INTERLAYER BONDING BEHAVIOURS OF CONTINUOUS CARBON FIBER REINFORCED PEEK COMPOSITES 3D PRINTED IN VACUUM

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ABSTRACT

Continuous fiber reinforced thermoplastic (CFRTP) 3D printing technology has become a popular topic in industries such as manufacturing and aerospace due to its exceptional flexibility, high conductivity, and low cost. However, the mechanical properties of CFRTP parts produced using 3D printers are weaker compared to parts produced using other composite manufacturing methods, which is one of the main disadvantages of this method. Researchers have focused intensively on developing methods to increase the strength of parts produced using various techniques. The main objective of this study is to investigate the mechanical performance of continuous carbon fiber reinforced PEEK (Polyether ether ketone) composites produced additively in a vacuum environment. To achieve vacuum levels of 0.5 Mbar, a vacuum chamber that can be placed inside the Fused Deposition Modeling (FDM) printer was designed. The CFRTP filament used in the 3D printer was produced by using a fiber-polymer impregnation platform to produce a continuous carbon fiber reinforced PEEK thermoplastic filament. CFRTP composite samples were produced at various vacuum levels (0.5 Mbar, 10 Mbar, 100 Mbar) inside the vacuum chamber, and three-point bending tests were conducted to investigate their mechanical properties. The bending stress and elastic modulus of the samples produced at 0.5 Mbar were 151.39% and 18.70% higher, respectively, than the samples produced at 100Mbar.

Keywords: Fused Deposition Modeling (FDM), Vacuum Environment , Additive Manufacturing, Mechanical Properties.

INTRODUCTION

It is known that selecting lightweight materials with high specific strength is one of the most important criteria in the aviation, space, and defense industries (Kaya et al.; Yılmaz and Evci). Composite materials are widely used in the aviation industry due to their long lifetimes, lighter weight compared to metals, high chemical and mechanical properties, and many other superior features (Y. Li et al.; Maria). Polymer-based composite materials, which are part of the composite materials family, are lightweight and high-strength materials and therefore have more areas of use in the aviation industry than metal-

based materials. Polymer-based composite materials are divided into two groups: thermoplastic and thermoset composites. In today's technology, thermoset composites have a wider range of applications because they have higher mechanical properties compared to thermoplastic composites. However, with the recent advancements in technology, the use of thermoplastic composite materials is increasing due to their increasing mechanical properties and lower costs (Hoang et al.; Lapena and Lopes; Siddique et al.). Producing fiber-reinforced thermoplastic composite materials by reinforcing chopped (Kumar et al.; Maqsood and Rimašauskas) and/or continuous fibers (Mosleh et al.; Shuto et al.) with thermoplastic resin has recently become very popular. Fiber-reinforced composites can be produced by multiple methods, such as extrusion, laminated object manufacturing, automated fiber placement (AFP), injection molding, etc. However, these methods limit the geometry of the part to be produced to specific molds and do not allow for complex geometries. Additive Manufacturing technologies that enable the production of fiber-reinforced parts with complex geometries, especially those used in aviation and other industrial areas, have become increasingly popular, and Fused Deposition Modeling (FDM) is one of them (Zhang et al.; Ye et al.). Also known as 3D printing, the FDM method is based on the principle of adding layers to the desired part. 3D printers provide many conveniences, such as easy use and portability, and make it possible to produce parts using 3D printers in laboratories, offices, production areas, etc. Using 3D printing, it is possible to produce many specific parts (such as aircraft wing support parts, components in unmanned aerial vehicles, complex lattice support parts, etc.) without the need for any molds and for the sake of geometric freedom.

FDM, which is one of the additive manufacturing methods, is widely used for producing parts in homes, offices, and laboratories due to its simplicity and portability. FDM method can be easily used for complex geometries without using molds, such as airplane wing parts, unmanned aerial vehicle parts, and many specific parts.

Despite the advantages of 3D printers, the mechanical properties of parts produced using 3D printers have weak mechanical properties and rough surface quality compared to parts produced by injection molding and other production methods, which are among the main disadvantages of the method.

Parts produced with 3D printers have weak mechanical properties. The main reasons for this are: i) the non-use of high-temperature thermoplastic polymers (such as PEEK, PPS), ii) defects in the internal structure of the produced composite structures (such as voids and porosity), iii) non-homogeneous mixing of fibers and polymers and a relatively low fiber ratio, iv) weak bonding interactions between layers, v) and problems such as anisotropy (Goh et al.). To overcome these defects, various methods have been intensely focused on by researchers to develop strong bonding in both medium-sized and nano-sized interfaces, including heat-based processes (laser, infrared lamp, microwave, etc.) (Uşun et al.; Du et al.), optimization of production parameters (Krajangsawasdi et al.), production under vibration (Tofangchi et al.), production under pressure (van de Werken et al.), production in a vacuum environment (O'Connor and Dowling; Maidin et al.), and the addition of nanoparticles (Chen et al.).

These methods aim to achieve lower porosity and higher strength parts.

Studies on additive manufacturing in a vacuum environment have only recently been started in the literature (O'Connor and Dowling).In these studies, where production is carried out in a vacuum environment, an improvement of approximately 12% in tensile strength was achieved for ABS polymers. This improvement in mechanical properties is due to the slowing down of cooling in the parts. In another study, carbon, glass, and Kevlar fiber-reinforced polymer composites were produced using a vacuum chamber. When compared to those printed at atmospheric pressure, an increase of 33%, 22%, and 12% in interlayer shear strength was obtained for carbon, glass, and Kevlar fiber-reinforced polymer composites, respectively, when printed under vacuum. It was determined in these studies that the reduction in heat

transfer due to vacuum resulted in an increase in interlayer strength and a significant decrease in porosity formation (O'Connor, Heather J. Dowling).

In this study, continuous fiber reinforced PEEK composite parts will be produced at various vacuum levels in order to investigate the contribution of the vacuum environment to the mechanical properties. The optimum point of the vacuum degree will be investigated by increasing the vacuum level. For this purpose, a conventional 3D printer will be placed in a vacuum chamber and the printing process will be carried out in a vacuum environment. Three-point bending tests were conducted to investigate the obtained mechanical properties.

MATERIALS AND METHODS

Materials

Continuous fiber reinforced PEEK filament was obtained from Dowaksa, consisting of 3K carbon fibers with a tensile strength of 3800 MPa, an elastic modulus of 245 GPa, and a density of 1.8 g/cm^3. Each fiber has a diameter of approximately 7 µm. VICTREX PEEK (Poly-ether-ether-ketone) filament was used as the polymer matrix, with a diameter of 1.75 mm. The vacuum chamber, with dimensions of 0.5*0.5*0.5 m^3, was constructed for this study, and a BSV40 vacuum pump with a flow rate of 40 m^3/h was used. Additionally, a VACUUM·VIEW EXTENDED device capable of measuring vacuum pressure from atmospheric pressure down to 0.002 Mbar was used to digitally measure the vacuum level in the chamber.

Design of a Vacuum Chamber and CFRTP Part Production

The vacuum environment reduces the contact of the part with air during production using a 3D printer, resulting in slower cooling of the parts. This situation will help to achieve a more even temperature distribution on the part.

More importantly, delayed cooling will cause the polymer inside the CFRTP composite filament to remain flowable for a longer period. As a result, new layers will adhere better to the non-cold lower layer, and warping will be prevented. Additionally, it is predicted that there will be less voids due to the suction effect of the vacuum during this process. Therefore, the aim is to produce high-strength parts using the vacuum environment. The vacuum chamber design is shown in Figure 1. The vacuum chamber consists of a vacuum pump, discharge valve, vacuum gauge, and FDM-type 3D printer. In the vacuum chamber design, attention was first paid to ensuring that the internal volume was large enough to accommodate the platform of the 3D printer to be placed inside. The thickness of the chamber and the numerical design of the lid were performed to achieve a value of 0.001 Mbar for the vacuum chamber. Sealing with gaskets was aimed to be achieved in the lid section. To achieve this vacuum value, a vacuum motor with a minimum pressure drop of 0.5 Mbar and a volumetric flow rate of 40m^3/h was used.

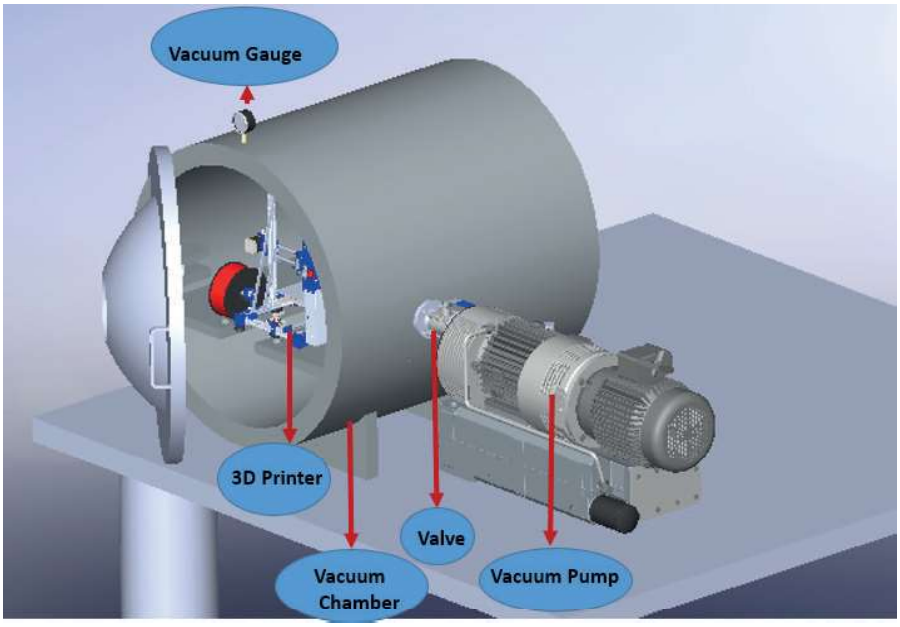
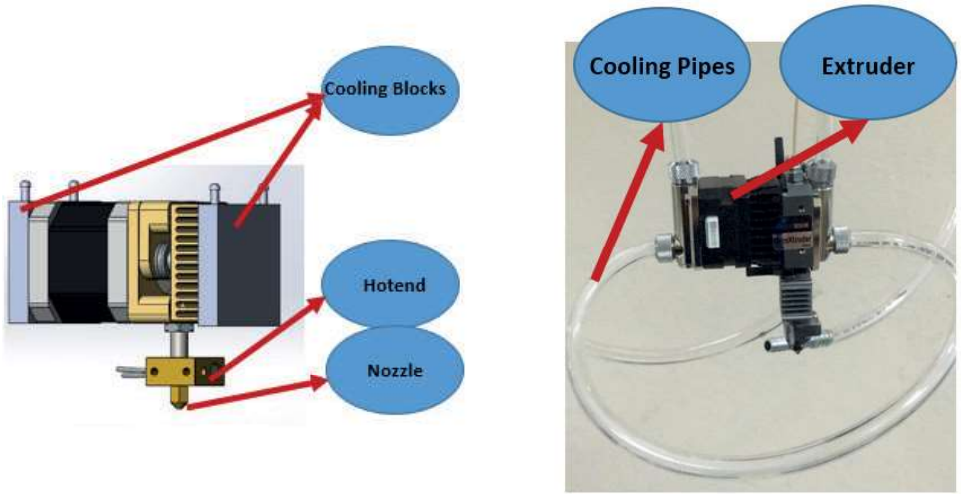


Fig 1. Design of a Vacuum Chamber

In addition, it is not feasible to cool the electronics and printer components with air during the printing process in a vacuum environment. Precautions must be taken in this regard. Generally, in FDM type 3D printers, cooling is achieved through a fan. When the heat in the hotend part of the 3D printer is not dissipated sufficiently, it can negatively affect the printer's calibration or movements. It can also cause the filament to swell prematurely and result in nozzle clogging. For this reason, a water-cooling system has been implemented in the designed vacuum system. The cooling of the print head and stepper motors is achieved by circulating water through the system using tap water. The sealing of the cooling pipes on the vacuum chamber has been ensured by adding inlet and outlet records. Figure 2 shows the extruder design of the 3D printer with added water-cooling blocks.



a) b)

Fig 2. The 3D printer nozzle modified with water cooling blocks; a) Design. b) The manufactured version

The CFRTP filament used in the vacuum environment was produced on the pultrusion-based platform used in our previous study (Vatandaş et al.). The platform consists of three main regions: the fiber spreading region, the polymer mixing region, and the die. The schematic visualization of the platforms is shown in Figure 3. The fiber ratios of the CFRTP filaments were calculated using the volume fraction. The equation for calculating the fiber ratio is given in Equation 1 below. The diameter of a single fiber is approximately 7 µm, and 3K fibers are used, so the fiber area is multiplied by 3000. As a result of these calculations, the fiber ratio was found to be 41%.

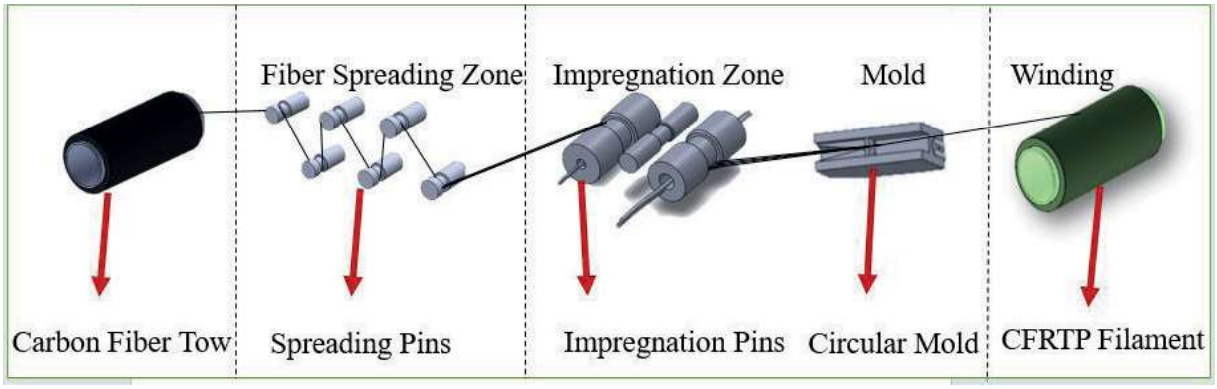


Fig 3. CFRTP filament production platform

$$Total\ Fiber\ Area = 3000 \times \frac{\pi \times (Fiber\ Diameter)^2}{4}$$

$$Nozzle\ Area = \frac{\pi \times (Nozzle\ Diameter)^2}{4}$$

$$Volume\ Fraction\ (\%) = \frac{Total\ Fiber\ Area}{Nozzle\ Area} \times 100$$

.....(1)

Test specimens were produced using the 3D printer placed in the vacuum chamber. All bending test specimens were produced in one piece in accordance with ASTM D7264 standards (100x15x2 mm). The production parameters under vacuum are shown in Table 1.

Table 1. Printing parameters

Vacuum Degree	Nozzle Temperature	Table Temperature	Chamber Temperature
0.5 Mbar	430	100	20
10 Mbar	430	100	20
100 Mbar	430	100	20

Three-Point Bending Tests

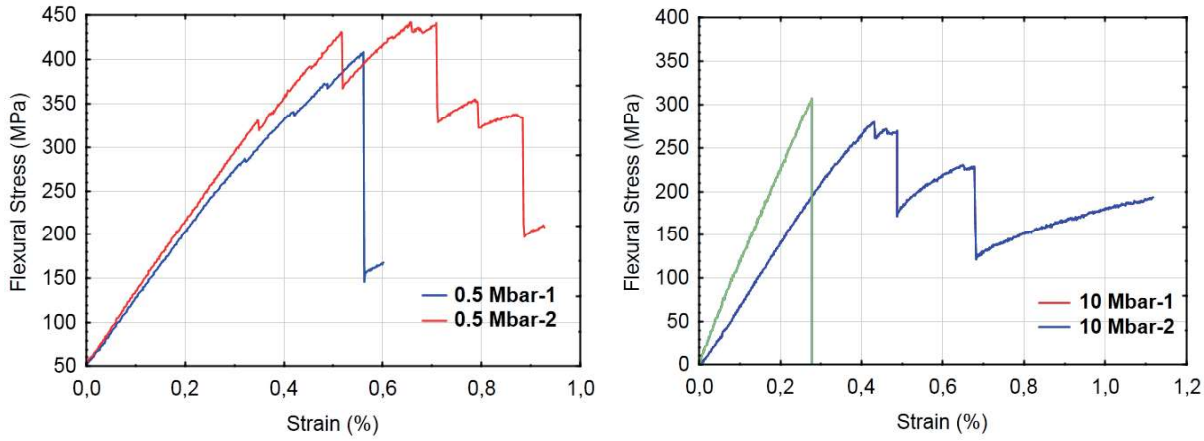
Automatic test machine with a capacity of 0.5 to 1200 kN from Instron was used for bending tests.

Bending specimens were produced in accordance with the ASTM D7264 standard "Flexural Properties of Fiber Reinforced Plastic Composites". A crosshead speed of 5 mm/min was used to apply the load. The support span distance was set at 80 mm. To observe the repeatability of the results, the three-point bending tests were repeated four times.

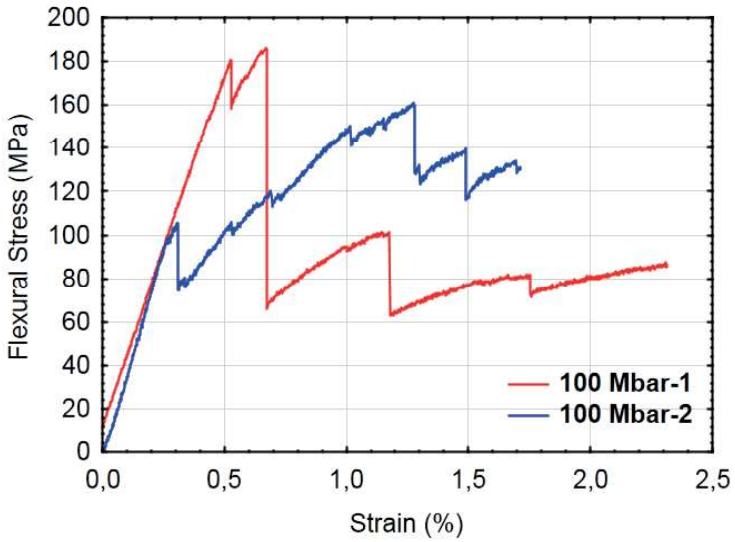
RESULTS

In the study, CFRTP composite parts were produced in vacuum at three different vacuum levels and subjected to bending tests. In the study, four samples were used for each vacuum level, and standard deviations were calculated. Firstly, the maximum bending stress and modulus of elasticity obtained from the test samples produced at 0.5 Mbar vacuum level were found to be 404.40±39.21 MPa and 93.46±20.03 GPa, respectively.

Then, the maximum bending stress and modulus of elasticity obtained from the test samples produced at 10 Mbar vacuum level were found to be 276.03±21.17 MPa and 86.11±9.83 GPa, respectively. Finally, the maximum bending stress and modulus of elasticity obtained from the test samples produced at 100 Mbar vacuum level were found to be 160.86±5.45 MPa and 78.73±0.19 GPa, respectively. When the results were examined, it was found that the bending stress and elasticity value of the samples produced at 0.5 Mbar were increased by 151.39% and 18.70%, respectively, compared to 100 Mbar. The study conducted by Li et al. has similarly proven that vacuum contributes positively to bending performance. Bending stress-strain curves obtained from the three-point bending test of CFRTP produced in vacuum are given in Figure 4.



(a) (b)



(c)

Fig 4. Unit stress-strain curves for three-point bending of CFRTP samples produced under different vacuum degrees are shown in Figure 4.; a) 0.5 Mbar, b) 10 Mbar c) 100 Mbar.

Upon detailed inspection of the unit strain curves, it was observed that delamination occurred between the layers during the fracture of composite parts. The sudden drops in the curves indicate the occurrence of delamination, which is a common flaw in composite parts (Tian et al.). The main reason for delamination is the insufficient strength of the adhesive bond between the layers. This flaw limits the mechanical strength of the part. A summary of the maximum mechanical properties obtained from the three-point bending tests applied is presented in Table 2.

Table 2 : Summary of three-point bending test results.

Vacuum Degree	Maximum Flexural Strength (MPa)	Increase in Flexural Strength (%)	Flexural Modulus of Elasticity (GPa)	Increase in Flexural Modulus of Elasticity (%)
0.5 Mbar	404,40	151,39	93,46	18,70
10 Mbar	276,03	71,59	86,11	9,37
100 Mbar	160,86	-	78,73	-

CONCLUSION & DISCUSSION

This study investigates the effect of different vacuum levels on the mechanical properties of continuous fiber reinforced thermoplastic composites (CFRTP) parts produced using a 3D printer in a vacuum environment.

For this purpose, a rigid vacuum chamber resistant to a vacuum pressure of 0.001 Mbar was designed and produced, and a modified 3D printer was installed inside. CFRTP parts were produced at three different vacuum levels (0.5 mbar, 10 mbar, 100 mbar), and the vacuum level with the highest mechanical properties was determined. The flexural stress and elastic modulus of the samples produced

at 0.5 Mbar were found to increase by 151.39% and 18.70%, respectively, compared to those produced at 100 mbar. As the vacuum level increased, the number of air molecules inside the chamber decreased. As a result, heat transfer through convection and conduction from the printer nozzle to the print platform was reduced compared to the atmosphere, and the CFRTP filament that was poured from the nozzle and solidified on the platform cooled more slowly. This strengthened the bond between the previous and new layers. Although the flexural strength increased when the stress-strain curves were examined, delamination defects persisted. Although the vacuum environment strengthened the adhesion between the layers, these defects could not be eliminated. To eliminate delamination, the vacuum chamber must be heated using external heaters. In future work, it is anticipated that gradually heating the vacuum chamber to the optimal 0.5 mbar vacuum level will further increase the strength.

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