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Improving the Flexural Properties of E-Glass Fibers/Epox y Isogrid tiffened Composites through Addition of 3-Glycidoxypropyltrimethoxysilane Functionalized Nanoclay

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Abstract

This research work investigates the effect of silane functionalized nanoclay on the flexural response of E-glass/epoxy isogrid stiffened composite (IGSC). To do so, the sodium montmorillonite nanoclay were functionalized by 3glycidoxypropyltrimethoxysilane as a silane coupling agent. In the following, by using the mechanical stirrer and ultrasonic waves, the surface functionalized nanoclay particles with the different weight percentages (0, 1, 3, and 5 wt.%) were dispersed into the epoxy resin. After that, the IGSC panels containing the functionalized nanoclay were made by combining the filament winding and hand lay-up methods. By using the Fourier transform infrared (FT-IR) analysis, the functionalizing trend of nanoclay particles was characterized. It was realized that by adding 5 wt.% functionalized nanoclay to IGSC, the flexural load, flexural stiffness and energy absorption of that were reached to 1402 MPa, 82.6 N/mm and 233.9 J/kg, respectively, which were the maximum improvement of those properties. By using electron microscopy examinations from the fracture surfaces of IGSC structures, it was found that the presence of silane functionalized nanoclay particles improved the interfacial adhesion between the epoxy matrix and E-glass fibers.

Keywords: Silane coupling agent, Surface modification, Isogrid stiffened composites, Multi-scale composites, Nanoclay particles

1. Introduction

Composite materials due to their especial properties such as high specific strength and stiffness are widely used in various industries [1–3]. One of the most famous composite structures is the grid stiffened composites (GSC), which have unique features like high specific strength and stiffness, high capability to absorb the energy and proper resistance in corrosion media. Therefore, they can be substituted to the different parts in the various applications like airplane wings, solar array substrates, ship and car frameworks [4–6]. It is worth noting that these structures have been composed of ribs and skin. The ribs act as a load bearing elements, whereas the skin satisfies the design requirements [7]. Given up the reticulation types of the ribs, the GSC structures can be classified into various geometries, which the major group of these geometric patterns are the isogrid and anisogrid skeleton [8].

One of the newest methods for improving the mechanical properties of the polymeric matrix composites is using nanoparticles, which improves the stiffness, strength and energy absorption of those. Generally, adding nanoparticles especially nanoclay particles in the polymeric matrixes improves the energy absorption under dynamic and static loadings [9, 10]. Kanny et al. [11] investigated the reinforcing effect of amine functionalized nanoclay particles on the flexural properties of glass fibers/epoxy composites. They reported that by adding 3 wt.% nanoclay, the flexural strength of glass fibers/epoxy composites approximately increased 12%. Anbusagar et al. [12] found that the inclusion of 4 wt.% of nanoclay in the glass fibers/polyester composite increased the 40% flexural strength and 57% flexural modulus. By

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increasing the content of nanoclay particles, the flexural properties of composite were decreased, due to the agglomeration of those.

In the study of Alamri et al. [13] was reported that the impact strength of the cellulose fibers/epoxy composite filled with 1 wt.% of nanoclay was 17% higher than that of pristine composite. Assaedi et al. [14] showed that maximum increment in the fracture toughness and flexural strength of flax fibers/ geo-polymer composite was obtained by adding 2 wt.% nanoclay into that. In the study of Mohan et al. [15], by adding 5 wt.% of nanoclay, the limit velocity of glass fibers/ epoxy composite was improved about 42%. Also, in the similar research [16], the highest absorbed energy under the impact test was reported for glass fibers-epoxy composite containing 5 wt.% nanoclay.

Up to now, investigating the geometrical parameters and optimizing of those in the GSC structures are the subjects, which have been done by different researchers. As far as we know, there are few studies addressing the effect of adding nanoparticles on the mechanical properties of isogrid stiffened composite (IGSC) structures. Therefore, in the present study, 3-glycidoxypropyltrimethoxysilane (3-GPTMS) was firstly employed to functionalize the nanoclay particles. After that, the effect of adding nanoclay particles on flexural properties of the E-glass/epoxy IGSC panels were assessed.

2. Experimental Section

2.1. Raw Materials

In order to fabricate the IGSC structures, the diglycidyl ether of bisphenol-F (epoxy resin) with the trade name of ML-506 was selected. To cure this resin, the polyamine harder with trade name of HA-11 was used. The epoxy resin and hardener of that were provided from Mokarrar Engineering CO as Iranian epoxy producer. It is worth noting that this resin was

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selected due to having the low viscosity (1450 cP) and proper gel time (60 min) for fabricating the IGSC structures at the ambient temperature. To reinforce the rib in this research, the glass fibers (diameter of 12 μm , density of 2.58 g/cm^3) in form of unidirectional was purchased from Lintex, China. Also, E-glass fabrics in form of plain weave (surface density of 400 g/m^2) was supplied from Lintex Company for using in the skin part of IGSC structures. The sodium montmorillonite nanoclay particles as filler were purchased from Sigma Aldrich Co, USA. The properties of supplied nanoclay particles are shown in Table 1. These nanoparticles were functionalized by 3-Glycidoxypropyltrimethoxysilane as silane coupling agent (Merk, Germany). This coupling agent has epoxy ring in its structure, which proper for using in the epoxy based composites. It is worth noting that the molecular structure and weight of that are $\text{C}_9\text{H}_{20}\text{O}_5\text{Si}$ and 236.4 g/mol , respectively.

Table 1. Details of the purchased nanoclay particles

Thickness (nm)	Length (μm)	Density (g/cm^3)	Moisture Content (wt.%)	Specific surface area (m^2/g)
1–5	7	2/86	4–9	270

2.2. Fabrication of Silicone Mold

By adding accelerator into silicon monomer, the flexible polymer is formed in contact with air. For fabricating the mold, the poly vinyl chloride (PVC) sheet as the initial template was manufactured by using CNC machine (Fig. 1a). In the next step, the silicon and accelerator mixture were poured onto the surface of template. Necessary time for curing the liquid silicone was approximately 6 h. After that, the fabricated silicone mold was separated from template (Fig. 1b).

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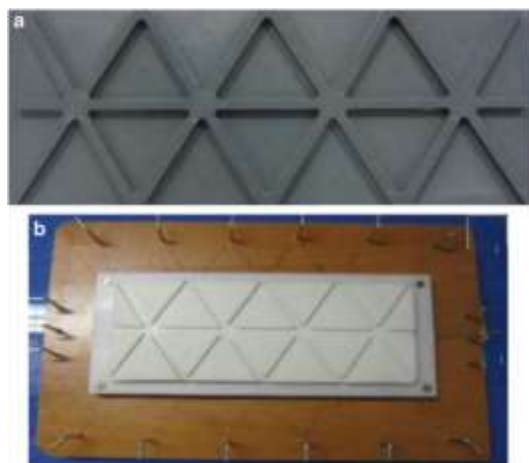


Fig. 1. a) Template used for making mold, b) Fabricated silicone mold.

2.3. Surface Functionalization of Nanoclay Particles

To improve the interaction between the nanoclay particles and epoxy, the surface functionalization of nanoparticles is essential. In the first step, 5 g of nanoclay particles were added to the 100 ml solution (consist of 95% ethanol and 5% distilled water). In the next step, the silane coupling agent with ratio of 1:1 was added to the mixture. The resulting mixture was subjected to the ultrasonic waves for 10 min and then reflux operation was conducted at 80 °C for 8 h. The pH of solution was set in 4–5 by adding hydrochloric acid. After performing the reflux process, the obtained mixture was placed on a centrifuge device under the speed of 4000 rpm for 30 min. In the final step, the obtained powders were washed three times with ethanol to remove the extra coupling agent and then were placed inside the oven at 80 °C for 12 h.

2.4. Fabrication of IGSC Panels

After the functionalizing process, the nanoclay particles were added into the epoxy resin with different percentage (0, 1, 3 and 5 wt.%). For achieving the high distribution of nanoclay particles and reducing the agglomeration of those in the epoxy resin, the nanoclay should be uniformly dispersed into the epoxy, which was previously confirmed in another research work

[17]. Based on the optimized parameters in distribution process, the 3-blade mechanical stirrer (Finetech, Korea) with the rotation rate of 2000 rpm for period time of 20 min was firstly used. After that, the sonication process with frequency of 24 kHz and power of 120 W was performed by a probe sonicator (Fapan, Iran) for 90 min. By using the vacuum oven for 60 min, the created bubbles during the mixing and sonicating processes were removed. Finally, as per the producer recommendation, the polyamine curing agent with a weight ratio of 100: 15 was added into the mixture. IGSC specimens were made by combining the filament winding and hand lay-up methods. For this purpose, as shown in Fig. 2a, the special fixture was used for fabrication of specimens. Then, 15 layers of glass fibers, which impregnated with the epoxy resin and nanoparticles mixture were placed inside the ribs (Fig. 2b). The ribs length, ribs angle and volume fraction of fibers in ribs were 60 mm, 60° and 44%, respectively. After that in order to formation of the skin, 4 layers of E-glass woven fabrics impregnated with the same mixture were placed upon the ribs. The volume fraction of fibers in skin was 48% and its thickness was 1.8 mm. The initial curing time of the specimens for bringing out IGSC from the mold was 3 h. To reach the highest strength and ultimate curing, according to the recommendation of resin producer, the flexural test of the IGSC panels was carried out after 7 days. The fabricated specimens with and without nanoclay particles addition are illustrated in Fig. 3.

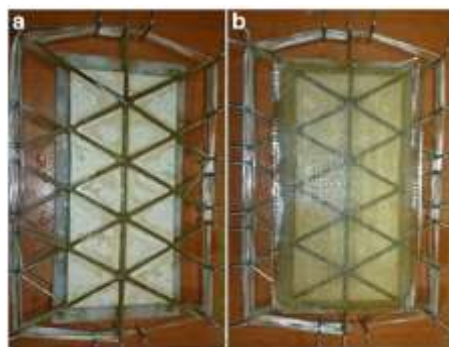


Fig. 2. The main stages for fabrication of IGSC panels, a) Laying up the ribs, b) Laying up the skin.

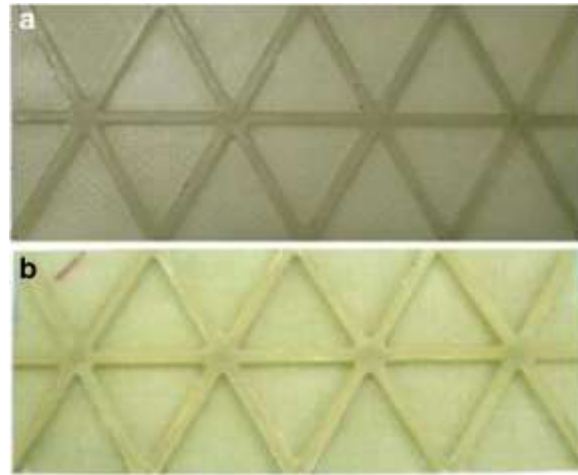


Fig. 3. IGSC specimens, a) without nanoclay, b) containing silane functionalized nanoclay.

One of the important factors in the ribs and skin of IGSC panels is the void content of those. In this regard, the void content was calculated according to ASTM D2734 standard. As per the rule of mixtures, the theoretical density can be calculated by eq. 1:

$$\rho_t = V_f \rho_f + V_e \rho_e \quad (1)$$

Where, the ρ_t , ρ_f , V_f , ρ_e and V_e are the theoretical density of composite, density of fibers, volume fraction of fibers, density of epoxy resin, and volume fraction of epoxy resin, respectively.

Based on ASTM D2734 standard, the void content can be calculated by eq. 2:

$$V = 100 \times \frac{\rho_t - \rho_m}{\rho_t} \quad (2)$$

Where, the V and ρ_m are the void content and the measured density of composite, respectively.

The density of composite was calculated by Archimedes' method. The density of glass fibers and epoxy resin were 2.58 and 1.11 g/cm³, respectively. Also, the measured density of ribs and skins by Archimedes' method were 1.73 and 1.79 g/cm³, respectively. Based on the eqs. 1 and 2, the void content of rib and skin were 1.70 and 1.65%, respectively.

2.5. FT-IR Test

In order to verify the surface modification, which was performed on nanoclay particles, the formation of functional groups on the surface of the nanoparticles and also check out the taken possible reactions, the FT-IR spectrometer instrument (Jasco, 460 plus) in the range of 400–4000 cm^{-1} with sensitivity of 4 cm^{-1} was used.

2.6. Three-Point Bending Test

In this study, to investigate the flexural behavior of the IGSC panels, three-point bending test based on the ASTM D7264 standard was performed. At the first, a fixture was designed and constructed. For all tests, the distance between supports was constant and considered equal to 250 mm, so that, the ratio of support span to thickness for all samples was 32:1. In the following, all samples from the middle part (according to Fig. 4) were exposed under quasi-static load. The load rate was set on 5 mm/min. In order to more accuracy in obtained data, the test was repeated five times for each sample.

Due to the inhomogeneous cross-section of IGSC specimens and based on conducted researches [18] after performing the bending test, the linear slope of the load displacement graphs for evaluating the stiffness, the obtained maximum load for assessment of the load bearing capacity and the area under load-displacement graphs for evaluating the energy absorption of the IGSC panels were used. As regards in IGSC structures, the weight of the structure is an effective parameter. Therefore, the obtained values for energy absorption were divided to the total mass of structure.

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Fig. 4. IGSC specimen in the fixture of three-point bending test.

2.7. Microscopic Studies

In order to examine the failure surface and characterize the effective mechanisms on the flexural properties of the IGSC panels, the field emission electron microscopy (FESEM-Hitachi S-4160) under voltage of 25 kV was used. To improve the conductivity of the specimens, a thin gold coating was applied to them in a vacuum chamber.

3. Results and Discussion

3.1. Results of FT-IR Analysis

The FT-IR results of unmodified nanoclay particles, silane coupling agent and surface modified nanoclay particles are shown in Fig. 5. In the FT-IR curve of un-functionalized nanoclay particles, a peak near the 3444 cm^{-1} is observed, which belongs to the stretching vibrations of hydroxyl groups. Also, the appeared peak in 1649 cm^{-1} can be attributed to the bending vibrations of H-O-H. It should be noted that the peak of 3636 cm^{-1} in this curve

corresponds to the stretching vibrations of bonding between OH and Al [19]. The appeared peak at 1058 cm^{-1} represents the stretching vibration of Si-O-Si and Si-O groups [20]. Also, the observed peaks at 526 and 471 cm^{-1} are due to the bending vibrations in Si-O-Si and Si-O-Al groups [21]. The visible bond in 802 cm^{-1} belongs to the symmetric stretching of Si-O-Si.

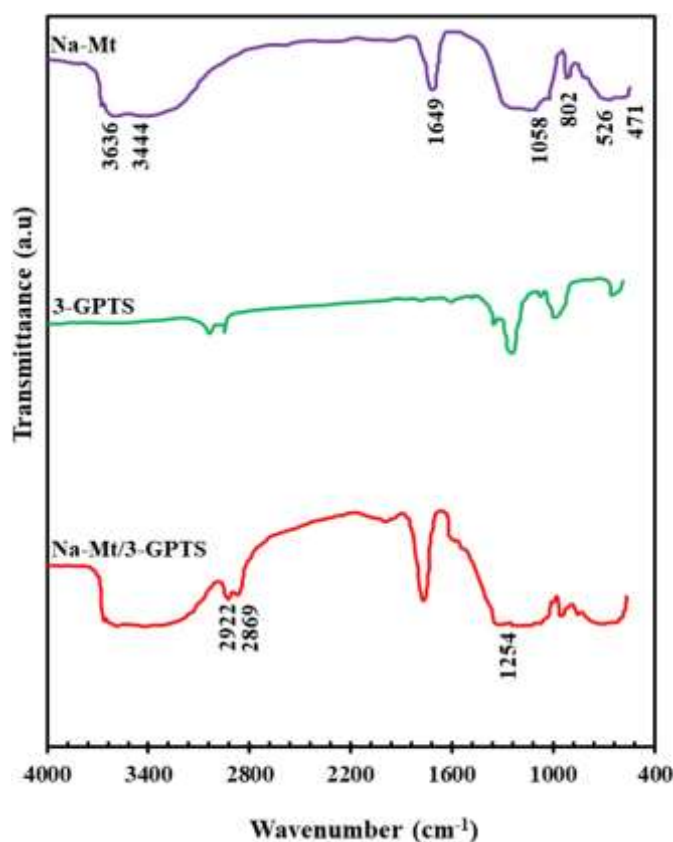


Fig. 5. FT-IR curves of the unmodified nanoclay particles, silane agent and modified nanoclay particles.

To realize the interaction between the silane coupling agent and nanoclay particles, the schematic reactions between them in the ethanol-water media including hydrolysis and condensation reactions have been shown in Fig. 6. Based on this figure, it was observed that the existing hydroxyl groups (OH) in the silane agent reacted with OH groups on the surface of nanoparticles. So when the surface modified nanoclay particles were dispersed into the

epoxy matrix, the reaction between existing functional groups on the surface of nanoparticles and epoxy matrix was performed in the presence of polyamine hardener. Accomplishment of these reactions was led to the formation of covalent bond between matrix and nanoparticles, which was caused the stronger interface between the matrix and nanoparticles [22].

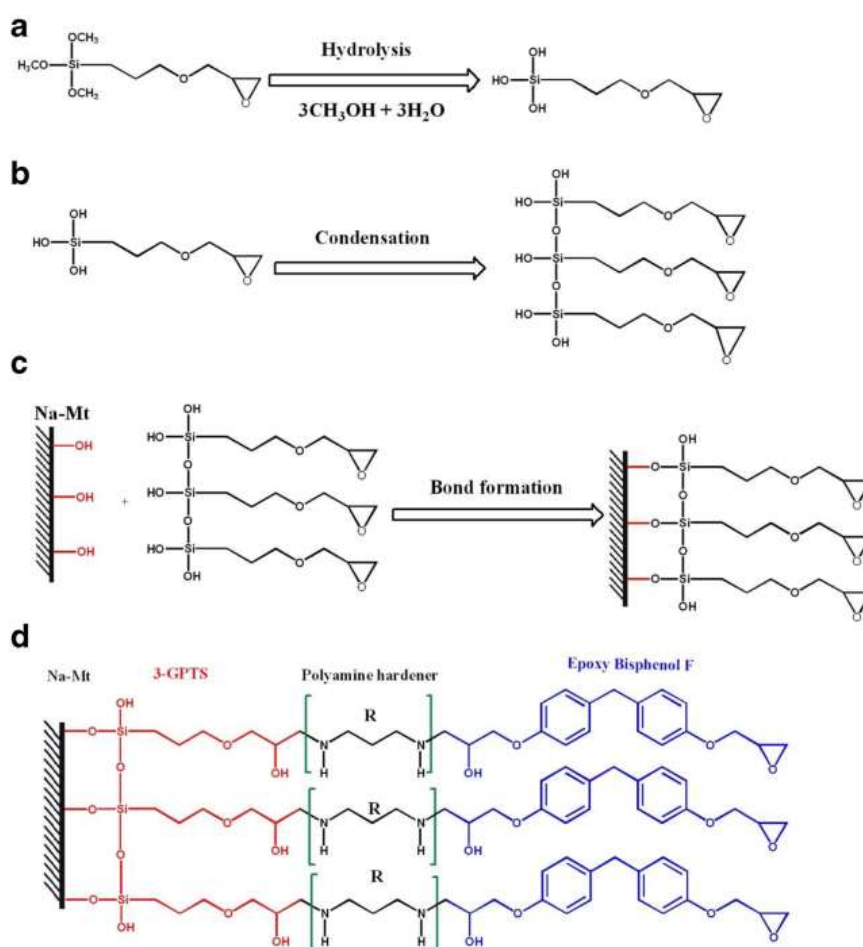


Fig. 6. Chemical reaction between nanoclay, silane coupling agent and epoxy matrix, a) hydrolysis, b) condensation, c) bond formation between nanoclay and coupling agent, d) chemical structure between 3-GPTS/Na-Mt and epoxy matrix in the presence of hardener.

3.2. Three-Point Bending Test Results

Three-point bending test results on IGSC panels with different weight percentages of surface modified nanoclay particles in the form of load-displacement curves has been

illustrated in Fig. 7. It is worth noting that all IGSC structures were loaded from the skin side up to displacement of 80 mm. According to Fig. 7, overall trend of the curves was similar to each other and addition of nanoclay particles only changed the maximum load, stiffness and energy absorption of IGSC panels. It is noteworthy that a significant proportion of energy absorption was occurred after the primary fracture at peak load, manifesting the high damage tolerance of the IGSC panels.

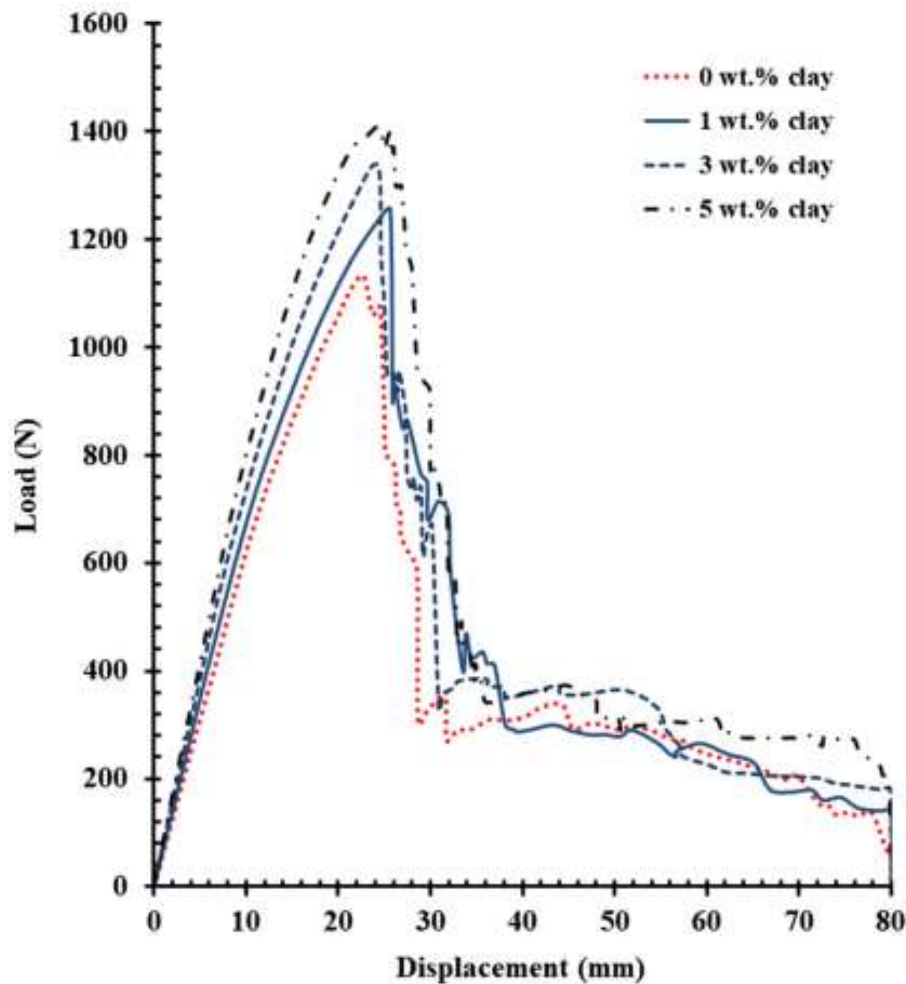


Fig. 7. Load-displacement curves of the IGSC panels with different contents of nanoclay particles under three-point bending test.

Different failure modes of IGSC panels under flexural loading from the skin side have been illustrated in Fig. 8. As previously mentioned, the ribs are main load bearing elements in IGSC structures. During the loading from the skin, longitudinal rib was firstly yielded and broken, which showed as maximum load in the load-displacement curve (Fig. 8a). After that, the complete failure of longitudinal rib as well as separation of helical ribs was occurred (Fig. 8b). After complete failure of the ribs, the applied load was tolerated by skin. Based on the other literature, the main failure mechanisms of IGSC panels under flexural loading were fibers breakage in ribs and fibers micro-buckling in skin [23].

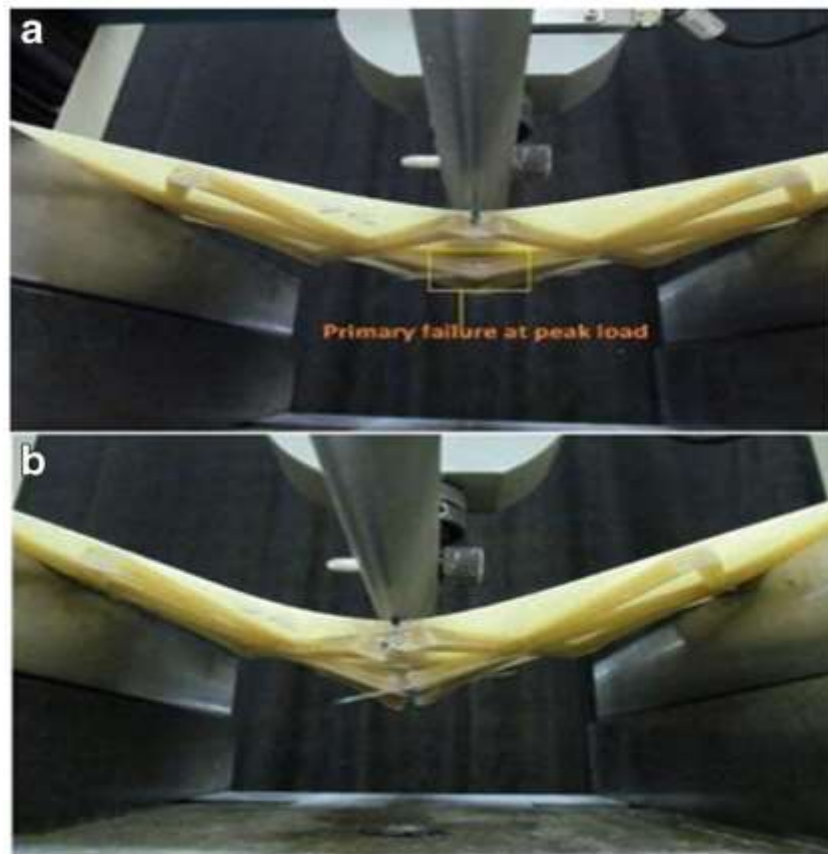


Fig. 8. Various failure modes of IGSC panels under flexural loading, a) Primary failure of the longitudinal rib, b) Complete failure of the ribs under loading nose.

In order to evaluate the effect of adding nanoclay particles on the flexural properties of IGSC panels, the related data to the maximum load, stiffness (slope of the curve in the linear region) and the specific energy absorption (area under the curve divided to the mass of the panel between the two support) were extracted from Fig. 7.

In Fig. 9, the maximum load graph versus different content of nanoclay particles has been shown. According to this graph, the addition of nanoclay particles into the epoxy matrix was caused a significant improvement in maximum load, so that by adding 5 wt.% of nanoclay particles, the maximum load was approximately 24% higher than that of without nanoclay sample. The improvement of the maximum load under flexural loading was caused by two factors. The first factor was due to the reinforcement effect of nanoclay particles in the polymer matrix of composite. In composites that have been reinforced with the fibers, a major part of the applied load is concentrated on the fibers [24]. The dispersion of nanoclay particles into the polymer matrix reduced the stress concentration on the surface of fibers and thus the requirement stress for fibers failure was increased. This is due to that with the reinforcing the matrix by nanoparticles, the greater part of the applied load was imposed by the matrix. The second factor was related to the characteristics of interface between matrix and glass fibers. When the nanoclay particles were added to the matrix of composite, the frictional slippage between matrix and fibers was limited, so that, the load transfer between matrix and fibers has more effectively occurred [17].

According to Fig. 10, by increasing the weight percentage of nanoclay particles, the specific energy absorption (SEA) of IGSC panels was continuously increased and the maximum improvement of SEA was related to the specimens containing 5 wt.% of nanoclay particles, which the SEA of that was 34% higher than SEA of pristine composite. It should be noted that when the crack reached to the nanoparticles, for spreading required the rotation or

torsion among the nanoparticles, which changed the crack growth path [25]. Therefore, the deviation of crack inside the matrix by nanoclay improved the energy absorption of composites.

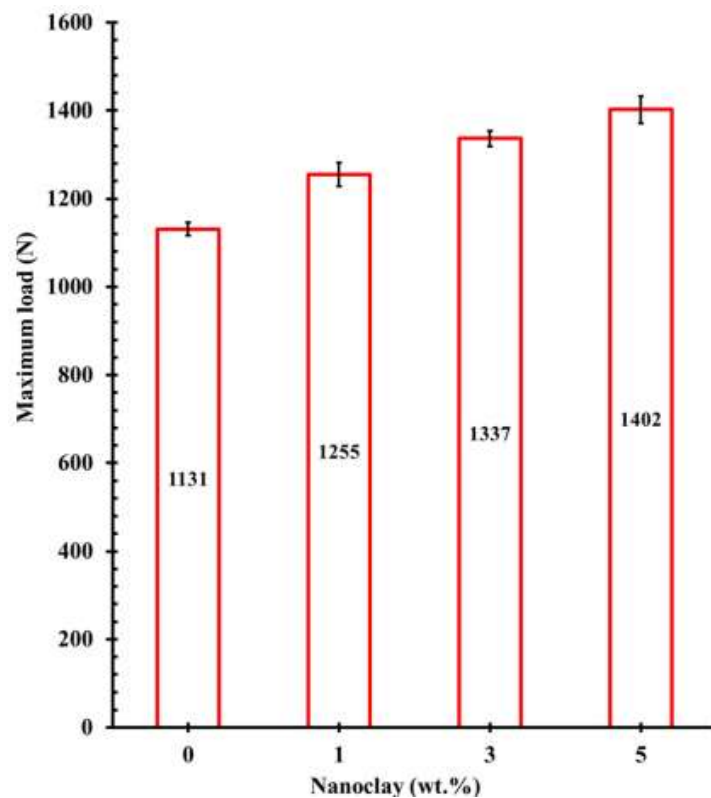


Fig. 9. Values of the maximum load for IGSC panels with different contents of nanoclay.

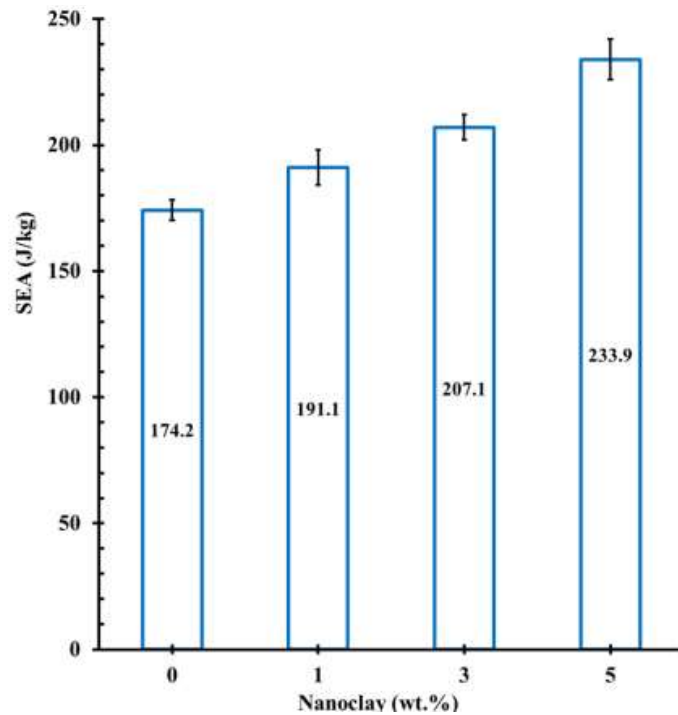


Fig. 10. Values of energy absorption for IGSC panels with different contents of nanoclay.

Figure 11 shows the relation of flexural stiffness of IGSC panels versus content of nanoclay particles. Given up Fig. 11, by including of 5 wt.% of nanoclay into the epoxy matrix, the flexural stiffness was increased about 32%. The reason of this improvement can be due to the much stiffer nature of nanoclay compared with the epoxy. The silane functionalized nanoclay can prevent the polymeric chains slippage. Therefore, the interaction of the silane functionalized nanoclay with epoxy chains can be another reason for improving stiffness.

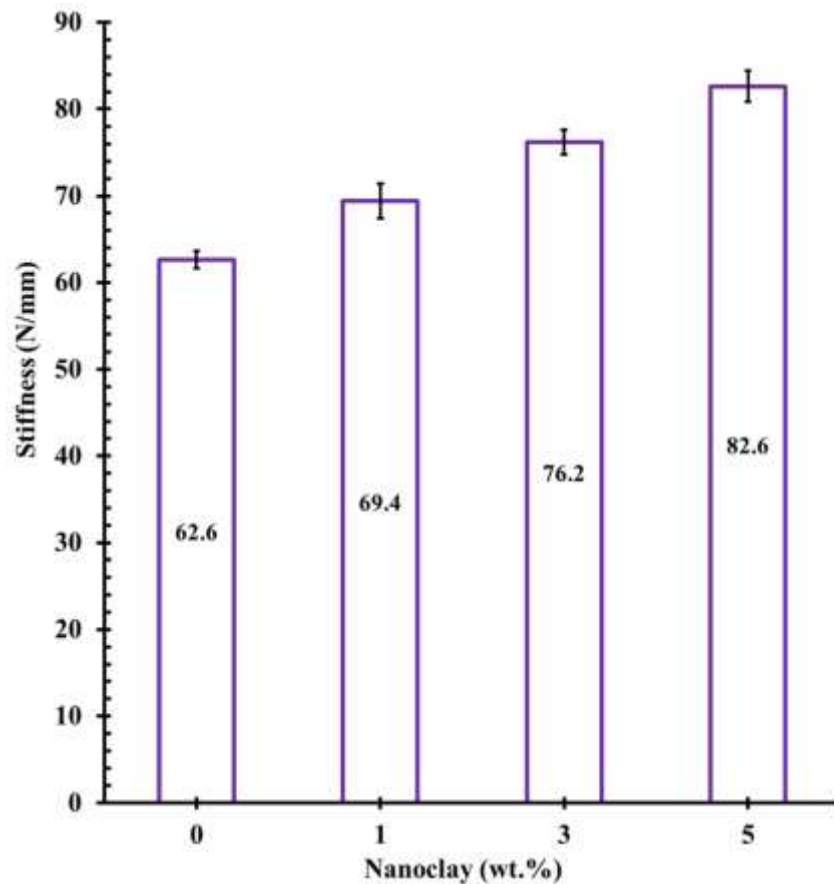


Fig. 11. Values of the flexural stiffness for IGSC panels with different contents of nanoclay.

3.3. Microstructural Analysis

Figure 12 represents the FESEM images from the longitudinal rib and the adhesion between fibers and matrix. In the pristine IGSC structure (Fig. 12a), the surface of glass fibers was so clean, so that, the epoxy was entirely separated from the glass fibers. This behavior could be attributed to the poor adhesion between epoxy and glass fibers. Given up the Fig. 12b, it can be seen that the epoxy has been attached to the glass fibers. This means that the functionalized nanoclay improved the adhesion between epoxy and glass fibers.

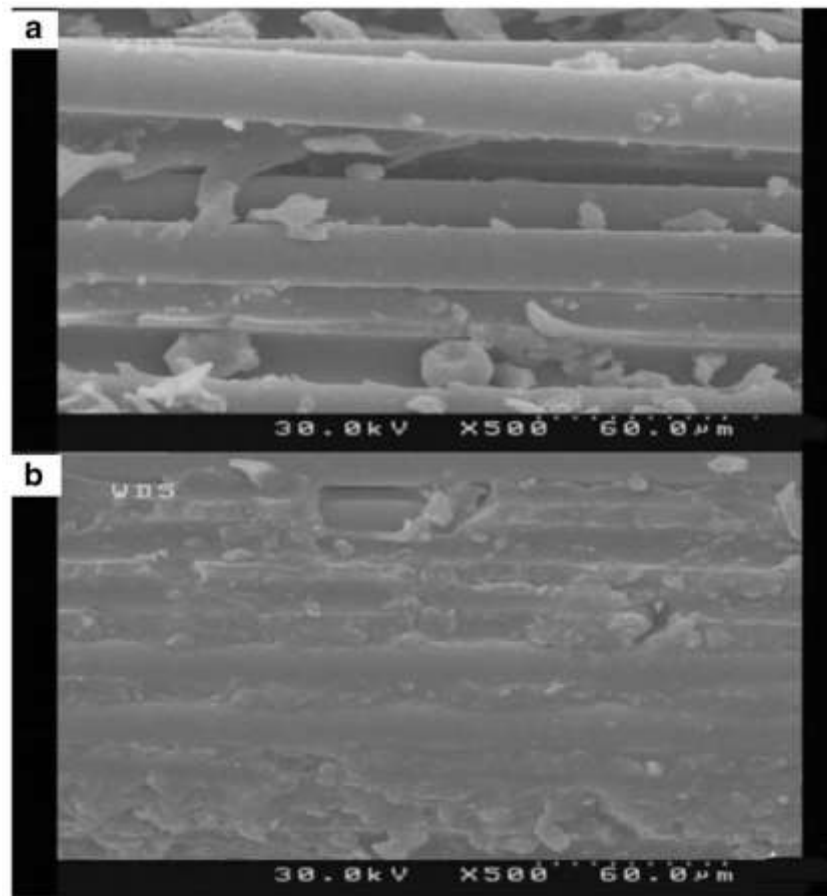


Fig. 12. Fracture surface of longitudinal rib of IGSC panel, a without nanoclay, b containing 5 wt.% of nanoclay particles.

In Fig. 13, the fracture surface of the matrix reinforced with 5 wt.% of silane functionalized nanoclay particles has been illustrated. Based on this figure, it could be observed that the fracture surface of sample including 5 wt.% nanoclay was rough. Also, the deflection of the crack path was observed. This was the main reason for improving the energy absorption of IGSC panels. In relation with specimens without nanoclay particles, the fracture surface of matrix was smooth, which is characteristic of brittle fracture (Fig. 14a). Improving in the adhesion between matrix and fibers and the failure mechanism of matrix has been shown in Fig. 14b.

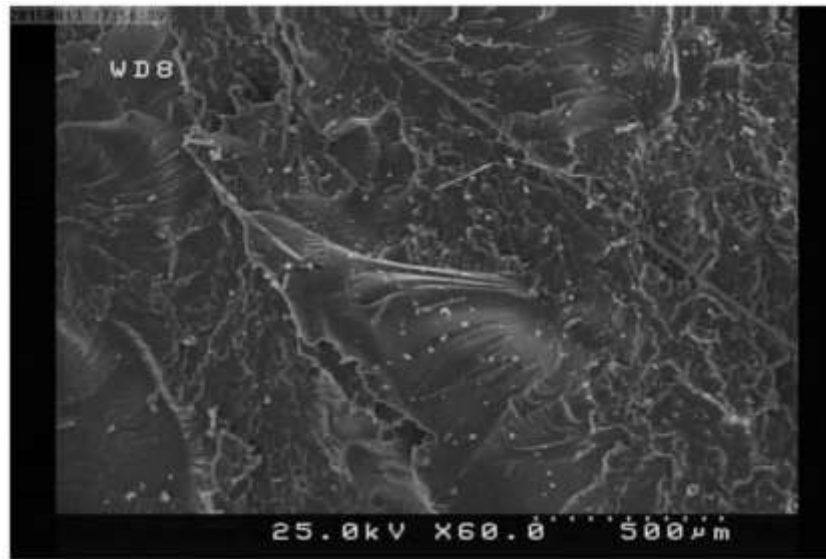


Fig. 13. Fracture surface of matrix of IGSC panel containing 5 wt.% of silane functionalized nanoparticles.

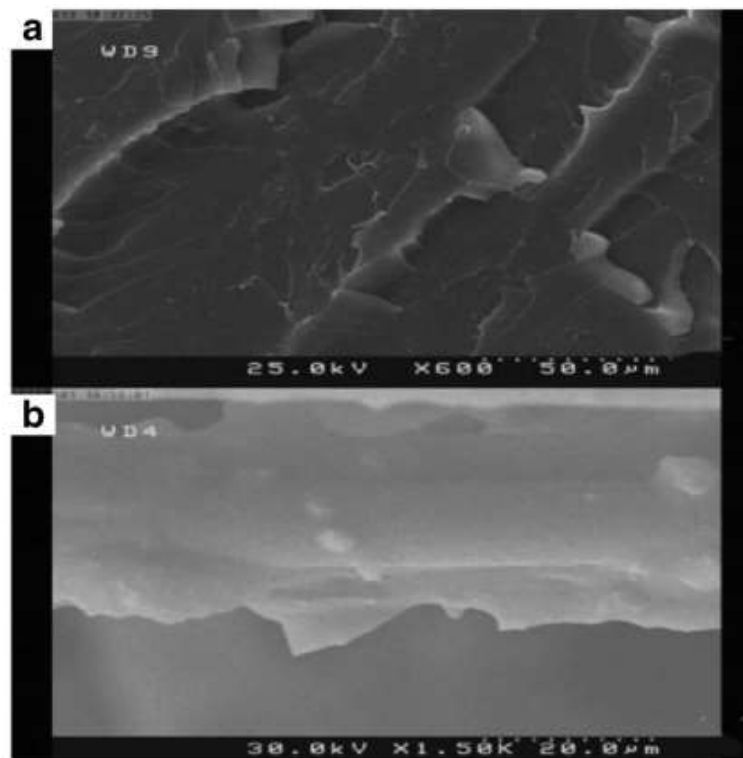


Fig. 14. A) Brittle fracture of epoxy matrix, b) failure mechanism of matrix in specimens containing nanoclay particles.

4. Conclusions

In the present study, the improvement of flexural properties of IGSC panels by the addition of 3-glycidoxypropyltrimethoxysilane functionalized nanoclay particles was investigated. Various weight fractions of nanoclay particles (0, 1, 3 and 5) were incorporated into the matrix. The results of this research work can be summarized as follows:

1. FTIR analysis results showed that the surface modification of the nanoclay was performed successfully.
2. Three-point bending test results showed that by adding nanoclay into the epoxy matrix, the flexural properties the IGSC panels were improved. The highest improvements in flexural stiffness, load-bearing capacity and the energy absorption were observed for sample containing 5 wt.% of nanoclay particles, which were 32, 24 and 34%, respectively.
3. The results of FESEM studies indicated that the addition of nanoclay particles led to better adhesion between matrix and fibers, which had a significant effect on the improvement of the flexural properties of IGSC panels.

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