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Experimental Investigation on Machine-Induced Damages during the Milling Test of Graphene/Carbon Incorporated Thermoset Polymer Nanocomposites

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Abstract: The fiber laminate composites are extensively used in aerospace, aircraft, automotive components due to their high stiffness, corrosion, moisture resistance, low weight, and durability features. These fiber composites are modified with nanomaterials to acquire the desired manufacturing properties. The complex structure and anisotropic features differ from metals and their alloys. Additionally, the machining principles of fiber laminates significantly differ from conventional engineering materials. The present work investigates the machining behavior and permeates the damage generated while milling of graphene-modified carbon-fiber reinforced polymer nanocomposites (G/C@FRNC). The surface damages and defects caused in the milling samples have been examined through the high-resolution spectroscopy test. The influence of machining constraints such as cutting speed (N), feed rate (F), depth of cut (D), and graphene oxide weight % (GO) has been investigated to achieve the desired milling performances viz. material removal rate (MRR), cutting force (Fc), surface roughness (Ra), and delamination factor (F_d). The outcomes indicated that the cutting parameters and graphene nanomaterial prominently affects the milling responses. The addition of graphene improves the machinability of proposed nanocomposites with lesser defects generated. However, its higher addition can lead to the phenomenon of agglomeration that can reduce the machining efficiency. The damages and delamination generated in the machined sample are low at a higher cutting speed. This work suggests a new system to control the damage and defects to enhance the laminate samples' quality and productivity.

Keywords: graphene oxide; polymer; composite; milling; carbon fiber

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1. Introduction

For the last two decades, fiber laminate composites have been highly utilized in the components of aerospace, naval, space, and automotive industries with better mechanical properties and modified fatigue life [1]. It becomes an adequate substitute for traditional engineering materials such as metallic alloys and non-metallic materials. The selected manufacturing materials must have unique physical and structural properties with a combination of low specific weight and high resistance to degradation. Under these multi-functional conditions, it can ensure economic performance and safety factors. In addition to thermoset polymers, cross-linked connection contributes to higher stiffness, thermal and mechanical properties. The cross-linked nature, anisotropic, and non-homogeneity of fiber composite creates challenging machining issues [2,3]. In the epoxy resin, nanofiller materials reinforcing were regarded as an effective technique to boost the strength and stiffness under different loading conditions. In addition to the feasible nanomaterial in the epoxy matrix could significantly improve the flexural and tensile behavior [4,5]. Various carbon nanomaterials (CNMs), such as fullerenes, nanotubes (NT), nanorods (NR), graphene derivatives, etc., have been used to enrich polymer composite mechanical behavior [6]. The mechanical properties of fullerene (C60) epoxy nanocomposites were characterized by Rafie et al. [7] at the different aspect ratio of fullerene nanoparticles in the polymer matrix. At relatively low nanofiller loading fractions, the epoxy (resin) mechanical characteristics improved substantially through the fullerene nanoparticle (0.1 to 1% of the resin). The effect of multi-walled carbon nanotubes (MWCNTs) on polymer nanocomposites' impact and bending properties at room temperature was examined by Liang et al. [8]. The study disclosed the nanomaterial weight fraction could increase it. The same findings (flexural strength) were explored by Gantayat et al. [9] to analyze the reinforced hybrid composite. Watson et al. [10] researched a modified dispersion method of graphene oxide using a sonic process. The vacuum resin infusion for nanocomposites reinforced by carbon fiber is performed for the fabrication phase. The research explored the effects of the developed carbon nanocomposites' tensile strength and flexural strength. It was noted that the tensile strength of the neat epoxy fiber composite was lower than the GO-modified composites. A similar trend was observed by Cho et al. [11] The positive impacts of graphene nanoparticles (GO) on epoxy resin-based composites were analyzed by Abdullah et al. [12] The GO/epoxy composite was cast and prepared at room temperature by the casting process. The finding decided that nanomaterial performs a vital position in enhancing mechanical properties such as tensile strength, impact strength, and hardenability. The morphological surface analysis confirmed that cracks in the composite were prevented from propagating. Additionally, the results claimed that a variety of industrial applications might use GO/epoxy composites. To produce highly conductive textiles with mechanically tunable, hydrophobic, and TASER protective properties, Ghosh et al. [13] demonstrated an easy-to-scale method of dipping and drying materials. Protective EM-treated organic wool composite smart textile with sensitive touch switches for household and wireless connectivity. With the help of the rGO nanosheets, a robust electrical and EMI shielding network was constructed in electrical and electromagnetic interference protecting application areas. Additionally, Ghosh et al. [14] developed silver nanoparticle-decorated graphene sheets (rGO/Ag) using a two-stage wet mixing technique. Using non-ionic polymer adhesive to create conductive coatings protects against radiation pollution from electronic technologies and equipment.

Milling is the primary machining process in industries to create the slots and channels for the assembly of products. For several years, the milling of isotropic metallic materials has been studied in depth, but these results can sometimes not apply to the machining of polymer laminate composites. According to the work of Davim et al. [15], delamination and fiber damages are generated in CFRP composites during machining in a significant way. It affects the machining efficacy and quality of the samples. It occurs in laminate polymer due to the anisotropic and non-homogeneous nature and similar remarks were observed in the finding of eminent scholars [16–18]. The macro-reinforced (fiber) composites' existing properties are prominently improved by supplementing nanomaterials. Doping theory is sometimes used in fiber composites to investigate polymers' physical and mechanical aspects. However, several valuable studies examined the cutting tool geometry constraints and tool materials for orthogonal cutting [19,20]. The fiber orientation, fiber size, and weave design also affect the machining performances [18,21,22].

The pioneer scholars limitedly attempt the studies on the use of graphene oxide in carbon/epoxy composites. It requires more attention to efficiently utilize polymer nanocomposites for multifunctional products. Studies show that graphene oxide has exceptional characteristics due to better mechanical properties, aspect ratio, and dispersion [5,23,24]. However, machining efficiency computations and control of damages are highly required in the polymer manufacturing sector to improve product quality and productivity.

From an exhaustive state of the art, it has been remarked that the machinability evaluation of laminate composites modified by carbon nanomaterials is passing through the preliminary phase. Researchers' attention, practicing engineers, and academic interest are needed to overcome these modified polymers' machining drawbacks and limitations. It can become an emerging area of research for the stakeholder to create cost-effective and stable products. Because of the emerging mechanical properties, the present work investigates the damages and defects generated during the milling of graphene-modified carbon-fiber reinforced polymer nanocomposites (G/C@FRNC). The control of process parameters, such as the cutting speed (N), feed rate (F), depth of cut (D) and graphene oxide weight % (GO%), is proposed to overcome the surface defects and damages that ensue during the milling procedure. These defects hamper the efficiency of product development by decreasing structural rigidity. The machined surface is examined with microstructural analysis, and the impact of varying constraints has been evaluated. The milling performance is estimated for the desired value of MRR, F_c, R_a and F_d through the control of process constraints.

The present work shows the defect's spectroscopy investigation on the slot wall of the unidirectional laminates composite. The issue of deterioration directly contributes to the breakage of fibers and occurs through the bending and shearing of carbon fibers. The outcomes of the literature work have demonstrated an improvement in the fundamental and functional properties of hybrid nanocomposites. This article proposed a pioneering method to suppress the delamination and damages versus machining parameters.

2. Experimental Work

2.1. Development of Graphene-Modified Carbon-Fiber Reinforced Polymer Nanocomposites

A matrix (epoxy 520) and straight woven 400 GSM carbon fibers were used for the nanocomposite fabrication. The graphene oxide with 255 m^2/g surface area > 99% carbon impurity is used. Due to epoxy's high viscosity, it is challenging to make a homogeneous mixture of graphene nanoparticles and epoxy. Epoxy (thermoset resin at higher viscosity value 1.162 gm/cm^3) was stirred with different amounts of dispersed graphene nanoparticle (1, 2, and 3%) up to 30 min at 60 °C. Finally, a binding agent (Hardener-D) was introduced with the ratio of 10:1 and stirred again with a temperature of 27 °C. The fabrication procedure of nanocomposite samples is described in the schematic diagram (Figure 1). For control of agglomeration level in the mixture, a balance addition of graphene nanomaterial (1, 2, and 3% weight ratio) is employed with reference to previous experimental results and theoretical study [25–27]. Three samples with a selected set of milling parameters were assessed to enhance the reliability of the experimental results. The Hand lay-up method prepares the laminate composite of 18 layers of woven fiber. For better cutting force analysis (in depth), dimensions of 100 mm in length, 100 mm in width, and 10 mm in thickness have been selected for the fabricated sample.

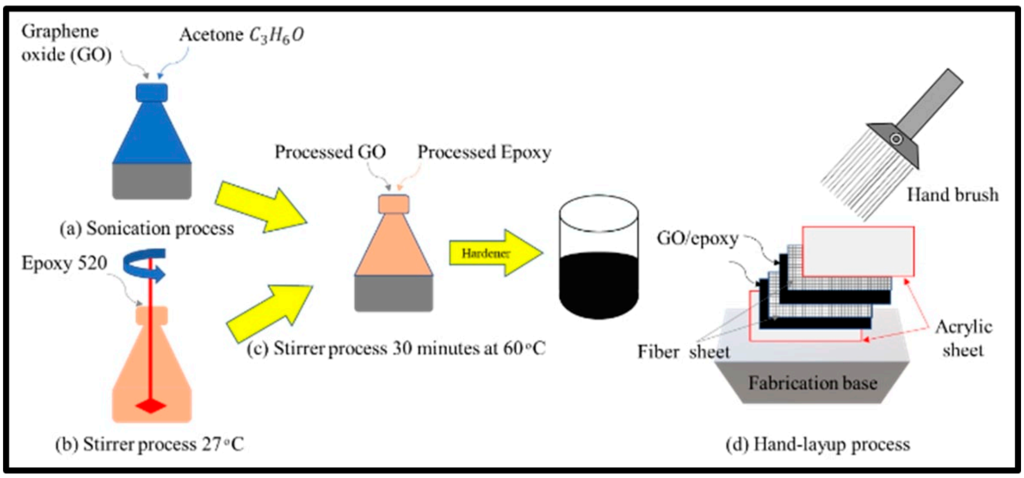

Figure 1. Nanocomposite fabrication procedure (**a**) Sonication process, (**b**) Stirrer process 27 °C, (**c**) Stirrer process 60 °C, (**d**) Hand-layup process.

Fiber thickness 400 GSM = 0.45 mm
Number of layers used in the development composite = 18 layers
One-layer area = 250 × 250 mm^2
One-layer weight = $\frac{250\times250}{400}$ = 25 g
Eighteen-layer weight = 25 × 18 = 450 g
Fiber: resin = 70:30
$\frac{450}{R} = \frac{70}{30}$
R = 192.85 g
1 wt.% of GO = 1.9285 g
2 wt.% of GO = 3.875 g
3 wt.% of GO = 5.7855 g
Epoxy: Hardener = 10:1
$\frac{192.85}{H} = \frac{10}{1}$
H = 19.285 g

GO/C@FRNC laminates nanocomposite with 10 mm thickness (18 plies) were examined in this study. The detailed mechanical properties of GO/C@FRNC are described in Table 1.

Table 1. Mechanical properties of G/C@FRNC laminates.

Sample	Tensile Strength (MPa)	Flexural Strength (MPa)
1	934.35	442.55
2	875.04	363.70
3	774.01	289.95

2.2. Experimental Setup and Response Measurement during Milling of Modified Composites

The fiber polymer nanocomposite plate was machined (milling) under dry environments to investigate the cutting process. A vertical milling machine computerized controlled setup (Model No. MBV 35 TC20) was employed for the milling test of composite samples. The milling cutter tool is made of SiC coated TiAlN, with a 5 mm diameter is selected for the experiment. A dynamometer (Model No. MLB-PML-300) for cutting force and precision balance meter weight was used for MRR calculation during experimentation. The surface roughness tester (Mitutoyo Model No. SJ 210) was measured using mathematical average at three different slot locations, as shown in Figure 2.

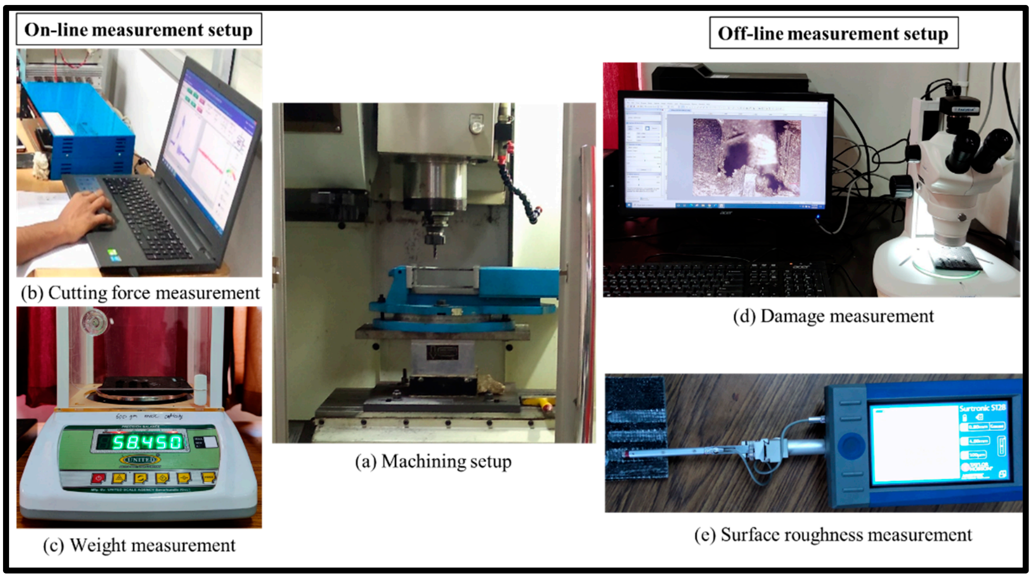

Figure 2. Experimentation setup. (**a**) Machining setup, (**b**) cutting force measurement, (**c**) weight measurement, (**d**) damage measurement and (**e**) surface roughness measurement.

The tomography microscope (Model No. ZSM3780 T2) has software ULTRA-CMOS5100 to estimate delamination value. The milling parameters are described in Table 2. The milling delamination factor (F_d) characterizes the machined slot delamination region (milling slot). F_d is expressed as the ratio of the maximum damage slot length of the machined zone to the nominal slot diameter. The F_d was computed by using the below formula:

$$Delamination\ factor\ (\mathrm{F_d}) = \frac{Maximum\ damage\ slot\ width\ (W_{max})}{Nominal\ damage\ slot\ width\ (W)} \quad (1)$$

Table 2. Process parameters and their working range.

Sr. No.	Factors	Nomenclature	Range/Unit
1	Cutting speed	N	12.56–37.68 m/min
2	Feed rate	F	80–240 mm/min
3	Depth of cut	D	0.5–1.5 mm
4	Graphene nanomaterial	G	1–3 wt.%

3. Result and Discussion

3.1. Influence of Process Parameters on MRR

The cutting speed increased noticeably at a control material removal rate, which is contradictorily associated with traditional machining. The material removal rate (MRR) showed a higher value at medium cutting speed (25.12 m/min). The higher feed value is preferred for MRR; however, its higher level would be more robust. The predominant MRR decreased at a lower cutting depth compared with the conventional materials. In contrast, the cutting depth changed at a higher value, increasing the rate of material removal similar to that of the feed. If the cutting is more thorough, the workpiece's fibers have been cut entirely, and less impact is put on the tool, and successful material removal could be achieved [28]. In turn, results in lower specific cutting pressure, and therefore, high cutting depth to achieve the best performance characteristics is preferred [28]. These may indicate alterations due to irregularities in cutting speed, depending on the cutting force's parameters. In representing the cutting force properties, epoxy/CFRP modifiers are likely to be more ductile because of increased temperature and greater molecular mobility than traditional materials at higher feed speeds. While the lack of effective heat loss from the machined surface and the composite chip accelerates tool wear. At this time, the material's low thermal conductivity contributes to the difficulties of the milling operation. Figure 3 demonstrates that with higher cutting depth (1.5 mm), feed rate (240 mm/min), medium cutting speed (25.12 m/min), and wt.% of graphene nano content (2%), the MRR substantially improved the machining process.

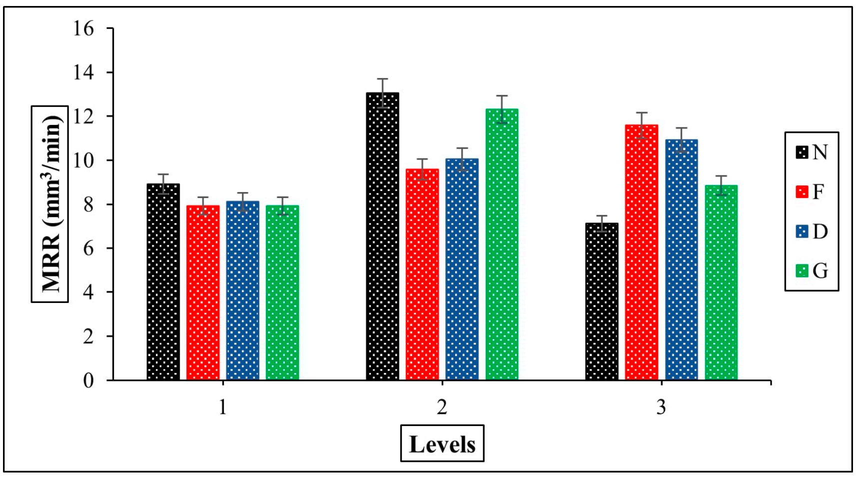

Figure 3. MRR vs. milling factors analysis.

3.2. Influence of Process Parameters on Cutting Force

Cutting forces are the primary factor affecting the quality of the machined surface. It greatly reduces generically with rising cutting speeds (N) and a control combination of the cutting depth and feed rate (F). There is variation in CFRP's cutting force conditions due to the fiber/polymeric material's anisotropy nature at different feed rates. At various cutting speeds (low, medium, and higher), the modified epoxy/CFRP composite material displayed contrasting cutting force features. The hybrid materials exhibit brittle fracture behavior at low cutting speeds that affects their machinability, but medium cutting speed influences the process. The fiber experiences a high strain rate under a high cutting speed, causing it to fail at a lower strain or in a brittle form [29]. It raises the strain rate leading to low-stress fractures and material degradation, thereby increasing the cutting region's temperature and improving the composite's molecular ductility. During machining, a large material flow was observed with the cut fibers compared to higher cutting speed to medium cutting speed, so moderate speed is recommended for lower cutting force [28]. Figure 4 displays the graphical representation of cutting forces versus process parameters at various cutting speeds, feed rates, cutting depths, and weight percent of the graphene filler, respectively. The cutting force is found to a minimum with the cutting speed up to 25.12 m/min and 160 mm/min feed. The reduction force is the minimum due to the ploughing impact at a high feed rate [28]. The cutting force is substantially lowest at a lower cut depth and weight % of graphene nanomaterial. Another remarkable aspect seems to be that the cutting force's intensity increased dramatically for higher cutting depth. It is possible due to the rate at which the cutting tool penetrates more with increased cutting depth and extracts more material, resulting in increased cutting force [30].

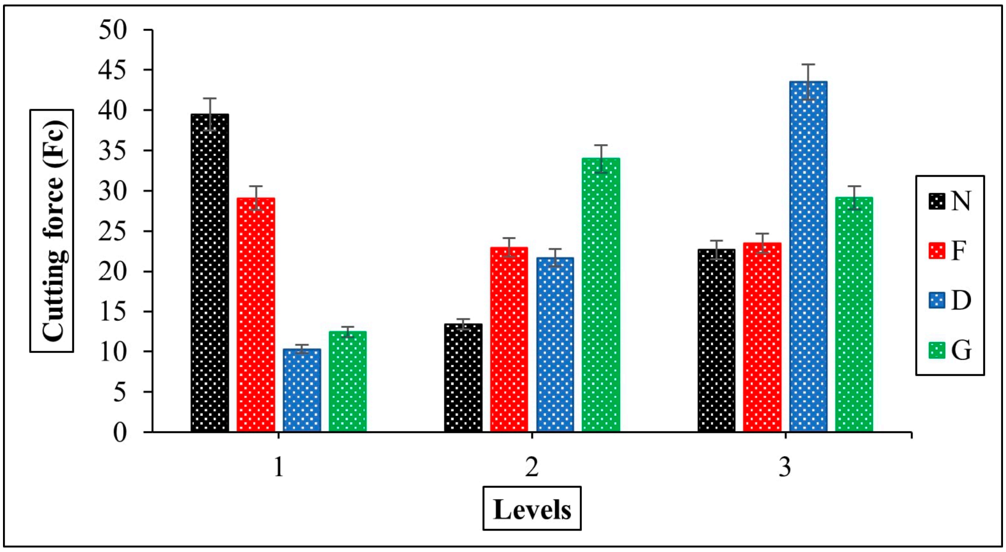

Figure 4. Cutting force vs. milling factors analysis here.

3.3. Influence of Process Parameters on Surface Roughness

Unlike in the traditional milling process where cutting is speedier, feed are more significant, machining surfaces of modified epoxy/CFRP. The explanation for the simultaneous presence of fibre and resin interfacial debonding and the weak resin solidification at high temperatures. Several investigations demonstrated that the polymer is softened by increasing the cutting temperature during the milling process. The degradation of temperatures above the matrix resin can occur, contributing to negative processing quality. The effect of this phenomenon is a poor resin support function for carbon fibers [31]. Alongside that, slightly higher surface roughness can be observed at a high feed level. In the machine tool, the feed rates and cut depth cause vibrations that influence the performance and surface quality of the milling. The reason might be that the rise in cutting force and higher tool wear also occurred with increased depth of cut and defected surface quality [30]. In polymeric materials, lower feed rates related to the material and composites' inhomogeneity create more vibrations during the cutting process. Figure 5 indicates the plots of surface roughness over different levels of process parameters.

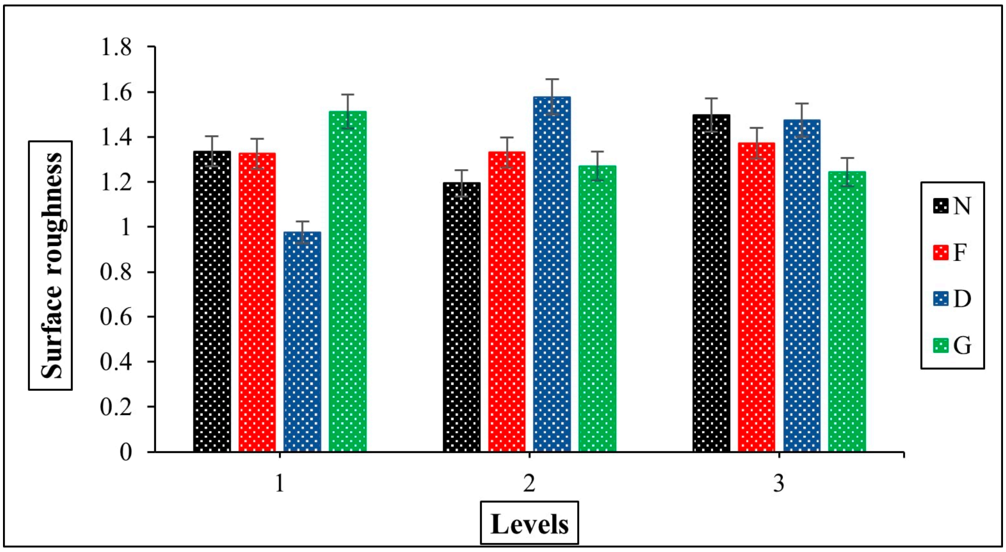

Figure 5. Surface roughness vs. milling factors analysis.

A smooth surface finish was remarked at 25.12 m/min cutting speed with a lower depth of cut. For minimal surface roughness in FRP composites' processing, medium cutting and lower feed rates are recommended [32]. The observation shows that all three weight % of graphene nanomaterials are impacted with a high aspect ratio. The tool–workpiece interface tends to communicate with lubricants due to graphene nanomaterial presence. It indicates the prevalence of ductile transformation in the polymer increases for improved machinability. However, cutting speed is consistent with graphene nanomaterial and is dominated by modified epoxy/CFRP soft machining. With increased nano content, the decrease in surface roughness through 25.12 m/min is indicative of the need to mill these materials at an improved surface finish. Figure 5 shows that with a cutting speed of 25.12 m/min, the surface roughness decreases significantly at lower cutting depth, feed rate, and higher graphene content of wt.%.

The machined sample was observed with a microscope image at different cutting conditions to demonstrate the fiber or composite interfaces' micro deterioration at different milling operation levels. Figures 6–8 show the typical damages incurred while conducting the machining trials. The microscope image of the fiber pullout, uncut fiber, fiber fraying, feed mark, cavity hole, fiber fracture, and crack regarding the different cutting speed conditions indicates higher surface roughness during the milling experiments. The appearance of surface damages may be because of high cutting speed and high feed level. Additionally, it leads to material defect encountered during the composite material process. The microscope images of the slot surfaces are shown in Figure 6a–c. It is observed that surface cavity, feed marks and fiber pullout damage have occurred at the slot region. This is due to interfacial debonding of fibers, higher feed, and the poor support of fiber. Medium cutting speed is recommended for minimal surface roughness in the machining of FRP composites [32]. From Figure 7a–c shows the very few cavity forms, fiber breakage, and pullout. Figure 8a–c shows that more surface damages occurred, propagating the cracks in the laminates. However, high cutting speed leads to higher breakage, pullout, serious cavity formation, and brittle fracture resulting in poor surface finish. This is because the resin is softened with increased cutting temperature, higher feed, and nanomaterials agglomeration.

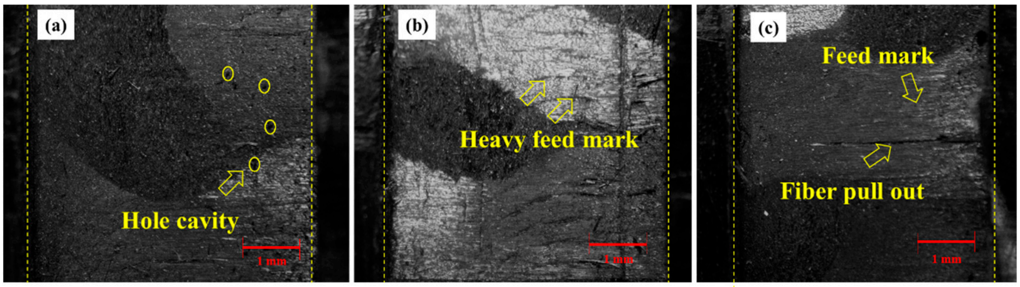

Figure 6. Microscope image of the machined slot at lower cutting speed for (**a**) 1 wt.% GO, (**b**) 2 wt.% GO, and (**c**) 3 wt.% GO.

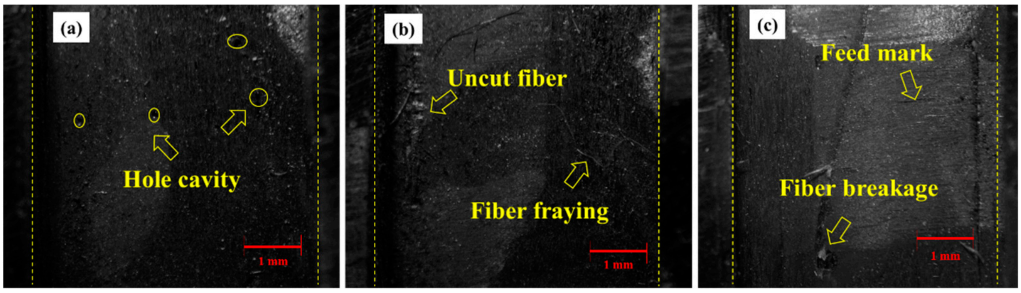

Figure 7. Microscope image of the machined slot at medium cutting speed for (**a**) 1 wt.% GO, (**b**) 2 wt.% GO, and (**c**) 3 wt.% GO.

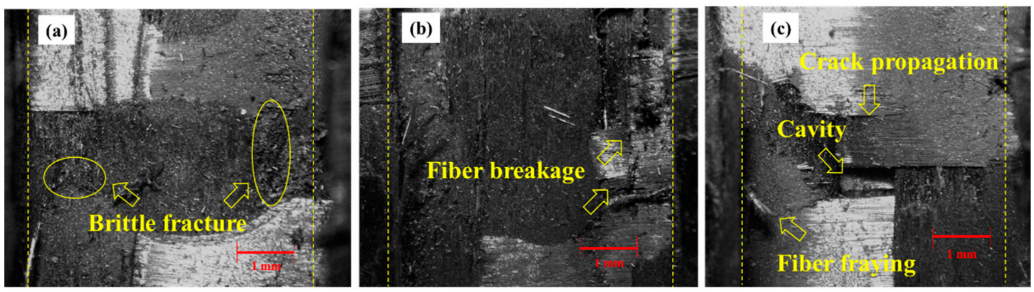

Figure 8. Microscope image of the machined slot at higher cutting speed for (**a**) 1 wt.% GO, (**b**) 2 wt.% GO, and (**c**) 3 wt.% GO.

3.4. Influence of Process Parameters on Milling-Induced Delamination

During the milling of fiber laminates, delamination is a severe and challenging machining issue in polymer composites. It affects product assembly, productivity, and the overall efficiency of the manufacturing process. The control of cutting conditions can overcome this machining-induced damage. The cutting force estimated through the computerized toolbox dynamometer is peaked when the maximum force occurred during the machining process. In ceramics, metallic chip formation is a vital source of information on the milling process's deformation characteristics, besides composites where fiber and resin have unique physical properties and perforation and damage analysis parameters are different. In comparison, the critical cutting force of the synergistic reinforced composite of graphene oxide was greater. The delamination region in the milling test of the proposed nanocomposite becomes more complex due to the plain-woven fiber properties and weaves. These properties cause variation in the cutting force. The high strain rates lead to chip fragmentation, and feed rates indicate a resin fracture early due to the modified epoxy/CFRP milling process combined with high cutting speed. However, the heat produced can increase the cutting zone's temperature with a higher combination of feed and cutting speed, increasing the material's molecular chains' long-range mobility, thus increasing its ductility. In this way, the fracture of fibers tends to increase, causing more severe damage [29]. At the higher cutting speed and feed rate, the chip microstructure is prominent in the long continuous carbon fiber strands' instantaneous fracturing, demonstrating substantial fiber crack propagation on the chip at intermittent lengths. It was found (Figure 9) that due to high rubbing at the work–tool interface at high cutting speed and feed, the bond strength between fiber and matrix decreased so that fibers are either peeling (delamination) or removed from the matrix.

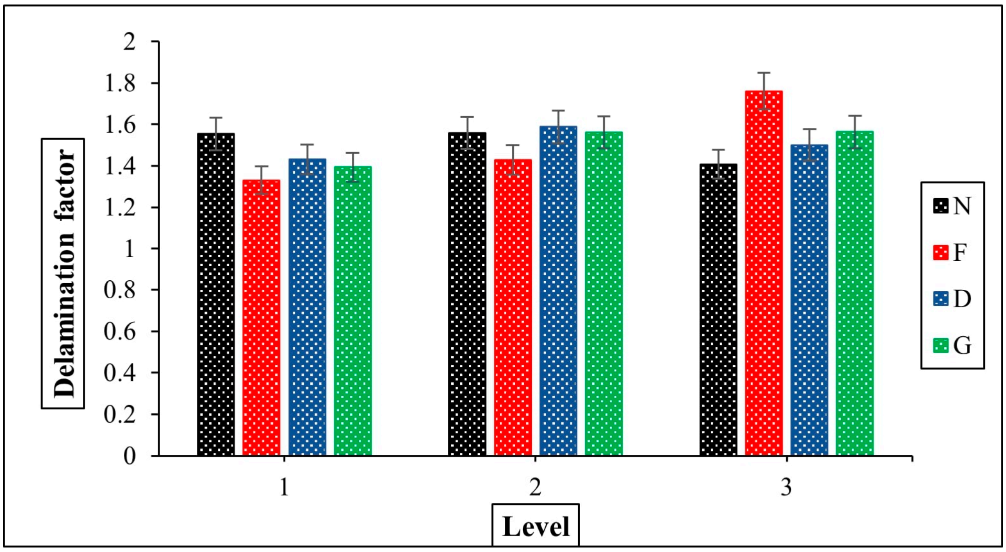

Figure 9. Delamination factor vs. milling factors analysis.

The machined sample microstructure images reveal serrations and segmentation with higher surface damage at the highest feed rate (240 mm/min). A high feed rate leads to more significant vibration during the cutting process, contributing to fracture failure. The higher feed rates resulted in damage due to lower interfacial strength caused by the breakage of the fiber/matrix [15]. The segmentation pattern is sufficient at the lower feed and higher cutting speed, indicating less failure, better machinability, and smooth finishing. At a higher cutting speed, a few polymeric resin crack propagations with fiber pullout is observed at a slightly lower feed rate. This would be due to the lack of bending of the cutting tool, which is accomplished by decreasing the cutting force, smooth discharge of the chip when the cutting speed of the cutting chips exceeds the cutting speed; and a decrease in uncut fiber yarn burrs [19,33]. However, with increased depth of cut, extensive machining surface damage, higher fiber pull can be confirmed of epoxy/CFRP composite [29]. In addition, the damage caused by improved mechanical properties and microstructure is diminished by loaded nanofiller samples [34]. Therefore, graphene nanomaterial affected the cutting force, contributing to more uniform surface topography in doped CFRPs without substantial fibers' breakage [35]. Carbon nanomaterials improve their high surface-to-volume ratio to the contact surface between the reinforcement and the matrix, which results in greater strain distribution and damage resistance [36]. As previously demonstrated by similar research, the G/carbon-fiber-reinforced composites can achieve less delamination damage with a lower feed rate [36,37]. Figure 10a–f show the machining effect on modified epoxy of carbon nanocomposite with 1–3 wt.% of graphene oxide. The aspect of nanomaterials the influence of machine composite is revealed with three different samples. Figure 10a sample containing 1 wt.% of nanomaterial with a 20 μm scale with minimal fiber debonding. Figure 10b sample containing 2 wt.% of nanomaterial with a 20 μm scale with micro-crack, crack propagation, and machined marks. Figure 10c sample containing 3 wt.% of nanomaterial with a 20 μm scale with severe breakage of the machined sample fiber. The current study results were compared to previous ones, and it was determined that the method was feasible in machining while responses were taken individually (Table 3).

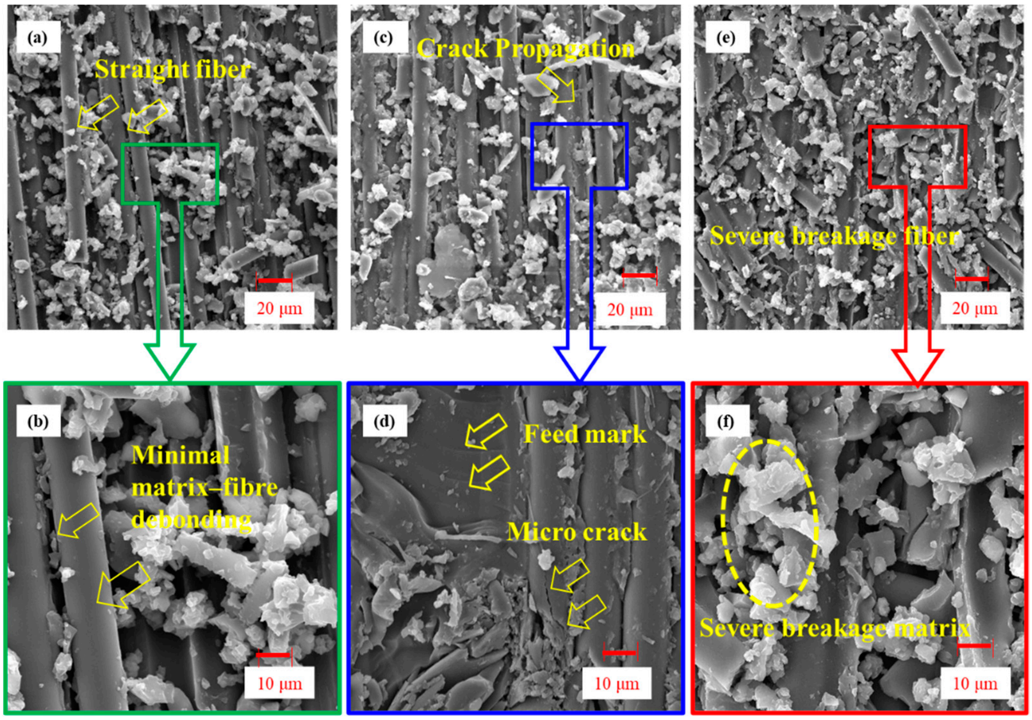

Figure 10. Spectroscopy analysis (SEM) of milling workpiece for (**a**) 1 wt.% GO at 20 μm, (**b**) 1 wt.% GO at 10 μm, (**c**) 2 wt.% GO at 20 μm, (**d**) 2 wt.% GO at 10 μm, (**e**) 3 wt.% GO at 20 μm, and (**f**) 3 wt.% GO at 10 μm .

Table 3. Comparative analysis with the previous result.

Sr. No.	Condition	Response	Present Study	Previous Study	Error%	Ref.
1	N2F3D3G2	MRR	18.768	17.0484	9.16%	
2	N2F2D1G1	Fc	4.706	4.706	-	[38]
3	N2F2D1G3	Ra	0.716	0.730	1.95%	
4	N3F1D1G1	Fd	1.043	1.043	-	[39]

4. Conclusions

The works focus on improving the quality and productivity concerns during the milling of graphene-modified carbon-fiber reinforced polymer nanocomposites (G/C@FRNC). The microstructural investigation through high-resolution results demonstrates the milling efficiency of the fabricated nanocomposites. The effect of cutting parameters on the MRR, Fc, Ra, and Fd has been explored to estimate the polymers' machinability features. The findings of the present article are as follow:

- The cutting force and MRR trend observed in the proposed laminate nanocomposite are similar to traditional materials. The value of MRR and Fc is higher at the combined effect of the higher feed and medium cutting speed;
- The quality of the machined surface can be controlled at a medium cutting speed, lower feed, lower depth of cut and a higher addition of nanomaterial;
- A significant amount of matrix (epoxy) damage is observed at lower feed rates and higher cutting speeds. Furthermore, chip separation around the edge of the machined sample (milling slot) was observed at lower cutting speed and higher value feed;
- The lower value of cutting speed and higher feed value was observed for fiber fraying, fiber pullout, and matrix smearing, with fractured fibers (damage) firmly embedded

in the matrix. However, medium cutting speed machining can be used to effectively machine modified epoxy/CFRP laminates at optimized lower feed rates, the depth of cut depth, and weight% of graphene nanoparticles;
- The addition of graphene oxide contributes significantly to the reduction of milling induce damages. A little addition of graphene improves the machining efficiency of the proposed nanocomposite.

The outcomes of the works show that the proposed nanocomposites can withstand structural component needs. Additionally, the machined surface quality at a medium cutting speed shows the higher application potential for an effectual milling test. The inclusion of other machining operations, such as drilling, turning, etc., can be used as the scope of future work.

Author Contributions: J.K.: Writing—original draft, Methodology, Conceptualization. K.A.: Writing—review & editing, formal analysis. J.X.: Writing—review & editing, formal analysis. R.K.V.: Writing—review & editing, Software, Supervision, Validation, Conceptualization. All authors have read and agreed to the published version of the manuscript.

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