Destructive and non-destructive testing of samples from PLA and PETG materials

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Abstract. This study deals with the properties of PLA and PETG materials, which are important for gaining knowledge in biomedical applications. The process of obtaining an ideal implant from a material with suitable mechanical and physical properties is a complex process considered to be one of the most difficult in tissue engineering. PLA and PETG material properties were determined based on performed non-destructive and destructive materials testing methods. Destructive testing was performed on Hegewald and Peschke equipment and non-destructive testing was performed on Carl Zeiss Metrotome 1500 (Germany). Commercial filaments from various companies were selected, from which type 5A dogbone samples were printed on a TRILAB printer. Subsequently, after carrying out the tests on dogbone samples, the results were evaluated and compared with the help of graphs and tables.

1. Introduction

3D printing allows you to produce bodies with a sophisticated shape in a short time. This innovative technology is useful for the production of prototypes in biomedical engineering. FDM printing using PLA and PETG materials is very common. It brings many advantages but also disadvantages in the form of microscopic defects that cannot be visible with the eye. To detect these defects, it is necessary to perform a scan on the device, which uses X-rays and CT tomography. In this study, we scanned the samples before and after the tensile test. We performed the tensile test on equipment from Hegewald and Peschke company. Detected defects and examination of porosity help with the selection of samples that are suitable for determining the mechanical properties [1] - [3].
2. Properties of PLA and PETG materials

2.1. PLA

At present, new knowledge in the field of polymer development and use is coming to the fore. The polylactic acid (PLA) biopolymer is a more well-known polymer that is produced from renewable raw materials, important from the point of view of ecology. It excels in its biocompatibility, and broad-spectrum usage, especially for biomedical applications (Figure 1) and additive production. Due to its properties such as biodegradability, mechanical strength, and biodegradability, it is a suitable material that can be used in the human body. PLA blends extensively with other polymers, eliminating worse PLA properties such as low toughness and hydrophobicity. PLA is widely used in the biomedical field, such as the production of scaffolds, covering membranes, implants, and sutures used, for example, in dermatology. Polymers are more suitable for scaffolds than metals because of their degradability. Another key factor is the adhesion of the cells to the polymer. PLA has a great potential for bone fixation because it has several advantages over metal fixations, such as high strength, similar to bone strength. PLA is the only polymer that can be useful in many applications with simple modifications of its physicochemical structure [4].

![Figure 1. Biomedical applications of PLA [4].](image)

2.2. PETG

Polyethylene terephthalate glycol (PETG) is a polyester thermoplastic derivative polymer made of polyethylene terephthalate (PET). It is used in implantology but also the food and packaging industry. PETG properties include good formability, chemical resistance, and low forming temperature, resistance to thermal changes, low moisture absorption, recyclability, and sustainability. PETG is a suitable material for additive production with optimal parameters [5].
3. Preparation of samples for mechanical testing

In this study, we selected filaments made of PLA and PETG materials, from which we printed dogbone type 5A samples for destructive and non-destructive testing. Modeling and testing were carried out according to the relevant standard EN ISO 527-2: 1996. Dogbone samples were modeled in SolidWorks. The dimensions of the dogbone sample are shown in Figure 2, where the total length of the sample is 75 mm, the thickness is 2 mm and the width at the edges is 12.5 mm [6] [7].

![Type 5A sample parameters.](image)

3.1 3D printing of samples

The samples were printed on a TRILAB printer. The individual printing parameters are listed in Table 1 for PLA and in Table 2 for PETG. 3D printing went smoothly (Figures 3 and 4). TRILAB DELTIQ 2 is the ideal printer for printing from PLA and PETG materials [8]. When printing from PETG material, the temperature of 1 layer was set to 235 degrees for better adhesion to the plate.

| MATERIAL                              | PLA                      |
|---------------------------------------|--------------------------|
| PRIMARY LAYER HEIGHT                  | 0.3mm                    |
| INTERIOR FILL PERCENTAGE              | 100%                     |
| HEATED BED                            | 55°C                     |
| HOTEND 1 LAYER                        | 215°C                    |
| HOTEND 2 LAYER                        | 205°C                    |
| DEFAULT PRINTING SPEED                | 3000 mm/min              |

| MATERIAL                              | PETG                     |
|---------------------------------------|--------------------------|
| PRIMARY LAYER HEIGHT                  | 0.3mm                    |
| INTERIOR FILL PERCENTAGE              | 100%                     |
| HEATED BED                            | 90°C                     |
| HOTEND 1 LAYER                        | 235°C                    |
| HOTEND 2 LAYER                        | 240°C                    |
| DEFAULT PRINTING SPEED                | 3000 mm/min              |
After printing the samples, we marked them, measured them with a caliper, and vacuumed them to prevent biodegradation.

4. Non-destructive testing of samples

Non-contact non-destructive measurement on a Carl Zeiss Metrotome 1500 (Germany) (Figure 5) was performed on 20 PLA samples and 20 PETG samples before and after the tensile test. A total of 80 samples were taken and evaluated. The parameters of the metrotome device that uses the application of X-rays and CT tomography are in Table 3 [9].

| Maximum dimensions of the measured component in 3 axes | 300x300x300 mm |
|--------------------------------------------------------|----------------|
| Maximum mass of the component to be measured | 50 kg |
| Illuminator power | 225kV/225W |
The purpose of this effective non-destructive scanning on a Carl Zeiss metrotome was to detect various sample defects that cannot be detected by a normal aspect. These defects affect the final test values of the samples for determining the mechanical properties. The porosity of the samples, the internal structure and the quality of the layers were monitored. Individual aspects were evaluated using VGStudio MAX software. A total of 40 different samples of PLA and PETG were evaluated after and before mechanical testing, where up to 80 scans had to be evaluated. The total porosity (%) of the samples (Table 4) gives us more accurate values, where sample no. 18 of PLA material had the highest value of 3.71% and, conversely, sample No. 14 of PETG material had the lowest porosity value of 1.15% [10] - [12].

Table 4. Porousness of all samples.

| SAMPLE | PLA [%] | PETG [%] |
|--------|---------|----------|
| 1      | 2.54    | 2        |
| 2      | 1.96    | 2.35     |
| 3      | 2.68    | 1.51     |
| 4      | 2.33    | 2.19     |
| 5      | 2.41    | 2.80     |
| 6      | 2.42    | 2.39     |
| 7      | 2.29    | 2.49     |
| 8      | 2.39    | 2.35     |
| 9      | 2.58    | 3.04     |
| 10     | 2.28    | 3.33     |
| 11     | 2.97    | 2.75     |
| 12     | 2.14    | 2.34     |
| 13     | 2.02    | 1.51     |
| 14     | 3.51    | 1.15     |
| 15     | 2.45    | 2.30     |
| 16     | 2.58    | 1.23     |
| 17     | 2.21    | 2.61     |
| 18     | 3.71    | 1.96     |
| 19     | 2.66    | 1.59     |
| 20     | 2.47    | 2.73     |
When testing type 5A dogbone samples with a tensile test, it is assumed that the tearing or stretching of the sample will occur in the area of interest indicated before testing. Due to various defects, which can only be detected using CT tomography or electron microscopy, we found that the cause of rupture of sample no. 12 of PETG material (Figure 6a) outside the area was a defect that can be seen in cross section (Figure 7a) and a higher porosity value at the rupture point of the sample.

![Figure 6a and 6b. Sample No.12 from PETG before and after tensile test.](image)

The point of rupture (Figure 6b) and the higher porosity rate (Figure 7b) are factors that affect the resulting mechanical properties of the samples. The findings showed that the use of CT tomography in the testing of samples from different materials has its necessary justification.

![Figure 7a and 7b. Cross section and porosity rate of sample No. 12 made of PETG material.](image)

When examining the individual layers of sample no. 18 of the PLA material showed layering defects that cannot be detected by the eye (Figure 8). This defect can affect the resulting area of interest and the results of mechanical testing.
5. Tensile test

The tensile test was carried out on a machine from Hegewald & Peschke. This machine is designed to perform destructive tests, and it is located at the Department of Biomedical Engineering and Measurement. The test speed was designated according to EN ISO 527-2: 1996 to 2 mm/min. Figure 10 shows the clamped specimen between the jaws of the testing machine. The evaluation of the tensile test consists of the evaluation of the tension $\sigma_{\text{MAX}}$ [MPa]. During the tensile test, the software of the traction machine recorded the value of the cross-section $S_0$, the value of the modulus of elasticity in tension $E$, the value of the maximum tension $\sigma_{\text{MAX}}$, or the value of the yield tension $\sigma_Y$. The initial measured length $L_0$, the relative elongation and the percentage elongation were documented using an additional extensometer.
Samples of PLA material after the tensile test are shown in Figure 11. Most of the samples were ruptured in the area of interest marked before testing. Samples made of PETG material (Figure 12) showed greater plasticity.
A total of 40 samples were tested. Graphical evaluation with supplemented tables serves for the best possible interpretation of the obtained results. Table 5 shows the tension values (MPa).

Table 5. Sample tension values.

| TEST SAMPLE | PLA [MPa] | PETG [MPa] |
|-------------|-----------|------------|
| 1           | 33.58     | 48.90      |
| 2           | 46.55     | 51.83      |
| 3           | 46.90     | 47.56      |
| 4           | 46.25     | 48.25      |
| 5           | 43.38     | 48.34      |
| 6           | 47.63     | 48.81      |
| 7           | 46.41     | 50.26      |
| 8           | 46.32     | 49.72      |
| 9           | 45.59     | 50.43      |
| 10          | 47.62     | 49.16      |
| 11          | 43.10     | 51.91      |
| 12          | 47.27     | 49.24      |
| 13          | 49.19     | 47.22      |
| 14          | 42.61     | 50.17      |
| 15          | 45.17     | 52.46      |
| 16          | 46.01     | 51.65      |
| 17          | 45.28     | 52.87      |
| 18          | 46.36     | 46.87      |
| 19          | 43.86     | 50.15      |
| 20          | 50.10     | 51.10      |
| AVERAGE     | 45.46     | 49.84      |
| STANDARD DEVIATION | 3.29 | 1.71 |

These values are graphically illustrated in the graph (Figure 13). It follows that the highest values of 52.87 MPa are shown by sample 17 from PETG material. On the contrary, the smallest values of 33.58 MPa were reached by sample 1 and 42.61 MPa by sample 14 made of PLA material. Sample 18 of PLA material with the highest porosity value had a tension value of 46.36 MPa, i.e. above average, and sample 12 of PETG material was 49.24 MPa. From the values found, it can be said that the lower the porosity value, the better the sample and the more relevant results from mechanical testing.
6. Conclusion

By using additive technology, produced components can be used in various fields. The additive manufacturing process can lead to the production of personalized components, but due to the lack of knowledge about the quality of production, it is necessary to proceed to the analysis of components using various imaging methods. In this study, we modeled, printed, labeled, scanned, and tested samples from the most widely used materials in the medical additive field. By scanning, we determined the degree of porosity of all samples, and thorough analysis of various defects in the structures of the samples. The result is tables and images from scanning on the Metrotrom 1500. From the properties obtained by tensile tests, it is clear that the PETG samples achieve a higher degree of plasticity and higher tension values. The study has a significant contribution to the testing of materials and provides scope for further research in the mechanical testing of samples. Based on the acquired knowledge, it is possible in the future to perform extended mechanical tests with statistical calculations and later for complex research to perform tests using an electron microscope. Another direction lies in widespread destructive and non-destructive research. The study is the basis for research into the mechanical properties of samples produced using additive technology and points to the need to use CT tomography to obtain relevant results.

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