NÖHÜ Müh. Bilim. Derg. / NOHU J. Eng. Sci., 2021; 10(2), 777-783

Niğde Ömer Halisdemir Üniversitesi Mühendislik Bilimleri Dergisi
Niğde Ömer Halisdemir University Journal of Engineering Sciences

Araştırma makalesi / Research article

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An experimental study on interlaminar shear strength and fracture toughness: Carbon fiber reinforced epoxy composites enhanced with the $CaCO_3$ nanoparticles

Tabakalararası kayma mukavemeti ve kırılma tokluğu üzerine deneysel bir çalışma: $CaCO_3$ nanoparçacıkları ile iyileştirilmiş karbon fiber takviyeli epoksi kompozitler

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Abstract

This study examines the effect of nano-$CaCO_3$ particles on interlaminar shear strength (ILSS) and fracture toughness properties for carbon fiber reinforced epoxy composites. The nano-$CaCO_3$ modified carbon fiber epoxy composites (nC-CFECs) were prepared using the vacuum assisted resin infusion (VARI) method. The short beam shear (SBS) test and single edge notch bending (SENB) test were conducted to calculate ILSS and fracture toughness, respectively. The results illustrate that the introduction of 2% wt. nano-$CaCO_3$ into the epoxy increased the ILSS by up to 24%. Moreover, the fracture toughness in the nanoreinforced composite was 32.3% higher than the unmodified composite. Overall, the nanocomposite material has shown better mechanical performance in terms of ILSS and SENB tests.

Keywords: Carbon fiber, Composite, Nano-$CaCO_3$, ILSS, SENB

Özet

Bu çalışma, nano-$CaCO_3$ partiküllerin, karbon fiber takviyeli epoksi kompozitlerin tabakalar arası kayma mukavemeti (ILSS) ve kırılma tokluğu özellikleri üzerindeki etkisini incelemek üzere gerçekleştirilmiştir. Nano-$CaCO_3$ ile modifiye edilmiş karbon fiber epoksi kompozit malzemeler (nC-CFEC) vakum destekli reçine infüzyon (VARI) yöntemi kullanılarak hazırlanmıştır. ILSS ve kırılma tokluğunu hesaplamak için sırasıyla kısa kiriş kesme (SBS) testi ve tek kenar çentik eğilme (SENB) testi yapılmıştır. Sonuçlar, epoksiye ağırlıkça %2 nano-$CaCO_3$ eklenmesinin ILSS 'yi %24'e kadar artırdığını göstermektedir. Dahası, nano-takviyeli kompozit malzemede kırılma tokluğu, nano takviyesiz kompozitten %32.3 daha yüksek bulunmuştur. Genel olarak, nanokompozit malzeme, ILSS ve SENB testleri açısından daha iyi bir mekanik performans göstermiştir.

Anahtar kelimeler: Karbon elyaf, Kompozit, Nano-$CaCO_3$, ILSS, SENB

1 Introduction

Carbon fiber reinforced composite materials have been widely preferred by various industries such as marine, defence, aviation, automotive, and sport due to their improved mechanical properties such as high strength, excellent stiffness [1]. Interlaminar shear strength (ILSS) and fracture behavior of carbon fiber reinforced composites play an important role in their design and performance [2, 3]. While the fracture behavior such as delamination resistance is outperformed on fiber dominated in the plane, it is shown a significant performance decrease in through the thickness direction (Z-axis) due to weak matrix/fiber interface bonding [1]. Therefore, improving the interface properties of composite materials is important to their performance of shear strength and fracture behavior.

Nanoparticles are emerged as a key reinforcing element to improve the fracture behavior of carbon fiber composites by means of advances in nanocomposite technology [1, 4-7]. Many researchers have investigated to enhance fracture resistance by using nanoparticles. Ulus et al. found that 2 wt% halloysite nanotube into fiber reinforced composites (FRC) increased the interlaminar shear strength (ILSS) by 19% [4] and, in their other study, increased the critical stress intensity factor (K_{Ic}) by 24% [8]. Fan et al. [3] achieved 33% enhancement in the short beam test (SBT) by adding multi-walled carbon nanotube (MWCNT) into FRC. The improved by 31% fracture toughness of MWCNT/Zr_2O_2-based hybrid epoxy nanocomposites (MNCs) is obtained by Rathi and Kundalwal [9]. Furthermore, carbon nanotubes (CNTs) can be delayed the onset of matrix cracking in the carbon fiber reinforced composites [10]. 2 wt.% melamine-functionalized graphene nanoplatelets (MGNPs) reinforced epoxy nanocomposite increased fracture toughness by 124% in the paper of Cha et al [11]. Naous et al. [12] gained that adding 2% vol Al_2O_3 into epoxy improved the stress intensity factor (K_Q) of single edge notch bending (SENB) by 40%. The prepared carbon fiber epoxy composite with the addition of 4 wt.% nano-$CaCO_3$ by hot press process has occurred 36.6% increment in the ILSS [13] and in another study of the continuation of this work, carbon fiber/epoxy composite

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Geliş / Recieved: 10.12.2020 Kabul / Accepted: 05.03.2021 Yayımlanma / Published: 27.07.2021
doi: 10.28948/ngumuh.838807

modified with silane coupled nano-$CaCO_3$ is even more increased the ILSS [14]. In our previous reports, we have addressed the efficiency of nano-$CaCO_3$ on the static and dynamic behavior of carbon fiber epoxy composites [15]. Notwithstanding, studies on the fracture properties of nano-$CaCO_3$ modified carbon fiber reinforced epoxy polymer composites are limited.

The inclusion of a certain value of nanoparticles into a composite is meant to improve fracture performance in many studies. However, particle agglomeration due to high particle ratio and poor dispersibility are limited the fracture resistance [16-18].

Up to now, the fracture toughness performance of nano-$CaCO_3$ modified carbon fiber epoxy composites (nC-CFECs) has been faintly discussed. Moreover, no studies have addressed on single edge notch bending of nC-CFECs. In this study, one of the first investigations is to evaluation in detail the effect of nano-$CaCO_3$ particles on interlaminar shear strength and fracture properties of carbon fiber epoxy composites (CFEC). ILSS and SENB tests are separately conducted to assess the fracture behavior for CFEC and n-CFEC samples. Moreover, the fracture surfaces are examined based on the optical and scanning electron microscopy (SEM).

2 Material and methods

2.1 Materials

Epoxy resin and its curing agent (MGS L160 and H160) were purchased from Momentive Hexion. Plain weave carbon fiber fabric (0/90) with 400 g/m^2 areal density was procured from DowAksa. Nano-$CaCO_3$ reinforcements (98% purity, 30~100 nm) were supplied by Chengdu Kelong Chemical Co., Ltd.

2.2 Production of Carbon Fiber Reinforced Nanocomposites

The required amount of $CaCO_3$ nanoparticles was scattered in acetone by tip sonicator (frequency of 20 kHz) for 15 minutes. Afterwards, the blend was poured into the epoxy and sonicated for 30 minutes. The acetone was then evaporated from a mixture in a vacuum furnace at 70 °C for 24 hours. After removing acetone, the curing agent was applied to the mixture and then mixed with a mechanical mixer for 5 min. The Vacuum Assisted Resin Infusion (VARI) process was used to manufacture carbon fiber (CF) reinforced epoxy nanocomposites composed of stacked sheets, which were set on a stainless steel tool plate. After the vacuum bagging process, the tool plate was preheated to 70 °C to minimize the viscosity of the mixtures during infusion. The wet fiber fabric infused with the mixture was cured at 70 °C for 1 h and cured at 120 °C for 4 h. The same method of curing was used for counterpart sample in the epoxy infusion of carbon fabric layers without $CaCO_3$s. By the way of this production, 1-5% nano- $CaCO_3$ reinforcement carbon fiber epoxy nanocomposite laminates were attained. To reduce the filtering effect of nanoparticles which formed in layers and fiber bundles, the nanocomposite laminates were produced as rectangular parts (300 x 150 mm^2).

2.3 Characterization

Two different fracture tests were conducted in the mechanical test procedures of the samples. The geometries of sample and test setups are schematically illustrated in Figure 1. Both of fracture tests were carried out using Shimadzu AGS-X testing machine at room temperature. According to ASTM D2344/2344M, interlaminar shear strength (ILSS) was performed by the short beam shear (SBS) method (Figure 1(a)). The test speed was adjusted at a rate of 1 mm/min. The short beam strength is the transverse shear stress as calculated from Equation (1) implemented at the mid-plane of sample, following;

$$F^{sbs} = 0.75\frac{P_{max}}{b\,h} \tag{1}$$

Where F^{sbs} is short-beam strength, P_{max} is the maximum load, b is width, and h is the sample thickness. The span length specified for the samples was 16 mm and the dimensions of the samples were determined 4 mm in thickness, 8 mm in width, and 24 mm in length.

To determine the fracture toughness (K_{Ic}), the single edge notch bend (SENB) test was implemented according to ASTM 5045. Considering the width of the samples to be 16 mm, the notches were carefully shaped by a circular saw to achieve a notch length of approximately 8 mm and the tip of the notch was sharpened by a razor blade (Figure 1(b)). A crosshead speed of 1 mm/min was identified for the SENB tests at room temperature and all of tests were repeated five times.

The stress intensity factor (K_Q) of SENB test was calculated by Equations (2) and (3).

$$K_Q = \left(\frac{P_Q}{B\sqrt{W}}\right) f(x) \tag{2}$$

$$f(x) = \frac{6x^{1/2}[1.99 - x(1-x)(2.15 - 3.93x + 2.7\,x^2]}{[(1+2x)(1-x)^{3/2}} \tag{3}$$

Where P_Q is the private load value (see in ASTM 5045) *B* is specimen thickness, "*W*" is specimen width, "*a*" is crack length and "*f(x)*" is a unitless calibration factor and its value depends on "*x=a/W*".

After the fracture tests, damage areas were observed with the FX 50 mm 1.8 d lens of Nikon DSLR camera and scanning electron microscopy (SEM) of Zeiss EVO LS 10.

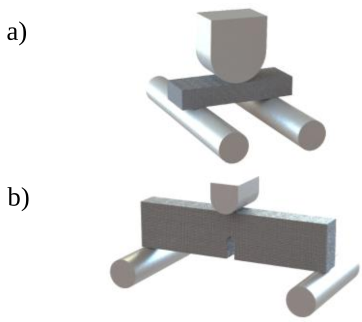


Figure 1. Schematic representation of test samples a) ILSS test sample b) SENB test sample

3 Result and discussion

3.1 ILSS tests

Figure 2 presents several experimental results of load-displacement and ILSS versus nano-$CaCO_3$ contents for CFEC and nC-CFEC samples. In Figure 2 (a), two different load-displacement behaviors have been appeared as (*I*) and (*II*). In Figure 2(a.*I*), the curve is gradually raised and then softly dropped, while in Figure 2(a.*II*), the curve is directly raised and then dropped suddenly. In case of the sample forming a curve such as Figure 2(a.*II*), interlaminar shear failure clearly can be appear with some failure mechanism. The curve form of Figure 2(a.*I*) is flattened for a while now after the dropping load. At the end of this present case, some fracture failures such as matrix cracking and fiber breakage can be observed [3]. Load-displacement curves of samples are illustrated that the shear load of the nC-CFEC samples is higher than that of 0% wt. CFEC except containing 5% wt. nC-CFEC sample. The addition of 2% wt $CaCO_3$ has exhibited the highest fracture resistance. Note that ILSS performance of the 1-4 % wt nC-$CaCO_3$ is better than the other counterparts. This indicates the nano reinforcement enhances the shear resistance due to nanoscale toughening mechanisms [4]. As summarized in Figure 2(b), the shear strength of unmodified CFEC laminate is calculated as 40.3 MPa. The addition of 2 % wt nano-$CaCO_3$ is gained to increase the shear strength up to 50.2 MPa, which is approximately 24% higher than that of the unmodified sample. Afterward, the increasing nano-$CaCO_3$ content from 2% to 5% wt. is decreased the shear strength. He and Gao [13] stated that the reason for this decreasing shear strength was the agglomeration of nano-$CaCO_3$ particles and the weak interaction between particle and epoxy.

Figure 3 shows the fracture surface versus different nano content after the ILSS test. Failure mechanisms of samples are represented by the symbols in the figure. When the fracture surface is examined, the severity of the interlaminar shear failure clearly can be seen in Figure 3 (a) and (f). This explains the reason of the low shear stress for unmodified and 5% wt reinforced samples. Figure 3 (b) shows the preforms of fiber breakages and shear failure in the compression regions under the loading punch. With the addition of 2% wt nano-$CaCO_3$, a decrease in fiber breakage compared with Figure 3(b) and matrix damage are observed in the regions under compression stress (Figure 3 (c)). The absence of clear shear failure in Figure 3 (c) is explained as a good interlaminar strength. Furthermore, damage forms of interlaminar shear failure, matrix crack, and fiber breakages can be seen in Figure 3 (d) and (e). The breakage of fibers, which occurred during the transfer of forces, illustrates that the enhanced matrix with nano-$CaCO_3$ has performed a good performance against shear stress.

3.2 SENB Test

The relationships of bending loads and displacement for samples obtained from SENB tests are drawn in Figure 4. The maximum bending loads are increased in the all of nC-CFEC samples apart from the addition of 5% wt content compared to unmodified samples. A representative critical load (P_{crt}) point on curves illustrates a new crack initiation in front of the preexisted notch [2]. While the value of P_{crt} is 4406.5 N in the unmodified sample, the value of 2% wt nano reinforcement nanocomposite is increased by 30% to 5753.6 N. The crack initiation load is decreased to 3556.8 N by 19% reduction in the 5% wt additive rate. On the other hand, the point P_{max} is denoted the maximum bending load. The samples are quickly lost their durability after the point P_{max}. P_{max} values and changes of the samples are given in Table 1. The increasing P_{max} value of composite specimens with nano reinforcement is reached 35%. When the fracture toughness of samples in the Table is examined, the increment of 2% wt nC-CFEC sample is eventuated as 32.3 % compared with unmodified counterpart. However, the reduction of the fracture toughness for the samples is about 12.3 % with the addition of 5% wt nano-$CaCO_3$.

Figure 5 demonstrates damaged samples after the SENB tests. When the fracture behavior is compared, the effect of nano reinforcement can be clearly seen in the bending angle of samples. It is observed that the bending angle (α) of unmodified samples is lower than that of the nano-reinforced samples due to the damage formation such as matrix crack, fiber breakage and mostly delamination.

a)

b)

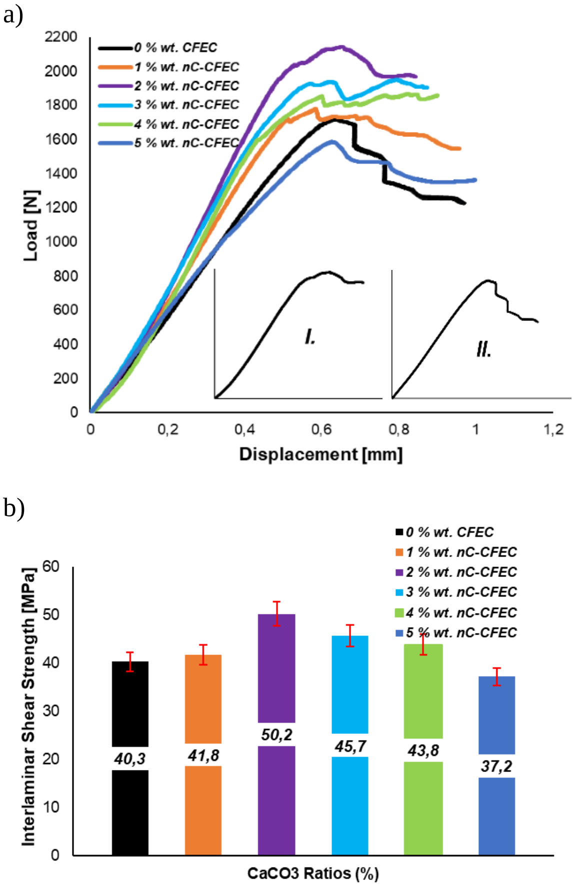


Figure 2. Short beam shear test results versus nano-$CaCO_3$ contents a) typical curves of load vs. displacement b) ILSS of CFEC and nC-CFEC samples

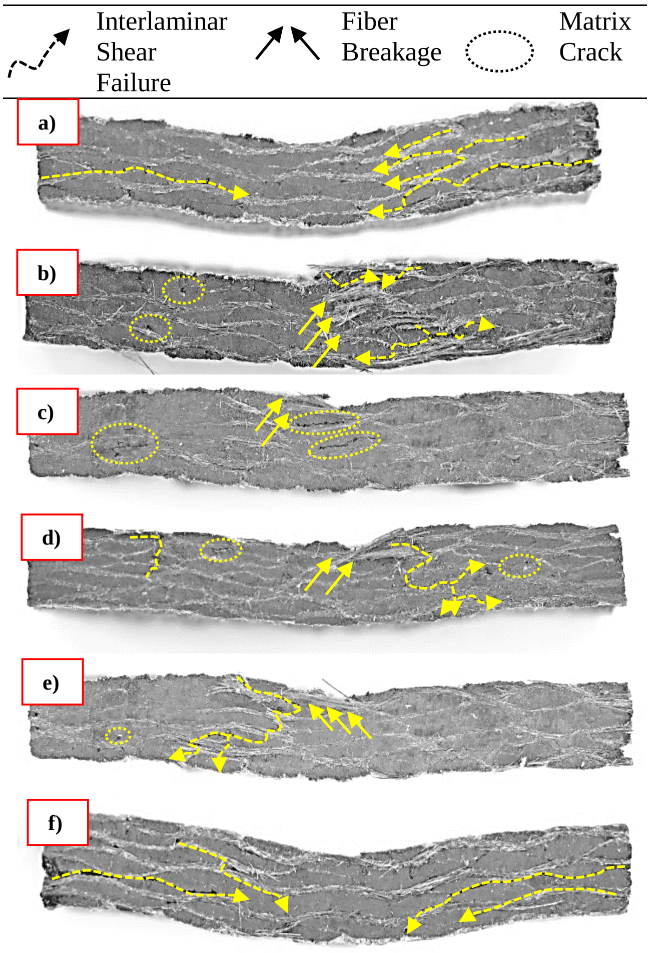


Figure 3. Optical images of fracture surface for ILSS test a) 0% wt. CFEC (b)-(f) 1-5% wt. nC-CFEC

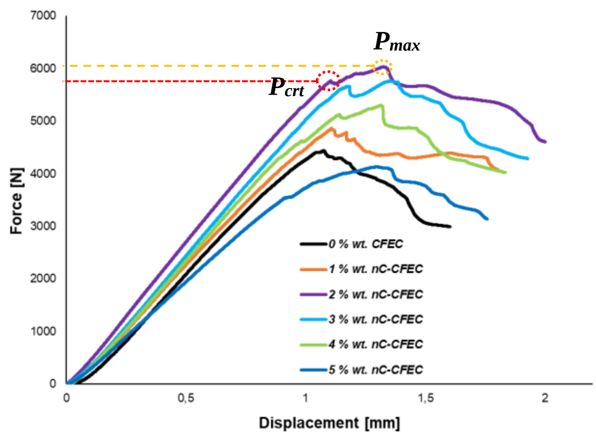


Figure 4. SENB curves of CFEC and nC-CFEC samples

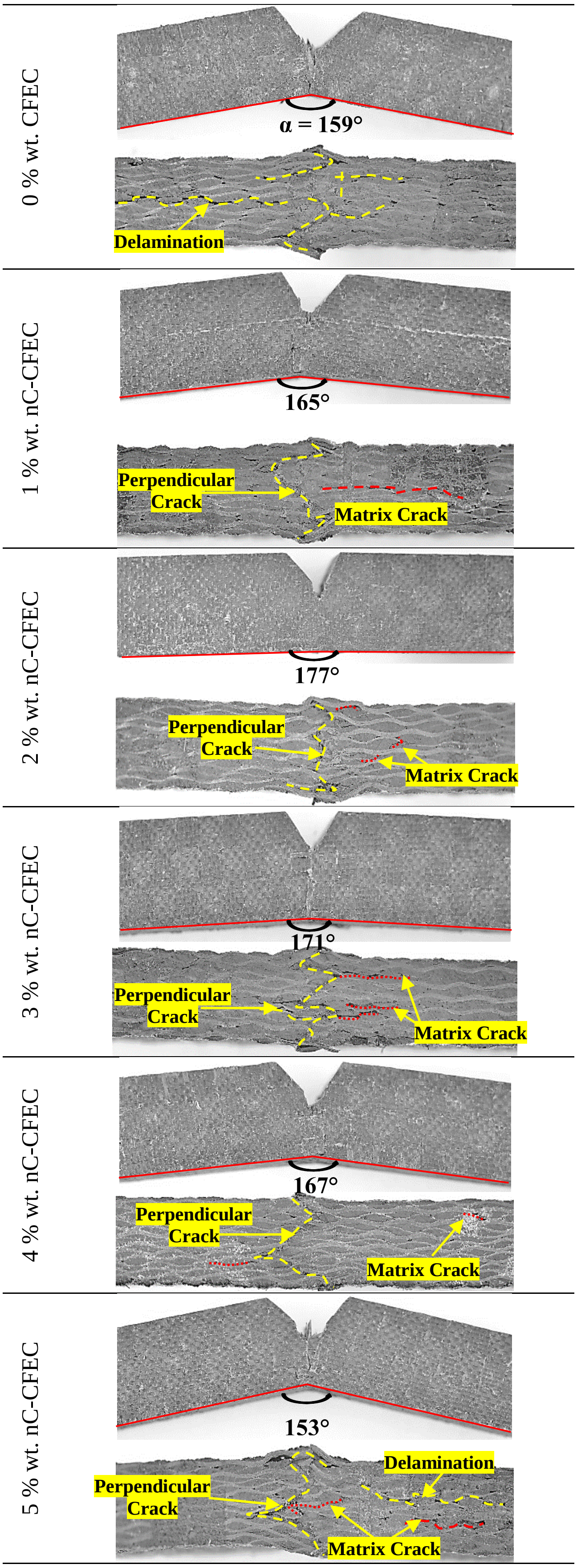


Figure 5. Optical image of damaged SENB specimens

Table 1. Fracture toughness of samples

Samples	P_{max} (N)	Change of P_{max} (%)	P_Q (N)	K_Q (MPa $m^{1/2}$)	Change of K_Q (%)
0 % wt. CFEC	4445.6	-	4405.5	46.4	-
1 % wt. nC-CFEC	4861.5	9.3	4717.0	49.6	6.9
2 % wt. nC-CFEC	6035.3	35.8	5834.3	61.4	32.3
3 % wt. nC-CFEC	5768.4	29.8	5553.1	58.4	25.9
4 % wt. nC-CFEC	5302.3	19.3	5076.6	53.4	15.1
5 % wt. nC-CFEC	4133.6	7 (-)	3867.6	40.7	12.3 (-)

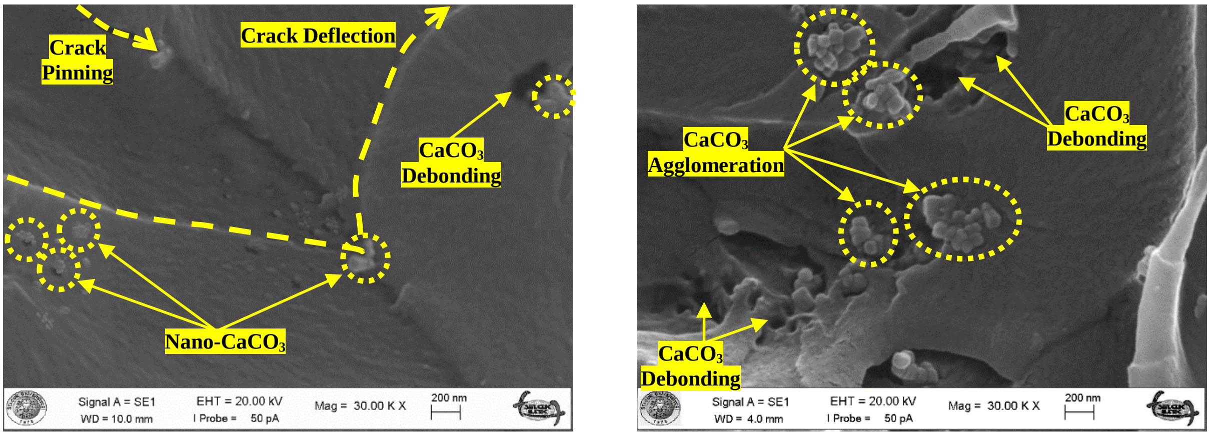
Increasing the nano reinforcement ratio has resulted in a decrease in the bending angle. In particular, the bending angle line of the 2% wt. nano doped sample is became an almost straight line. Accordingly, the 2% wt. nano reinforced sample is not illustrated any significant delamination damage other than matrix cracking and perpendicular crack to the layer. The effectiveness of delamination damage appears to be reduced in the nano-doped samples. In addition, the matrix cracks are formed. However, in the sample with 5% nano reinforced, delamination is reappeared.

3.3 SEM observations of fracture morphology of nC-CFEC

The observation of fracture surfaces of the nanocomposites samples is shown in Figure 6 for 2% and 5% wt. nC-CFEC samples.

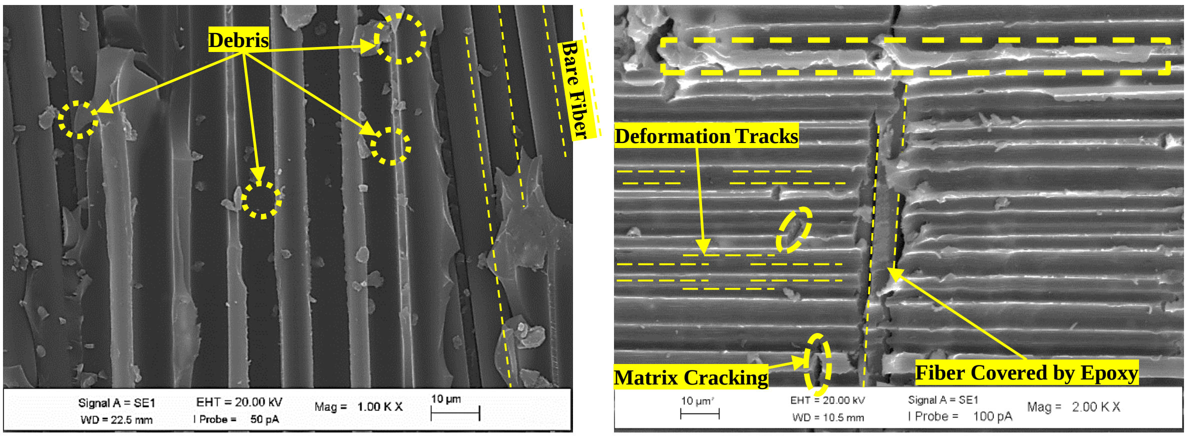
The improved ILSS and fracture behavior performances of nanocomposite samples are related to the toughness mechanisms tracked in Figure 6 (a). The nano mechanisms such as crack deflection, debonding, pinning are increased the fracture energies and interlaminar strength with the homogenous distribution of nano reinforce into the matrix [3, 4]. The distribution of nanoparticles non-homogeneously, which is call the agglomeration, in the matrix may be caused the initiation of cracks due to the residual stresses [4]. It can be said that the worsening of nanocomposites performance containing 5 % wt. $CaCO_3$ is owing to these huge agglomerations in Figure 6 (b).

Figure 7 illustrates a view of the fracture surfaces of the unmodified and modified samples. A number of the fibers are debonded from the unmodified epoxy with smooth fracture areas which indicate poor adhesion at the interface



(a) (b)

Figure 6. Toughness mechanisms of nanocomposites samples a) 2% wt. nC-CFEC b) 5% wt. nC-CFEC



(a) (b)

Figure 7. Fracture surfaces of CFEC samples a) 0% wt. CFEC b) 2% wt. nC-CFEC

for the carbon-epoxy sample (Figure 7(a)). As seen in Figure 7(b), the deformation tracks in the fiber slots are proof of the better interfacial interactions in the 2% wt. nC-CFEC compared to unmodified counterpart sample. Furthermore, the carbon fibers partially covered with epoxy are sighted plastic deformation which shows enhancement of interfacial strength.

As a result, the redesign of epoxy matrix with the $CaCO_3$, which causes an improvement in the fracturing properties of the composite, improves the load-carrying capacity in the interfacial regions by increasing the interfacial interaction of CFEC.

4 Conclusions

The aim of the present research was to examine the effect of nano-$CaCO_3$ particles at different weight ratios on the interlaminar shear strength and fracture behavior of carbon fiber epoxy composite laminates. Resultants from ILSS and SENB tests revealed that the performance of CFEC was remarkably improved with 2 % wt. nano-$CaCO_3$ particles into epoxy. A drastic enhancement at the interlaminar strength was found as nearly 24% increase. Also, the SENB load and K_Q performances of 2 % wt. $CaCO_3$ introduced CFEC improved up 35.8 % and 32.3 %, respectively. Further, optical microscopy analysis revealed the shear failure formation was minimized with $CaCO_3$ reinforcement except for 5% wt. ratio after the ILSS test. In the optical examination of the SENB test, the bending angles of the nano-reinforced samples were decreased based on not catastrophic damage formation such as delamination especially compared to neat counterparts. The SEM examinations demonstrated that the increment of fracture performance was related to the several toughening mechanisms of nano-$CaCO_3$ such as crack deflection, crack pinning, and particle debonding. When the damages on the interface were examined, traces of plastic deformation in the nano-reinforced sample were evidence of a strong carbon fiber / epoxy interaction. These findings provide important insights into the role of nano-$CaCO_3$ reinforcement help to improve the interlaminar shear strength and fracture behavior of carbon fiber epoxy composites.

Acknowledgment

Technical support from Amasya University and Necmettin Erbakan University is much appreciated.

Declaration of interests

The author declares that no known competing financial interests or personal relationships could have appeared to influence the work reported in this paper.

Similarity Index (iThenticate): 8%

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