# Evaluation of delamination in the drilling of CFRP composites

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Abstract. Carbon Fiber Reinforced Polymer (CFRP) composite provides outstanding mechanical capabilities and is therefore popular in the automotive and aerospace industries. Drilling is a common final production technique for composite laminates however, drilling high-strength composite laminates is extremely complex and challenging. The delamination of composites during the drilling at the entry and exit of the hole has a severe impact on the results of the holes surface and the material properties. The major goal of this research is to investigate contemporary industry solutions for drilling CFRP composites: enhanced edge geometries of cutting tools. This study examined the occurrence of delamination at the entry and exit of the hole during the drilling. For each of the 80°, 90°, and 118° point angle uncoated Brad point, Dagger, and Twist solid carbide drills, Taguchi design of experiments were undertaken. Cutting parameters included three variable cutting speeds (100-125-150 m/min) and feed rates (0.1-0.2-0.3 mm/rev). Brad point drills induced less delamination than dagger and twist drills, according to the research, and the best cutting parameters were found to be a combination of maximum cutting speed, minimum feed rate, and low drill point angle (V:150 m/min, f: 0.1 mm/rev,  $\theta$ : 80°). The feed rate was determined to be the most efficient factor in preventing hole entry and exit delamination using analysis of variance (ANOVA). Regression analysis was used to create first-degree mathematical models for each cutting tool's entrance and exit delamination components. The results of optimization, mathematical modelling, and experimental tests are thought to be reasonably coherent based on the information obtained.

Keywords: ANOVA; delamination; drilling; regression

## 1. Introduction

CFRP composites are in more demand than ever before in the aerospace sector. The aerospace industry is now concentrating its efforts on creating high-performance carbon fibre composites to address the major issues of lightweight aero-components/structures, cost reduction, and enhanced

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durability. Because high-temperature resistance is necessary for such products, aerospace industries demand high-end structural composites. The majority of high-quality composite components are manufactured with epoxy resin-based prepreg laminates cured in an autoclave at high temperatures and pressures. The fibre volume and void content fraction are critical for aerospace composites performance Shaik *et al.* (2021).

The variability, heat susceptibility, and abrasive processes make machining composite materials challenging Davim *et al.* (2003). One of the major operations in composite machining is drilling, which requires machining with precise hole quality and a short cycle time. Fasteners with holes are frequently used to assemble Composite Structures. Drilling composite materials is crucial for joining their structures as well as bolting and riveting them together. Fiber/matrix debonding micro-cracks, matrix burning, delamination, and deterioration in surface integrity are factors that differentiate composite drilling from metal drilling Iliescu *et al.* (2010).

Drilling geometry should be optimised as well as cutting parameters to decrease delamination occurrences. Delamination is often measured as the ratio of the maximum delaminated area diameter area to the nominal hole diameter Karnik *et al.* (2008). Within the aerospace sector, 60% of parts made from composites are rejected during the last stage of assembly owing to delamination caused by drilling Stone *et al.* (1996). As a result, any drilling-induced delamination that leads to component rejection is extremely expensive. Composite drilling is still a source of concern for the industry and researchers. There is numerous research in the literature that highlights decreasing delamination. Previous literature mainly focuses on the effect of the delamination factor by cutting tool shape and cutting parameters. Durao *et al.* (2008) stated that selecting an appropriate drill bit is useful in decreasing delamination.

Palanikumar et al. (2011) reported that ideal drilling conditions for CFRP material with cementide carbide drills were minimum feed rate, drill point angle and maximum cutting speed. The influence of five various drill geometries (Step, Twist, Brad and Dagger) on delamination and thrust force was investigated by Durao et al. (2010). They stated that the twist 120 drills may minimise the delamination, and that in the Dagger and Step drills the thrust was lower than in the other ones. Qiu et al. (2019) utilised a sophisticated design for drill bit (candlestick, double pint angle and dragger) to eradicate the exit delamination during CFRP drilling. Bayraktar and Turgut (2020) investigated the influence of drill tip angle on delamination of C/C/Al 6013-T651 stacks using coated and uncoated drills and discovered that uncoated drills outperform coated drills in terms of delamination. Krishnamoorthy et al. (2012) examined delamination at hole entry and exit in drilling CFRP composite material with HSS drills with various tip angles (100°-118°-135°) using the Taguchi method and grey fuzzy mechanism. They determined that maximum cutting speed (3000 rpm), minimum feed rate (100 mm/min) and drill tip angle (100 °) were the optimum cutting parameters. The geometry of tools plays an important role in CFRP composite drilling. Gaitonde et al. (2011) have used cemented carbide twist drills for drilling on unidirectional CFRPs with high spindle speed (40000 rpm) and low feed rate (1000 mm/min) and found that drills with a narrower drill tip angle of 85° work better than others. According to Abrao et al. (2008), Brad and Spur drills for CFRP drilling produced better results concerning mechanical stresses and quality of hole surface. In order to assess the performance characteristics in the drilling of CFRP composites using a carbide bit, Abhishek et al. (2016) introduced a regression model based upon the harmonic search (HS) method. Jai et al. (2020) provided an analytical analysis of delamination damage as well as a delamination-free drilling method for CFRP composites. In thick CFRP composites, Sobri et al. (2020) set out a novel approach for assessing drilled hole delamination. For multilayer CFRP/Ti6Al4V stacks of diamond-coated and uncoated carbide twist drills using various drilling

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sequence approaches, Xu et al. (2020) has studied the tool wear signs, drilling forces and temperatures associated with it. As per Jawahir and Van Luttervelt (1993), coatings are preferred with high thermal conductivity as heat barriers with poor thermal conductivity. The usage of various coatings affects not only the thermal characteristics of the drills but also their friction characteristics. Finally, it is necessary to address the characteristics of the workpiece material. It is acknowledged here that increasing the generation of heat makes the polymer matrix smooth and hence reduces the thrust forces. This leads in reduced delamination by decreasing thrust forces. Even if the temperature increase is minimal, it should be kept below the glass transition temperature. This temperature is about 180 degrees Celsius for CFRPs based on epoxy. The CFRP strength will decrease as the temperature exceeds the glass transition temperature and defects such as delamination arise per Yashiro et al. (2013). Wang et al. (20108) state that a diamond coating on the cutting tool will minimise delamination and thrust force. They also reported that diamond coating improves life and tool wear resistance. Diamond-coated twist drills have been shown to be successful. In addition, dagger drills provide great solutions for drilling small diameter holes, while spur and brad drills are effective for minimising hole exit defects in CFRPs per Norbert Geiera et al. (2019). Xu et al. (2022) used diamond-coated candlestick and step tools to drill CFRP and indicated that candlestick drill lowers drill temperature drastically while consistent hole diameters and better surface morphology are achieved when step drills are used. Progressive abrasion wear was confirmed as a dominant wear pattern in both drill types while the step drill was found to chip before the candlestick drill starts chipping acc to Xu et al. (2021). Lin et al. (2021) observed that when brad spur tools were used delamination occurred inside the inner plies and uneven surfaces and cavities were formed among the carbon epoxy composite plies. When uncoated carbide brad spur drills were used by Lin et al. (2021) the cylindricity errors increased while the average hole diameters declined as the tool abrasion wear increased. This indicated that the drilling process and machined hole surface quality depend on the tool wear. According to Xu et al. (2018), the brad spur drill reduces the drilling-induced damage shown by dagger and twist drills. Usage of the non-destructive ultrasonic C-scan technique to quantify the interlaminar delamination inside the composite laminate was proposed by Xu et al. (2018) which enables a three-dimensional criterion.

Previous research revealed that limited work has been reported on the delamination formation in conventional CFRP composites drilling using advanced drill tools with varying drill point angles, coatings, and technologies. To fill this literature gap, factors contributing to the delamination were evaluated at the entry and exit of the hole of bidirectional carbon prepreg fabricated through an "Out-of-Autoclave" process. The processing of OOA prepreg specimens in an oven is the authenticity in this experiment due to its lower manufacturing costs to obtain products of the same quality. According to Shaik *et al.* (2021), Pre-cure techniques like vacuum bag compaction, bagging sequence, vacuum pressure, and vacuum application time are viewed as key variables in producing acceptable quality parts, especially aero structure parts, in the out-ofautoclave. Statistical analyses were done in accordance with the experimental data using the Taguchi L9 (3<sup>3</sup>) OA. As a result, mathematical models were created, and the study's effectiveness was verified. The ultrasonic C-scan technique and drill tool wear effect were not considered in this experiment as there is no specific interest to examine its effect on drilling. Thus, it was considered to be appropriate as it reduces the simulations and computational costs.

# 2. Materials and methods



Fig. 1 Image of CFRP laminate used in drilling tests

Fiber density (g/cm <sup>3</sup> )	Glass transition Temp (°C)	Ũ	Compression strength (MPa)	Compression modulus (GPa)	Interlaminar shear strength (MPa)
1.79	219	924	848	61.5	128

# Table 1(b) Laminate (Specimen) details

No of Layers	Sequence	Approximate Dimensions L×W×T (mm)		
16	[90°/45°/-45°/0°] <sub>4S</sub>	150×150×4		



Fig. 2 Drilling set-up overview

# 2.1 Experimental setup and drilling tests

 $150 \times 150 \times 4$  mm composite specimen shown in Fig. 1 with Out-of-autoclave carbon prepreg HexPly® M56/40%/193PW/AS4-3K high-performance epoxy matrix and  $[90^{\circ}/45^{\circ}/-45^{\circ}/0^{\circ}]_{4s}$  woven fiber orientation was used for drilling tests. Tables 1(a) and (b) show the CFRP composite



Fig. 3 Solid carbide drills (a) Brad point (b) Dagger (c) Twist



Fig. 4 Schema of delamination in drilling CFRP

laminate physical properties and dimensions fabricated for this work.

Tests were carried out in a Leadwell-MCV-610P CNC-VMC under dry cutting circumstances as depicted in Fig. 2. For the machining of CFRP composites, carbide tools with various cutting geometries and coatings were developed. These cutting tools are widely utilised as they generate high-quality holes, cut efficiently and are highly long-lasting. Cutting tool coatings are popular due to their high rigidity, resistance to wear, and low frictional coefficient; nevertheless, they are not encouraged, because of the coating, the radius of the cutting-edge increases which decreases sharpness and makes penetration of the material more difficult for the drill cutting edge. Higher cutting forces and heat generation causes tool wear and reduces surface integrity according to Caliskan *et al.* (2017). An uncoated tool with sharp edges should be employed while drilling aluminum alloys. The coating of the drill makes the heat distribution in the cutting zone difficult due to the low thermal conductivity of the coating.

High precision drilling is required for the installation of fasteners in aerospace and automotive applications. According to Singh *et al.* (2022), brad and dagger drill tools appeared to be better



Fig. 5 Delamination Mechanisms (a) Peelup at the entrance and (b) Pushout at exit

suited for the drilling of CFRP composites. Also, the drill angle is the most essential element which affects the drill's material penetration. This angle changes depending on the material and the method of cutting. As a result, the right drill point angle must be determined for optimal cutting conditions. So apparently in this study, three different 6 mm diameter uncoated Brad point, Dagger and Twist Solid carbide drills were utilised as shown in Fig.3 having angles of 80°, 90° and 118° respectively to explore the drill point angle impact on delamination during drilling. A total of 27 coupons were drilled and tested in this study.

#### 2.2 Delamination factor analysis

The ratio of the hole's maximum damage diameter ( $D_{max}$ ) to the drill diameter ( $D_{drill}$ ) is utilized to calculate the delamination factor,  $F_D$  (Fig. 4). This is frequently utilized to analyse the damage, and it is seen during the drilling of these materials in the form of a peelup and pushout at the entrance and exit of the hole (Fig. 5). The damage diameter was measured using a VEGA 3 SBH scanning electron microscope. The delamination factor's value is calculated as follows

$$F_D = \frac{D_{\text{max}}}{D_{\text{drill}}} \tag{1}$$

# 2.3 Taguchi technique

The Taguchi approach is a very effective method for dealing with responses that are influenced by a lot of variables. The Taguchi method uses a one-of-a-kind orthogonal array design to look at the whole domain with a minimum number of trials. This technique is based on a design of trials in which the factors are distributed across various rows in an orthogonal array. The Taguchi technique is utilized to design the experimental layout, assess the effects of every parameter, and forecast the best process parameter combination. In the experiment, all control parameters were used as feed rate, drill point angle and cutting speed, with three levels defined. Drilling experiments were conducted using the L9 (3<sup>3</sup>) orthogonal experiment design (Table 2). In the

				Levels	
Parameters	Symbol	Unit	Ι	II	III
Cutting speed	v	m/min	100	125	150
Feed rate	f	mm/rev	0.1	0.2	0.3
Drill point angle	$\theta$	(°)	80	90	118

Table 2 Process parameter levels

trials, cutting rates of 100, 125, and 150 m/min were used, with spindle speeds of 4000, 5000, and 6000 rpm, respectively.

After the tests are completed with an orthogonal array, the results are transformed into a signal to noise (S/N) ratio data. This ratio is used to analyse critical process and quality characteristics variables using analysis of variance (ANOVA). To accomplish so, S/N ratios were calculated using the Eq. (2) for smaller is better performance characteristic.

$$S/N = -10\log(\frac{1}{n}\sum_{i=0}^{n}F_{d,i}^{2})$$
(2)

Where  $F_d$  is the value of delamination factor for the *i*<sup>th</sup> trial out of n tests. Based on the experimental data, the effect levels of the variables on the delamination factor were estimated using ANOVA with a 95 percent confidence interval. MINITAB®. 19 was used to carry out the optimization and ANOVA analyses.

#### 2.4 Regression analysis

Regression analysis was utilised in numerous investigations to determine the relationship between experimental results and control parameters as stated by Sarıkaya *et al.* (2018). The relationship between the entrance and exit delamination factor and cutting parameters during the drilling of CFRP Laminate specimens was established using a first-degree mathematical model created using multi-variable linear regression analysis. As a result, control factors such as cutting speed (V), feed (f), and drill point angle ( $\theta$ ) were calculated, as well as entry and exit delamination factors.

#### 3. Results and discussion

#### 3.1 Experimental results analysis

For the present investigation, three different uncoated solid carbide drills of Brad point, Dagger and Twist types are being explored to reduce delamination at entry and exit in the drilling of CFRP composites. This experiment also evaluated the influence of parameters of cutting on delamination.

SEM is used to detect delamination failure and identify the failure mechanism in CFRP coupons. Additionally, energy-dispersive X-ray spectroscopy (EDX) is used to determine ingress and chemical changes in the fiber-epoxy interface and CFRP constituent elements. Following that,



Fig. 6 Typical holes of laminates observed in SEM, drilled with different drills (a) Brad point (b) Dagger (c) Twist



Fig. 7 Images of the drilled surface captured with a scanning electron microscope (SEM) (a) Brad point (b) Dagger (c) Twist

an analytical model for determining delamination reduction factor is proposed. For high-resolution imaging of surface topology, the machine employs Schottky field emission gun (FEG) SEM. This allows for a detailed examination of microcracking and fracture propagation in material surfaces and cross-sections. Element dispersion within sample surfaces is identified and mapped using EDX. In addition, the chemical composition of coupons was obtained. An electron beam is created and bombarded on the substance during EDX. Secondary and backscattered electrons emit from the excitation of electrons within atomic molecules as the electron beam interacts with molecules in the material. These collated electrons' wavelengths and intensities are then used to determine chemical composition.

Fig. 6 illustrates the holes macrograph seen when CFRP composites were drilled with Brad point, Dagger, and Twist drills. Fig. 7 shows a scanning electron microscope surface image of the workpiece drilled with these drills. The figures clearly show that using a Brad point drill enhances



Fig. 8 Sectional view of CFRP drilled with different drills (a) Brad point (b) Dagger (c) Twist



Fig. 9 EDX spectrum of CFRP composite



Fig. 10 S/N graph for delamination factor ( $F_d$ ) (a) Entry, (b) Exit

Test —	v	v f		Ent	Entry		Exit	
	Ι	II	III	$F_d$	S/N	$F_d$	S/N	
1	100	0.1	80	1.152	-1.229	1.048	-0.403	
2	100	0.2	90	1.382	-2.810	1.121	-0.988	
3	100	0.3	118	1.390	-2.860	1.375	-2.766	
4	125	0.1	90	1.231	-1.805	1.054	-0.450	
5	125	0.2	118	1.247	-1.917	1.126	-1.03	
6	125	0.3	80	1.290	-2.211	1.353	-2.62	
7	150	0.1	118	1.112	-0.922	1.103	-0.849	
8	150	0.2	80	1.259	-2.005	1.135	-1.09	
9	150	0.3	90	1.370	-2.734	1.482	-3.410	

Table 3 Test results and corresponding *S/N* ratios for L9 (3<sup>3</sup>) orthogonal array

Table 4 Delamination factor- S/N response table

		Entry			Exit	
Level	v	f	Θ	v	f	$\theta$
Ι	-2.300	-1.319	-1.814	-1.386	-0.570	-1.375
II	-1.978	-2.243	-2.450	-1.371	-1.038	-1.621
III	-1.886	-2.602	-1.900	-1.788	-2.936	-1.549
Delta	0.414	1.283	0.636	0.417	2.366	0.246
Rank	3	1	2.000	2	1	3

Table 5 ANOVA for factors in drilling using uncoated Brad point, dagger, and twist solid carbide drills

Parameters	Degrees of freedom	Seq. Sum Sq	Adj. Sum Sq	Adj. Mean Sq	F	Р	% Contribution
Entry							
Cutting Speed	2	0.00651	0.00651	0.00325	2.69	0.271	8.30
Feed rate	2	0.05430	0.05430	0.02715	22.46	0.043	69.26
Drill point angle	2	0.01518	0.01518	0.00759	6.28	0.137	19.36
Error	2	0.00242	0.00242	0.00121			3.08
Total	8	0.07840					100
Exit							
Cutting Speed	2	0.00732	0.00732	0.00366	4.42	0.184	3.59
Feed rate	2	0.19221	0.19221	0.09611	116.10	0.009	94.38
Drill point angle	2	0.00248	0.00248	0.00124	1.49	0.401	1.22
Error	2	0.00166	0.00166	0.00083			0.81
Total	8	0.20366					100

the hole surface profile. The sectional view of the laminate is shown in Fig. 8. The energydispersive X-ray spectroscopy (EDX) spectra analyses were conducted on the CFRP samples to ensure the distribution and presence of reinforcement and the results are shown in Fig. 9. A compositional analysis using EDS was performed on CFRP samples.

#### 3.2 ANOVA

The S/N graphs in Fig. 10 demonstrate that as the cutting speed increases the delamination formation gradually decreases in all of the experimentations done with three different cutting angle carbide drills (Figs. 10(a) and (b)). When the angle of the drill point increased, it was noticed that the delamination of the entry and exit points also increases. Drilling CFRPs with smaller point angles allows the drill to become stuck on the component more easily than drilling metallic materials. A drill with a lower point angle is recommended to minimise delamination.

As a result of drilling trials using Brad point, Dagger, and Twist solid carbide drills, delamination factors and S/N rates at the hole entry and exit of the CFRP composites were calculated using Eqs. (1) and (2), and are depicted in Tables 3 and 4 respectively. Table 5 illustrates the ANOVA results for the delamination factor at the hole entry and exit while drilling the CFRP composite using Brad point, Dagger, and Twist solid carbide drills. Table 5 demonstrates that feed rate was found to be the most important variable affecting the entrance and exit delamination (P=69.26% and P=94.38%). The optimum values were therefore v3, f1 and  $\theta1$  (Fig. 10(a)) at the hole entrance and v2, f1, and  $\theta1$  (Fig. 10(b)) at the hole exit.

#### 3.3 Regression

Statistical models are influential in evaluating delaminating factors during drilling. This paper used multivariable linear regression analysis to develop a first-degree mathematical model that showed the relation between the entry and exit delamination factors and cutting parameters during the drilling of CFRP composite. As a result, control parameters such as cutting speed (v), feed rate (f), and drill point angle ( $\theta$ ) were calculated, and also entry and exit delamination factors. The first-degree model equation is indicated as Eq. (3).

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 \tag{3}$$

When Y is the response,  $\beta$  is the predicted variation in a mean response of each unit in prediction variables,  $\beta$  is the coefficient of regression. Where  $X_1, X_2, X_3$  are the predictor variables. In the current study, the selected model covers the effects and interaction of key cutting factors. The relation between the response variable and the parameters of cutting is, therefore

$$F_d = \beta_0 + \beta_1 V + \beta_2 f + \beta_3 \theta \tag{4}$$

The model is created and presented as follows based on the above relationship

$$F_{d_{ent-pred}} = 1.264 - (0.00122 * v) + (0.925 * f) - (0.00027 * \theta)R - Sq = 87.66\%$$
(5)

$$F_{d exit-pred} = 0.685 - (0.00118 * v) + (1.676 * f) - (0.00034 * \theta)R - Sq = 99.19\%$$
(6)

#### 3.4 Validation of experiment

The validation of the estimated model is used to verify the results with the experimental results. It is necessary to validate the estimated model and the findings in optimum points. Table 6 shows

	Euroninaant	Taguchi		Regression		
	Experiment	Predicted	Error (%)	Predicted	Error (%)	
Entrance						
<i>v</i> 3- <i>f</i> 1-θ1 (Optimum)	1.125	1.105	1.8	1.152	2.3	
$v3-f2-\theta2$ (Random)	1.256	1.330	6.7	1.242	1.2	
$v1$ - $f3$ - $\theta1$ (Random)	1.346	1.351	0.5	1.398	3.9	
Exit						
<i>v</i> 2 <i>-f</i> 1 <i>-θ</i> 1 (Optimum)	1.030	1.025	0.5	0.973	5.9	
$v3-f2-\theta2$ (Random)	1.156	1.186	2.5	1.167	0.9	
$v1$ - $f3$ - $\theta1$ (Random)	1.333	1.363	2.2	1.279	5.8	

Table 6 Experimental results validation



Fig. 11 Interaction effect of cutting speed and feed rate on (a)  $F_d$  entry and (b)  $F_d$  exit, Interaction effect of drill point angle and feed rate on (c)  $F_d$  entry and (d)  $F_d$  exit

the estimated findings based on the Taguchi technique and mathematical models (Eqs. (5) and (6)) as well as data from the experiments. Table analysis indicates that test values are quite like the estimated values and that this model may thus be used in the CFRP composite drilling process to estimate the delamination factor. According to Cetin *et al.* (2011), statistical analysis errors should be below 20%.

# 3.5 Process parameter interaction effects

The interaction effect of the cutting speed along with drill point angle and feed rate on the delamination factor for both entrance and exit points is shown in Fig. 11. From Figs. 11(a) to (d), In all of the solid carbide drill trials, it was found that as the cutting speed increased, the formation of delamination decreases. As the temperature goes up in the cutting zone by cutting speed, the matrix phase is softening and the cutting process is easier (Figs. 11(a) and (b)). Delamination at the entrance and exit points were established to increase with higher feed rates and higher drill point angles (Figs. 11(c) and (d)). Drilling FRPs with smaller point angles makes it easier for the drill to become stuck on the component than drilling metallic materials. To minimise delamination, a drill with a smaller point angle must be utilized. Protective coatings were not considered for the drill bits used in this experiment as there is no specific interest to examine its effect on drilling.

## 4. Conclusions

The Brad point, Dagger, and Twist solid carbide drill types were used in this study to drill CFRP composites at high speeds. The study focuses on minimising the damage resulting from delamination at the entry and exit of the hole with Taguchi Design. ANOVA was utilized to determine the best process parameter levels for reducing delamination. Furthermore, utilising the regression analysis approach, the trials with each cutting tool resulted in the creation of first-order mathematical models for hole entry and exit delamination. The experimental results of this study draw the following conclusions.

• Uncoated drills performed better in terms of minimizing delamination than coated drills. Due to coating, penetration of the drill into the CFRP becomes difficult due to the reduction in sharpness of the cutting edge.

• The studies indicated that a combination of minimum feed rate, maximum spindle speed and lowest drill point angle should be utilised to minimise hole entry and exit delamination.

• In the drilling of CFRP composites, the feed rate is a critical factor that affects delamination. Lower feed rates are suggested to achieve minimum delamination on the CFRP workpiece. In all drills, the cutting speed and the interactions of the parameters have only a minor impact.

• Drilling of CFRP composites with Brad point drills shows better results than Dagger and Twist drills.

• The optimal parameters of cutting in the entry delamination factor have been determined for all drills at a higher cutting speed (150 m/min) with a low feed rate (0.1 mm/rev) and low drill angle (80°), while the exit delamination factor was determined to be of medium cutting speed (125 m/min) with low feed rate (0.1 mm/rev) and low drill angle (80°).

• Regression models have shown that the experimental results are extremely reliable with statistical outcomes. The measured values were determined in 95 percent CI following

validation trials.

#### Credit authorship contribution statement

Mr. Feroz Shaik conducted experiments and gathered data, as well as assisted in the optimization of process parameters and the preparation of the manuscript. Dr. Ramakrishna Malkapuram developed the experimental strategy and monitored the trials. Dr. Ramakrishna was also involved in the process parameter optimization and manuscript drafting. Dr. K. Chandra Shekar and Dhaval Varma P. actively participated in the revision process and also double-checked the manuscript. During the revision of the text, all three writers collaborated extensively.

#### **Declaration of competing interest**

The authors state that they have no known competing financial interests or personal ties that could have influenced the research presented in this study.

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