

# FDM 3D PRINTING IN HIGH-PRESSURE OXYGEN AND PURE NITROGEN ATMOSPHERES & EVALUATION OF MECHANICAL PROPERTIES

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### ABSTRACT

3D printing is still advancing rapidly in academic and industrial research. Fused deposition modelling (FDM) is an additive manufacturing technology that uses filaments as input material. The filaments are arranged adjacent to and above each other. The bonding strength of the contact between these filaments determines the mechanical properties of FDM products. These parts have the lowest dimensional accuracy and resolution of any 3D printing technology. Despite its many uses, FDM rapid prototyping is ineffective for manufacturing structural parts due to anisotropic mechanical properties. The consolidation of layers in additive manufacturing procedures does not need pressure, in contrast to traditional polymer processing techniques. This study investigates the effect of high ambient pressure on the consolidation of layers during the FDM process and their characterization of mechanical properties. To attain high strength qualities for 3D printed items as similar to injection-moulded specimens, an experimental setup was built up using a 3D printer incorporated into a customized Autoclave. A maximum temperature of 185 °C and 135 bar of pressure may both be maintained in the autoclave. Atmospheres of compressed air at 0 bar, 5 bar, 10 bar, 15 bar, and 20 bar as well as nitrogen at 5 bar were used for PLA 3D printing in the autoclave. The effects of pressure and temperature on 3Dprinted samples were examined, and tensile, flexural, and Charpy tests were performed on printed specimens as well as on specimens that had been injection moulded. It could be demonstrated that autoclave preheating before to printing and autoclave pressure during printing greatly enhance layer consolidation. Increased yield strength, Young's modulus, and impact strength are produced as a result of closer contact between the layer surfaces caused by the pressure within the autoclave. Most experiments produced better results when the autoclave pressure was 15 bar.

### **1 INTRODUCTION**

The newest plastic manufacturing technique, additive manufacturing (AM), promises to produce complex and multifunctional parts and products in a single processing step utilizing a CAD model [1]. According to the definition given in [2], it is the "Process of combining materials such as polymers, metals, concrete, ceramics, or rubber in the shape of consecutive layers on top of each other." Rapid prototyping and additive manufacturing, often known as 3D printing, have been around for a while. Stereo Lithography (SL), the first 3D printing method, was created in 1984 by Charles W. Hull of 3D Systems Corporation and was highly costly [3]. Originally, designers and architects mostly employed fast prototyping due to their ability to create working prototypes. Eventually, substantial study was conducted, and tremendous progress was made. This resulted in the invention of several different AM techniques such as fused deposition modelling (FDM), materials jetting, inkjet printing, powder bed fusion, and so on. These advances in AM have lowered prices, productivity, and waste while improving printing quality, accessibility, sustainability, and usability. These advancements have increased its applicability in automotive, aviation, medical, construction, and other fields [4].

Because of its cheap cost and practicality, FDM has a prominent position in all types of enterprises, from small-scale to large-scale, and may be controlled by individuals. A layer-by-layer specimen is constructed using the FDM technique, which uses a circular cross-sectional filament with a predetermined diameter that is pushed into the hot end through a feeder at a certain speed. In addition to low-cost equipment and basic technological procedures, FDM is a practical solution to make prototypes and functional components fast and at a reasonable cost. FDM components, on the other hand, have issues with their mechanical characteristics. Of all 3D printing methods, these parts have the lowest dimensional accuracy and resolution [5]. A smooth, flat surface and less uniform behaviour must be achieved by post-processing because an FDM model's pieces contain visible layer lines. While FDM fast prototyping has several uses, producing structural elements is not one of them because of the anisotropic mechanical characteristics [6]. Its use is limited in many applications due to this flaw. According to research, a common FDM printer that prints parts in a nitrogen environment increases the tensile strength of such parts by 30% [7]. Processing parameters such as nozzle material, nozzle diameter, extrusion temperature, bed temperature, incoming materials (whether neat or recycled), and fan speed, among others, influence the strength of FDM parts. The printing instructions also have an impact. Inadequate adhesion between the deposited layer and the incoming extruding material during printing could account for the poor mechanical properties. This could be due to the temperature difference between the previously deposited layers and the incoming layers, as it relies on extruding and cooling heated material [8]. According to another study, heat treatment of 3D-printed components improves interlayer adhesion and lowers internal tensions [9]. Another study found that 3D-printed FDM parts have more enclosed voids than injection-moulded parts due to pressure during the process and tight dimensional control [10]. These voids work in tandem with mechanical strength. Pressure is important in controlling the isotropic behaviour of parts. Downturns in layer thickness and varying infill density can be used to control voids. Although microvoids are available in such cases, they cannot be eliminated. However, an annealing process has constraints, as some polymers are temperature-sensitive and leading to thermal shrinkage or warping. The effect of pressure and temperature on 3D-printed samples were analysed. In previous studies, the post-processing treatment of 3D-printed and injection-moulded specimens with autoclaving pressure and temperature treatment increased properties in all areas, including modulus and strength. This is because the samples' internal tensions were released during the post-treatment process. This helped with modulus and the development of strength. The combined effect of pressure and temperature, which relived internal stresses, increased grain structure, enhanced their mechanical properties by approximately 20%, and the results were published [11, 12]. The goal of this study is to 3D-print specimens in an autoclave with the same infill density and process parameters at 0 bar, 5 bar, 10 bar, 15 bar and 20 bar compressed air, and 5 bar Nitrogen gas atmosphere in the transverse, longitudinal direction to the hot-end nozzle and to investigate the effect of ambient pressures and inert gas environment on layer consolidations. On all the specimens Tensile, Flexural, and Charpy tests were performed and properties such as yield strength, yield strain, and Young's modulus, Flexural, and Impact strength are determined. The test results are compared with injection-moulded specimens and conclusions are drawn.

### 2 MATERIALS AND METHODS

#### 2.1 Material

In this research work, pure PLA (Polylactic Acid) filament of high quality and PLA granules from Herz GmbH, Germany was used. PLA filaments usage in FDM is common because of its low melting point (180°C-220°C), and it supports quality surface prints, is non-toxic, has high UV resistance, and low moisture adsorption allows easy handling.

#### 2.2 FDM 3D-Printer

In this research, an Ender-3 as shown in Fig.1, V2 model FDM 3D-Printer from Creality-2020 was used. The maximum possible dimensions are  $220 \times 220 \times 250$  mm (L×B×H), and the total weight of the machine is 7.8kgs. General specifications like Maximum bed temperature, maximum extruder temperature, and maximum printing Speed are 100°C, 250°C, and 180 mm/sec, respectively.



Figure 1: Creality Ender 3 V2 FDM 3D-Printer.

## 2.3 Autoclave

A customized autoclave chamber from Haage Anagram GmbH, Germany, which had been specially designed to support polymer-processing methods, was used in this research. This autoclave maintains a maximum of 135 bar and 185°C and weighs about 1300 kg (including a front lid with a weight of 300 kg).

## 2.4 Fabrication of specimens

In the autoclave, the testing specimens were printed at 0 bar, 5 bar, 10 bar, 15 bar and 20 bar of additional pressure and in 5 bar nitrogen atmosphere. Because the material was PLA, the printing procedure used a 205°C nozzle temperature and a bedplate temperature of 60°C, with the temperature inside the autoclave fixed at 50°C while printing. For each test, 5 samples were printed in an autoclave in two distinct printing patterns (longitudinal and transverse to the printing direction). The test parameters given include nozzle diameter, layer thickness, printer voltage capacity, and sample printing environment conditions as shown in table 1.

|                                      | 3D                      | 3D printing    | 3D printing | 3D printing    | 3D printing    | 3D printing    |
|--------------------------------------|-------------------------|----------------|-------------|----------------|----------------|----------------|
|                                      | printing<br>at<br>0 bar | at             | at          | at             | at             | at             |
|                                      |                         | 5 bar          | 5 bar       | 10 bar         | 15 bar         | 20 bar         |
| SIno                                 |                         | pressure in    | pressure in | pressure in    | pressure in    | pressure in    |
|                                      |                         | autoclave      | autoclave   | autoclave      | autoclave      | autoclave      |
| 51110                                | n                       | [Compresse     | [Nitrogen   | [Compressed    | [Compressed    | [Compresse     |
|                                      | п                       | d air]         | atmosphere] | air]           | air]           | d air]         |
| Nozzle<br>diameter                   | 0.5mm                   | 0.5mm          | 0.5mm       | 0.5mm          | 0.5mm          | 0.5mm          |
| Printing<br>speed                    | 100%                    | 100%           | 100%        | 100%           | 100%           | 100%           |
| Layer<br>thickness                   | 0.15mm                  | 0.15mm         | 0.15mm      | 0.15mm         | 0.15mm         | 0.15mm         |
| Hot end,<br>bed<br>temperature       | 200°C,<br>60°C          | 200°C,<br>60°C | 200°C, 60°C | 200°C,<br>60°C | 200°C,<br>60°C | 200°C,<br>60°C |
| Voltage<br>capacity of<br>hot end(V) | 24V                     | 24V            | 24V         | 24V            | 24V            | 24V            |

Table 1. 3D Printer parameters while printing in an autoclave

#### **3 EXPERIMENTAL SETUP**

The 3D printing was carried out in Autoclave. The pressure was build up inside the Autoclave by sending compressed air into it using a compressor. Autoclave integrated with a 3D-printer as shown below in Fig. 2. In this research work, different mechanical tests like tensile, flexural, and Charpy impact were conducted on the printed samples, based on tests results, conclusions were drawn.



Figure 2: 3D-Printer setup in autoclave

#### 3.1 Tests on samples

The tensile strength, stiffness, and elongation properties were measured according to DIN ISO 527, using a tensile testing machine from Zwick (Proline-Z005) along with Zwick's Test Expert software. The strain measurement was done optically with the Video extensions system also from Zwick.

Flexural properties of materials are examined through the 3-point bending test according to DIN ISO 178 using the Zwick -UTM mentioned previously.

Charpy impact test DIN ISO 179 was carried out with a Ray-Ran pendulum with an impact energy of 4 joules and an impact velocity of 2.9 m/sec.

### **4 RESULTS**

#### 4.1 Tensile test results

The specimens were put through the aforementioned tests, yielding the results below, which were then analyzed. According to Fig. 3, the young's modulus of PLA material is 2435 MPa for the specimen printed in a longitudinal direction under 5bar nitrogen pressure, which is lightly higher than the 2403 MPa for the Injection molded sample. It also concludes that at 10bar compressed air pressure, young's modulus rises to 2124 MPa and then decreases as pressure increases in 5bar intervals.

As shown in Fig. 4, the young's modulus of PLA material for the specimen printed transversely under a 15 bar compressed air environment is 1813 MPa, which is higher than the 1682 MPa for the injection molded sample and other atmospheres. Also, it shows that young's modulus decreases as pressure rises in steps of 5 bar, with 5 bar of nitrogen atmosphere being about equivalent to 20 bar of compressed air atmosphere. It is possible to draw the conclusion that PLA material printed longitudinally has a higher young's modulus than a sample printed transversely. The sample with the highest values is the one printed longitudinally at 5 bar nitrogen atmospheres.



Figure 3: Young's modulus comparison of samples printed in the longitudinal direction in different pressure conditions with injection moulded sample.



Figure 4: Young's modulus comparison of samples printed in the transverse direction in different pressure conditions with injection moulded sample.

According to Fig. 5, the yield strength of PLA material specimens printed in a longitudinal direction under a 15bar compression air atmosphere has the greatest value of all, 74.9 MPa, while yield strain also attains the maximum value of 3.5%, virtually equivalent to injection moulded sample.

The yield strength of PLA material specimens printed in a transverse direction under a 5bar nitrogen atmosphere has the highest value of all, 57.4 MPa, as depicted in Fig 6. At the same pressure situation, yield strain reaches a maximum of 3%, which is practically identical to the injection moulded sample. As pressure is increased in 5 bar intervals, the yield strength decreases when compared to the injection moulded sample.



Figure 5: Yield strength comparison of samples printed in the longitudinal direction in different pressure conditions with injection molded sample.



Figure 6: Yield strength comparison of samples printed in the transverse direction in different pressure conditions with injection molded sample.

### 4.2 Flexural test results

The flexural modulus of a PLA material specimen printed in a longitudinal direction under a 15 bar compressed air environment has the maximum value of 2338 MPa in Fig. 7, which is approximately similar to the 2548 MPa of an injection moulded sample. A specimen produced in a 5bar nitrogen environment has a flexural modulus of 2248 MPa, which is roughly comparable to a 15bar compressed air atmosphere.

According to Fig. 8, the highest value for the flexural modulus of PLA material specimen produced in a transverse direction in a 20 bar compressed air environment is 2375 MPa, which is nearly equivalent to a 15 bar compressed air atmosphere. That leads to the conclusion that the flexural modulus increases as pressure does. In this case, a 20 bar compressed air atmosphere in the transverse direction and a 15 bar compressed air atmosphere in the longitudinal direction have nearly identical flexural moduli.



Figure 7: Flexural modulus comparison of samples printed in the longitudinal direction in different pressure conditions with injection moulded sample.



Figure 8: Flexural modulus comparison of samples printed in the transverse direction in different pressure conditions with injection moulded sample.

The highest value of 80.7 MPa for the flexural strength of PLA material printed in the longitudinal direction in a 20 bar compressed air environment shown in Fig. 9, followed by 88.2 MPa for an injection-moulded sample. Ultimately, the flexural strength steadily rises with increasing pressure and approaches that of an injection-moulded sample.

In Fig. 10, the flexural strength of PLA material specimen printed in a transverse direction under 10 bar compressed air environment is 63.5 MPa, which is somewhat higher than the injection moulded sample of 61.7 MPa. The specimen was printed in a 5bar nitrogen environment at 59 MPa strength. It acknowledges that flexural strength is maximum when the material is printed longitudinally under pressure conditions, but provides relatively low results when printed transversely.



Figure 9: Flexural strength comparison of samples printed in the longitudinal direction in different pressure conditions with injection moulded sample.



Figure 10: Flexural strength comparison of samples printed in the transverse direction in different pressure conditions with injection moulded sample.

### 4.3 Impact test results

The impact strength of PLA material printed in longitudinal direction under 5 bar nitrogen atmosphere has the maximum value of 24.6 kJ/m2 as depicted in Fig. 11, followed by 20 bar compressed air atmosphere with a value of 23.1 kJ/m2, which is higher than the injection moulded sample. It was also discovered that increasing the pressure causes an increase in the impact strength.

The impact strength of PLA material specimen printed in transverse direction under 15 bar compressed air atmosphere has the highest value of 20.3 kJ/m2 as shown in Fig. 12. Then comes 10 bar compressed air, which has 18.2 kJ/m2 when compared to the injection moulded sample. Finally, it concludes at 15 bar compressed air atmosphere in both longitudinal and transverse directions has the exactly almost same value.



Figure 11: Impact strength comparison of samples printed in the longitudinal direction in different pressure conditions with injection moulded sample.



Figure 12: Impact strength comparison of samples printed in the transverse direction in different pressure conditions with injection moulded sample.

### **5** CONCLUSION

The autoclave is used for this research to print PLA specimens in two different orientations, longitudinal and transverse, under different pressure conditions. In order to compare the results of the testing, injection molding was also performed using the same PLA material. The printing environment is the most important factor in the 3D printing process, influencing surface polish, printing quality, and specimen strength. Under typical air conditions, the filament layers may oxidize, resulting in a divergence in the attachment of fresh layers and, inevitably, wider gaps between the layers. A few samples were printed at 5 bar nitrogen pressure to examine this. All tests reveal that the nitrogen-printed materials have gained strength and are roughly similar to the strength of injection-molded samples. The young modulus was enhanced by 30% and 50% in longitudinal and transverse orientations, respectively, in a nitrogen environment, and is now equal to injection-molded specimens.

The nitrogen gas atmosphere in the chamber inhibits layer oxidation, promoting improved layer adhesion. The autoclave was set at 0 bar, 5 bar, 10 bar, 15 bar, and 20 bar of compressed air for this research. The autoclaving pressure and temperature treatment have undoubtedly improved specimen modulus and strength. This occurs as a result of the procedure causing internal tensions in the samples being released. Layer consolidation for 3D printing was enhanced by autoclave preheating and pressure. By employing the right autoclave pressure and temperature, the void content is decreased. Tensile test findings show that under 15 bar compressed air conditions, the samples perform better, with a young's modulus of 2010 MPa. The yield strength and flexural modulus are greater in the longitudinal direction at 15 bar than in the injection moulded sample. Flexural and impact strength increase correspondingly as autoclave pressure increases.

The study comes to the conclusion that specimens printed in 3D in the longitudinal direction at autoclave pressure and temperature have superior qualities than specimens produced in the transverse manner. The cause may be improved consolidation, which leads to more secondary bonding's and an increase in shear strength between layers. Another explanation may be a sharp decline in voids. Higher values for the recrystallization temperature, glass transition temperature, melting temperature, and density are obtained for 3D printed objects when autoclave pressure and temperature are used.

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