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EFFECT OF NANOGRAPHENE ON THE MILLING PROCESS OF EPOXY BASED COMPOSITE

UTICAJ NANOGRAFENA NA PROCES OBRADE REZANJEM KOMPOZITA NA BAZI EPOKSI SMOLE

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- milling process
- composite damage
- deformation
- mechanical properties

Ključne reči
- nanografen
- obrada rezanjem
- oštećenje kompozita
- deformacija
- mehaničke osobine

Abstract

The paper studies nanographene oxide fracture properties in different content (stress intensity and energy release rates). These nanoparticles are added to thermosetting polymer EPON 862 and its nano-graphene reinforced counterparts. Deficient (0.2 and 0.3) weight percent nano-graphene specimens are dispersed in EPON 862 matrix. Tensile tests are conducted with the universal testing machine; using displacement control, significant enhancement 23% and 22 %, respectively is recorded in fracture, roughness, and energy. The content of more than 0.15 wt.% has had a defection into the desired mechanical results. Electron microscopy (SEM) was conducted to visualize and understand the mechanism of crack, fracture, energy, and to enhance these results. SEM has been utilized to study crack deflection due to increased surface roughness, NGO specimen's pull-out, and plastic deformation of the matrix causing filler-matrix debonding. The machining process and damage effect over the process, the role of machining, hole over the micro-cracks using SEM method are analysed and discussed entirely.

Izvod

U radu se proučavaju osobine loma oksida nanografena i to sa aspekata parametara (intenziteta napona i brzine oslobađanja energije). Ove nanočestice se dodaju termoreaktivnom polimeru EPON 862 koji sadrži ojačavajuće komponente nanografena. Uzorci sa osiromašenim težinskim procentom (0,2 i 0,3) nanografena su disperzno rastvoreni u matrici EPON 862. Izvedena su ispitivanja zatezanjem na univerzalnoj kidalici u režimu kontrolisanih pomeranja, gde se javlja značajno povećanje od 23% i 22%, respektivno praćenjem loma, hrapavosti i energije. U sastavu od preko 0,15 tež.% javljaju se veći uticaji na zahtevane mehaničke osobine. Elektronskom mikroskopijom (SEM) dat je uvid u razumevanje mehanizama razvoja prsline, loma, energije, kao i za poboljšanje ovih osobina. SEM je omogućio proučavanje skretanja prsline usled povećanja površinske hrapavosti, nagomilavanja oksida nanografena (NGO) u epruveti, plastičnih deformacija matrice, što je izazvalo raskidanje ispune-matrice. Analizirani su uticaji mašinske obrade i oštećenja na postupak, kao i rupe iznad mikroprslina primenom SEM.

INTRODUCTION

Epoxy resin is very popular and can be used in many applications, such as manufacturing polymeric base composite which is important in novel engineering design /1/. Graphene's wide range of applications, from energy storage applications to immune sensors, has made it an ideal test candidate as a filler for polymer systems due to its superior mechanical, electrical, and thermal properties, /2/. Nanographene oxide widespread industries are attracted to this new material due to its unique material properties. Research due to superior mechanical enhancements at very low filler volume fractions nanographene oxide (NGO) has also been widely researched for similar purposes due to their compatibility with various polymer systems and possible content modification of NGO, /3/. But the higher cost of manufacturing and grade NGO has made researchers lean toward graphene as a better option for large-scale applications. Excellent epoxy-based composite materials epoxy base nanocomposite due to their adhesive and well weight-to-strength ratio have been populated into the world of aircraft, auto industry, boat industry et al. This material due to their 3D net-shape has gotten the low strength-to-damage fail, /1, 3, 4/. This material is maximally oxidised; NGO is a crystalline allotrope of carbon with carbon 2-dimensional properties. Carbon atoms are highly dense in a regular atomic-scale and hexagonal pattern. This nanostructure turns NGO into a great material while maintaining its features, /4/. Promoting mechanical features by attaching oxide to the compound turns this material into a strong structure. They have unusual properties. These nanographene tracted and became valuable for different scientific fields such as nanotechnology, electronics, optics, etc.; they have incredible and exceptional mechanical features such as high stiffness, /5/. They have over 200 GPa strength, which means 200 times more than steel. With this knowledge, NGO can be a great additive to cover and improve some of the epoxy features, /5/. Several studies have been carried out from the residual strength of the composite. Researchers are interested in the relationship between velocity and structural failure. Many types of research have been done to analyse the microcracks that cause failure in micromechanical structures. Damage in micromechanical structures will be introduced by modelling under load composites due to superior mechanical features. Enhancement at a meagre filler volume fraction of polymer/nanographene has drawn widespread interest in industry and research /6-8/. Carbon nanotubes have been widely utilised and researched for similar purposes due to surface modification and their compatibility. Nevertheless, high-cost manufacturing and high-grade CNTs, also better mechanical response has made a preference to put better option for large scale application and superior epoxy base material /9-11/. Some nanocomposite macro scale properties depend on thermodynamic parameters. These structural properties include interfacial polymer compatibility. Nano-filler size, phase dispersion, and distribution depend on the filler aspect ratio. Also, the dispersion technique, mixing time, filler volume fraction, applied shear, bonding strength between filler and matrix are fundamental parameters /12, 13/. Full filler NGO advantage can only be taken by considering all the above processing factors. These specifications lead to better load transfer between the polymer matrix and the filler surface with superior mechanical features. Recent studies are reported that the precursors generate NGO in improving the elastic modulus (100 %), fracture toughness (65 %), fracture energy (115 %). For damaged specimens of very low 0.1-1 wt.%, major improvement in these reports is ascribed to surface roughness, crack spinning, and crack deflection. Due to the presence of NGO, plastic deformation and matrix debonding can be a great potential mechanism for a drastic increase in mechanical features /14, 15/. Thus, NGO is a great pioneer future nanocomposite to its best weight-to-ratio aspect. With its remarkable mechanical properties, here in this paper, some of the mechanical properties of thermoset polymer/NGO are observed. The result is discussed on EPON 862. The research has emerged from epoxy system used for applications due to its low processing viscosity and better mechanical properties for composite applications /16, 17/. EPON 862 is an aerospace-grade di-functional epoxy resin with low molecular weight. When mixed with curing agent 'W' it is categorised as a thermoset polymer system with high crosslink density. Although the polymer is strong, it is relatively brittle with low stiffness. In this study, the thermoset epoxy EPON 862 is selected to investigate the changes in damage to fracture properties (toughness and energy) of its nano-graphene reinforced counterpart. Compact tension (CT) specimens with nanometre-sized starter cracks are used in the study, with four replicate specimens per nanographene polymer (NGP), initiate load was of 0 wt.% (neat), 0.1 wt.%, and 0.5 wt.% employed. ASTM standard 5045 is used to perform fracture testing under room temperature and quasi-static loading conditions. Nano-graphene reinforced epoxy specimens are observed to increase the fracture toughness by 141 and 200 % for 0.1 and 0.5 wt.%, respectively, and fracture energy by 347 and 567 % for 0.1 and 0.5 wt.% NGP, in respect, as compared to the neat the major toughening studied using experimental test and figures /6, 18-21/.

EXPERIMENTAL SECTION

Material details and preparation

NGO with an average diameter of 5 nm is purchased from XG-Sciences Inc. with 99.9 % purity see Fig. 1. With a specific surface area of 120 m^2/g, the epoxy utilised in this study is EPON 826, a di-glycidyl ether of bisphenol-F epoxy (DGEBF) from Momentive Inc. The curing agent is ‘W’ (DETDA-diethyl toluene diamine). According to Fig. 3, the thermoset polymer and curing system is utilised in this study, where the TDA amine group acts as crosslink centres. Reinforcing the epoxy resin is done to prepare the homogenous mixture and making of the NGO and epoxy resin mixture. The nanoparticles of 0.1 to 0.2 wt.% of composite are mixed well with acetone by a mechanical stirrer for 2 hours and with 1000 min^{-1}. In the current study, the mixture was home by ultrasonicating (SONOPLUS-HD3200, 50 % amplitude, 20 kHz and pulsation; on for 10 s and off for 3 s) for 8 minutes. Figure 2(a-d) shows the level of dispersion of NGOs. At this stage, 23 per (per hundred resins) of diethyl toluene diamine is added as a hardener based on a stoichiometric ratio. The epoxy to curing agent ratio according to the manufacturing data sheet is 100 : 26.4. The nano mixture is set at room temperature for 24 hours and then poured into specific silicon casts. Finally, they were cured in an oven from 50 to 120 °C each two hours with a 20 °C temperature enhancement interval. All tensile test specimens are prepared according to ASTM D:6641. Specimen size is 12×140×4.5 mm in width×length×thickness, and at least five samples are prepared for each test.

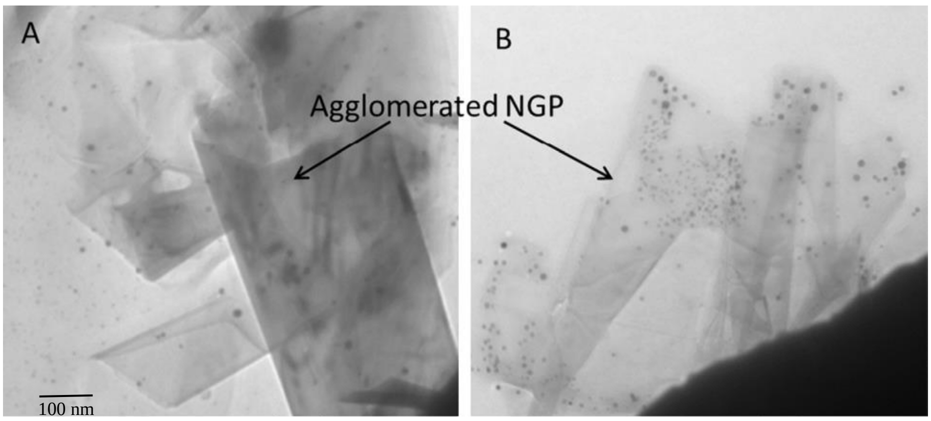


Figure 1. Agglomeration scheme.

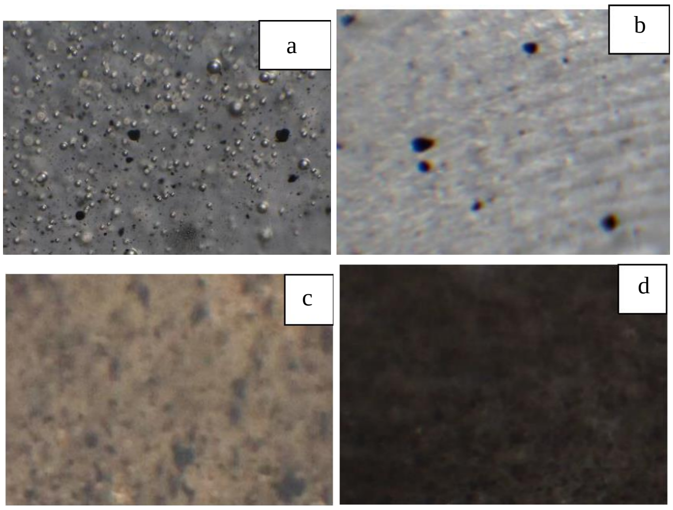


Figure 2. Microscopic pictures show the dispersion level of NGO after sonication (a-b-c-d).

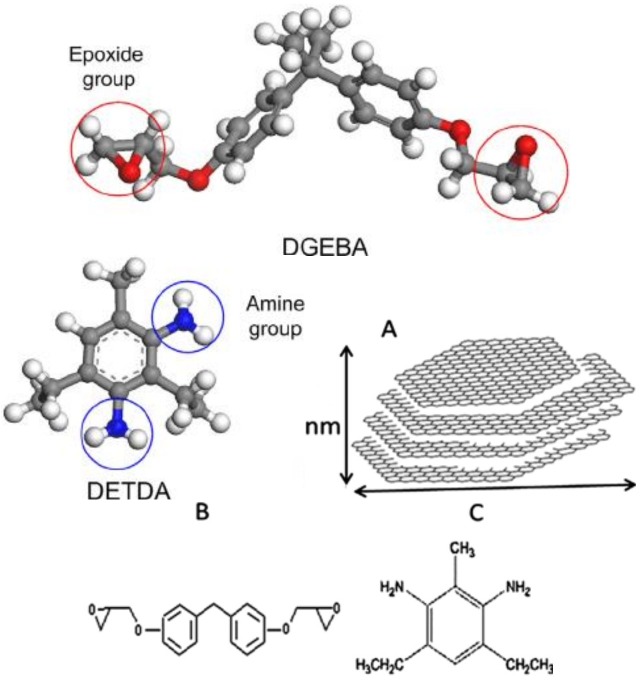


Figure 3. Reaction of epoxy to the amine group.

Applying damage by CNC machine

The machining process is the critical part of the application composite. This process is an essential part of engineering which with different applications of nanocomposite in the industry will classify the type of machining process. By this knowledge, the machining process is done due to making a hole as shown in Fig. 4. According to Figs. 25-28, after applying machining damage, different microcracks are revealed over machining area Fig. 5. It may also be said that machining forces and the machining process will lead to residual stress. This can decrease the ultimate tensile stress point.

Testing

- Undamaged and damaged test result

Tensile stress is chosen to understand fracture behaviour. Based on ASTM D:6641 test protocol, the size of specimen

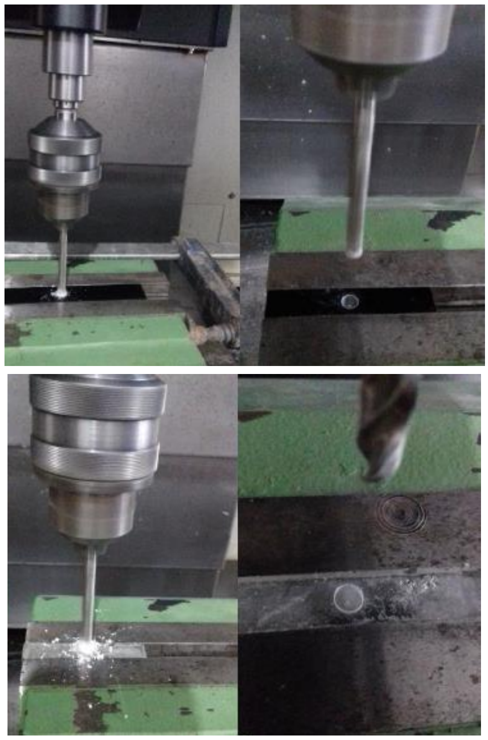


Figure 4. Machining process.

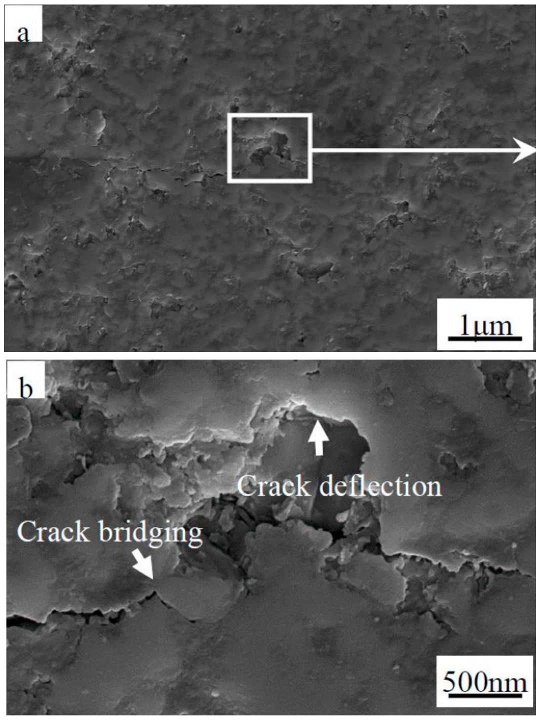


Figure 5. Microcracks after machining over machining area.

is 12×140×4.5. Manufactured specimens are shown in Fig. 4. As Fig. 6 shows, the change in colour of epoxy due to the presence of NGO is very noticeable. In each test, an average of 5 samples are tested (damaged and undamaged). All tensile tests are performed on the STM-150 testing machine by Santam company Iran, as seen in Fig. 7. After testing, the force-displacement curve is derived as seen in Fig. 8. The effect of NGO in the tensile result is noticeable. As seen in the result, NGOs increase the ultimate tensile stress (UTS).

The level of NGO shows that increasing NGO up to 0.15 wt.% can enhance the UTS. Still, after the aimed content, the UTS will decrease because of accumulation in higher contents. Figure 9 shows the testing of different content of NGO neat and specimens that contain NGO. As see in Figs. 9-10 in the next level, machined (damaged) specimens are tested to specify the result and effect of microcracks in the nano composite. These cracks lead nano composite into unexpected failure and cause residual stress that can decrease UTS. The result shows that after machining with depth of 3 mm, the UTS compared to undamaged specimens decreases gradually to reach the highest UTS about 47 MPa for 0.15 wt.%, while the highest rate of UTS for undamaged specimens is 55 MPa at 0.15 wt.%. As seen in Fig. 11, the compared results for both damaged and undamaged show that increasing the NGO wt.% above 0.15 wt.% will lead to reverse result which decreases the UTS even less than 0.1 wt.%, this incident can be because of agglomeration, in other words the crystal bonding with higher content of NGO in epoxy will be weak and cause unexpected failure.

(a) (b)

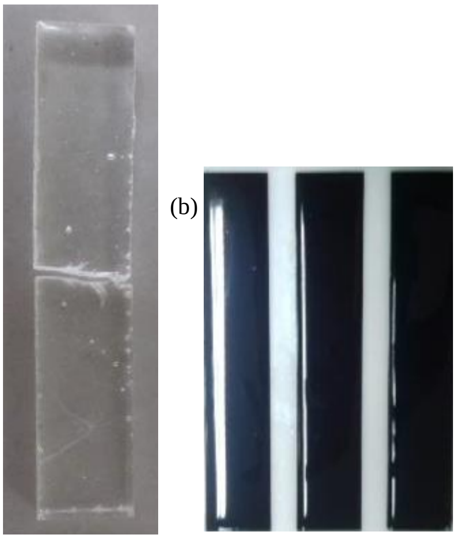


Figure 6. Samples: a) neat epoxy; b) NGO.

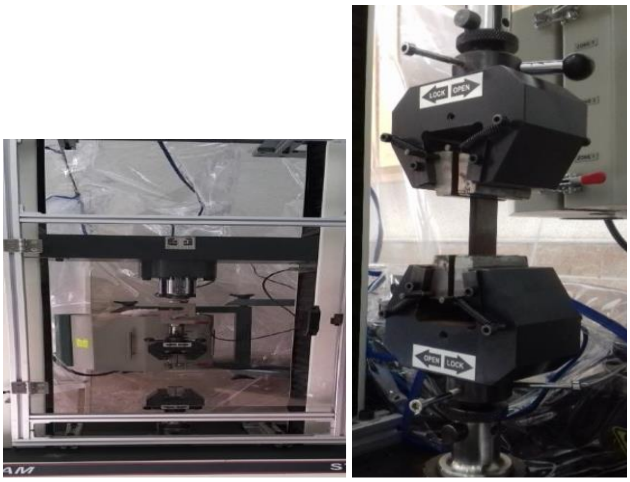


Figure 7. Tensile testing machine.

As shown in Fig. 5 the crack growth path deviates and creates deflection and crack bridging. According to Fig. 12 after comparing two specimens damaged and undamaged, it can be found that for neat (0 wt.%), 22.96 % of decrease appears in UTS because the residual stress develops from the machining step, thus for the respective UTS decrease of -21.58, -16.58, and -21.13 % for 0.1, 0.15, and 0.2 wt.%. Also, Figs. 13 and 14 show different UTS levels compared to neat for both damaged and undamaged.

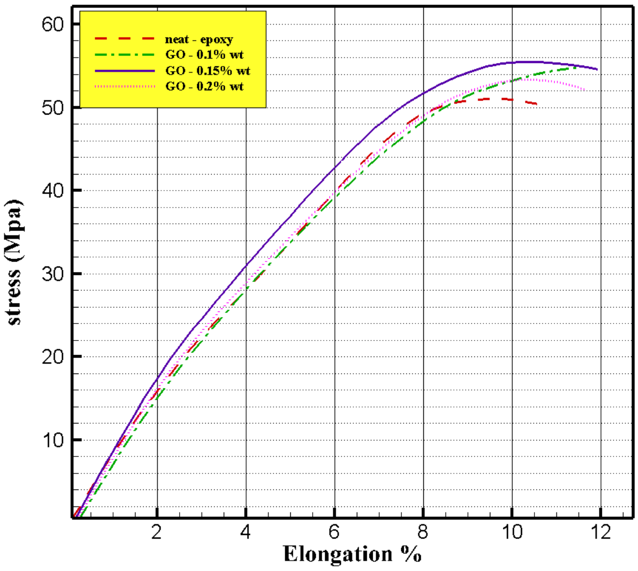


Figure 8. Undamaged specimen tensile result.

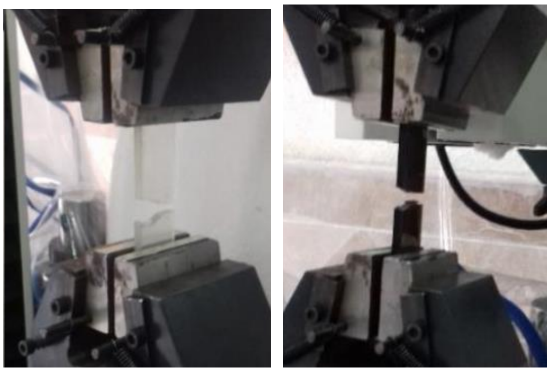


Figure 9. The test result of the NGO composite.

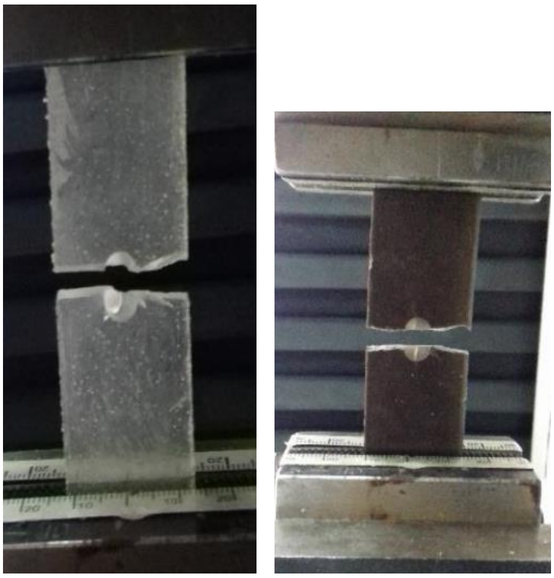


Figure 10. Damaged nanocomposite with different content of NGO.

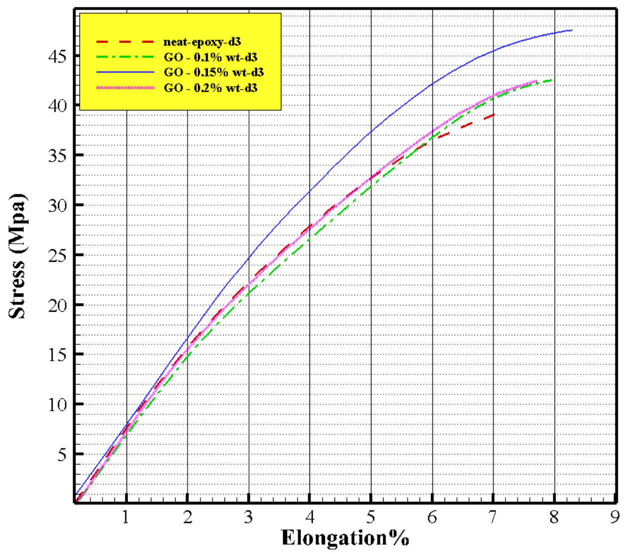


Figure 11. Result of damaged nanocomposite after the tensile test.

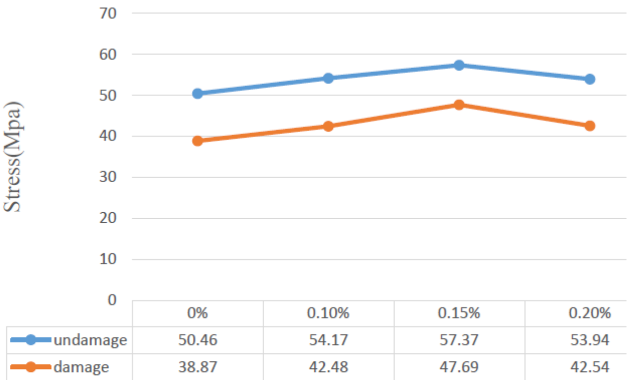


Figure 12. Different levels of stress for undamaged and damaged composite (wt.%).

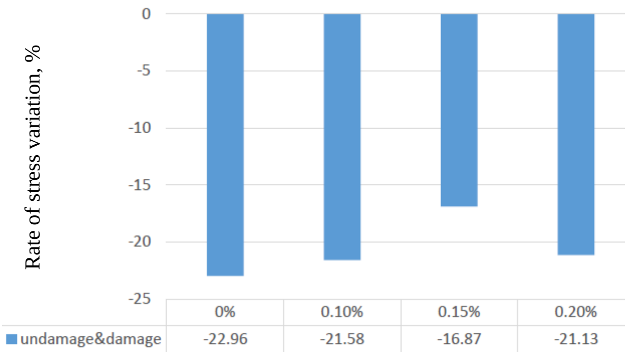


Figure 13. Rate of stress change (wt.%).

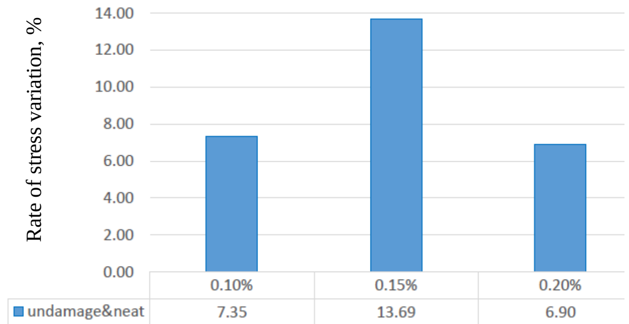


Figure 14. Rate of stress change toward neat for undamaged specimens (wt.%).

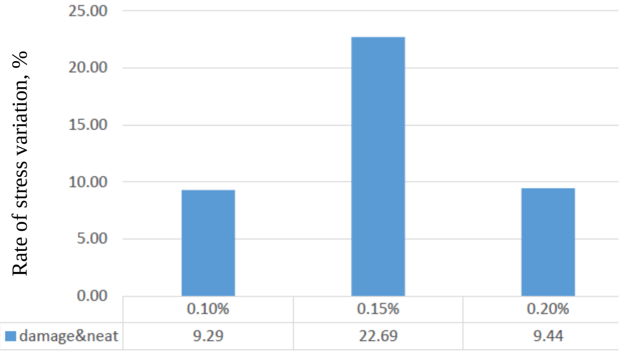


Figure 15. Rate of stress change toward neat for damaged specimens (wt.%).

- Elasticity modules analysis results

As seen in Fig. 15, it shows that the maximum rate of elasticity belongs to 0.15 wt.%; thus, by analysing the following diagram, the total elasticity can be obtained due to accumulation in contents above 0.15 wt.%, and the module decreases gradually. As seen in Figs. 16-20, the elasticity module decreases after damage, because of microcrack growth. As seen in the figures, the elasticity module rate changes gradually. By adding NGO content, the rate changes from 12.77 for neat, to 12.97 for 0.15 wt.% NGO. A growth in more than desired content was not observed for 0.2 wt.% NGO, and the rate of the elasticity module decreases considerably, by considering both damaged. The undamaged specimens have the highest rate of elasticity module growth again at 0.15 wt.% with 8.76 % growth. This is because the well bonding crystalline with the epoxy causes a better response in mechanical results.

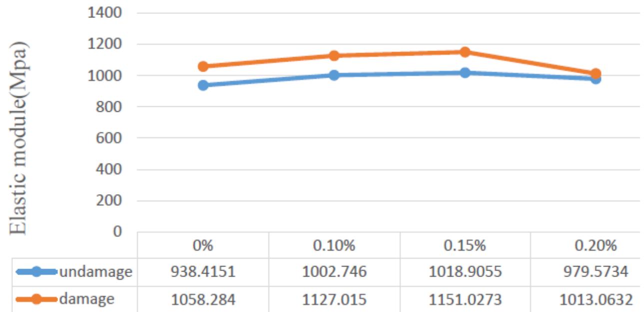


Figure 16. Elasticity modulus (MPa) for damaged and undamaged specimens (wt.%).

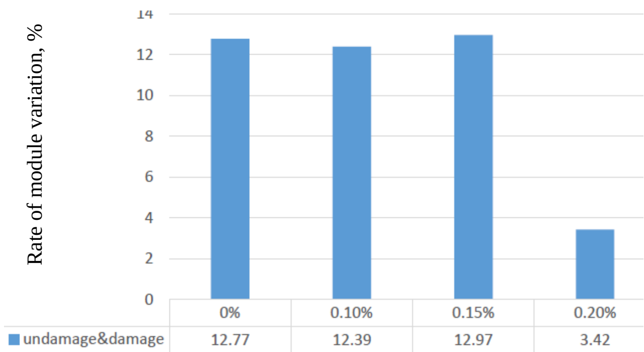


Figure 17. Elasticity modulus (MPa) rate for damaged and undamaged specimens (wt.%).

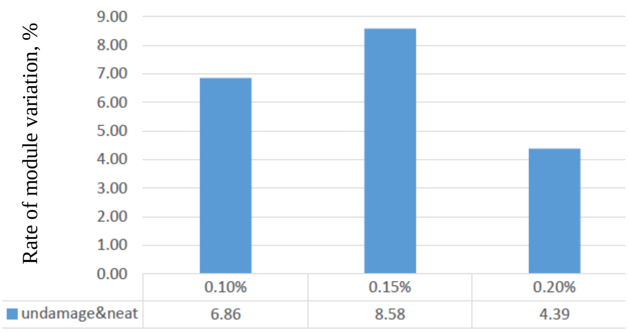


Figure 18. Elasticity modulus (MPa) rate for damaged and undamaged specimens compared with neat (wt.%).

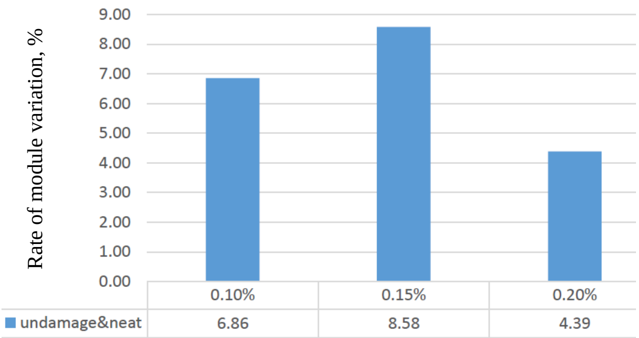


Figure 19. Elasticity modulus (MPa) rate for undamaged specimens compared with neat (wt.%).

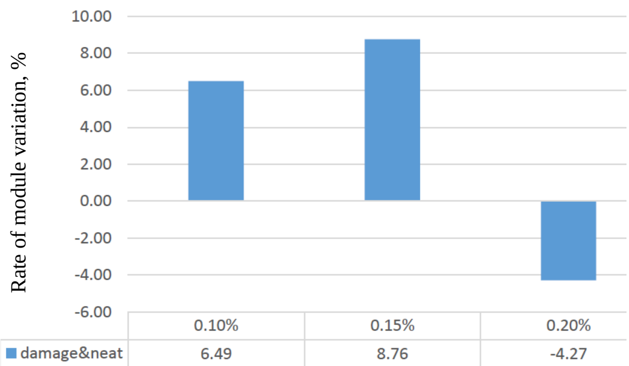


Figure 20. Elasticity modulus (MPa) rate for damaged specimens compared with neat (wt.%).

- IZOD test result

According to Fig. 24, after tensile test specimens are prepared for IZOD test (ASTM D:256), specimen sizes are 12×25. The IZOD test is applied with STM-230 from Santam Co., Iran. As seen in Fig. 21, toughness behaviour of NGO is considerable. Adding NGO content up to 0.2 wt.% the toughness grows, and while continuing contents over 0.2 wt.%, the toughness decreases. Because of better bonding with epoxy crystals, for 0.2 wt.%, a 23 % of toughness growth is observed. But for over 0.2 wt.% it will increase the pothole where even this incident has a significant defect over the mechanical test, but here packing and presence of NGO to 2 wt.% helps improve toughness. However, according to Figs. 25-28, this pothole increases microcracks, intensifying this incident when machining is applied. As Figs. 22-23 show, after deep study of NGO toughness behaviour, it is found that agglomeration incident increases the pothole and most of the microcracks appear around these potholes that lead to unexpected failure, where 0.15 wt.% becomes a good choice and gives a better response in increasing toughness, because of better bonding with epoxy.

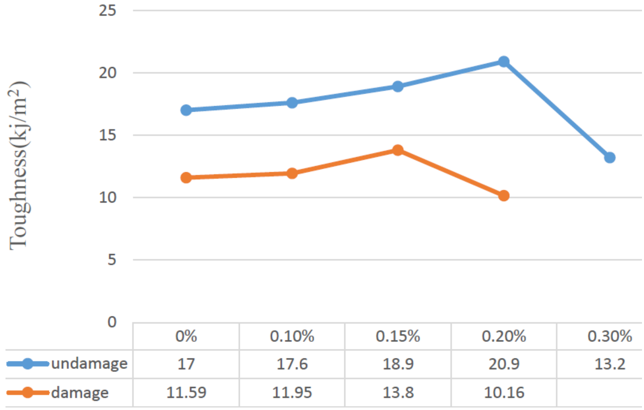


Figure 21. Toughness result for damaged and undamaged specimens (wt.%).

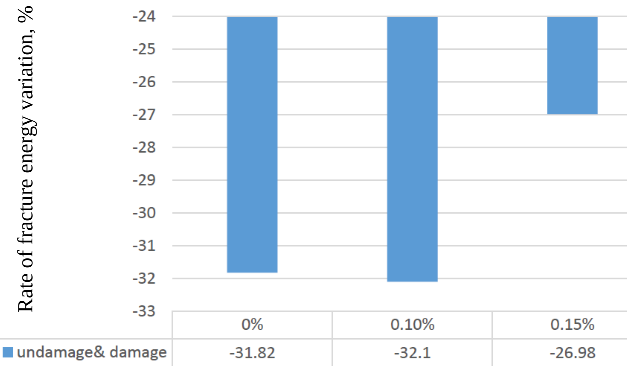


Figure 22. Rate of toughness result changes for damaged specimens (wt.%).

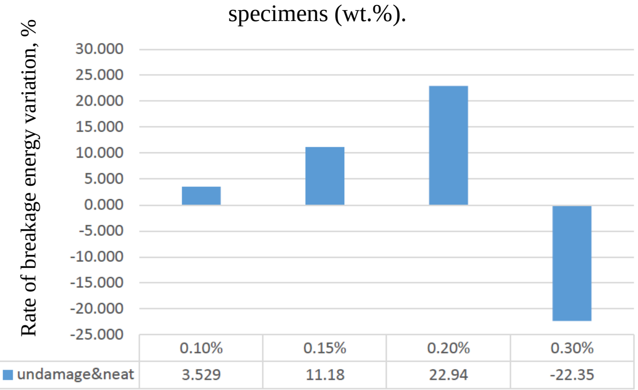
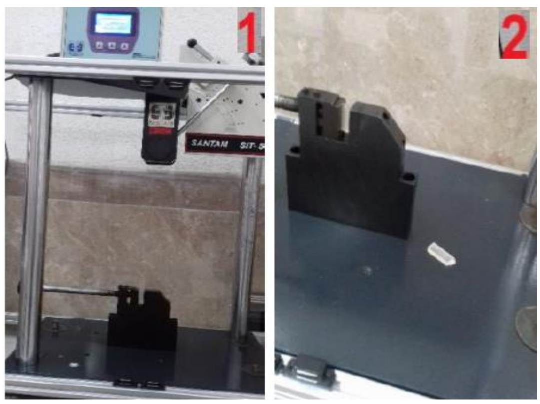


Figure 23. Rate of toughness result changes for undamaged specimens (wt.%).



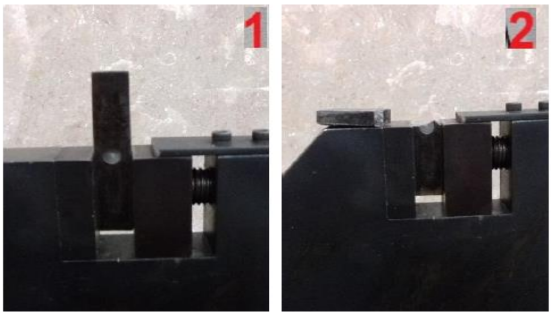


Figure 24. Izod test procedures with standard samples.

- SEM analysis results

After analysing SEM images, agglomeration and pothole are more significant, where agglomeration in higher content of NGO causes more increase in microcracks whose role of accumulation is undeniable even after the machining process, while pothole at higher NGO contents causes an increase of microcracks. As seen in Figs. 25-28 after mechanical tests and before, there aren't any cracks, which means machining has less impact on increasing microcracks, leading the nano-composite to unexpected failure. However, accumulation and potholes become intense after the machining process and decrease the mechanical properties at higher contents.

Figures 25-28 show crack growth around the end mill and cylindrical radius end, in respect. The growth of cracks in milled specimens with the end mill tool caused more microcracks than the cylindrical. As mentioned in previous sections, this manner can be explained by the nature of the tool and its more destructive effects on specimens, which caused more residual stress that increased the number of microcracks compared to the cylindrical radius end tool. Also, the NGO around the hole has a restraining role against crack growth. The reason might be the well-dispersed NGO particles into matrices, where NGO particles play as curing agent into crack areas which cure the cracks and make the crystalline connection to cover the cracks. This polymeric bonding structure can make the structure stronger than in milled neat epoxy specimens, NGO fills the crack gaps and crack lines which can be mentioned as a curing agent.

As soon as cracks grow because of applying concentrated force on mentioned areas, the healing role of NGO tries to hold the chain of polymer compound, which by filling the crack zone tries to increase the mechanical behaviour and prevent unexpected failure. As seen in Figs. 25-28 some of these micro cracks can have different origin. Here the main reason in this study are micro cracks made by the milling process. After understanding the effect of NGO in undamaged specimens with 0.2 % of NGO hit the most. In other words it enhanced mechanical features up to 38 % with the undeniable role of NGO. By comparing obtained result from Figs. 25-28 it can be found that all factors effective in toughness, here are also effective, which by comparing two tools and the hole depth helps understand the tool type effect in the machining process on specimens. Also by surveying the first tools and obtained results from tests recorded in Figs. 25-28, the milled specimens of 1 mm depth, have only 20% decrease in mechanical features recorded, but when the depth of the machining process increases to 3 mm, the effect of depth is very significant, which in other words is a 40 % decrease in mechanical features that is recorded.

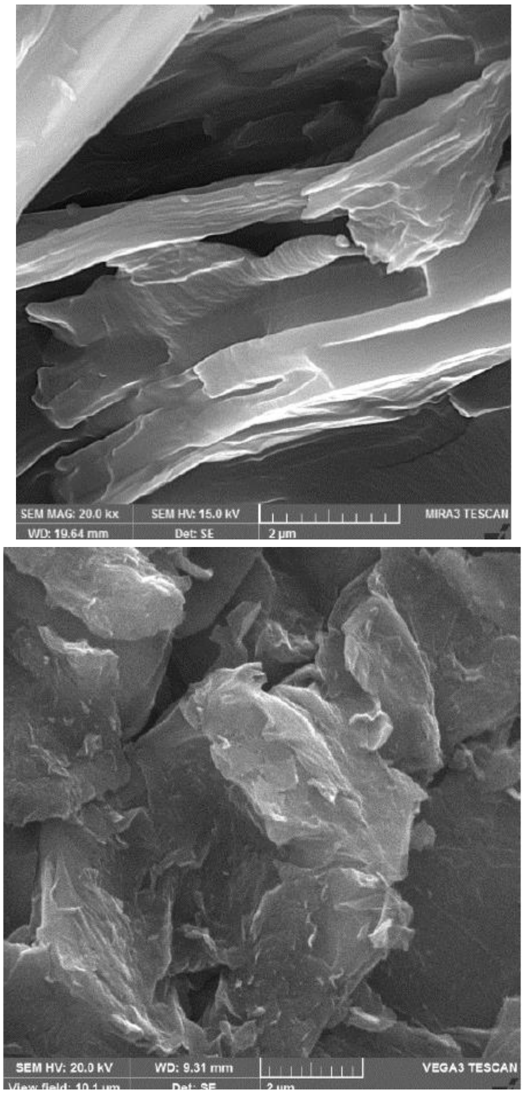
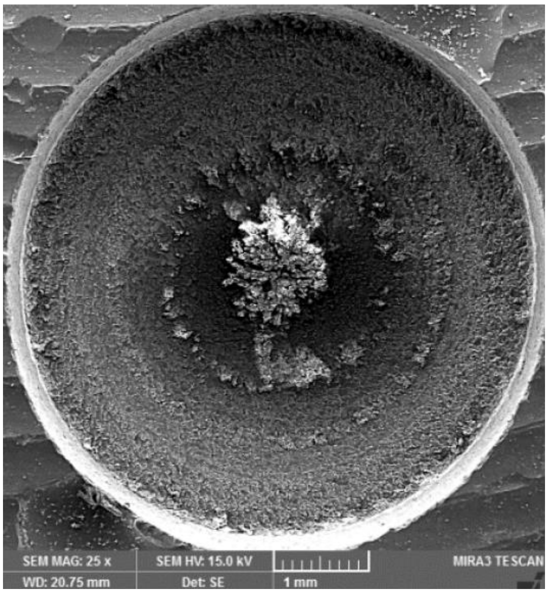


Figure 25. SEM image of NGO.

(a)



(b)

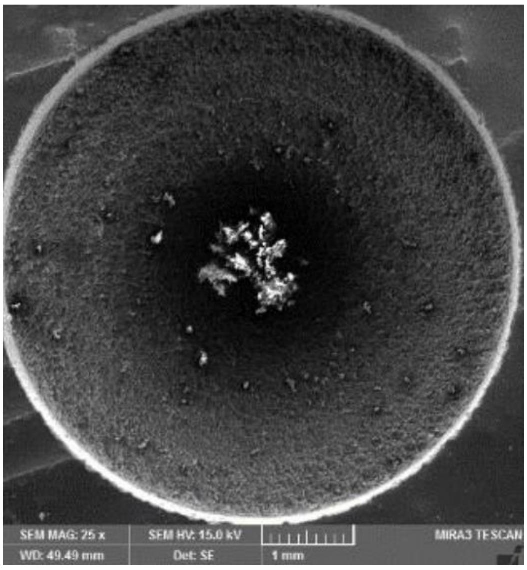


Figure 26. SEM image of NGO specimens after machining process for: a) neat; and b) contains NGO.

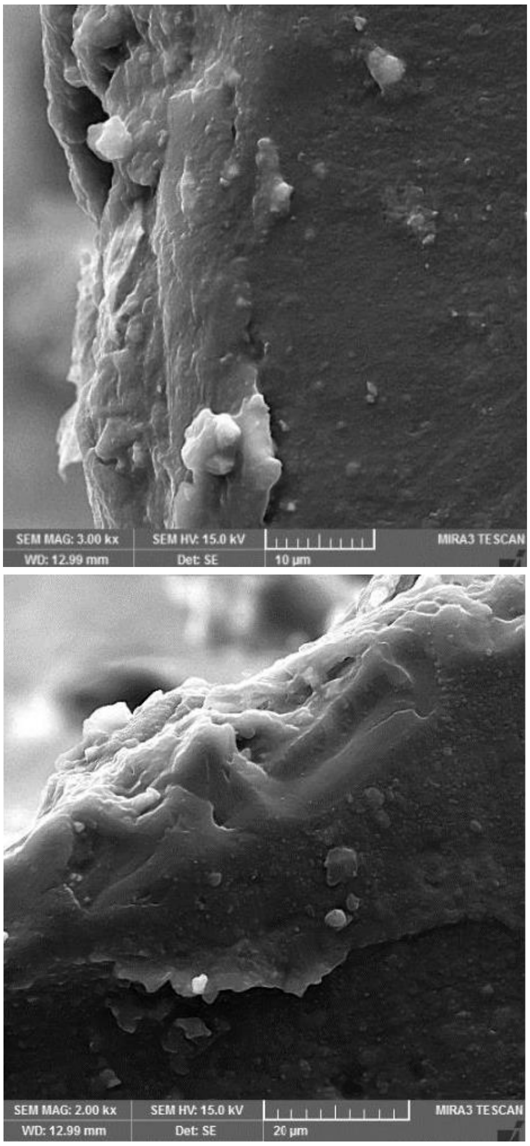


Figure 27. SEM image of undamaged neat specimens around machined hole before the test.

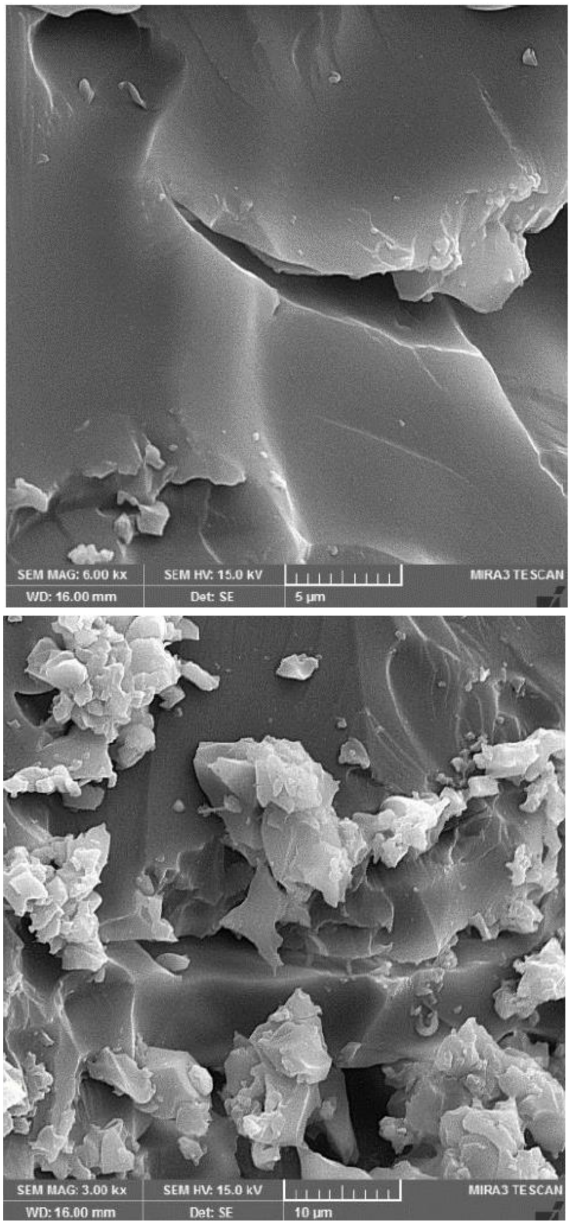


Figure 28. SEM image of undamaged neat specimens around machined hole after test.

CONCLUSION

In summary, a thermosetting polymer (EPON 862) reinforced with NGO is shown to dramatically enhance the fracture properties (fracture toughness and energy release rate) compared to the significant neat improvements, 23 % values are recorded for up to 0.15 wt.% NGO loading. The likely mechanisms for such high increase are explained through a crack deflection mechanism corroborated with evidence of graphene pull-out and void formation by using SEM, which are deemed significant contributors to the increase in fracture properties. It would be helpful to study the changes in the fracture properties of nanocomposite specimens after better dispersions of NGO in organic solvents, which might lead to even better results. The EPON 862 thermoset epoxy system shows significant potential to be modified with small NGO loadings for delamination toughness improvements in fibre-reinforced composite specimens. Work is currently underway to use NGO reinforced EPON 862 system in conjunction with carbon fibres to produce high toughness lightweight composite laminate systems for macro scale aerospace and automobile applications.

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