Investigation of Experimental Setup for Interlaminar Fracture Toughness of Additively Manufactured Composites

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This study highlights Mode I fracture toughness in additively manufactured composites. Initially employing a design of experiments approach, the investigation aimed to discern the impacts of three variables on interlaminate fracture toughness. However, the initial experiment approach did not yield optimal results. Consequently, a combated approach is taken to explore the setup of a design of experiments and test fracture toughness. Data detailing the relationships between fracture toughness and the printing parameters of print speed, nozzle temperature, and bed temperature are explored. The findings not only elucidate the individual effects of each variable on fracture toughness but also reveal compounding effects when parameters are varied in tandem. Despite the initial setback, this research contributes to the understanding of Mode I fracture toughness in additively manufactured composites and provides key insight into developing a design of experiments approach.

Nomenclature

a	=	delamination length.
b	=	width of the DCB specimen.
F	=	large displacement correction factor.
G_{IC}	=	mode I interlaminar fracture toughness.
L'	=	horizontal distance from the center of loading-block pinhole to edge of the loading block.
m	=	slope of plot log(C/N) versus log(a).
N	=	large displacement and loading block correction factor.
P_c	=	critical force for mode I fracture.
t	=	vertical distance from the center of the pin hole to the midplane of the specimen arm.
δ_c	=	critical load point displacement for mode I fracture.

I. Introduction

LIMITED literature and testing data exist on the Mode I fracture toughness of additively manufactured composites. This paper explores how various factors influence the material properties of high-temperature nylon filament reinforced with carbon fiber. Additive manufacturing technology has made a significant impact on composite materials. Essentially, additive manufacturing creates a three-dimensional (3D) object through layer-by-layer deposition of materials. The layer-by-layer composition influences the mechanical behavior of the part under loading conditions. However, advancements of these new manufacturing capabilities introduce multiple unknowns in the properties of additively manufactured composites. One of these unknowns includes fracture toughness, which indicates a material's resistance to fracturing due to stress. This is particularly relevant for composites used in high-stress environments such as aerospace, automotive, and structural engineering. In the context of composites, Mode I fracture toughness is the measurement of the material's resistance to crack propagation in a direction perpendicular to an applied tensile load on the composite. Several factors within the printing parameters can influence the Mode I fracture toughness of these additively manufactured composites. The research in this paper is intended to follow a

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Design of Experiments (DOE) approach to investigate how the fracture toughness of the composite material behaves when forces are applied perpendicular to the layers of the composite.

A study testing every combination of variables affecting fracture toughness would take years to complete; DOE is a method of statistical analysis that employs analyzing and interpreting experimental data. The advantage of this approach is the ability to combine multiple variables to see how intervariable interaction affects the experiment. The experiment discussed in this document involves a fractional factorial approach where only a portion of possible combinations of variables is analyzed. Additionally, the study employs response surface methodology to systematically explore the effects of various factors on the inter-laminar fracture toughness of these composites. Based on an investigation into previous studies, several printing parameters were considered including printing orientation, layer height, and thickness ratio. After further consideration and a review of recent publications within the space, nozzle temperature, bed temperature, and print speed were selected as the final variables. Previous studies suggest that these factors are the most influential on Mode I fracture toughness. With this DOE approach, the goal is to optimize the printing parameters to achieve higher fracture toughness, which is crucial for ensuring the structural integrity and reliability of the composite components.

II. Methodology

For manufacturing, the specimens will be printed on the Bambu Lab X1-Carbon printer, as shown in Fig. 1., due to its reliability, speed, and affordability. The printer is designed to print higher temperature filaments such as the carbon-reinforced nylon that is this study's focus. Additionally, this printer is a fused deposition modeling (FDM) printer that uses a build platform and a heated nozzle to deposit filament layer by layer. As a result, these layers fuse together and form a 3D model. The printing dimensions for each specimen are 140mm long x 20mm wide x 7.2mm thick. Additionally, a piece of 12.7-micrometer thick polytetrafluoroethylene tape is inserted into the test specimen to induce crack propagation halfway through specimen printing. The initial delamination length in the specimens is 20mm. Further, an additional 20mm will be added to the length of the specimen past the crack starter to ensure printing can resume without warpage after the tape is inserted. The extra 20mm is then removed before double cantilever beam (DCB) testing. Details for the printing parameters can be found in Table 1.

Figure 1. Bambu Lab X1-Carbon FDM printer

Metal hinges are bonded to the specimen on the top and bottom where the length deformation begins. Further, the pin of the hinge is oriented towards the cracked end of the specimen. The bonding area of the specimen and the hinge are lightly sanded with 120-grit sandpaper. The hinges will then be bonded with JB-Weld to the specimens. The JB-Weld is a two-part epoxy system that fully cures in 24 hours and has a tensile strength of 34,611 kPa. Attention to detail must be enforced to ensure that the epoxy does not seep into the crack of the specimen or the hinge. To mitigate this risk, the specimen's crack will be taped off with blue mylar flash tape. Furthermore, it is important to ensure that the specimens will have the same hinge components on top and bottom, as this allows the testing procedure to be conducted more efficiently. For this study, the hinges used have 3-prong and 2-prong variants which will interface together. The 3-prong part is used on the top of every specimen and the 2-prong part is on the bottom. By doing so, the testing will be done effectively and efficiently by unpinning the specimens from the machine without replacing the hinges inside the machine grips. A diagram of the specimen with the hinges can be seen in Fig. 2.

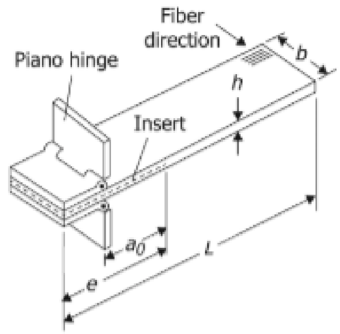

Figure 2. DCB specimen diagram from ASTM D5528

To avoid printing every possible specimen combination, a system of experiments will be developed using the DOE approach. Table 1 illustrates the different printing conditions initially chosen for this experiment. The limits of the printing parameters are chosen based on literature reviews and limitations of resources. Table 2 details the exact parameters at which each initial test specimen will be printed. Three specimens will be printed for each combination, adding up to a total of 45 specimen prints.

Table 1: Non-dimensional coded levels and the corresponding actual levels

Coded Level	Actual Levels		
	Print Speed	Nozzle Temperature	Bed Temperature
X_i	X_1, mm/s	X_2, °C	X_3, °C
-1	35	270	90
0	50	285	105
1	65	300	120

Table 2: Specimen specifications

Specimen Number	DOE Weights			Experimental Variable Values		
				Print Speed, mm/s	Nozzle Temperature, °C	Bed Temperature, °C
1	-1	-1	-1	35	270	90
2	1	-1	-1	65	270	90
3	-1	1	-1	35	300	90
4	1	1	-1	65	300	90
5	-1	-1	1	35	270	120
6	1	-1	1	65	270	120
7	-1	1	1	35	300	120
8	1	1	1	65	300	120
9	-1	0	0	35	285	105
10	1	0	0	65	285	105
11	0	-1	0	50	270	105
12	0	1	0	50	300	105
13	0	0	-1	50	285	90
14	0	0	1	50	285	120
15	0	0	0	50	285	105

Each specimen will undergo a DCB test. The fixture will move at a constant crosshead rate of 0.5 mm/min. This is accomplished using an Instrong load frame as seen in Fig 3. The machine's grips hold the match for hinges bonded to the specimens. When testing is complete for one specimen, it is unpinned, and another specimen is loaded into the machine without loosening the grips. Highspeed and resolution cameras will be aimed at the specimen to capture images as load and displacement changes. The load frame outputs the displacement and load of the frame as the test progresses. As the crack progresses past the cameras' view (around 100 mm from the beginning of the specimen), the test resides.

Figure 3. Instrong Testing Machine

III. Results

The initial Mode I fracture toughness DOE conducted encountered challenges, primarily due to inconsistencies arising from the manufacturing and processing of the specimens. The inconsistencies discovered included the painting of the specimens, as well as the crack propagation technique during printing. Due to the resources available and advisement, the specimens were spray painted with a gloss white color. Despite attention to detail during the printing process, the application of spray paint introduced unexpected variables that influenced the fracture toughness measurements. It was observed that the paint not only infiltrated the crack itself but was also present between the print layers as seen in Fig. 4 and Fig. 5. As a result, spray painting the specimens contributed to inconsistent data for the original prints. This presence of paint between layers altered the bonding properties, contributing to an overall increase in strength rather than providing a true representation of the fracture toughness of the material.

Figure 4. Paint affecting crack propagation

Figure 5. Paint between print layers

In addition to the challenges posed by the application of paint, the experimental setup encountered complications related to the use of polytetrafluoroethylene tape to propagate the initial crack. While intended to facilitate controlled crack propagation for fracture toughness testing, the tape introduced defects that affected the integrity of the printed specimens. Specifically, the tape hindered the fusion and adhesion of the top layer of the print. As a result, the top layer allowed inadequate bonding to the layers, compromising the overall structural integrity of the specimens. The compromised adhesion introduced variability in the mechanical properties of the specimens, further complicating the interpretation of experimental data. The top layer that bonded to the tape can be seen in Fig. 6.

Figure 6. Layer adhesion to polytetrafluoroethylene tape

These observed discrepancies in fracture toughness underscore the importance of controlling all variables in the experimental setup. In response to these challenges, a decision was made to print three additional specimens to test. This adjustment aimed to isolate the effects of printing parameters on fracture toughness without the influence of experimental error variables. To do so, no paint was applied to the specimens and mitigation techniques for the layer adhesion to the polytetrafluoroethylene tape were enforced. The new specimens are 160mm long x 25.4mm wide x 7.2mm thick and the parameters for which the specimens were manufactured are listed below in Table 3.

Table 3: Non-dimensional coded levels and the corresponding actual levels for new specimens

Coded Level	Actual Levels		
	Print Speed	Nozzle Temperature	Bed Temperature
X_i	X_1, mm/s	X_2, °C	X_3, °C
-1	65	285	120
0	65	285	120
1	65	285	120

After printing the new specimens with adjusted parameters, testing was conducted to evaluate the fracture toughness. The fracture toughness testing involved subjecting the specimens to controlled loading conditions designed to induce crack propagation and measure the resistance to fracture. The testing equipment and setup can be seen below in Fig. 7.

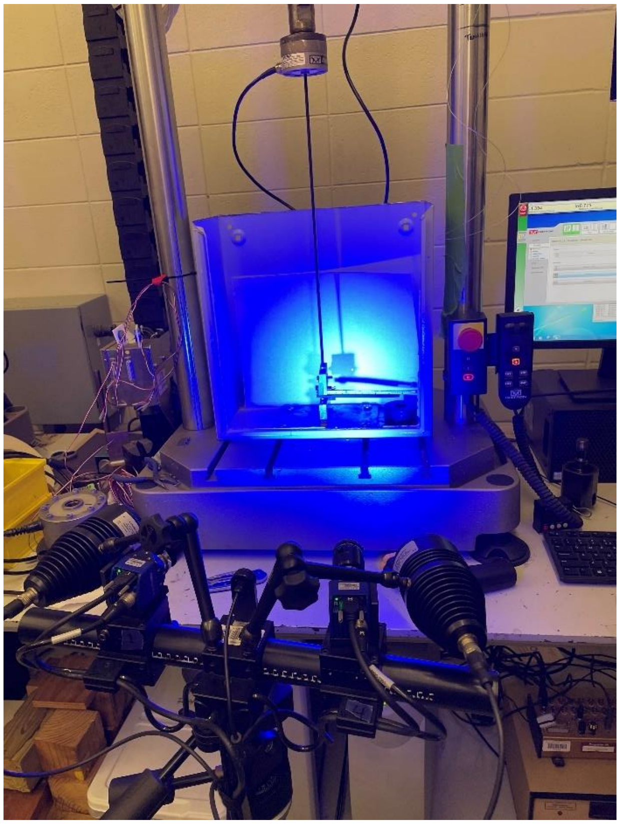

Figure 7. Testing setup

Data gathered from the testing of the new specimens revealed notable improvements in fracture toughness compared to the initial experiments. The absence of paint and the optimization of printing manufacturing led to enhanced layer adhesion and structural integrity, resulting in more consistent and reliable fracture toughness measurements. The specimen being tested can be shown below in Fig. 8.

Figure 8. Specimen being tested

The data collected from the Instrong testing machine for each specimen was recorded which can be seen in Fig. 9 and Fig. 10. The data from the testing procedure provided the displacement, time, and loading parameters for the analysis of the specimen's response to the applied forces and its fracture behavior. Displacement measurements track the extent of the crack propagation and growth as the specimen undergoes loading. The time was also recorded as it captures the duration of loading and the rate of crack propagation. Time-dependent phenomena like creep and stress relaxation can significantly influence fracture behavior and must be considered in the analysis. Monitoring the time-to-failure also offers valuable information about the material's durability and resistance to crack propagation over some time. Additionally, the loading data provides a direct measure of the applied force and stress on the specimen. By analyzing the relationship between loading and displacement over time, statistical analysis can be employed to extract the mechanical properties such as fracture toughness, which can be used for assessing the material's structural integrity and performance.

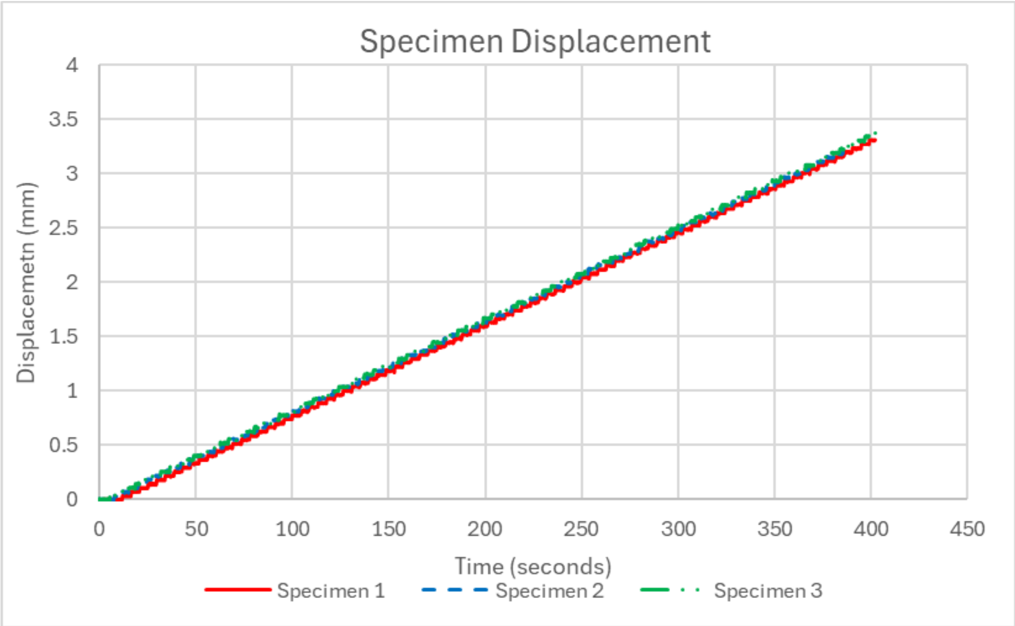

Figure 9. Specimen displacement

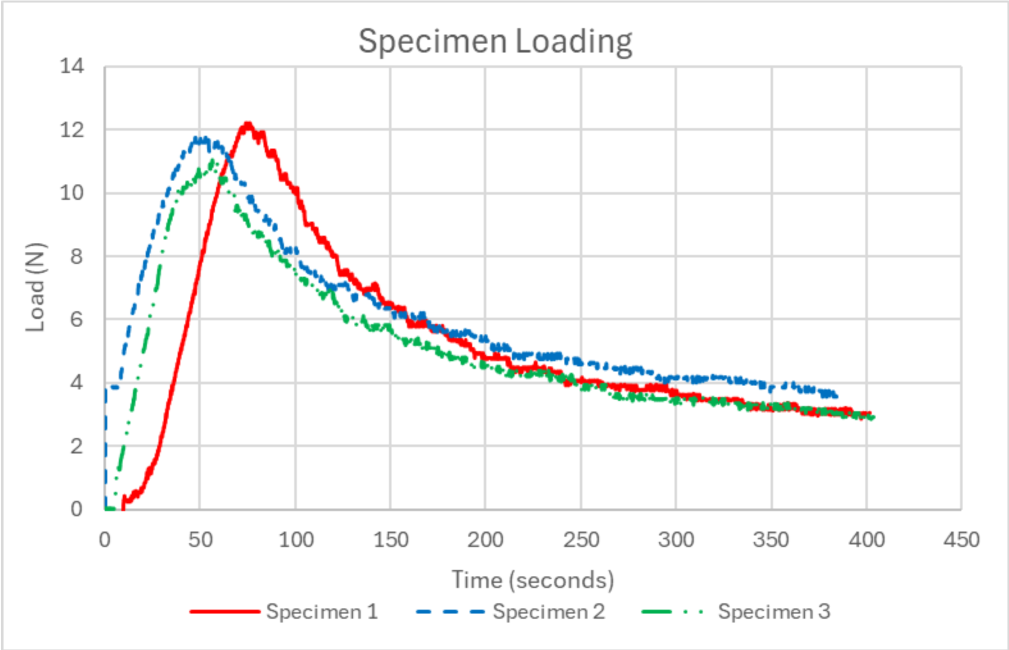

Figure 10. Specimen loading

With a fractional factorial design for the statistical analysis of the fracture toughness, a subset of the potential combinations of factors is systematically chosen to reduce the number of experimental runs while still capturing essential information about factor effects and interactions. This approach utilizes resources while maintaining the ability to assess the influence of selected factors on fracture toughness. The initial factors selected included the print speed, nozzle temperature, and bed temperature. By carefully selecting these factors to include in the fractional factorial design, identifying the significant effects that these factors have and contribute to fracture toughness can be explored. Statistical analysis techniques can be employed to interpret the experimental data and determine the most influential factors affecting fracture toughness. For this study, once the test data has been analyzed, a comprehensive DOE model will be created with a fractional factorial design. Using Eq. 1, the Mode I interlaminar fracture toughness, G_{IC}, of each specimen will be calculated with the compliance calibration (CC) method, which adheres to ASTM D5528. For Eq. 1, F is a parameter that corrects for the effects of large displacement at fracture and is calculated using Eq. 2. Additionally, N is the parameter that accounts for the stiffening of the specimen by the load blocks or piano hinges which is calculated using Eq. 3.

$$G_{IC} = \frac{mP_c\delta_c}{2ba}\frac{F}{N} \tag{1}$$

$$F = 1 = \frac{3}{10}\left(\frac{\delta_c}{a}\right)^2 - \frac{3}{2}\left(\frac{\delta_c t}{a^2}\right) \tag{2}$$

$$N = 1 - \left(\frac{L'}{a}\right)^3 - \frac{9}{8}\left[1 - \left(\frac{L'}{a}\right)^2\right]\left(\frac{\delta_c t}{a^2}\right) - \frac{9}{35}\left(\frac{\delta_c}{a}\right)^2 \tag{3}$$

The statistical calculations based on the interlaminar fracture toughness include sample mean, sample standard deviation, and sample coefficient of variation, and will be calculated after further analysis of the data collected from the three specimens. Any results that appear to deviate from predicted outcomes, such as crack migration, early-onset crack propagation, and delamination values outside of 3-5 mm, will be explored and discussed in the future iterations of this study.

IV. Conclusion

Data gathered from the second set of samples shows an average maximum load of 11.682 N with an average displacement of 0.446 mm. The data of the three specimens is consistent and shows similar trends, which is promising for future work with this study. Before an analysis can be fully completed, more specimens will need to be printed. Future work includes creating 45 more specimens that will be printed according to the specifications found in Table 2. Changes to these specimens include a 76mm delamination insert, a coating of typewriter correction fluid, and the introduction of a controlled chamber for the filament roll. These changes will allow the experiment to adhere to ASTM D5528 and mitigate risk of unexpected variation in the samples. The specimens may also need to be printed longer and trimmed down to mitigate warpage and print failure on top of the delamination insert. Data must be gathered from these specimens, and an average Mode I fracture toughness can be found. The data would need to be analyzed to ensure that variation due to unconsidered variables has been eliminated. The previously mentioned processes are ultimately the result of countless hours of a lesson-learning process that spanned over the course of several semesters. The team encountered many challenges including printing failures, unexpected ASTM deviations, and inconsistencies with testing. Though these were difficult to overcome, the finalized product underscores the importance of proper variable control and specimen preparation and hopefully paves the way for future work in the study of additively manufactured composite materials.

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