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Research Article

Enhanced out-of-plane loading performance of multi-scale glass/epoxy composites doped with HNTs

Mahmut Özer[a] Hasan Ulus[b] Halil Burak Kaybal[ac]

[a] Department of Mechanical Engineering, Engineering Faculty, Amasya University, Amasya,Türkiye

[b] Huglu Vocational School, Selcuk University, Konya,Türkiye

[c] Department of Mechanical Engineering, Engineering Faculty, Wichita State University, Wichita, Kansas, USA

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ABSTRACT

Glass fiber reinforced composites have attracted great, widely used specific industrial areas such as defense, aerospace, etc. However, composite plates are defenseless to damage accumulation such as matrix cracks, fiber delamination, and delamination, which limits the application of glass composites in specific industrial areas at limited strength levels. Therefore, analysis of the behavior of composites under the out-of-plane loads is essential to optimize such material. This study examines the out-of-plane loading performance of multi-scale glass/epoxy composite laminate. To improve the load-carrying performance in the direction of out-of-plane, the halloysite nanotube (HNT) particle reinforcement was introduced to the epoxy matrix. The three-point bending tests were conducted to attain the out-of-plane load-carrying performance. The findings show that the flexural strength increases by almost 20% for the HNT-modified glass/epoxy composite compared to the unmodified counterpart; meanwhile, the toughness is effectively improved with the HNT addition. Moreover, the damage process of specimens in three-point bending tests was detected by microscopic examination.

1. Introduction

Fiber-reinforced polymer (FRP) composites are gaining popularity in the engineering fields of the defense, aerospace, automotive, and marine industries due to their superior mechanical performance [1]. Mainly glass fibers are commonly used in the composite industries owing to their low production costs and high mechanical properties [2, 3]. However, fiber-reinforced laminated composites are susceptible to out-of-plane loading as they are weaker in the thickness direction than in the lamination plane [4]. Therefore, improving interlayer and fiber/matrix interfacial properties indirectly affects out-of-plane load-carrying performance. Recent studies show significant property enhancements can be achieved in glass/epoxy composites, especially for the fiber-matrix interface and out-of-plane loading performance [5, 6].

In light of developing nanotechnology applications, it has been seen that modifying polymer matrix composite materials with nanoparticle reinforcements can lead to significant improvements in mechanical properties. Research groups discovered the positive effects of nano-fillers such as carbon nanotube (CNT), graphene, nano-silica, nanoclay, $CaCO_3$, and Al_2O_3 in improving the mechanical properties of composite materials and observed significant improvements in tensile and bending properties, fracture toughness, and thermal properties. Nayak et al. investigated the effects of nanoclay and nano-silica additives on the bending and thermal properties of glass fiber epoxy composites [7]. They found a 10% increase in flexural strength after hybrid doping. The addition of the fillers such as Al_2O_3 and $CaCO_3$ affects the composite properties such as interlaminar shear strength, tensile properties, flexural strength fracture toughness, and dynamic loading response [8-11]. Moreover, Pathak et al. observed the enhancement of flexural strength and modulus by 66% and %72, respectively, in the graphene oxide modified to epoxy composite [12]. The strengthening effect of CNTs on the performance of polymer matrices provides an opportunity to enhance novel composite materials with outstanding mechanical and physical properties [13]. Zhang et al. revealed that fiber-reinforced polymer (FRP)composite material with CNT addition improved interlayer shear and bending strength by 27% and 59%, respectively [14]. However, some disadvantages of CNT, such as high cost, toxicity, and sedimentation shortly after mixing, limit its wide-scale application [15-17]. On the other hand, halloysite nanotubes, similar to CNTs with their nanotubular geometry, are aluminosilicate with a hollow structure [18]. HNTs have been the focus of industrial and academic research because they can reach micron length or diameter at the nanometer scale, can be extracted from natural deposits cheaply, and are well dispersed in polymer matrices [19]. Scientific studies stated that the mechanical properties of FRP composite could be improved by doping HNTs, such as tensile and flexural loading performance, toughness and fracture properties, and elastic modulus [20-23]. So far, very little research has been conducted on fiber-reinforced composite's enhanced static loading performance via HNTs contribution in the out-of-plane direction. Moreover, the extent to which HNT reinforcement plays a role in the three-point bending performance of multi-scale glass/epoxy composites remains unclear.

In this study, HNT-doped glass/epoxy composite materials have been manufactured via hand lay-up and vacuum bagging

***Corresponding author:** Halil Burak Kaybal
*E-mail address: hburak@amasya.edu.tr
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methods. The effect of HNTs reinforcing on static loading in the direction of out-of-plane has been examined and discussed in terms of three-point bending properties and optical fracture surfaces.

2. Materials and Methods

2.1.Materials

The composite laminates were made from glass fiber fabric, epoxy resin, and HNT nanoparticle reinforcement. In this respect, 200 gr/m2 woven glass fiber fabric (0/90º) and low-viscosity epoxy suitable for lamination and hardener (MGS L/H 160) were purchased from Bilge Lab-Company Firm. Halloysite nano reinforcement with properties of 20 – 40 nm diameter, 10 µm length, and 98% purity was bought from Eczacıbaşı Esan.

2.2.Multi-Scale Composite Laminate Production

Homogenous dispersion of nano reinforcement in composite laminate is vital for producing specimens. In our previous works, optimized HNT addition was determined as 2% by weight (Ulus et al. 2019, 2020, 2021). The first production phase is mixing HNT nanoparticles in acetone in an ultrasonic mixer for 15 minutes. Secondly, an appropriate amount of epoxy was added to the mixture. Epoxy/HNT/Acetone mixture was blended for 60 minutes with 30 kHz frequency, and it was done in an ice bath to prevent heating. Following this, in order to remove acetone from the mixture, acetone was vaporized from the blend by processing it for 24 hours at 70˚ in a vacuum oven. After removing acetone from the mixture, hardener was added and mechanically stirred for 5 minutes at room temperature.

Multi-scale glass fiber composite laminate manufacture includes a combination of two different methods called hand lay-up and vacuum bagging. Production comprises impregnating fabrics with the epoxy/HNT mixture and curing composite laminates. Glass fibers were layered on a metallic surface. Each layer was impregnated by the epoxy/resin mixture inside a vacuum bag and cured with proper conditions related to the epoxy matrix (Figure 1). A detailed explanation of multi-scale composite laminate production was made in our previous work (Özer and Kaybal 2022). Composite laminates were produced in dimensions of 500 x 450 mm2 as a rectangle and had a 51% fiber content ratio.

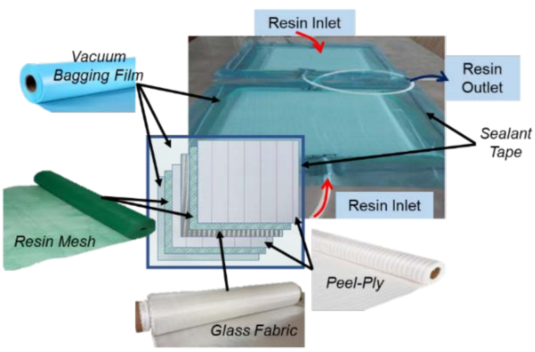

Fig. 1. Vacuum infusion process and equipment

2.3.Three – Point Bending Test

The three-point bending test by ASTM D7264 standard with the Shimadzu AGS test device investigated the flexural properties of multi-scale glass fiber composite laminates (Figure 2). The span length to thickness ratio is indicated as 32:1 in the standard. Therefore, the span length was maintained at 115 mm, and the average thickness of the specimen was around 3.6 mm. All tests were repeated five times, and the mean values were considered. After the three-point bending tests, the damaged specimens were monitored with a DSLR camera.

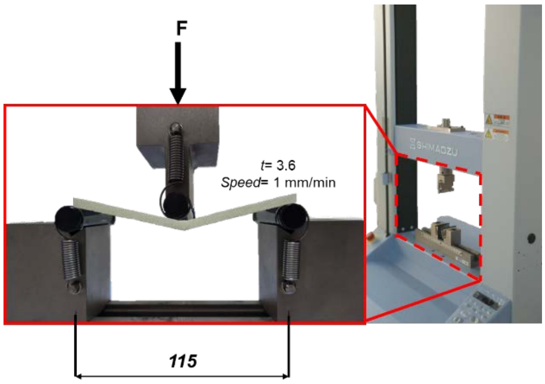

Fig. 2. Three-point bending test process

3. Results and Discussions

Flexural stress-strain curves of composite samples can be seen in Figure 3 (a). According to the graph, the HNT-doped nanocomposite material has a superior load-carrying performance under flexural loading compared to the unmodified sample. It is also observed that the elastic and plastic deformation capability under the loading is higher in the modified material in comparison with the pure counterpart. These results reflect those of Khan et al. who also found that adding of HNTs to epoxy nanocomposites had effect on their flexural properties [24]. A significant increase in flexural strength and strain is apparent for HNT-modified composites (Figure 3 (b)). After the three – point bending tests, the flexural strength values of 332.5 MPa and 276 MPa were calculated for the HNT-modified and unmodified composites, respectively. Therefore, approximately 20% enhancement in flexural strength is provided by HNT addition into glass fiber epoxy composites. This finding can be linked to occurring better adhesion between fiber-matrix components with HNT loading as well as the better dispersion of nanotubes. This also accords with our earlier observations, which showed that the HNTs modification in epoxy matrix enhances the flexural strength by 20.8% over neat epoxy [25].

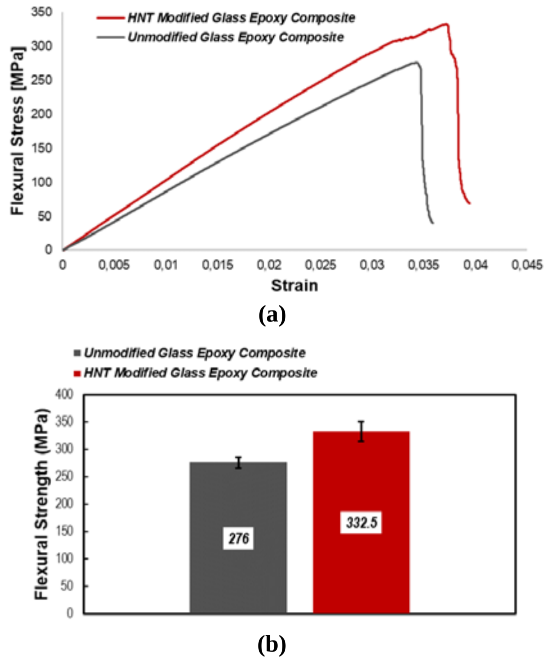

Fig. 3. a) Stress-strain curves of glass/epoxy composites b) Flexural Strengths of glass/epoxy composites

Static bending rigidities versus deformation of multi-scale composites are given in Figure 4(a). The rigidity values for HNT modified and unmodified specimens are 10.3 GPa and 8.6 GPa, respectively. Hence, with the HNT modification, there is a 19.8% increment for flexural rigidities of composites. The energy absorbed until the failure of specimens is stated as toughness. The toughness values of multi-scale composite specimens are shown in Figure 4(b). While the flexural toughness value for HNT modified composite was 7.34 J/mm3, it was 5.25 J/mm3 for unmodified composite samples. As a result, almost 40% improvement is gained with HNT modification. These outcomes are in agreement with Ulus' findings which showed the HNTs reinforcing in fiber composites exhibited remarkably improved flexural properties [26].

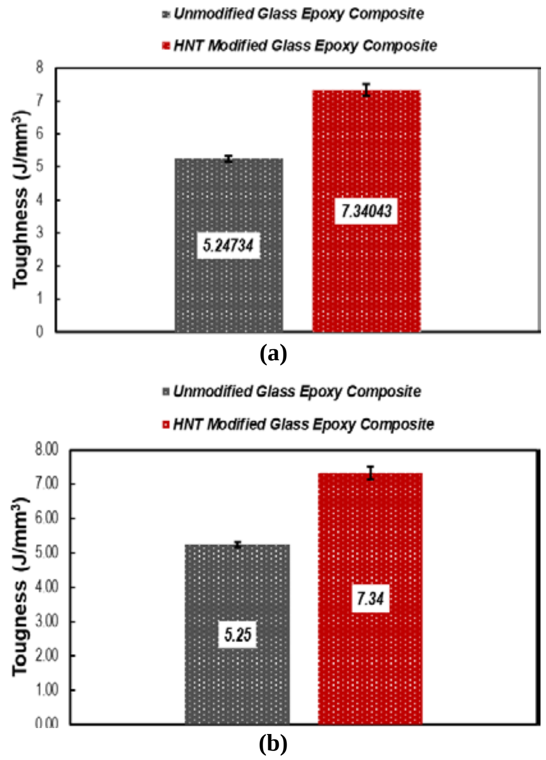

Fig. 4. Mechanic performance values of HNT modified and unmodified composites a) Rigidity b) Toughness

Similar results for glass fiber-reinforced epoxy composites can be found in the literature. In terms of the flexural strength of unmodified glass fiber composites, values differ from 240 MPa to 400 MPa in the studies [27-31]. The closest results can be found in studies of Singh et al. and Seretis et al. [28, 29]. This can be a result of differences in composite production techniques among the works. On the other hand, since there is no study related to the HNT effect on flexural properties of glass fiber composites, the studies that investigated the CNT effect can be compared because of the similar structure of HNT and CNT. Rahman et al. found that flexural strength and modulus of glass fiber composite was increased by 38% and %22, respectively, with 0.3% by weight addition of MWCNT [31]. In another work, Singh et al. (2018) indicated that flexural strength and modulus increased by 13.8% and 20.4 %, respectively, with 0.5% by weight addition of MWCNT [29]. Therefore, the HNTs' similar improvements achieved in this study, the use of HNT, which is more inexpensive, can be an excellent alternative to CNT addition.

Micro images of the composite specimens after the three-point bending test showing the damage mechanisms are given in Figure 5. From Figure 5a, it can be seen that the more propagated delamination occurring is for nonmodified composites. Delamination propagation indicates a weak fiber matrix bonding in pure composites. In addition, rarely occurred matrix cracks in unmodified samples have mainly transformed into delamination. Moreover, the out-of-plane damage progression path is shorter than that of the HNT-modified nanocomposite sample. On the other hand, a clear benefit of HNT modification in preventing delamination is identified in this damage analysis (Figure 5b). In the out-of-plane direction, the longer damage propagation path compared to the unreinforced specimen has indicated more absorbed energy and, so, better load-carrying capacity. In accordance with the present results, our previous studies have demonstrated that HNTs doping in the epoxy matrix improves the performance of the fiber-matrix interface, and so nano modification causes enhances load-carrying performance of nanocomposites [32, 33]. The observed enhancement in the mechanical performance of HNT-modified composites could be attributed to HNT crack pinning and bridging, deflection, and particle debonding fundamentally are the main toughening mechanisms [33, 34].

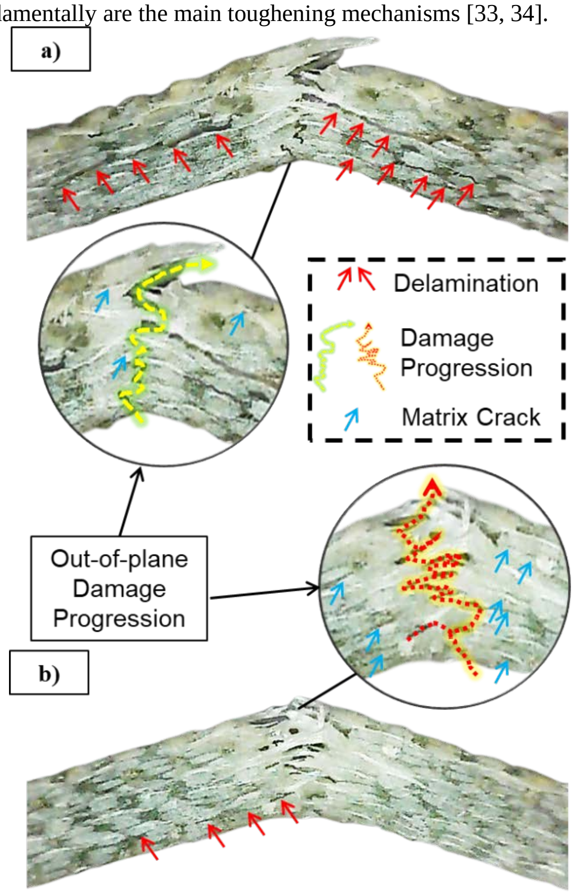

Fig. 5. Composites` damage analysis after bending test a) Unmodified b) HNTs modified

4. Conclusion

This study investigates the effects of HNT particle addition into glass fiber-reinforced epoxy composites in terms of flexural properties. In order to see the effects of HNT reinforcement, three–point bending test was applied to the specimens. 20% improvement in flexural strength was achieved with HNT addition compared with unmodified samples. On the other hand, when flexural rigidities of multi-scale composite specimens were compared, 19.8% of enhancement was determined with HNT reinforcement. Moreover, the toughness value of unmodified composites was increased by almost 40% with the HNT addition. As a result, HNT reinforcement clearly has improved the bending performance of glass fiber epoxy composites in terms of flexural strength, rigidity, and toughness.

Declaration of conflicting interests

The authors declare no competing interests.

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