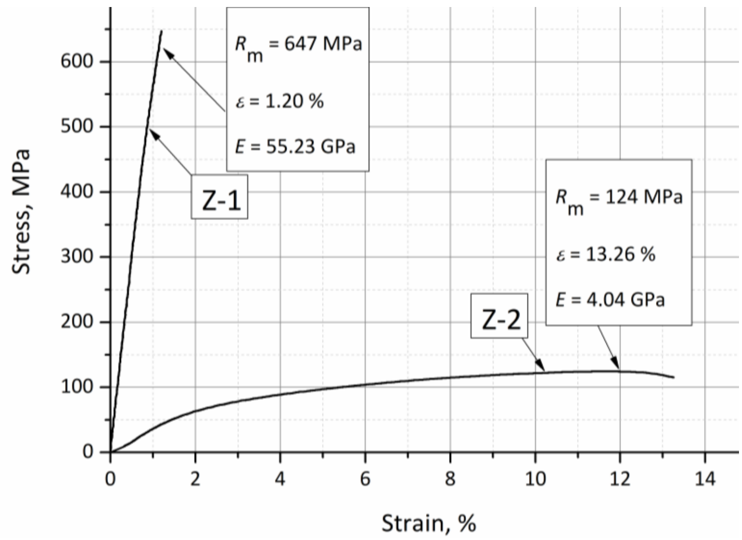

Figure 1. Typical comparative stress – strain curves for specimen series of 0°/90° (Z-1) and ±45° (Z-2) orientations with measured values of tensile strength (R_m), strain (ε), and modulus of elasticity (E)

Table 2. Average values of tensile strength, strain, and modulus of elasticity for specimen series Z-1 and Z-2

Specimen	Tensile strength, MPa	Strain, %	Modulus of elasticity, GPa
Z - 1	647 ± 11.81	1.2 ± 0.00	55.23 ± 0.99
Z - 2	124 ± 6.81	13.26 ± 2.06	4.04 ± 0.54

3. 2. Fatigue strength

The results determined for specimens obtained from the two-lay-up patterns are illustrated in Figure 2. in the form of stress amplitude (S) – number of cycles (N) curves. It was identified during the experiment that the stress amplitude continually decreased with a growing number of cycles. Fatigue strength S_f was defined as the strength after10^7 cycles. The *S-N* curves are rather smooth for both the Z-1 and Z-2 specimen series, meaning that small variations in the stress level resulted in significant cycle differences. The curve flatness was anticipated as the tested carbon-epoxy laminates had a fiber-dominated strength. The obtained values of S_f were 425 MPa and 95 MPa for Z-1 and Z-2 specimens, respectively. Since the specimen with ±45° fiber orientation did not contain fibers along the load, higher values were observed for the specimen with 0°/90° fiber orientation (Z-1).

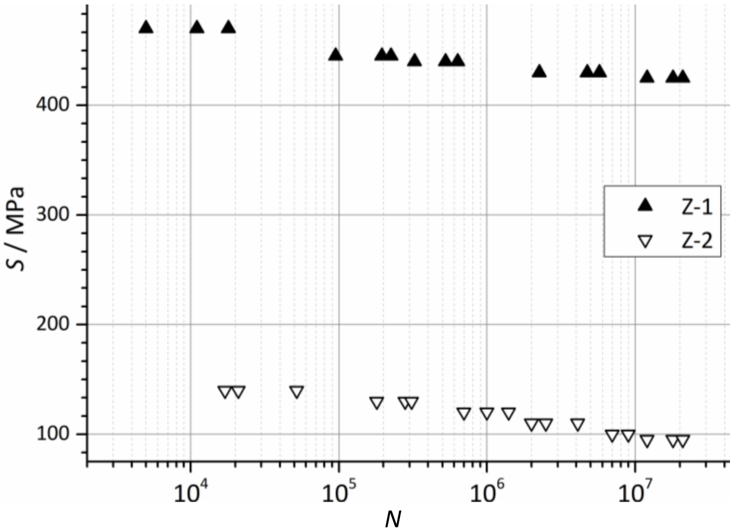

Figure 2. Comparative presentation of S -N *curves for specimen series of 0°/90° (Z-1) and ±45° (Z-2) orientations*

3. 3. Fatigue crack growth

Based on the size and the location of the reinforcement as well as the amount of applied mechanical loading, different stages in the damage process arise sooner or later. The initial damage stages comprise failure of network interfaces requiring low energy usage while the final phases comprising fiber breakage need a higher energy level [18]. At an early loading stage, microcracks are introduced in composite materials but these materials still have the ability to carry the loads before the final fracture. Damage is a complex combination of transverse breaks, cuts, fiber-fractures, and delamination [19]. It is very challenging to examine the growth of damage, but strategies to fracture mechanics provide an alternative where the damage growth is determined by the evolution of a single crack, such as delamination. The crack density can be considered as a single distributed damage, and it can be analyzed via various approaches by fracture mechanics as a single crack.

Crack growth measurements (crack length, *a*, against the number of fatigue cycles, *N*) are presented in Figure 3 for both examined lay-ups, which were further used for determination of the slopes d*a*/d*N*. At the same loading rate, the number of cycles needed for the formation of cracks for specimens Z-1 (0°/90°) was higher than that needed for specimens Z-2 (±45°).

It can be concluded that the higher tensile strength of the specimen demonstrated greater resistance to crack formation. The sudden change from the linear portion of the *a-N* dependence for the Z-1 specimen suggested an increase in the crack growth rate indicating lower number of cycles needed for the initial crack formation. Given the specificity of ±45 stacking in Z-2 specimens, the occurrence of cracks has a different further development compared to Z-1 specimens with 0°/90° fiber orientation. The difference is reflected in the fact that the crack propagation path for Z-2 specimens is longer and more varied in trajectory than at Z-1 specimens where the crack moves directly in the initiated direction. That is clearly seen in Figure 3 obtained after investigating the dependence of crack length, *a*, and the number of cycles, *N*. A sharp curve transition for Z-1 specimens confirms the previous claim. When it comes to Z-2 specimens, the crack occurred at a smaller number of cycles, but its further development was slowed down (as opposed to Z-1 specimens; Fig. 3), which unequivocally indicates that the crack growth was not linear.

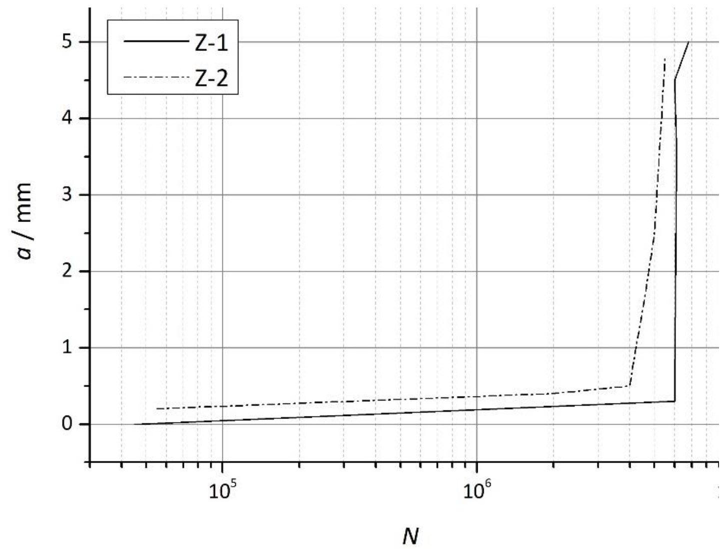

Figure 3. Fatigue crack growth: crack length, a, versus the number of cycles, N, for specimen series of 0°/90° (Z-1) and ±45° (Z-2) orientations

A large number of data was acquired during one specimen testing. The slope da/dn = log ΔK is related to the stress intensity factor range (ΔK) as:

$$\frac{\mathrm{d}a}{\mathrm{d}N} = \log \Delta K \tag{1}$$

This relation is generally demonstrated in Figure 4 showing the lower damage growth limit, or the effectively the absence of the growth rate. The higher fatigue threshold value was found for the Z-1 specimens, while in the case of the Z-2 specimen, a the higher crack growth rate and comparatively lower fatigue threshold values were found, supporting the assumption that in these systems the design is compromised by unusually high shearing interlaminar stresses. The differences observed were due to different micromechanisms of the crack growth.

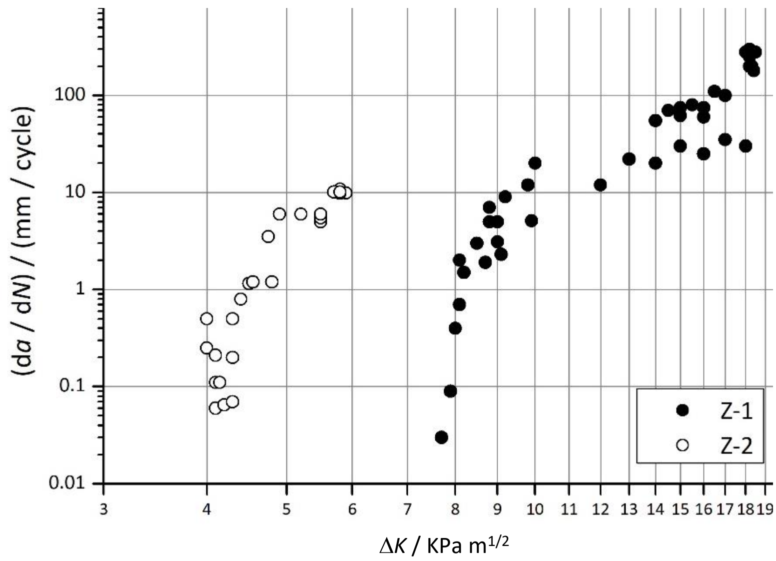

Figure 4. Measured constant-amplitude fatigue crack growth curves for Z-1 and Z-2 lay-ups

3. 4. SEM analysis

To determine the cause of the observed fatigue fracture cases, scanning electron microscopy (SEM) was used after fatigue testing to study the fracture surfaces of the specimens.

Figure 5. demonstrates the fracture surface of a Z-1 specimen. The composite material fracture occurred at the moment when it was compromised by longitudinal cracks and cracks created by delamination of layers of opposite orientations, allowing a proper transition of stress between them (Fig. 5a). Progressive degradation of the fibers in remaining layers was accompanied by fiber breakage within the weakest layer. Delamination of the layers below the fracture surface with opposite fiber orientations at a higher magnification is shown in Figure 5b. Based on the obtained results, it could be concluded that the successive breakage of fibers in the lay-up containing 0° oriented fibers resulted in the main crack growth [20]. The initial crack was normal to the tensile stress. Delamination and matrix crack are presented in Figure 5c.

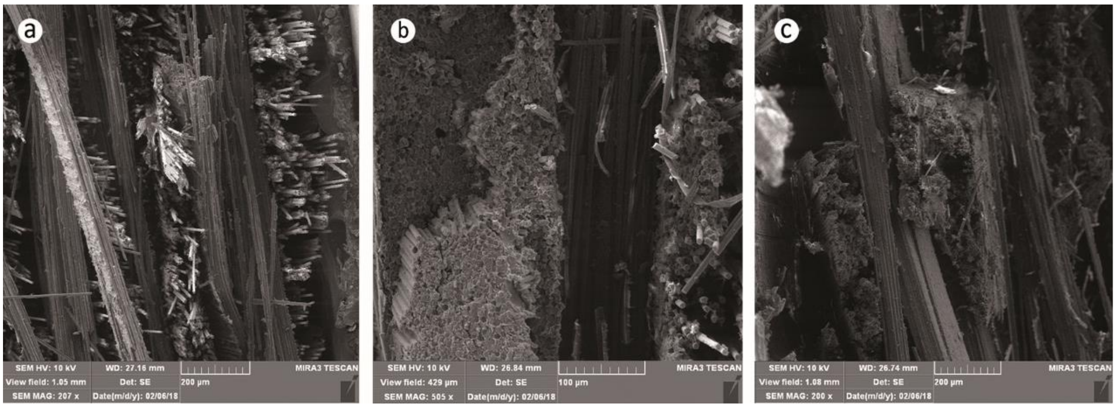

Figure 5. SEM micrograph of the fracture surface of the specimen Z-1: a) matrix cracking in 0° layers; (b) delamination of the layers below the fracture surface; c) fiber delamination and a matrix crack

In the case of crossply specimens (Z-2), failure occurred from breakage of most fibers in the layers and from the matrix's macroscopically visible fiber pullout from the matrix. This was followed by delamination, reflecting the additional failure condition during fatigue testing. The initial cracks started in one layer and, as a result of micro crack accumulation, turned into macro cracks after a certain number of cycles. The presence of such a crack triggered a dynamic condition of stress which was passed to adjacent layers in opposite orientation *via* shear stress, weakening the fiber-matrix bonds and, finally, transferring the entire load to fibers causing the fiber failure (Fig. 6.).

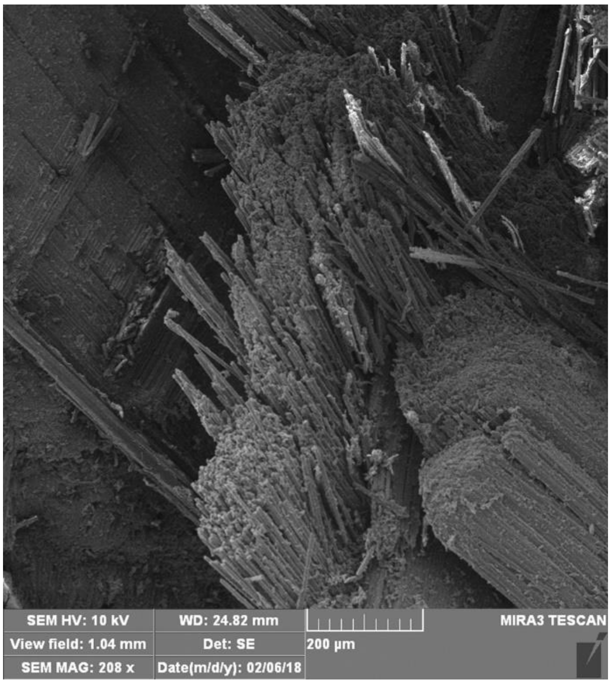

Figure 6. SEM micrograph of the fracture surface of the Z-2 specimen

Similar mechanism of fatigue damage growth was found in *a-N* tested specimens investigated the crack growth (section 3.3). The key difference was that subcritical cracks concurrently formed in lay-ups comprising fibers oriented at +45° and -45°. In addition, erratic orientation of the key crack growth was found since the fiber breakage occurred at different locations but often at around 45° to the tensile axis.

4. CONCLUSIONS

The results obtained after tensile testing of carbon fiber reinforce epoxy resin laminate structures were within the expected limits and values. Typical diagrams demonstrated higher tensile forces for the 0°/90° orientation arrangement. Larger deformations were also found with a decrease in strength in the samples with the ±45° orientation. The interpretation of the obtained results is linked to the models and fracture mechanisms in the systems, determined by the SEM analysis of fracture surfaces.

Higher values of fatigue strength were obtained for the 0°/90° orientation confirming the significance of the fiber orientation. In the case of angleply specimens (±45° orientation), the high crack growth rate and a relatively low fatigue threshold values were obtained, as compared to crossply specimens (0°/90° orientation) confirming the contribution of high shearing interlaminar stresses to additional weakening of the construction.

SEM analysis confirmed the initial crack development in Z-1 specimens (0°/90° orientation). The dominant crack development was in the layers of 0° fiber orientation which led to the final fracture. In the samples of both structures (Z-1 and Z-2), the development of a crack in the layers caused its expansion in the thickness of the layers and thus, delamination occurred which weakened the strength of the sample and caused the final fracture.

The aim of these investigations was to produce a newly obtained composite material that could be used in new structures exposed to variable loads, structures in which the crack growth should be slowed down primarily after the appearance of the initial crack.

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SAŽETAK

Mikromehanička analiza zamora i rasta prsline u ugljenik-epoksi kompozitima na osnovu mehaničkog ispitivanja

Aleksandra N. Jelić[1], Milica M. Sekulić[2], Marina R. Stamenović[3], Vukašin Đ. Ugrinović[1] i Slaviša S. Putić[4]

[1]*Inovacioni centar Tehnološko-metalurškog fakulteta Beograd, Beograd, Srbija*
[2]*Institut za nuklearne nauke "Vinča", Univerzitet u Beogradu, Beograd, Srbija*
[3]*Beogradska politehnika, Beograd, Srbija*
[4]*Tehnološko-metalurški fakultet, Univerzitet u Beogradu, Beograd, Srbija*

(Naučni rad)

U radu su prikazana statička i dinamička ispitivanja ugljenik/epoksi kompozitnih materijala strukture 0°/90° i ±45°. Određene su osnovna zatezna svojstva kao osnov za dalja dinamička ispitivanja i određivanje trajne dinamičke čvrstoće, rasta prsline i brzine rasta prsline u materijalu usled dejstva zamornog opterećenja. Poređenja su izvođena u odnosu na strukturu ispitanih uzoraka. Uzorci su dobijeni iz prepregа specifične gustine 1600 kg/m^3. Testovi su izvedeni na sobnoj temperaturi. U cilju analize nastalih oštećenja u materijalu pri ovim ispitivanjima, mehanizama njihovog daljeg razvoja i uticaja na rast i brzinu rasta inicijalne prsline u materijalu, korišćena je skenirajuća elektronska mikroskopija. Analizom numeričkih rezultata i mikromehaničkom analizom potvrđena je dominantna uloga ojačavajućeg strukturnog elementa u materijalu kod svih izvedenih ispitivanja. Dobijeni rezultati su od velikog značaja u primeni kompozitnih materijala ovakvih struktura u različitim uslovima eksploatacije i režima opterećenja.

Ključne reči: kompozitni materijal, statička ispitivanja; dinamička ispitivanja; rast prsline; SEM