

## Shear Test Characterization of 3D Printed Polyamide Reinforced Carbon Fiber Composites

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Received 18 May 2022, Received in revised form 23 September 2022

Accepted 23 October 2022, Available online 30 March 2023

### ABSTRACT

The issue of delamination occurring on PACF materials is a structural application failure due to poor mechanical strength. The bonding process of composite materials between layers using fused deposition modeling (FDM) printing affects the tensile strength of the material. The main objective of this study was to study the shear strength of polyamide reinforced carbon composite (PACF) materials by using shear test method and to study the effect of fractured or delamination surfaces during shear testing. In this study, PACF material was printed using FDM technique through Ultimate S3 machine. A total of 20 printed PACF specimens underwent a semi-overlapping adhesion process using Loctite E-20HP epoxy adhesive which required curing for 24 hours at room temperature. Shear test methods were performed on PACF composite specimens using Universal Testing Machine. Furthermore, PACF samples were conducted experiments to determine the physical properties of the composites of the material as well as Scanning Electron Microscopy (SEM) experiments through fractured or eliminated surfaces. Based on the results obtained, the PACF material has a high shear tensile strength compared to that of epoxy adhesives with an average rate of 1.6 MPa respectively. This is may due to the low curing temperature and the thick epoxy layer. It was observed that the FDM printing method produces a porous print layer that can facilitate delamination to occur.

**Keywords:** Fused Deposition Modeling (FDM); PACF composite; Delamination; Scanning Electron Microscopy (SEM)

### INTRODUCTION

The bonding of composite materials between layers has been applied in 3-dimensional (3-D) printing. 3D printing is referred to as additive manufacturing (AM) or rapid prototyping (RP), which is the process of making an object into a 3D model by using filaments in a layer-by-layer construction process automatically (Sanei and Popescu 2020). This is able to eliminate some of the problems associated with the conventional manufacturing process of formative manufacturing production techniques (Ye et al. 2019).

Now, there are many technologies that use additive manufacturing methods as well as several others including Fused Deposition Modeling (FDM) (Kulkarni et al. 2000). FDM is the best 3D printing method for polymer fabrication because of its relatively low cost, low material wastage and ease of used (Dickson et al. 2020; Wahid et al. 2019). FDM shapes 3D geometries through the successive deposition of layers of extruded thermoplastic filaments to form geometries (Yap et al. 2020).

However, the issue of exfoliated surface or delamination is the most critical defect issue on composites that can occur on polymer printing on FDM. This is because composite laminate layers have weak bonding or no bonding between

layers (Safri et al. 2018). Banea and Da Silva (2009) reviewed delamination seen as a major issue that can occur and needs to be addressed before the use of more representative composite materials in structural applications.

There are many adhesives on the market that have different adhesive strengths and are used to bind different materials as well as make it easier to connect components without compromising the mechanical performance of the structure (Yap et al. 2020). Past research has mentioned the adhesive such as epoxy, urethane, vinyl ester, phenolic, poly- ester, and polyurethane was used to holding materials together (Balla et al. 2019; Othman and Zainordin 2021).

According to Brito et al. (2021), using inappropriate adhesives will lead to major structural failures that should be avoided. The main advantages of using adhesive as a connection method are that it has better static and dynamic fatigue resistance, more uniform stress distribution, peeling and impact resistance, as well as structural weight reduction and load sharing (Quattrocchi et al. 2021).

Based on the previous studies, the used of adhesives such as epoxies in carbon composite laminate was the most effective way for a joint and connection details (Bagherpour 2012). Epoxy also served as a bonding agent for the reinforcing fibres because of its superior adhesion, low shrinkage after curing, and great chemical resistance

(Jayakrishnan and Ragul 2016). Other research has found that the use of epoxy in fiberglass adhesives shows excellent tensile strength in the range of 40-85 MPa (Bagherpour 2012).

In this paper, the mechanical and physical characteristics of PACF composites were examined, and a kind of commercially available adhesive, epoxy, was used to assess the adhesive performance of specimen and adhesive combinations. Polyamide 6 (PA6) is a thermoplastic polymer with exceptional thermal stability, a low dielectric constant, and a high tensile strength, according to a study by Karsli & Aytac (2013) and Khalid et al. (2022). Carbon fibre (CF) is a material with excellent mechanical, thermal, and electrical qualities. Epoxy was selected as the study’s preferred adhesive because of its outstanding bonding adherence and capacity for operation at high temperatures (up to 350°F).

METHODOLOGY

Methods and types of studied have been carried out on carbon fiber reinforced polyamide (PACF) composites manufactured by FDM. Polyamide composite (PA) and carbon fiber had been employed. Following the adhesion procedure, lap shear tests and SEM were carried out using polyamide reinforced carbon fibre (PACF). Smaller contaminated regions were examined using SEM at electron acceleration voltages suitable with energy scattering spectrometry (EDS). The Ultimate S3 printer was used to build and print the 3D design. In the composite manufacturing process, temperature and time in the FDM process were considered. To examine 20 specimens, two characterization studied with same printing parameters had been employed. This characterization was carried out on 10 samples of PACF composite with epoxy adhesive and another 10 samples of PACF composite in single lap shear condition.

Mechanical testing was performed on the specimens. The mechanical tests specified by ASTM-D5868-01 focus on the lap shear strength of the adhesives for joining the 3D printing polymers without heat. The SEM was used to compare the outcomes of physical experiments. This could be used to validate the studied’s findings. SEM was utilized in this worked to investigate the influence of surface and core structure on fracture and fracture regions.

PREPARATION OF PACF SAMPLES

Polyamide composites are macromolecules that are joined together by amide bonds. Because of its extremely crystalline structure, Polyamide 6 is a thermoplastic with a high melting point (Barkoula et al. 2008). The size of PACF samples followed ASTM D3163-01 is depicted in Figure 1. The mechanical parameters of PACF samples produced on the Ultimate S3 machine are listed in Table 1.

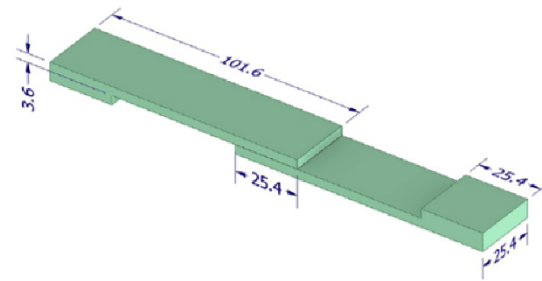


FIGURE 1. Dimension of PACF Samples

TABLE 1. Mechanical Properties PACF Sample by Standard

Characteristics	Standard	Specifications
Tensile Strength	ISO 527	103.2 MPa
Elongation at Break	ISO 527	1.8%
Young’s Modulus	ISO 527	8386 MPa
Flexural Strength	ISO 178	160.7 MPa
Flexural Modulus	ISO 178	8258 MPa
Flexural Strain at Break	ISO 178	2.4%

Processing parameters for 3D objects made using FDM techniques should be appropriately regulated to guarantee enough internal pressure and preserve bond quality between polymers (Wickramasinghe et al. 2020). The parameters used to build PACF composite samples on the Ultimate S3 machine are listed in Table 2. Figure 2 depicts the printed PACF sample that will be tested.

TABLE 2. FDM Printing Parameters for PACF Specimens

Characteristics	Specifications
Nozzle Temperature	270°C
Bed Temperature	100°C
Layer Height	0.3 cm
Printing Speed	50 mm/s
Printing Pattern	Printing Pattern

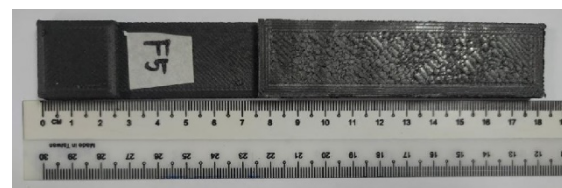


FIGURE 2. PACF Sample

ADHESION PROCESS

Loctite E-20HP epoxy (Loctite Corporation, Dusseldorf, Germany) was used to join the specimens. The Loctite E-20HP is a two-part epoxy that cures at room temperature and has a high tensile strength. For each specimen material

and adhesive, ten pairs of half specimens were made. During curing, all of the specimens were held together with spacers to guarantee alignment and a consistent bond line thickness of 0.06 mm, an overlapping length of 25.4 mm with a curing time of 24 hours to ensure the adhesive was truly durable and strong. The adhesion process parameters for PACF composites are listed in Table 3. A quarter-length overlap area is used to bind it. The specimen design was modified extensively to include an additional tab at the ends, allowing the specimens to be attached using normal fittings without introducing rotational moments. As illustrated in Figure 3, each single lap shear joint specimen was printed in two sections and glued together with an overlapping length of 25.4 mm.

TABLE 3. Adhesion Process Parameters

Characteristics	Specifications
Overlapping Length	25.4 mm
Curing Period	24 Hours
Thickness	0.06 mm
Temperature	Room Temperature (27°C-29°C)



FIGURE 3. Unbonded half of single lap shear test specimen with extra tab printed and final geometry of specimen after bonding.

#### SINGLE LAP SHEAR TEST

The single lap shear joint test was carried out in the same manner as the tensile test (ASTM D638). The ASTM D3163-01 single lap shear test was performed at room temperature. Single lap shear specimens were exposed to shearing stress by applying a tensile load axially to the lapped substrates with the Instron universal testing equipment at a crosshead displacement rate of 1.3 mm/min.



FIGURE 4. Universal Testing Machine Instron

The Instron Bluehill programme, which can create data in the form of graphs, may be used to generate the findings of this lap shear experiment. Experiments using Instron Bluehill can provide hundreds of data points. To generate data arrangements and graph plots, the data will be transformed from raw data to Excel files. The parameters for the lap shear test for PACF samples are listed in Table 4.

TABLE 4. Parameters for Lap Shear test of PACF

Characteristics	Specifications
Speed Rate	1.3 mm/min
Grip	75 mm
Temperature	Room Temperature (27 °C -29°C)
Load	1 kN

#### SCANNING ELECTRON MICROSCOPY (SEM)

Scanning Electron Microscopy (SEM) was utilised to investigate the distribution of microstructures and fibers in the matrix for PACF. To examine morphology and homogeneity, SEM analysis was done on matrix samples in the form of micrographic pictures. Delamination and fractures at the surface and cross section of the shear test samples evaluated on PACF were studied using SEM.

#### RESULTS AND DISCUSSION

##### FAILURE MODES AND EFFECTS OF ADHESIVE ON PACF

Typical failure mechanisms were detected on the full specimens to determine the inherent material responses with the effect of adhesion. All PACF specimens had adhesive failure. The failure mode is determined by the relative strengths of the specimen material and the adhesive. When the shear strength of the adhesive employed is greater than the strength of the material, the adhesive fails by slipping in the region just next to the adhesion region.

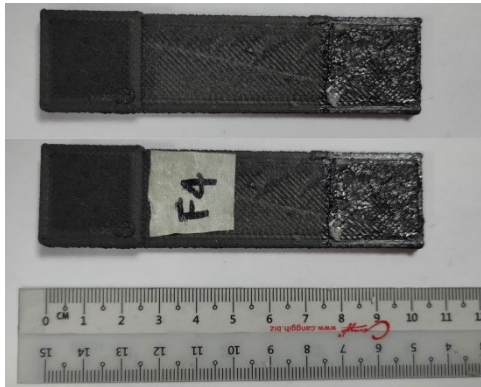


FIGURE 5. Failure modes observed in single lap shear test specimens of PACF adherend with epoxy exhibiting adhesive failure

The failure mechanisms of the single lap shear specimens are likewise comparable to those of the tensile specimens. The adhesive failure mode, on the other hand, indicates inadequate bonding ability between the substrate surface and the adhesive. As a result, the shear strength of the adhesive is less than the material strength, and failure occurs as indicated in Figure 5.

SINGLE LAP SHEAR TEST

This single lap shear test conducted for PACF samples is to measure and determine the shear strength of the sample and whether it increases or decreases when the adhesion process is performed on FDM material. There are 10 samples that have underwent this lap shear test. Shear test which have been conducted on 10 samples give an average of shear strength with 1.347 MPa. Five of ten samples have been chosen based on the best value of tensile strength at maximum load, which exceeds 1 MPa and above. Figure 6 shows the results for a 5 chosen sample that has undergone the lap shear experiment while Figure 8 shows the results for sample F1 which has the highest result of tensile strength at maximum load.

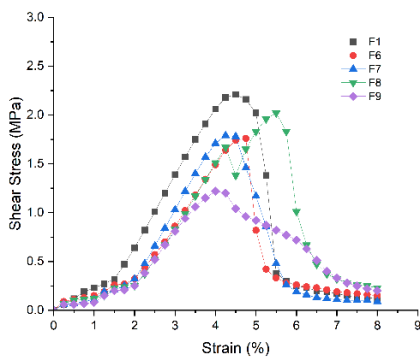


FIGURE 6. Stress-Strain Curve for 5 Chosen Samples

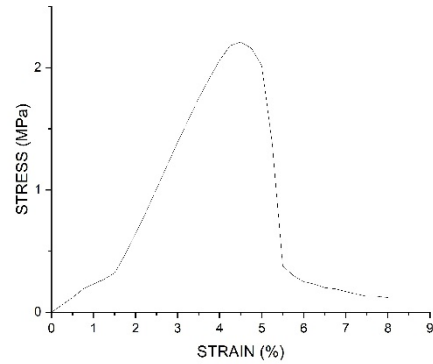


FIGURE 7. Stress-Strain Curve for Sample F1

Shear stresses were applied to specimens with a single overlap by applying axial tensile loads to the substrate (Yap et al. 2020). According to Figure 6 and Figure 7, it can be seen that at 423.063 N for sample F1 PACF composite starts to decrease after reaching 2.221 MPa of tensile stress value and 0.044% of tensile strain. This indicates that the F1 sample has the highest data rate compared to the others. Sample F9 is the sample that has the lowest data out of 5 selected sample data with the findings of 1.226 MPa tensile stress at maximum load and 0.041% tensile strain at maximum load as listed in Table 5.

TABLE 5. Parameters for Lap Shear test of PACF

Sample	Maximum Load (N)	Tensile Stress at Maximum Load (MPa)	Tensile Strain at Maximum Load (mm/mm)
F1	423.063	2.221	0.044
F6	337.511	1.772	0.046
F7	345.751	1.815	0.044
F8	385.799	2.025	0.055
F9	233.557	1.226	0.041

Furthermore, according to (Wan and Takahashi 2021), both the polymer matrix and the fiber length impact the mechanical properties and manufacturing features of the final carbon fiber polyamide. This shows that PACF has a much higher material strength than epoxy, as seen by the significantly lower failure load of 233.557 N for epoxy. The adhesive failure modes were also seen in the PACF single lap shear specimens. To produce a strong bond, the bonding method should be selected appropriately based on the unique qualities of the plastic, particularly the adhesive bond.

The shear strength of PACF specimen with an epoxy adhesive bond reaches 1.598 MPa. The thickness of the adhesive layer, which determines shear strength is a widely discussed issue. The study's findings Ochi et al. (2001), show that adhesion strength rises with increasing curing temperature and that epoxy containing unaltered resin has very poor bond strength. Epoxy bonded with PACF specimens failed at much lower stresses, demonstrating



insufficient lap shear strength between epoxy and the adherend materials. This study is similar to the findings of Espalin et al. (2010), who discovered that most epoxy-bonded FDM polymers had considerably lower ultimate tensile strengths than those bonded with other adhesives and techniques.

#### SCANNING ELECTRON MICROSCOPY (SEM)

The microstructure of PACF composites was examined using a scanning electron microscope (SEM) at magnifications of 100x and 300x. Figures 8 and 9 depict the microstructure of PACF composite samples manufactured using FDM. According to the image, there are multiple forms of porous structures that make the surface rougher.

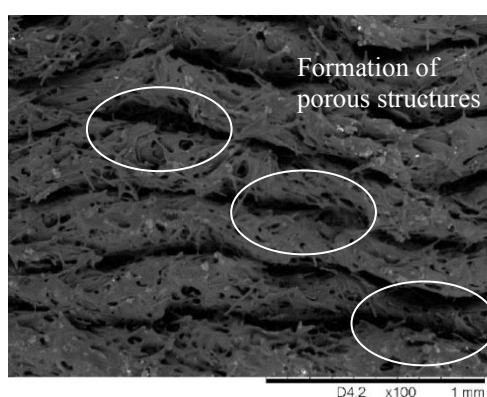


FIGURE 8. Microstructure image of PACF composite with magnification 100x

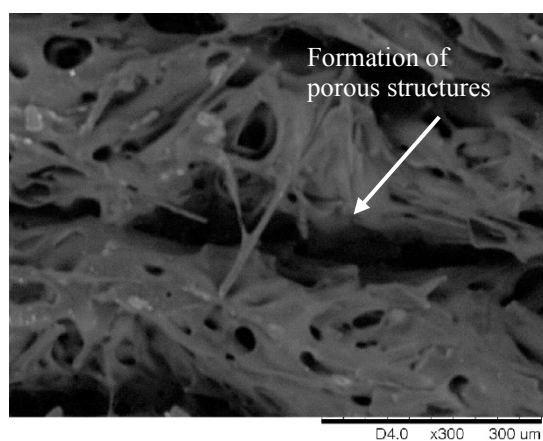


FIGURE 9. Microstructure image of PACF composite with magnification 300x

Continuous carbon fiber/polyamide filament damage often happens due to imperfections that may occur during the bonding and FDM printing process, or due to misalignment during specimen mounting on the machine. According to the study of Mazzanti et al. (2019), low bond quality, such as the formation of porous internal structures in fabricated components, leads to poor mechanical strength and a lack of surface finish on the final product, as seen in Figures 10 and 11.

As a result of the absence of binding strength on the overall structure, the layered production process during 3-Dimensional (3D) printing of carbon fiber reinforced polyamide (PACF) composite materials frequently creates inter-layer dissolving difficulties (Pereira, Kennedy, and Potgieter 2019). Therefore, if a smooth surface of the printing material is desired, the finishing process must be completed, which is a long and entirely compact operation. It can take days to construct a substantial portion of a complex. Some models employ a sparse coating mode to save time, however this clearly decreases mechanical qualities.

#### CONCLUSION

Single lap shear tests were performed on 3D printed polyamide reinforced carbon fiber (PACF) specimens, which were bonded using epoxy adhesives. The lap shear test on the adhesive strength of epoxy that was tested using a room-temperature adhesion technique definitely reveal that epoxy at room temperature is not a suitable adhesive for the adherend PACF materials evaluated since epoxy has a substantially lower adhesive strength and is entirely cured.

Adhesion strength rises with increasing curing temperature and that epoxy containing unaltered resin has very poor bond strength. Furthermore, carbon fiber is employed as a filler and bonding agent on the thermoplastic polymer layer, namely the polyamide matrix, to reinforce the FDM print layer's binding. When the bond strength of the material is weaker than the shear strength of the adhesive used, failure occurs on the specimen due to cracking or numerous porous formations. Carbon fiber/polyamide filament damage often happens during the FDM printing process. This decreases the coating's strength and mechanical qualities.

#### ACKNOWLEDGEMENT

The authors would like to gratefully thank and acknowledge the Centre for Research and Instrumentation Management (CRIM), Universiti Kebangsaan Malaysia for their financial support to complete this study under the grant number GGPM-2020-002.

#### DECLARATION OF COMPETING INTEREST

None

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