

Research Article

Experimental Investigations on the Drilling Performance of Carbon Nanotubes Reinforced Glass/Epoxy Multi-scale Composites

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DOI : 10.31202/ecjse.1366208

Received: 25.09.2023 Accepted: 19.01.2024

How to cite this article:

Murat Koyunbakan, Volkan Eskizeybek, Ali Ünüvar, Ahmet Avcı, "Experimental Investigations on the Drilling Performance of Carbon Nanotubes Reinforced Glass/Epoxy Multi-scale Composites", El-Cezeri Journal of Science and Engineering, Vol: 11, Iss:2, (2024), pp.(160-159).

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Abstract : Modifying fiber-reinforced polymers (FRPs) with carbon nanotubes (CNTs) becomes an effective strategy to improve the mechanical performance of the structural parts and add multifunctionality. However, this strategy increases the costs of such new-generation multi-scale composites. Machining of FRPs is challenging due to their susceptibility to machining-driven damages, leading to high-cost wastes. To prevent high-cost waste, the machinability of new-generation multi-scale composites with minimum damage becomes a vital processing issue. This work investigates the impact of CNTs on the drilling performance and hole quality of glass/epoxy multi-scale composites. Multi-scale composite laminates were drilled with high-speed steel drills under dry conditions. Cutting speed and feed rate were parametrically optimized, considering the deformation factor, delamination, and thrust force. The change in thrust force was recorded in situ, and deformation factors were calculated using image processing techniques. Moreover, the damage assessment of drilled holes was carried out with scanning electron microscope analysis to reveal the drilling-induced micro-scale damages. The addition of CNTs within the epoxy matrix increased thrust forces; however, lower delamination failures around exit sides were observed for multi-scale composites. Taguchi technique and analysis of variance were utilized to explore the contributions of drilling parameters and material type to the thrust force and deformation factor. Feed rate and material type were major factors affecting the deformation factor.

Keywords : Carbon Nanotube, Delamination, Multi-scale Composite, Machinability.

1 Introduction

Glass fiber-reinforced polymer composites (FRPs) have been employed intensively in various industries due to their high strength and stiffness, corrosive stability, and low thermal expansion [1, 2]. Although composite structures are designed to be produced to near-net shape, additional finishing operations like drilling are frequently preferred, especially for assembly [3]. The drilling process is utilized intensively to create bolted or riveted assemblies in composite structures, mainly employed in the aerospace and automotive industries [4, 5]. However, the drilling process of FRPs may raise severe defects that lead to the failure of composite structures, such as delamination, debonding, hole shrinkage, and fiber pull-out [6, 7]. Selecting optimum drilling parameters is crucial in achieving low thrust force and good hole quality and avoiding delamination [8].

Drilling fiber-reinforced composite materials can lead to delamination, which impairs the material's service life [5, 9]. Therefore, the drilling-dominated delamination failure of laminated composites has been investigated extensively in experimental and analytical methods [10, 11]. Capello[12] stated that drilling-dominated delamination failure is the primary responsible failure mechanism affecting the lifetime of the material. Mohan et al. [13] demonstrated that drilling parameters such as feed rate, cutting speed, and laminate thickness are the main parameters of delamination failure. Latha and Senthilkumar [14] recorded 3D images of the hole after drilling and established that the feed rate and drill diameter affect the delamination failure.

Different approaches have been suggested to reduce the delamination failure of FRPs. Recently, adding nanoscale fillers within FRPs to improve mechanical and physical properties has become an exciting point in material science. Carbon nanotubes (CNTs) offer to tailor interlaminar regions, well known as the "weak link" in laminated composites, without damaging the structure of FRPs due to their superior mechanical and physical properties [15, 16]. While much work has been carried out to

understand the mechanical properties of CNTs-modified FRPs, little has been carried out in machinability, particularly in the drilling process. Thus, the machinability of the new generation nanocomposites becomes a vital processing issue. Li et al. [17] investigated the drilling performance of CNTs-modified carbon fiber/epoxy composites and provided a reduced delamination factor of about 16% with the addition of CNTs. Kaybal et al. examined the effects of cutting parameters upon thrust force and delamination in carbon nanotube-modified carbon fiber-reinforced plastics. The impacts of the drilling parameters and degrees of influence were determined using the response surface analysis and Taguchi method. They reported that the machinability of Epoxy / CF is better than CNT-Epoxy/CF [18]. Kaybal et al. investigated the thrust force and delamination in drilling carbon-epoxy composites reinforced with boron nitride nanoparticles. According to the experimental results, boron nitride nanoparticles aided in reducing the delamination factor in the machining of the composite material [19]. Depending on the matrix materials, the impact of CNTs on the cutting forces varies. For instance, Mahmoodi et al. [20] and Le et al. [21] reported enhanced cutting forces and tool wear at high MWCNT weight percent in polystyrene (PS) or epoxy-based nanocomposites. It was reported that the cutting force of MWCNTs reinforced polycarbonate composite was less than that of neat polycarbonate. MWCNT/epoxy nanocomposites also produced higher thrust force with increasing CNT wt% in drilling. Kharwar and Verma [22] presented that the enhanced CNT ratio in the epoxy matrix resulted in higher thrust forces. Moreover, in drilling, it has been shown by Çelik et al. [23] and Kumar et al. [24, 25] that CFRPs treated with graphene or graphene oxide (GO) typically result in greater cutting forces, delamination, surface roughness, and circularity error. Once more, there are disparities between research regarding how CNT nanofillers affect drilling of fiber reinforced polymer composites. While some studies found poor machinability with higher thrust forces and delamination in multiscale composites compared with the neat CFRPs [18], others found superior holes in MWCNT modified carbon fiber or glass fiber reinforced composites in terms of reduced thrust force, delamination, and surface cracks, as well as higher residual flexural strength [26, 27, 28].

The disparity in the impacts of CNTs on cutting forces in multiscale composite machining reported in the literature is likely a result of the various materials, CNT weight ratios, and cutting parameters employed in the various experiments. The main goal of this study is to investigate the drilling performance of CNTs-modified FRPs. Different drilling parameters are applied to neat and CNT-modified FRPs under the same conditions to consider the drilling performances of CNT-modified FRPs using high-speed steel drills. Taguchi technique and analysis of variance (ANOVA) were utilized to explore the contributions of drilling parameters and material type to the thrust force and deformation factor. A scanning electron microscopy (SEM) analysis was performed to assess the damage to drilled holes and reveal the drilling-induced micro-scale damages. According to the findings, the current study can fill a gap in the literature on machining CNT-modified composite laminates with minimum waste.

2 Experimental Methods

2.1 Materials

The woven glass fabric (Metyx Composite, 300 g/m²) and diglycidyl ether bisphenol A (DGEBA) epoxy with an aliphatic amine curing agent (Momentive Hexion L285 and H285) were preferred to fabricate composite laminates. Multi-walled CNTs (MWCNTs) used in this study (Cheap Tube Inc., wt 95%) were synthesized by catalytic chemical vapor deposition process with lengths between 0.2 and 2 µm and diameters about 30-50 nm.

2.2 Oxidation of CNTs

The increase in mechanical properties by adding CNTs within polymer matrices is mainly governed by the effectiveness of dispersion within polymer matrix [29]. It is well known that the oxidation of CNTs improves dispersion and interfacial bonding with polymer matrix [30]. Hence, KMnO₄/H₂SO₄ solution was applied to create oxygen-containing functional groups on the CNTs' sidewalls [31]. A desired amount of CNTs/KMnO₄/H₂SO₄ mixture was prepared, and it was bath-sonicated for one hour at room temperature. After refluxing at 150°C for 5 h, concentrated HCl (10 ml) was introduced within the mixture. Finally, the mixture was filtered with polar solvents and dried at 100°C overnight.

2.3 Manufacturing of Multi-scale Composite Laminates

Multi-scale composite laminates are produced using vacuum-assisted resin infusion (VARIM) [32]. The matrix resin was prepared by dispersing 0.3 wt% of oxidized CNTs in the epoxy matrix without hardener using tip sonication (Bandelin Sonoplus HD 2070) for 15 min. The mixture was mixed manually for 5 min after the added curing agent and then degassed at 50°C for 30 min. The neat epoxy mixtures were prepared without utilizing dispersing and degassing processes. The mixtures were slowly infused into a vacuum bag containing six plies of woven glass fabric under negative pressure, cured at 80 °C for 60 min, and then post-cured at 120 °C for 180 min. The laminates cooled to room temperature slowly. The thickness of the composite laminates was 3±0.25 mm. The fiber volume fraction (vf) of the composite laminate was obtained experimentally by the acid bath dissolution process according to ASTM D-3171/15, which was around 52%. Tensile strengths of composite laminates were measured according to ASTM D 3039 standard using an Instron 3369 universal tensile testing machine with a crosshead speed of 5 mm/min at room temperature. At least five specimens were tested for each composition. The typical tensile features of prepared composites are given in Supplementary Table S1.

Table 1: Experimental design using L18 orthogonal array and results.

Exp. No	Material	CS (m/min)	FR (mm/rev)	TF (N)	DF Entrance	DF Exit
1	Epoxy	50	0.05	20.2325	1.1007	1.251
2	Epoxy	50	0.10	37.7433	1.1042	1.2824
3	Epoxy	50	0.20	62.9150	1.1618	1.4191
4	Epoxy	75	0.05	16.5100	1.1128	1.26
5	Epoxy	75	0.10	30.9825	1.1268	1.2834
6	Epoxy	75	0.20	61.2350	1.1642	1.4751
7	Epoxy	90	0.05	14.4750	1.1144	1.329
8	Epoxy	90	0.10	29.8300	1.1324	1.2939
9	Epoxy	90	0.20	49.1850	1.1715	1.3536
10	CNT/Epoxy	50	0.05	21.3033	1.1195	1.132
11	CNT/Epoxy	50	0.10	45.1475	1.1204	1.1338
12	CNT/Epoxy	50	0.20	56.2200	1.202	1.3191
13	CNT/Epoxy	75	0.05	18.5100	1.1279	1.143
14	CNT/Epoxy	75	0.10	39.3975	1.1323	1.1855
15	CNT/Epoxy	75	0.20	52.2400	1.2307	1.3354
16	CNT/Epoxy	90	0.05	17.5725	1.1609	1.169
17	CNT/Epoxy	90	0.10	34.9575	1.1617	1.2186
18	CNT/Epoxy	90	0.20	45.3550	1.2457	1.3699

2.4 Experimental Drilling Procedure

The drilling process of the fabricated composite laminates was utilized at a Mazak Variaxis 500 machining center. A piezoelectric dynamometer (Kistler 9257B) with a charge amplifier and data acquisition board was used to measure thrust forces during drilling. A high-speed steel (HSS) twist drill with two cutting edges (Ø8 mm and a point angle of 118°) was used to drill composite laminates. During the drilling process, the samples were fixed on the dynamometer between two steel supporting blocks with twelve holes (Ø12 mm) (Figure S1). The supporting blocks were preferred due to their advantages during drilling FRPs at high feed rates [33, 34]. Four holes were drilled for each processing parameter (see Supplementary Table 2), and the average thrust force values were calculated.

In the drilling of composite materials, the delamination at the entry and exit of the hole was evaluated via the delamination factor, FD, using the maximum crack length from the hole center. The deformation factor was determined by formula 1. [35]; where Dmax is the maximum diameter of the damage zone, and D is the hole diameter.

A Leica DM2700 M model optical microscope visualized the delamination zones at the drill entrance and exit. Scanning electron microscopy (SEM) (A Zeiss Evo LS 10) was also used to monitor the cross-sections of holes to reveal delamination failure between layers of the laminates. Taguchi technique is used to design high-quality experimental systems [35, 36]. Notably, the drilling parameters were designed to have three levels, while the material type has two. The L18 array was applied to design experiments, as shown in Table 1.

Taguchi method uses signal-to-noise (S/N) ratios to analyze mean response and variation derived from the quadratic loss function. The widely applicable S/N ratios are given in Eqs. 2–4;

$$F_D = \frac{D_{max}}{D} \tag{1}$$

$$\text{Nominal is the best: } \frac{S}{N} = 10 \log \left(\frac{y^2}{s^2} \right) \tag{2}$$

$$\text{Lower is the best: } \frac{S}{N} = -10 \log \left(\frac{1}{n} \sum_{i=0}^n \frac{1}{y_i^2} \right) \tag{3}$$

$$\text{Higher is the best: } \frac{S}{N} = - \log \left(\frac{1}{n} \sum_{i=0}^n \frac{1}{y_i^2} \right) \tag{4}$$

Where y' is the average of measured data, s² is the variation of y, n is the number of measurements, and y is the measured data [13]. The signal-to-noise response tables of the thrust force and deformation factor for each experiment are shown in Table 2.

2.5 Thrust force and delamination factors

The impact of drilling parameters on the recorded thrust forces and calculated deformation factors is shown in Figure 1. In general, the thrust force increases with increasing feed rates for both composite laminate samples at fixed cutting speeds (Figure 1a). Note that higher feed rates result in the increased contact area and load on the tool. The lowest thrust forces were recorded at the highest cutting speed (90 m/min) for fixed feed rates for all cases. This result is attributed to the softening of the polymer

Table 2: S/N response table for thrust force and delamination factor.

Trial No	Thrust Force (S/N Ratio)	Delamination Factor of Entrance (S/N Ratio)	Delamination Factor of Exit (S/N Ratio)
1	-26.120991	-0.83338	-1.94515
2	-31.536804	-0.86095	-2.16047
3	-35.975084	-1.30263	-3.04026
4	-24.354941	-0.92834	-2.00741
5	-29.822329	-1.03694	-2.16724
6	-35.739994	-1.32055	-3.37643
7	-23.212371	-0.94082	-2.47050
8	-29.493065	-1.08000	-2.23801
9	-33.836654	-1.37485	-2.62981
10	-26.568950	-0.98048	-1.07693
11	-33.092674	-0.98746	-1.09073
12	-34.997817	-1.59809	-2.40555
13	-25.348128	-1.04541	-1.16092
14	-31.909373	-1.07923	-1.47803
15	-34.360063	-1.80304	-2.51223
16	-24.896671	-1.29590	-1.35629
17	-30.870807	-1.30188	-1.71722
18	-33.132503	-1.90827	-2.73378

matrix with increasing cutting speed since higher temperatures occur at higher cutting speeds [37]. The high cutting speed and low feed rate result in minimized thrust forces for both composite laminates. Interestingly, relatively lower thrust force values were measured for CNTs/epoxy composites in the case of the highest feed rate. In contrast, relatively higher thrust forces were recorded for other cases, as shown in the figure. Kumar and Singh [38] reported that the thrust force was decreased by adding CNT nanoparticles. Similarly, Soleymani et al. [39] and Rajakumar et al. [40] revealed the diminishing trend in the thrust force by adding nanoparticles into the matrix. This result can be attributed to thermal relaxation of the CNT modified epoxy matrix due to the increased thermal conductivity with the addition of CNTs. Drilling-triggered delamination is an interlaminar failure problem for laminated composites and is fundamentally governed by interlaminar fracture toughness. Delamination damage for drilled composite laminates is expressed numerically by the deformation factor, which is the highest diameter in the damaged area to the diameter of the drill. Several observations can be made regarding calculated deformation factors for the entrance and exit sides, as shown in Figure 1b-c. Deformation factors increase with increasing feed rates and cutting rates as expected. The calculated deformation factors at entrance sides for CNT-modified composite laminates are relatively higher than neat composite laminates. However, an opposite delamination trend was observed for the exit holes, as higher deformation factors were found for neat composites rather than CNT-modified laminates at the processing conditions. The increase in deformation factors regarding increasing cutting speeds and feed rates is around 8 and 18% for entrance and exit holes, respectively. The higher deformation factors at the exit holes can be attributed to the bending effect of thrust forces and burr-dominated delamination. Note that lower deformation factors for CNT-modified laminates were obtained at exit holes, indicating the positive contribution of CNTs on delamination properties.

During drilling, the surface delamination types of laminated composites can be grouped as peel-up delamination at the entrance and push-down delamination at the hole's exit. The CNTs-modified glass fiber-reinforced epoxy composites provide higher Mode I interlaminar fracture toughness than traditional glass fiber-reinforced composite laminates. Our previous studies proved that CNT addition within the epoxy matrix for glass fiber-reinforced composites increased Mode I interlaminar fracture toughness by more than 20% of traditional glass fiber-reinforced composite laminates [41]. Results indicate that CNT modification of the epoxy matrix specifically enhances the delamination resistance of the composite laminate at the exit hole despite higher thrust forces being recorded [18]. These results suggest that adding CNTs increases fiber-polymer interfacial properties and mechanical toughening mechanisms [42, 43].

Generally, the deformation factor increases with increasing cutting speeds at fixed feed rates for both composite laminates. Maximum deformation factors were obtained at the highest feed rates. An almost linear increasing trend for the deformation factor with increasing cutting speeds at fixed feed rates was measured for CNT-modified composite laminates.

2.6 SEM analysis

The effect of CNT modification on the delamination of composite laminates during the drilling process was investigated by SEM analysis. The cross-sections of holes drilled at different cutting parameters were visualized and represented in Figure 2.

SEM investigations revealed that the fibers perpendicular to the tool axis were cut uniformly. Fiber pull-out was not observed for these layers (Figure 2a). Delaminated layers can be seen advancing around the hole surface (Figure 2b). The thrust force plays a vital role in the delamination of layers since the stress originated by thrust force leads to interlaminar crack propagation. Besides, bending and shear occur around the hole, and a combined stress problem appears during the drilling of layered composite laminates.

The uncut fibers as burr for laminated composites can be seen at the laminate cross-section in Figure 3a. Note that the

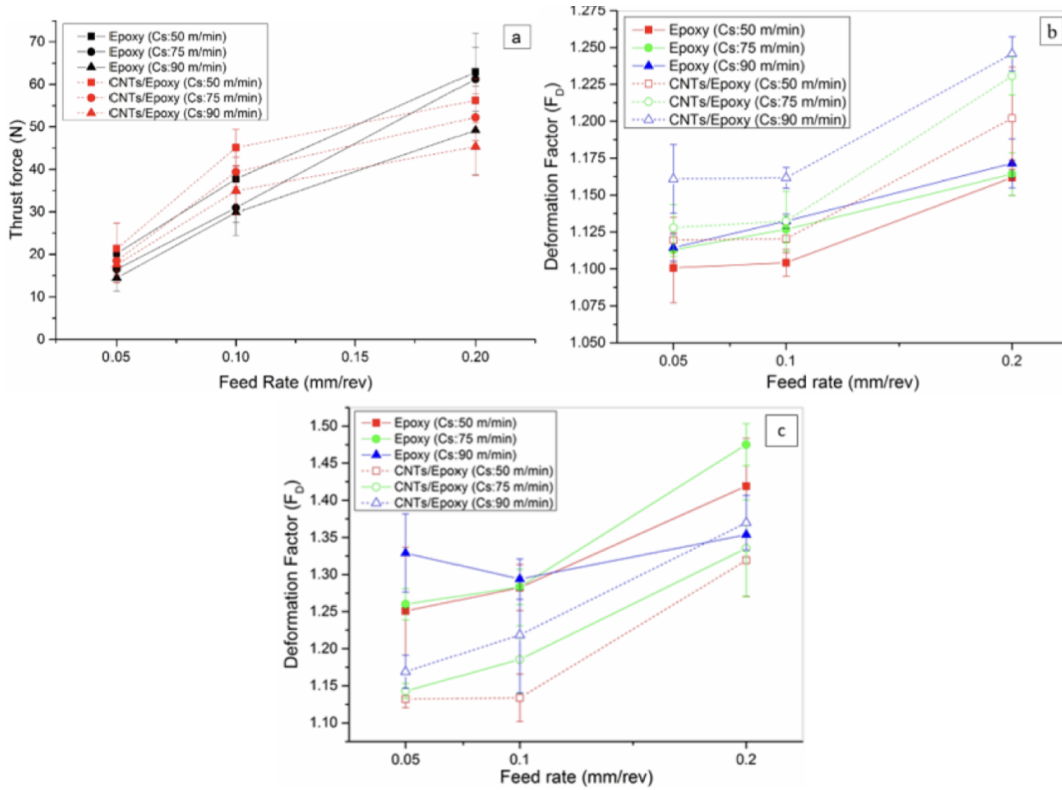


Figure 1: a) Measured thrust forces for epoxy and CNTs modified epoxy GF reinforced laminated composites at different drilling parameters, deformation factors b) entrance, c) exit

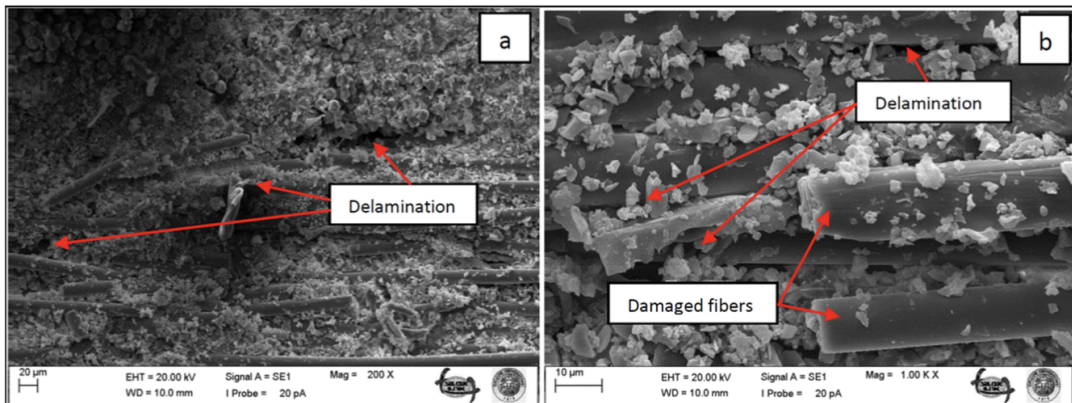


Figure 2: High magnification SEM images of the hole cross-section of GF/epoxy laminates (Feed Rate:0.1 mm/rev; Cutting speed: 75 m/min) a) delamination from resin-rich region of the lamina (the drill entry is located at the upside of the image), b) Fiber damage around delamination path of the laminate (the drill entry is located at the upside of the image)

composite laminates were fixed between two parallel tools to reduce vibration during drilling. However, the uncut fibers were observed mainly at the exits of holes (Figure 3b). Also, the figure represents the delamination damage between fabric layers initiated during drilling, as shown in Figure 3c. CNT pulled out as indicated by arrows, improves interlaminar fracture toughness by bridging and pull-out.

The impact of CNTs on the drilling performance is represented schematically in Supplementary Figure S2. Randomly oriented CNTs within the epoxy matrix were placed between glass fabric plies as resin-rich regions. The resin-rich regions in laminated composites are known to be very sensitive to the formation and propagation of cracks. During drilling, the thrust force and rotation of the drill bit lead to delamination failure by generating compression, bending, and shear stresses in the interlaminar region of the plies. CNTs between the plies within the resin-rich region enhance the contact area and adhesion between the matrix and fibers, increasing interlaminar fracture toughness.

Moreover, the bridging and pull-out of CNTs after crack formation increases the absorption of the fracture energy, which

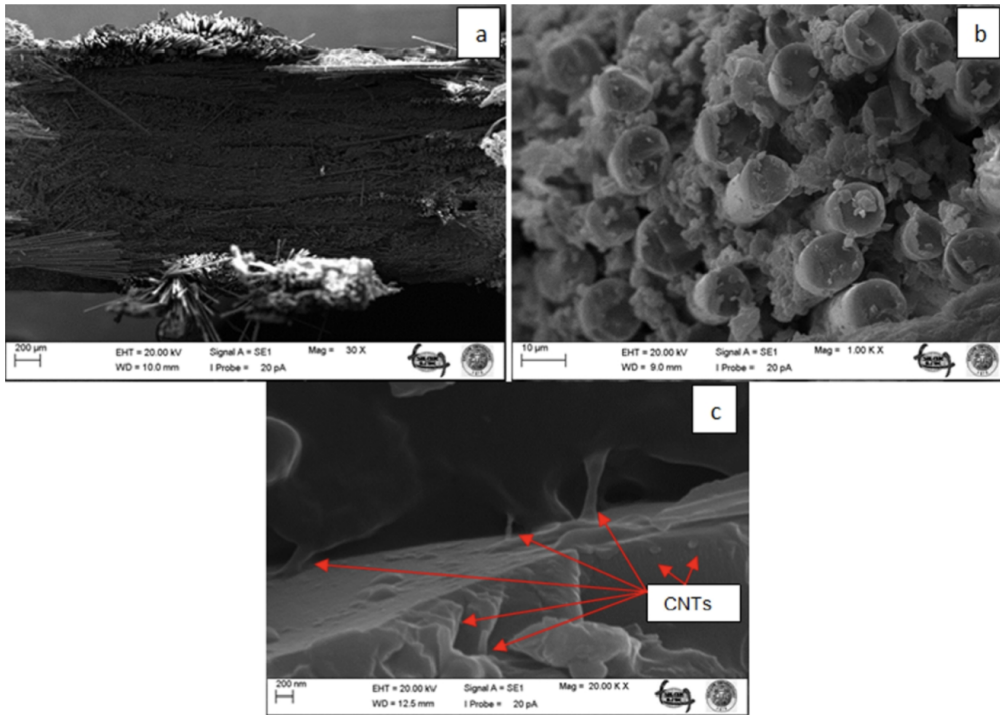


Figure 3: SEM images of the hole cross-section of CNT modified GF/epoxy laminates (Feed Rate:0.1 mm/rev; Cutting speed: 75 m/min) a) low magnification of hole cross-section (the drill entry is located at the upside of the image), b) uncut fiber around drill exit, c) CNTs contributing on delamination toughing of the composite laminate by bridging and pull-out

Table 3: S/N response table for thrust force and deformation factors.

Level	Thrust Force			Deformation Factor of Entrance			Deformation Factor of Exit		
	Material	C.Speed	F. Rate	Material	C.Speed	F. Rate	Material	C.Speed	F. Rate
1	-30.01	-31.38	-25.08	-1.075	-1.094	-1.004	-2.448	-1.953	-1.670
2	-30.58	-30.26	-31.12	-1.333	-1.202	-1.058	-1.726	-2.117	-1.809
3	-	-29.24	-34.67	-	-1.317	-1.551	-	-2.191	-2.783
Delta	-24.354941	-0.92834	-2.00741						
Rank	-	3	2	1	2	3	1	2	3

drives the crack and limits the crack’s propagation between the plies. Thereby, deformation factors were explicitly decreased at the exit holes for CNT-modified composite laminates by considering increased interlaminar fracture toughness with the addition of CNTs. The calculated S/N values for each experiment are represented in Table 3. The S/N ratio response graphs for thrust force and delamination factors at the workpiece entrance and exit are given in Figure 4. Based on the S/N ratio response results, the optimum drilling parameters were obtained as Level 3 (90 m/min) and Level 1 (0.05 mm/rev) for the cutting speed and feed rate, respectively. These results are consistent with our discussions derived from Figure 3.

On the other hand, the optimal material type found is epoxy and CNTs/epoxy for entrance and exit hole surfaces, respectively. Cutting speed and feed rate at Level 1 were obtained as the optimum drilling parameters for the deformation factor at the entrance

Table 4: Table ANOVA for the deformation factors at the entrance.

Source	DF	SS	MS	F	PCR
Material	1	0.2994	0.29936	29.59	18.0116
Cutting Speed (m/min)	2	0.1494	0.07469	7.38	8.9902
Feed Rate (mm/rev)	2	1.0914	0.54583	53.94	65.6915
Residual Error	12	0.1214	0.01012		
Total	17	1.6618			

Table 5: Table ANOVA for the deformation factors at the exit.

Source	DF	SS	MS	F	PCR
Material	1	2.3498	2.34982	38.00	30.5696
Cutting Speed (m/min)	2	0.1777	0.08884	1.44	2.3117
Feed Rate (mm/rev)	2	4.4172	2.20861	35.72	57.4654
Residual Error	12	0.7420	0.06183		
Total	17	7.6867			

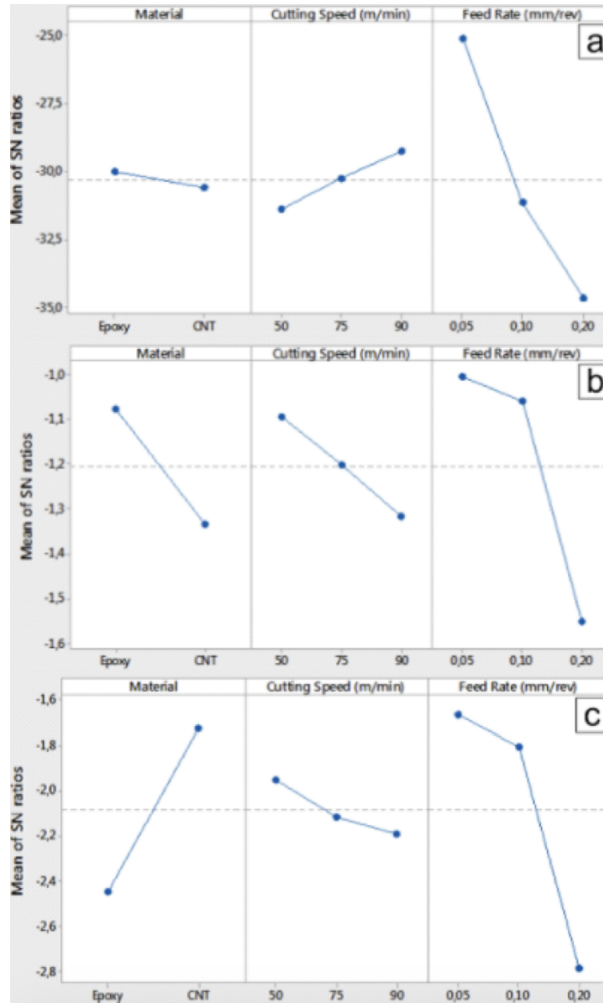


Figure 4: Main effects plots for S/N ratios a) thrust force, b) deformation factor at entrance, c) deformation factor at exit

Table 6: Table ANOVA for the thrust force.

Source	DF	SS	MS	F	PCR
Material	1	1.436	1.436	2.33	0.4713
Cutting Speed (m/min)	2	13.773	6.887	11.16	4.5203
Feed Rate (mm/rev)	2	282.077	141.038	228.66	92.5789
Residual Error	12	7.402	0.617		
Total	17	304.688			

and exit holes.

The analysis of variance (ANOVA) was implemented to realize the effects of material type and drilling parameters on the thrust force (Table 4) and delamination factors (Table 5 and Table 6). Based on the results in Tables 4-6, the feed rates govern the thrust force and delamination. Feed rate dominates the measured thrust force values with 92.57% PCR value, as seen in Table 4. According to ANOVA analysis, the thrust force is not affected by material type. The contribution of feed rate on deformation factors is calculated as 65.69 and 57.46% at the workpiece entrance and exit, respectively. ANOVA results revealed that cutting speed is the less critical parameter affecting the deformation factor, with contributions of 8.99 and 2.31%. In comparison, material type becomes the second one with contributions of 18.01 and 30.56% at the entrance and exit, respectively. The results indicate that CNT modification of the epoxy matrix contributes to decreasing deformation factors at the hole’s exit side by improving the matrix’s mechanical performance and interlaminar properties between resin-rich fiber layers.

3 Conclusions

The CNT-modified multi-scale epoxy/glass fiber composite laminates were drilled with HSS drills under different processing conditions. The CNT modification reduces delamination damages at the exit holes but increases thrust forces. The processing parameters were evaluated to optimize conditions for CNT-modified polymer composite laminates. However, detailed studies

should be carried out with different drills and drill geometries to clarify the effect of CNT modification on the drilling performance of the laminated composites. We believe this study will help researchers working on similar areas zoom in on nanoscale contribution to polymer composite processing. The ANOVA analysis exposed that the delamination factor is mainly controlled by feed rate, as high feed rates lead to maximum damage. However, material type also contributes as the second major factor in the deformation factor, which shows the aid of CNTs in modifying the epoxy matrix.

Acknowledgments

The authors thank the Department of Mechanical Engineering, Selcuk University, for their valuable support.

Authors' Contributions

In this study, MK: Project administration, Conceptualisation, Investigation, Writing - original draft, Visualisation. VE: Conceptualisation, Methodology, Writing - review & editing, Validation. AÜ: Investigation, Methodology, Writing - review & editing, Validation, Supervision. AA: Writing - review & editing, Supervision.

Competing Interests

The authors declare that they have no conflict of interest.

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