RESEARCH PAPER

An Experimental and Analytical Investigation of Novel Nanocomposite Reinforced with Nanoclay with Enhanced Properties for Low Velocity Impact Test

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ARTICLE INFO

Article History:
Received 06 October 2019
Accepted 03 December 2019
Published 01 January 2020

Keywords:
Epoxy
Impact
Hybrid composite
Mechanical properties
Nanoclay
Woven glass fiber

ABSTRACT

The application of nanoparticles in order to enhance the composites properties has been recently attracted many researchers' attentions. To increase the mechanical and physical properties of the composites, the nanoparticles have no significant effect on the weight and nanostructure of composites. One of the well-known nanoparticles is the Nanoclay (NC) that have been widely used in industries due to its unique geometric shape and some specific chemical properties. In this research, the effect of NC on the mechanical behavior of epoxy hybrid/glass fibers composite has been investigated. Samples containing (1, 2, 3, 5 and 7) wt% of NC with constant amount of epoxy hybrid /glass fibers composite were produced. Samples with 3 wt% NC has shown proper impact, tensile properties. The scanning electron microscopy (SEM) and X-ray diffraction (XRD) technique were used to analysis the morphology and phase characterization of the samples. The sample with 3 wt% NC shows elastic modulus, ultimate tensile strength and ultimate flexural strength as 2.5 GPa, 20 MPa and 50 MPa, respectively. Ballistic impact test on the samples demonstrated that initially as the NC increased, the mechanical properties also increased. The results of tensile, bending and impact tests on hybrid samples showed improvement in these properties compared to the primary sample without nanoparticles incorporated. On the other hand, the energy absorbed by the target was increased, due to the sensitivity of the glass fiber to the strain rate. The novel hybrid epoxy/glass fibers composite reinforced with 3 wt% NC present proper mechanical properties compared to another specimen.

How to cite this article
Bagheri Baba-Ahmadi S, Hashemian M, Khosravi M, Khandan A. An Experimental and Analytical Investigation of Novel Nanocomposite Reinforced with Nanoclay with Enhanced Properties for Low Velocity Impact Test. J Nanostruct, 2020; 10(1): 92-106. DOI: 10.22052/JNS.2020.01.011

INTRODUCTION

Non-metallic composites or polymeric base materials are one of the most advanced materials in mechanical and materials engineering products. Reason for the following excellent physical and mechanical properties is low weight, high strength, high stiffness [1-5]. During recent years, a lot of efforts have been done to make composite materials due to the improvement of mechanical properties [6-7]. Over the past few years, studies on the effects of nanoparticles on polymer composite have been done by Gilbert et al. [1-2]. By adding different volumetric percentages of iron nanoparticles to the polymer matrix or carbon

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nanotube (CNT) the fracture toughness and the cutting strength of the interlayer increases [7-14]. The effect of the addition of clay nanoparticles was investigated by Timmerman et al. [3], which significantly improved the resistance to thermal cycles and the thermal expansion of carbon/epoxy composites by adding Nano layers of clay. The addition of nanoclay (NC) caused the reduction of cracks in width. Haque et al. [4] examined compound properties of nanocomposite and CNT/epoxy. They showed that the composite material with 3 wt% additives have proper flexural modulus and flexural toughness on static breakdown which increased from 26% and 60%, respectively. Shim et al. [5] studied the effect of NC on the mechanical properties of two different types of epoxy resin. The first type is a rubber type (glass transition temperature below the ambient temperature) and the second type of glass (glass transition temperature above the ambient temperature). The results of the X-ray diffraction and transmission electron microscope indicated that the diffusion and epoxy resin is better than glass epoxy because of its low buckling rate and longer, hardening molecules. Boohung-gu et al. [6] studied the thermal and rheological properties of epoxy/NC for use in the VARTM (Vacuum Assisted Resin Transfer Molding) method. Three types of NC (C30B, C15A, C25A), along with an epoxy resin specially has been used. The results showed that the type of NC has no effect on the properties studied. Although the presence of NC has increased its viscosity. Tan et al. [7] examined the effect of adding NC particles on the mechanical properties of carbon fiber-reinforced epoxy-coated composites. They reported the effect of adding NC particles on the initial fracture toughness and crack expansion mode of these composites. According to their research results, the addition of NC has doubled the fracture toughness of the samples. The NC also have good potential application to use as low cost and ease of use in polymer composites [8-9]. Shim et al. [5] also added CNTs to the composite matrix to enhance the interlayer breakthrough toughness. The CNTs increase the specific electrical and mechanical behavior, such as the long-length modulus variation and high tensile strength of up to 150 GPs [10-13]. The unique properties of CNT with its low weight make it a good augmentation factor in nanocomposites. Cheeseman et al. [14] studied the mechanical properties of these materials by adding various types of Nano materials, including nan silica and carbon nanotubes, to composites with epoxy and glass fibers. They reported less impact of Nano materials on the interlayer-tensile strength of composites compared to other mechanical properties. Carlsson et al. [15] investigated the effect of high nanosilica percent on tensile properties, interlayer shear and first and second modes of fracture toughness between layers of composites with epoxy and carbon fibers. In their reports, they reported improvement in the fracture toughness of the first mode for increasing the nan silica percent. Various technique was used like sol-gel, combustion and mechanical activation technique for preparation of nanomaterials [16-18]. However, the reduction of the interfacial fracture toughness was achieved by increasing the amount of Nano silica and synergistic component [19-21]. There is various technique to estimate and predict the mechanical properties of the nanocomposite containing different metallic and non-metallic nanoparticles using molecular dynamic (MD) simulation [22-28]. The dynamic stability of the double-walled carbon nanotube (DWCNT) under axial loading has been investigated in several works [29,30]. Several researchers concluded that addition of nanosilica, nanoclay and carbon nanotube (CNT) has a good effect on the tensile properties as well as the toughness of the failure of nanocomposites epoxy using multi scale modelling [31-37]. They also improved the elastic modulus and tensile strength by adding nanomaterials to epoxy resin. They investigated the effect of adding nanomaterials on the first interlayer of the composite with basalt fiber and epoxy coatings. They reported the effect of nanoparticles on lower percentages of fiber volumes. Some works used organic silicate to strengthen epoxy/carbon fiber and with addition 2 wt% Nano-filler they could improve 10-15% hardness compare to pure epoxy [38-42]. In the current study, all mechanical properties of novel hybrid epoxy/glass fibers composite reinforced with nanoclay with enhanced properties for low velocity impact test were investigated for the first time.

MATERIALS AND METHODS

Materials preparation

In this study, the fabrication of compound nanomaterials, an Epoxy resin EPONE 828 produced by the Shell Chemical Company is

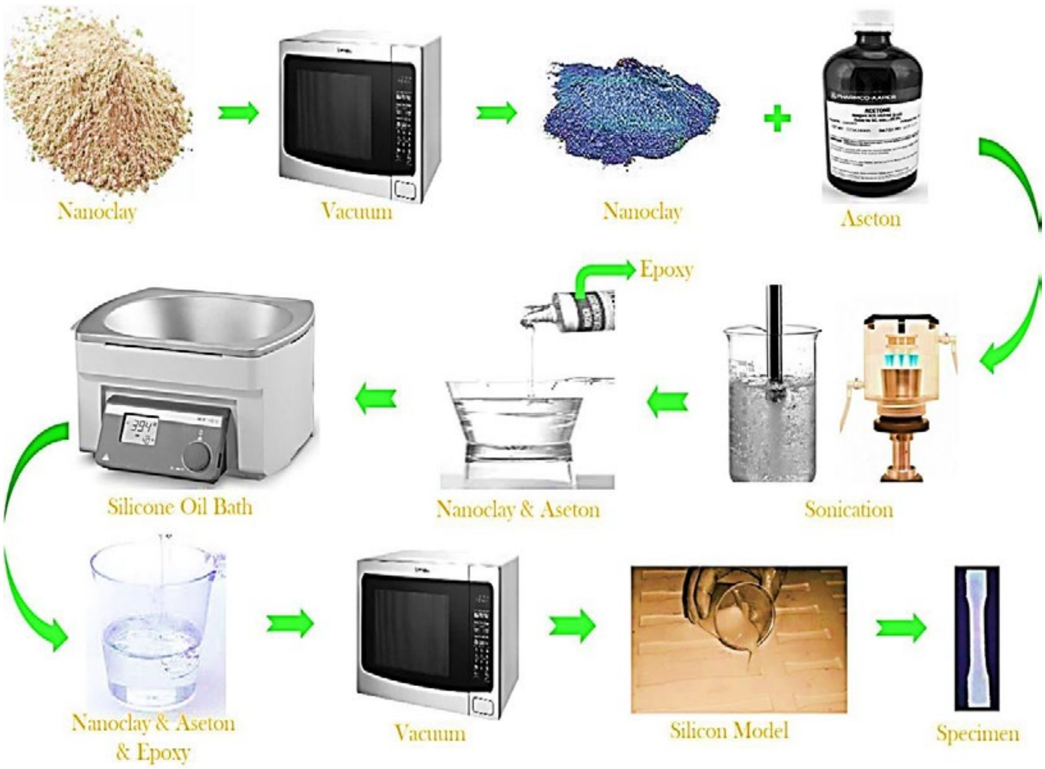

Fig. 1. Schematic of samples preparation for fabrication of NC-epoxy composite

used. Also, EPON 828 has two epoxy groups with an epoxy equivalent weight (EEW), 192 g/mol for each epoxide group. The cooking factor was set as D-400, with a molecular weight of 400 g/mol, which is a Huntsman Chemical Company product. Also, the materials mixed with a weight ratio of 55:100 to the epoxy base. The nanoclay (NC) proposed in the current study is CLOISITE 30B (natural modified montmorillonite with ammonium salt type IV). The following materials were purchased from American company Southern Clay Products Inc. This modified NC has the least degree of hydrophobicity among other types modified in this company.

Fabrication procedure

Two techniques were used for dispersion of NC in the resin and laminates. The first method is a soluble mixing with acetone solvent. First, the NC powders was placed in the oven for 24 (h) at a temperature of 80°C. Then, a certain amount of dried NC was poured into acetone. The acetone/clay mixture was sonicated for 30 minutes. The next step was addition of epoxy to the base materials. The mixture of epoxy/acetone/clay was mixed for 2 (h) in a bath of silicone oil at a temperature of 80°C. After stirrer the products for 2 (h), the mixture became clearer and homogenized. Then, for complete removal of acetone, the mixture was taken for 24 (h) at a temperature of 80°C in the vacuum oven. The second method is direct mixing, in which acetone solvent was removed. The cooking agent was first heated to 40°C to reduce its viscosity. Then, dried NC was added and the mixture was exposed to ultrasonic waves for a maximum of 35 minutes. The mixture was stirred at a rate of approximately 1000 rpm every 10 minutes to become homogeneous. Then, the mixture clay/cooking agent was added to the epoxy with 80°C and then was stirred for 5 to 15 minutes at 1000 rpm. The vacuum molding method (VARTM) was used to make a glass fiber composite. The schematic of samples preparation is shown in Fig. 1.

Mechanical testing

The experiments carried out in this study include the impact test using a gas gun at the Polymer Research Institute. Then, the uniaxial tensile test was performed according to ASTM-D3039

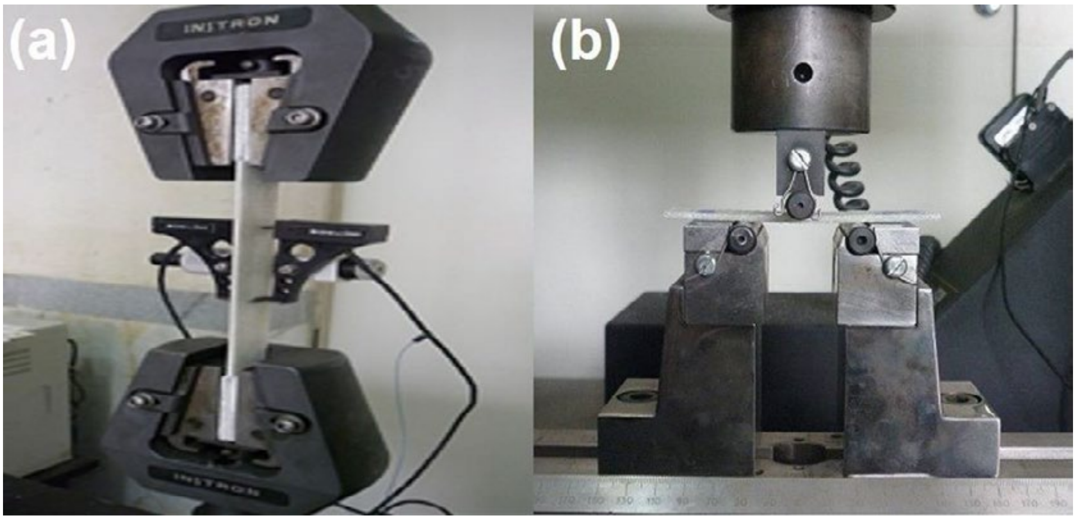

Fig. 2. (a) Tensile specimen measured using strain gauge and (b) Three-point bending test by INSTRON 5500R

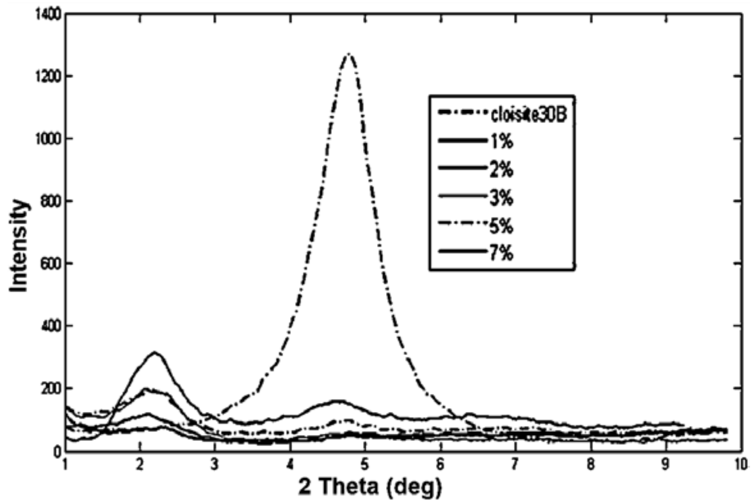

Fig. 3. The XRD patterns for resin samples produced using second method

standard. Finally, the specimens were produced and a tensile test was carried out at a loading rate of 2 mm/min (Fig. 2-a). These analyses were performed by the INSTRON 5500R machine at Tarbiat Modarres University. On the other hand, the three-point bending test was subjected to a bending load in accordance with ISO 178 standard (Fig. 2-b), which were done by the INSTRON 5500R test device of Tarbiat Modarres University. Finally, the scanning electron microscopy (SEM) and X-ray diffraction (XRD) technique were used to analysis the morphology and phase characterization of the samples at Tarbiat Modarres University.

RESULT AND DISCUSSION

XRD analysis

The results obtained from the XRD analysis for pure nanoparticles and epoxy resins show the nanomaterials inside the epoxy resin have Intercoolet shape as shown in Fig. 3. In this type of nanocomposite, clay particles, irrespective of their percentage, are dispersed in the form of regular layers with more interlayer heights than pure NC by placing their interlayer polymeric coatings. Fig. 3 shows the XRD for nanocomposite samples made by the second method (direct mixing method). The graph with a large peak at 2θ=4.75˚

Table 1. The XRD results for samples with two manufacturing methods

Nano clay percentage	%1		2%		3%		5%		7%	
	θ٢	d* (Å)	θ٢	d (Å)	θ٢	d (Å)	θ٢	d (Å)	θ٢	d (Å)
Soluble mixing method		-	-	-	-	-	-	-	2.16	40.76
Direct mixing method	2.16	40.76	2.06	43.63	2.19	40.36	2.09	42.23	2.17	40.69

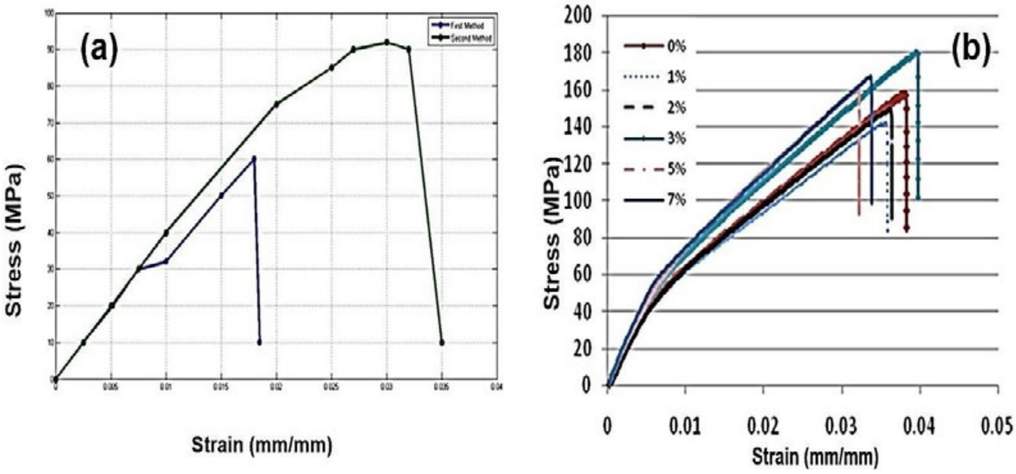

Fig. 4. (a) stress-strain diagram for a resin sample with 2 wt% nanoclay using two different fabrication methods and (b) stress-strain diagram for different weight percentages of nanoclay

Table 2. Mechanical properties of pure samples and specimens reinforced with NC powder

	Percentage increase in length	Ultimate strength (MPa)	Elastic Modulus (GPa)
Pure sample	4.7	55.1	2.91
Soluble mixing method	1.85	58.33	3.35
Direct mixing method	3.42	93.17	4.06

is for pure NC cloisite30B, which according to the Bragg equation has an interpolated spacing of 18.61 Å. Nanocomposite samples with different weight percentages of NC have a smaller peak and are transported to smaller angles and indicating that the NC plates are embedded. As shown in Table 1, the samples obtained with the first method, it means the soluble mixing in the acetone solvent, have reached the laminate state (except for the 7 wt% specimen that has a peak) but all the samples in the second method are adhered to the embedded state and have peaks in the XRD pattern. Among the samples fabricated with second technique, the sample with 2 wt% has the highest intensity value. Therefore, the second way was more appropriate method because the nanoparticles can locate within the resin spacing and leads to better spreading. After the tensile test, the stress-strain graph of nanomaterial containing 2 wt% NC obtained as Fig. 4(a). The stress-strain chart of nanomaterials with different percentages of NC also shown in Fig. 4(b). In Table 2, the mechanical properties of two samples with 0 wt% and 2 wt% NC were compared. As the resin samples made with the second method have better mechanical properties also the second method was selected as easier and faster technique for fabrication of resins hybrid specimens.

Effect of abrupt burning of resin (VARTM Method)

To prepare the resin, three epoxy/clay/baking agents has been mixed. As mentioned in the previous section, two methods were used for this work. In both methods, the NC mixed with one of the epoxy/baking agent components (in the first method, the clay first mixed in the epoxy, then the curing agent was added and mixed in the second clay in the cooking agent, at the end the epoxy was

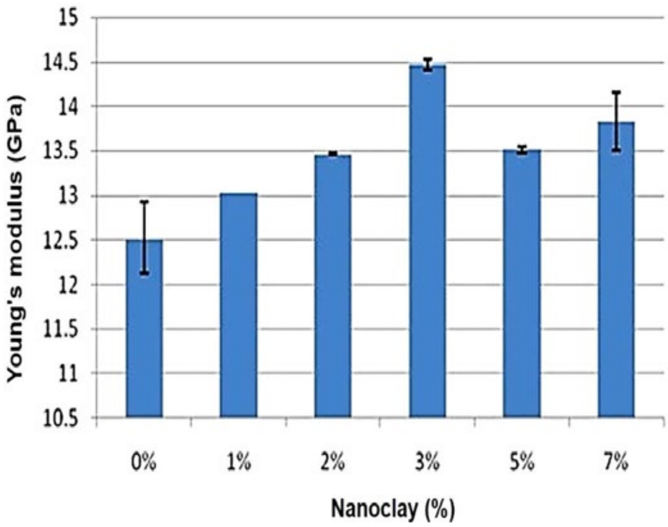

Fig. 5. Elastic modulus variation for various weight percentages of NC

added), but in the general state, with the addition of a third factor, the cooking mechanism started and the polymeric networks were formed. On the other hand, this stage (the baking step) is the most important step in creating a layer of lamination and NC network.

Effect of nanoclay on hybrid nanocomposites
Tensile test results

Fig. 5 (a-b) represent the elastic modulus of all the samples with various content of NC powder. Increasing the percentage of NC powder up to 3 wt% may leads to increase in elastic modulus value. Also, addition of NC powder can increase the resin modulus and increase the adhesion between the resin and the fibers. Although with sample containing 3 wt% NC powder, the resin elastic modulus increased, the adhesion between the resin and the fibers were decreased due to the increase in the resin viscosity. The sample with 7 wt% NC shows a higher elastic modulus compared to the sample with 5 wt% NC powder. The following changes occurred due to the high elastic modulus of the resin for the sample with 7 wt% higher than 5 wt%. The highest increase in the Young modulus in the sample with 3 wt% NC was increased by 15% and the final strength increased by 11%. The stress-strain patterns of the samples show that the area below the curve of sample 3 wt% NC, which indicates the fracture of the material is higher than other samples. Tensile strength of the samples almost has linear behavior before reaching to the highest tensile stress and failure occurs suddenly. Hence, the failure of nanocomposite samples can be considered as a brittle failure, which is one of the characteristics of composite materials reinforced fiber polymer. Due to the fact that the contribution of the fibers in the results of the tensile tests is greater than the sample matrix, the effect of adding nanoparticles on some of the tensile properties of these materials is justified. Fig. 6 shows the elastic modulus of all the samples containing various amount of NC powder (0, 1, 2, 3, 5 and 7 wt%)

Three-Point bending test results

Fig. 7 (a-b) show the flexural modulus and bending strength for samples with different weight percentages of NC powder. By increasing the percentage of NC nanoparticles, the porosity in nanocomposites increases, which prevents the formation of a continuous matrix. As a result, materials with high porosity may encounter with weak mechanical properties. Accordingly, the presence of some pore size may result in stress concentration and increase of degradation in mechanical properties [24-27]. The following event may result in reduction in the composite failure strain. However, in the sample with 5 wt% NC powder, due to the increase in porosity value, as well as the increased load in bending, these empty spaces (pore size) are filled and the continuous matrix is formed and the elastic modulus (E) is increased, which is evident in the sample with 5 wt % NC powder. As shown in Fig. 8 (a-b), by increasing the percentage of nanoparticles up to 1 wt%, the

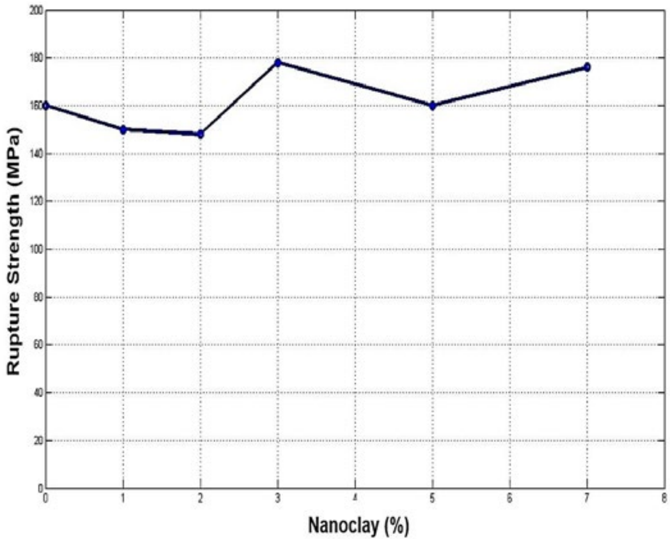

Fig. 6. Ultimate strength for different weight percentages of NC

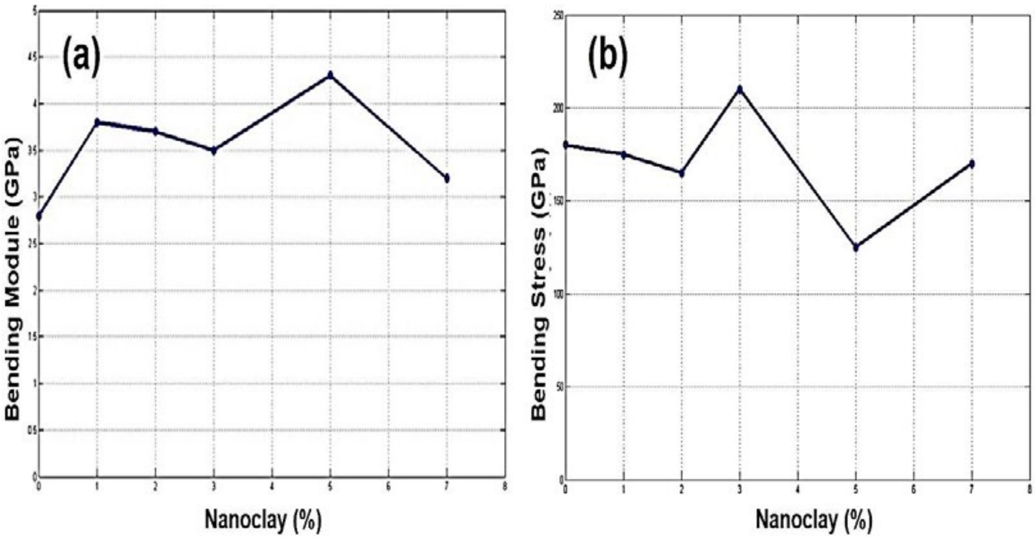

Fig. 7. (a) Flexural modulus for different weight percentages of NC and (b) Flexural strength for different weight percentages of NC

flexural modulus decreases to a very low value. Then, in the sample with 3 wt% NC powder the flexural modulus increases and after a significant drop in the sample with 5 wt%, it will increase again in the sample with 7 wt% NC powder. Fig. 8 shows the creep force in the three points bending test at different weight percentages. In general, taking into account all the results of elongation and bending, it can be concluded that the sample with 3 wt% has better mechanical properties due to the optimum amount of NC powder. The NC nanoparticles can influence the mechanical properties of nanocomposites through various mechanisms, and the final output of the experiments is a result of the positive and negative effects of various mechanisms involved in the effect of nanoparticles on the mechanical properties of composites. Fig. 9 shows how the nanocomposite parts are broken during the ballistic impact evaluation.

Impact test results

In the rupture of a plaguing, a strip with a high shear strain has been created radially near the radius of the projectile and detached from the target. Rupture is a state in which the radial cracks and the subsequent rotation of the target material

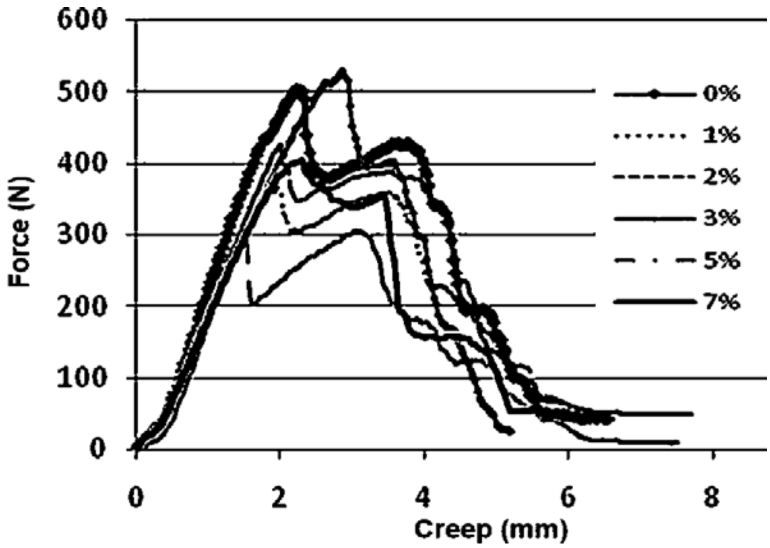

Fig. 8. Three-point bending test for different weights

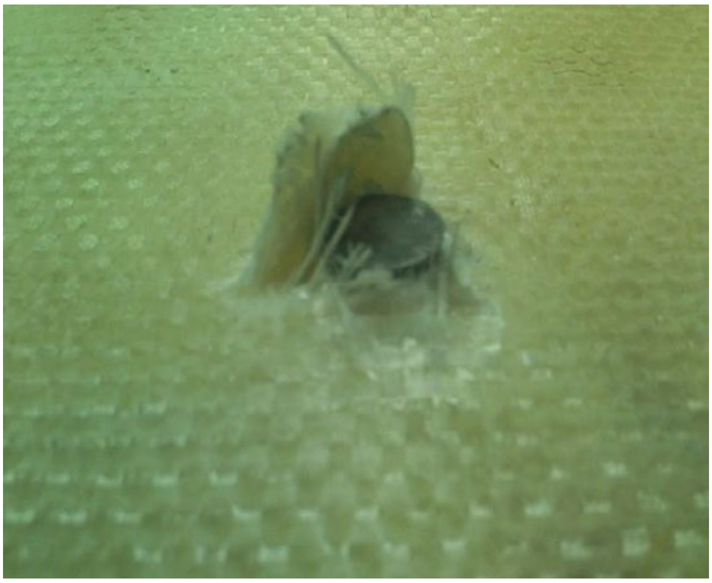

Fig. 9. Fragmentation of composite parts in the test

are created. The projectile deviates after the penetration into the target and the reason for this event can be attributed to the fact that the impact conditions are not ideal [28-31]. The improper possibility of homogenization of the target at the point of impact, the probability that the projectile will be completely non-perpendicular when entering the target, the possibility that the projectile will not collide precisely with the target center, the lack of a completely identical growth of the canals and etc. can be unavoidable factors that cause projectile deviation during the infiltration of the target. The energy absorption rate increases with an increase in the collision velocity, which is the same as the initial energy level, which means that the increase in the strain rate has a positive effect on the energy absorption of the epoxy glass composite. Comparison of strain rate effects on various composite materials showed that glass-reinforced composites exhibit a higher resonant coefficient than carbon-fiber-reinforced composites [32-38]. The greater the level of the target will be ruled out or, in other words, the larger the energy absorption mechanisms will be,

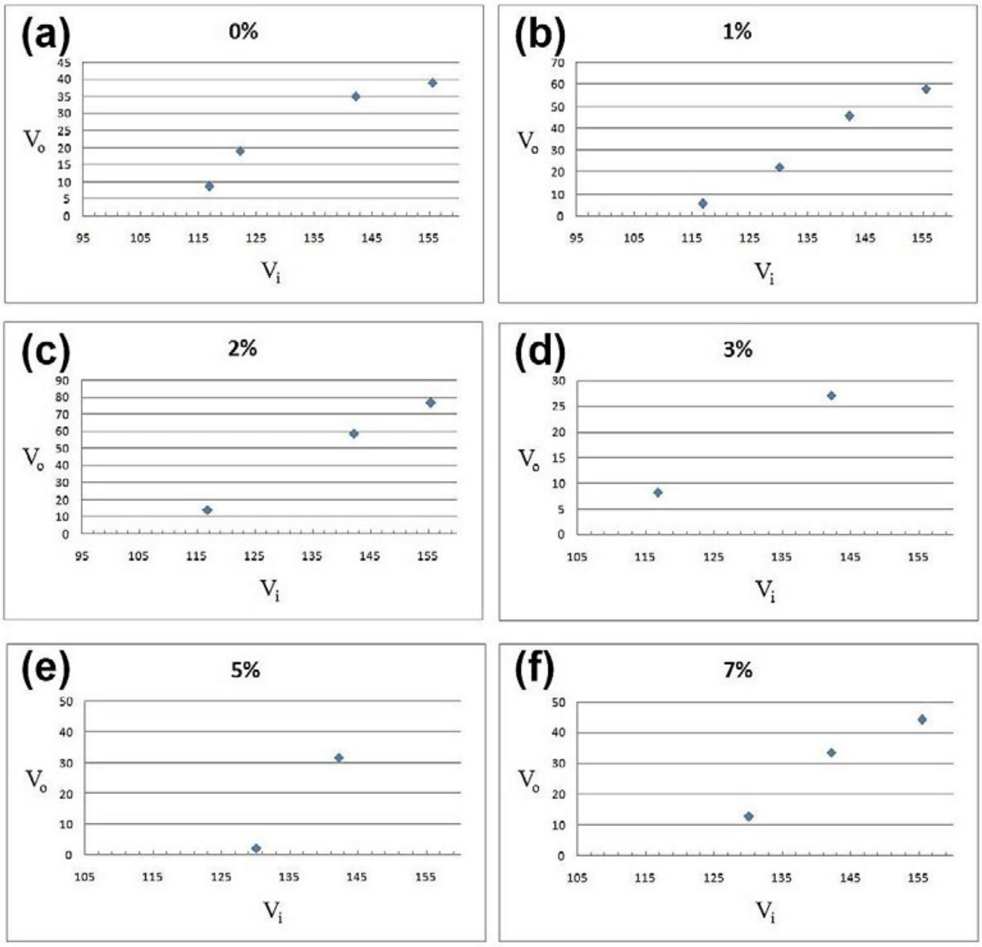

Fig. 10. Output velocity graph in terms of input speed (units m/s). Projectiles for different weight percentages of nanoscale (a) 0 wt%, (b) 1 wt%, (c) 2 wt%, (d) 3 wt%, (e) 5 wt% and (f) 7 wt%

the effects of dynamic will be more. In general, the amount of absorbed energy is equal to half the difference between the input and output kinetic energy shown in Fig. 10. Fig. 11 (a-f) shows the projectile output velocity for different NC proportions. In Fig. 11, a sample of the target has been shown from front of the projectile is clear (a) and a part of the sample is cut to the diameter of the projectile and forms the plug. However, it is not clear from the rear view of the target (b) the passing of the projectile, but the fiber is broken. In most of the samples, it is observed that the lamina is closer to the back of the target. Another point is the gradient of the graph Fig. 12(a) after the ballistic speed limit. As can be seen in this figure, the ballistic speed is close to 0 wt%, 1 wt%, 2 wt%, and 3 wt%, and is almost equal to 116 m/s and less than 5 wt% and 7 wt%, which is 130 m/s. Another point is the slope of the graph after the ballistic speed limit. It is found that this slope is less than 1 for all specimens, indicating the sensitivity of the glass fiber to the strain rate, unlike carbon fiber, that is increasing the strain rate increases the fiber strength. This slope increased with increasing NC percentage but decreased by 3 wt%, which is due to the high toughness of this sample compared to other specimens. Fig. 12(b) shows the output energy in terms of input energy for different NC percentages. In general, from this diagram, it can be concluded that with increasing energy input, the energy absorbed by the target increases, and this, as has been said, is due to the sensitivity of the glass fiber to the strain rate. If the primary energy of the projectile in the diagram is divided into two regions with energy less than 120 Jules and a high energy region of more than 120 Jules, it can be seen that there is no significant difference in the energy absorption for different samples in

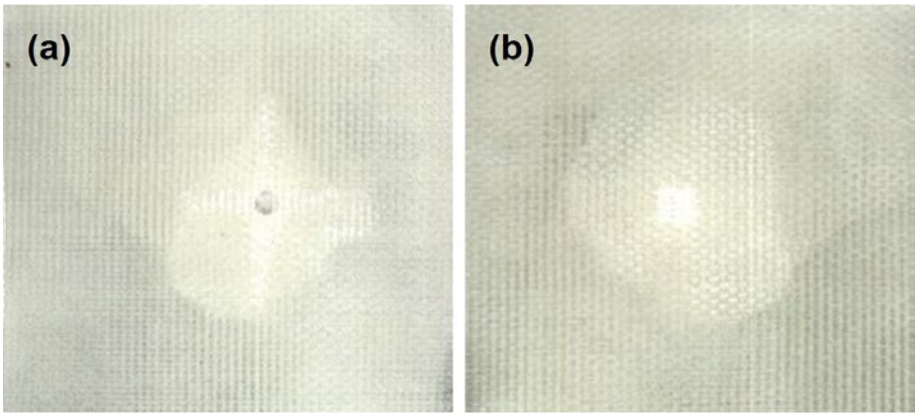

Fig. 11. The object after the projectile collides and crosses, (a) Front view and (b) rear view

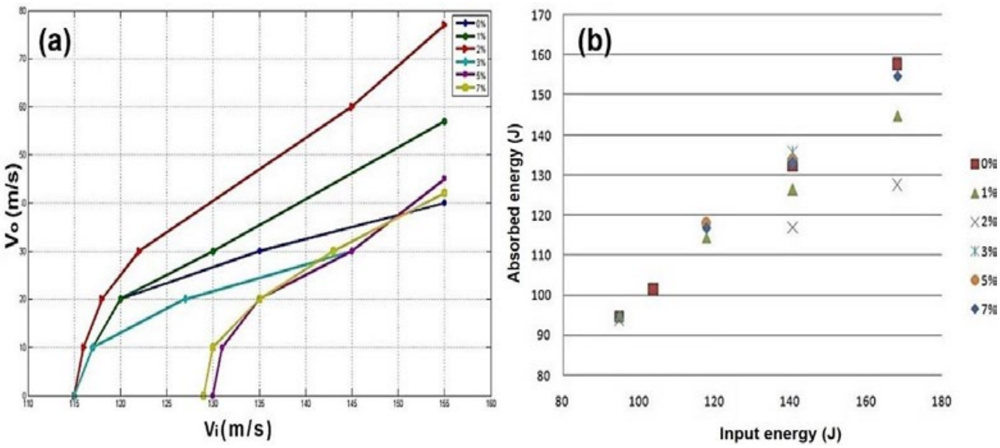

Fig. 12. (a) Output velocity graph in terms of input speed (units m/s) and (b) absorbed energy in terms of projectile input energy

the energy zone. Differences are significant in an input area of more than 120 Jules. In this region, samples of 0 wt, 5 wt% and 7 wt% of absorbed energy are higher and 1 wt% and 2 wt% less of the absorbed energy. The absorbed energy of samples 1 wt% and 2 wt% is low, thanks to good adhesion between fibers and resin due to the presence of NC. The proper adhesion between fibers and resin causes the target to drop under the impact of a projectile. This event means that as the fiber amount increased the tensile fracture has been reduced. Also, one can say that the projectile energy is simply decreased the fibers, which ultimately reduces the absorption energy by the target. In the sample with 5 wt% and 7 wt% NC powders as the NC powder increasing in resin, the nanoclay agglomerates (the residual masses of the clay plates that could not penetrate into the polymer chain) have been increased, which also reduces fiber adhesion and resin. Another reason is the reduction in resin toughness. This leads to multiple laminations in the thickness direction. Also, the absorbed energy rises and ultimately increases their ballistic velocity as shown in Fig. 13 (a-b). It is observed that with increasing nanoparticle adhesion between the resin and weakened fibers, the separation between resin and fiber is increased. In Fig. 14 (a-c), the target thickness is divided into two regions. Thickness I of the fibers are cut and the thickness II of the regions where the fibers are disintegrated. The longitudinal line in the shape of the overall laminar surface of the target is shown to be exactly at the end of the cut-off layers, and the shorter line of laminates with a smaller area, which is probably the result of a tensile tension reversal wave. Fig. 15 (a-c) and Fig. 16 (a-c) shows the samples of 0 wt%, 3 wt% and 7 wt% of the NC, which the projectile

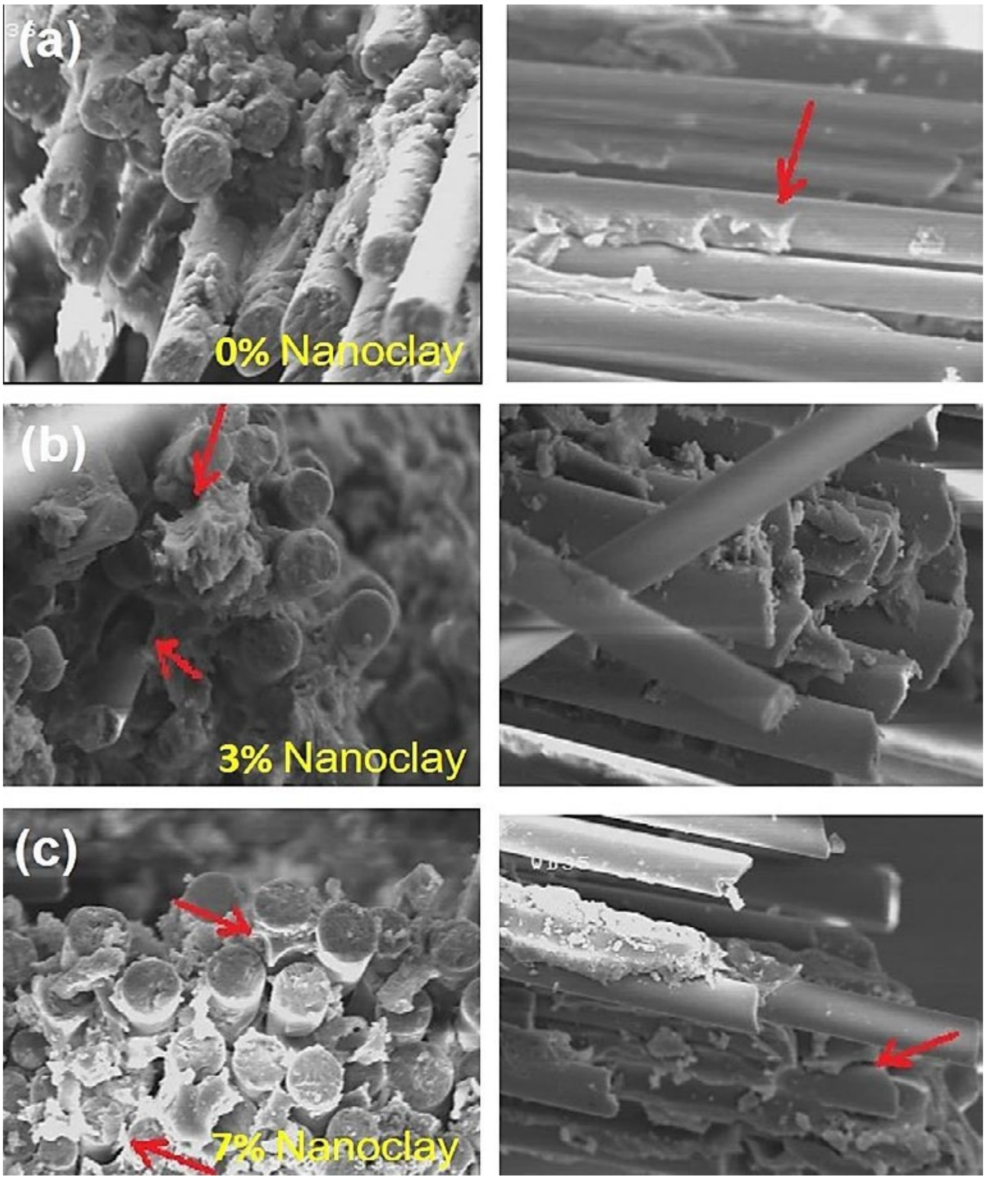

Fig. 13. SEM image of the sections of the three samples (a) 0 wt%, (b) 3 wt%, and (c) 7 wt% of the NC. The left column of the fiber is cut off and the right side of the fiber is broken into pieces. In the 7 wt% sample, there are several distinctions between resin and fibers.

has crossed. As it can be seen from the figures, the overall lamina of the composite plate is just in the substrate that has been cut and made up of the plug. Other laminates are found along different thicknesses and layers, which have less area. The thickness of the created plug can be detected from the shear layers. Fig. 16 (a) is a sample without NC. The thickness of the plug formed in this sample is small, and the number of layers of layering is slightly observed in the thickness direction. Fig. 16 (b) shows a sample with 1 wt% NC with a plug thickness of more than the other specimens, but fewer fibers on the back of the target have been broken by stretching. Some delamination's are seen in the thickness direction but due to the good toughness of the matrix, which has a direct effect on the composite laminate, they have a smaller area. The sample containing 7 wt% of NC powder is shown in Fig. 16 (c). The figure shows total lamellar is more in-depth and due to the weak adhesion of the fiber-matrix, there are also intra-layer injuries.

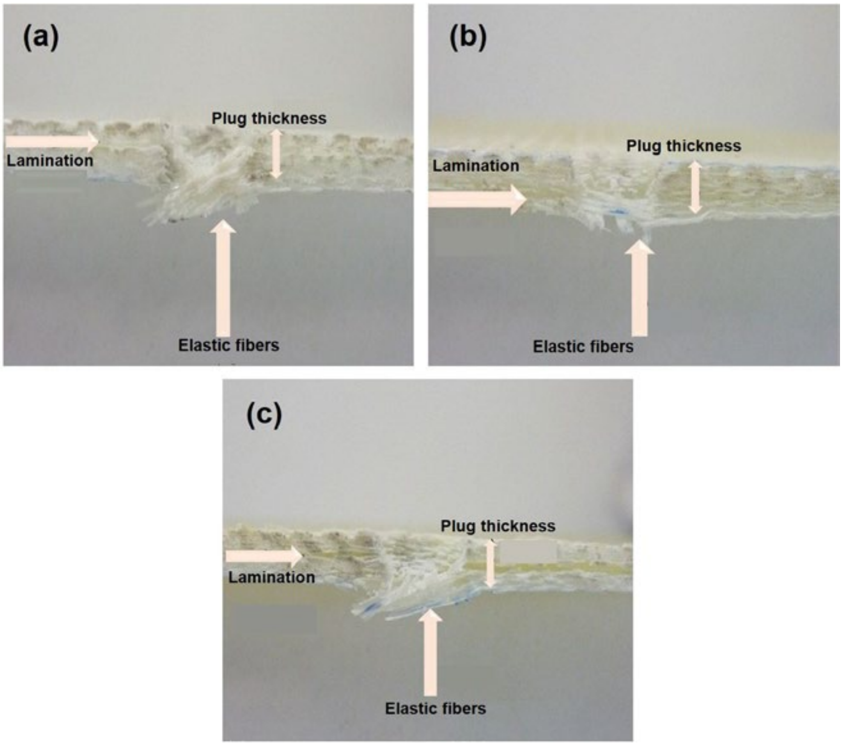

Fig. 14. Schematic of the cross-section of the composite target, which is located under two cutting mechanisms (thickness I) and fiber elongation (thickness II), lines are a sign of laminating.

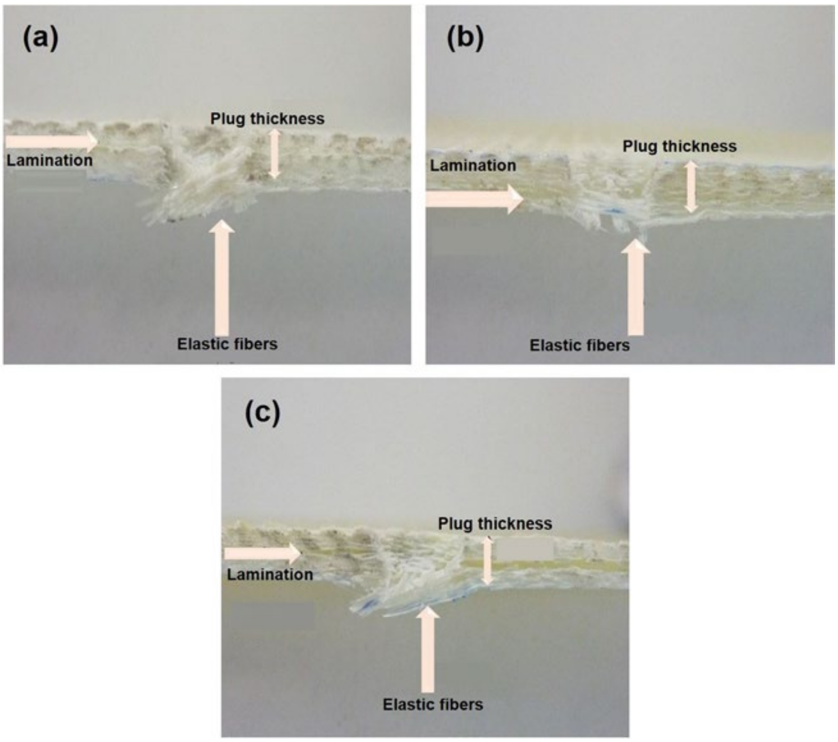

Fig. 15. The image taken from the cross-section shows three samples (a) 0 wt%, (b) 3 wt%, and (c) 7 wt% NC. The location of the overall lamina is completely clear.

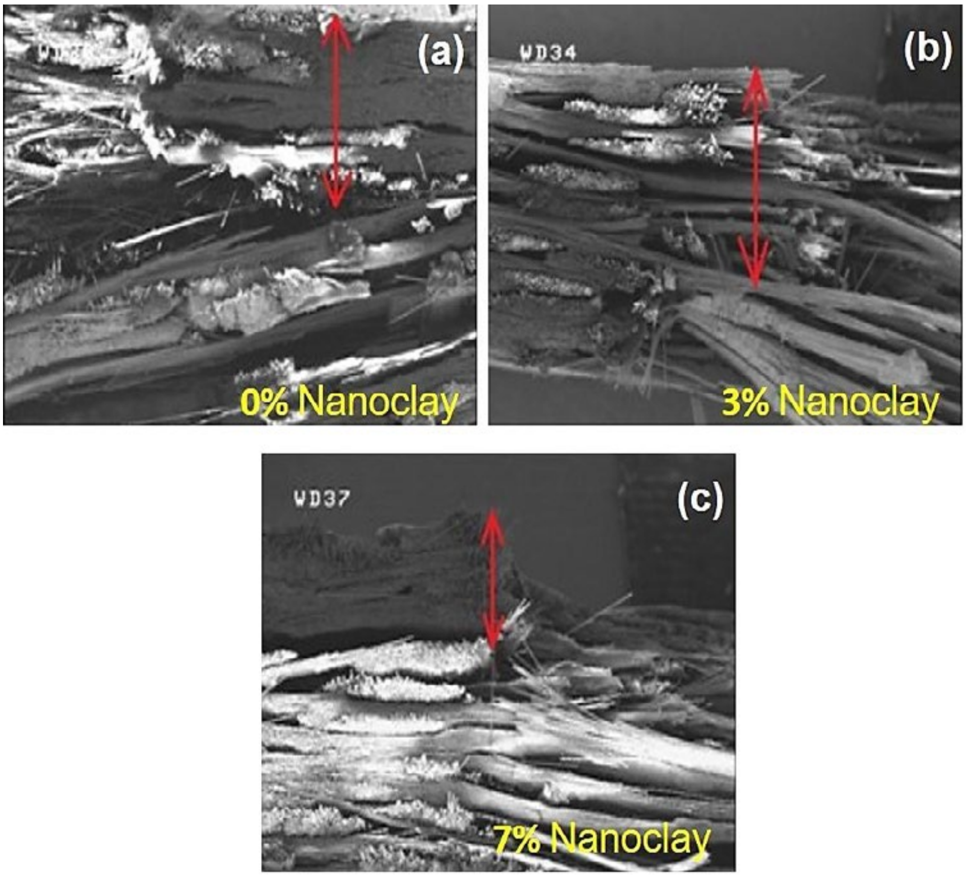

Fig. 16. The SEM image of the sections of the three samples (a) 0 wt%, (b) 3 wt%, and (c) 7 wt% NC that the projectile crosses, and the ratio of the thickness of the cut fiber to the thickness of the fibers pulled in the sample is 7 wt% less than the other two samples (units m/s).

CONCLUSION

In the current paper, the effect of adding NC powder to glass/epoxy composite with woven fibers was investigated. The NC nanoparticles with 1, 2, 3, 5 and 7 wt% were added to the glass/epoxy compound and their effect on the mechanical properties of the composite material was studied subsequently. Investigations have shown that the molding method with resin transferred by vacuum is a suitable method for the fabrication of hybrid nanocomposites. In addition, for cook of the resin properly at the beginning of the molding process in high nanoscale percentage, the NC should be injected immediately after epoxy penetration. The following treatment occurred due to the presence of NC nanoparticles with hardening properties. Other results for the pure resin sample made by the second method showed a noticeable increase of 60% and 21%, in tensile strength and elastic modulus, respectively. On the other hand, the elastic modulus and tensile strength for the specimen hybrid with 3 wt% NC were also improved by 11% and 15%, respectively. The bending strength of the sample containing 3 wt% NC was increased by 11% as compared to the NC sample, the flexural modulus of the sample with 5 wt% NC increased by 48% compared to the sample without NC powder. The ballistic speed limit for the sample is 116 m/s and for samples with 5 wt% and 7 wt% NC 130 m/s (12% more than the pure sample). First, the toughness of the resin was reduced at high velocities while the lamination was increased. The total laminating area of the sample is about 5% more than the other samples. Second, increasing the nanoparticle weight percentage, reduced the adhesion between the resin and the fibers. Third, the fracture of the fibers from shear to stretching, which also increased the energy absorption of samples with high NC percentage. In general, by increasing NC, the overall structural efficiency of ballistic was enhanced.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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