Thermo-mechanical properties of fused filament fabricated PLA at elevated temperatures

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DOI: 10.13111/2066-8201.2023.15.1.6

Received: 15 November 2022/ Accepted: 03 February 2023/ Published: March 2023

Abstract: *This study aims to present the authors' recent research investigating the mechanical and thermo-mechanical properties of commercial polylactic acid (PLA) polymer. Samples were manufactured by 3D printing of fused filament fabrication (FFF) and tests were performed according to ASTM International standards for polymers D638, D695 and D790. All test samples were made using the same printing process parameters. The static mechanical tests consisted of tensile and flexural loadings at various temperature ranges, from room temperature to elevated temperature (25°C, 40°C and 50°C, respectively). For ensuring that the additively manufactured products can resist severities of real-life applications, thermal stability under mechanical load tests (HDT - heat deflection temperature) were carried out. The temperature influence on the mechanical and thermomechanical properties was determined and presented, and a synthesis of the characteristics was made in accordance with the applications of products based on the studied material.*

Key Words: *polylactic acid, mechanical and thermo-mechanical properties, 3D printing, fused filament fabrication.*

1. INTRODUCTION

Polylactic acid (PLA), used in a wide variety of applications as part of a composite, and common in the food and medical industries, is among the most common plastics that can be 3D printed on a large scale. Unlike most plastics that are produced using fossil fuels, PLA is a thermoplastic polymer that is found and can be extracted from renewable organic sources (e.g. corn starch or sugar cane), making it a biodegradable polymer [1, 2]. In addition to the biodegradable properties, it also shows good mechanical resistance, properties being much needed in the production of new components through 3D printing [3, 4].

For some time, 3D printing has already been an established process for obtaining structural parts from metals, ceramics and polymers. Unlike the usual technologies for obtaining composite structures, 3D printing is superior in terms of customized design and the use of multiple materials can be applied in structural applications to benefit from modified, superior properties [5, 6]. Besides the customized and complex geometries and design, 3D printing-based technologies using polymers offer cost and time effective manufacturing solutions for a wide range of engineering applications from very divers fields ranging from consumer-good, sports, electronics to medical, automotive and aerospace industries [7]. Some of the most important factors that influence 3D printing of polymers are materials, processes, and design strategies, all of which influence the performance of a manufactured part.

Research in this area led to the synthesis and development of polymers with advantageous characteristics in different directions (such as mechanics, biocompatibility, etc.) by adjusting mechanical properties obtained by altering the printing process parameters [8].

In order to increase the mechanical properties, the printing direction, temperature condition and slicing parameters must be taken into account in the printing process. All these parameters can affect the mechanical properties in different ways─ some more significant than others [4, 9]. In the literature, there are many studies presenting the mechanical properties of materials based on the 3D printed PLA polymer with different additions in the composition [10-12], but few studies are available on the mechanical behaviour at various temperatures and the behaviour of PLA in these temperature ranges.

The novelty of this research is to calculate the mechanical properties such as the strength and modulus of elasticity under tension and bending stress in various temperature ranges of 3D printed PLA and to discover the maximum deflection temperature that this polymer can sustain.

2. EXPERIMENTAL

The polymeric polylactic acid filament used in laser 3D printing was SMARTFIL PLA having the technical specifications presented in table 1 [13].

Table 1. - Polylactic acid filament – technical specifications [13]

Chemical Name	Polylactic Acid
Material Density	1.24 g/cm^3
Filament diameter	1.75 mm (±0.03mm)
Print Temperature	210 ± 10 °C

Two sets of samples were developed (rectangular and dog bone) with specific shapes and sizes for mechanical and thermo-mechanical test according to the international standard ASTM D638 Standard Test Method for Tensile Properties of Plastics [14], ASTM D790 Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials [15] and ASTM 648 Standard Test Method for Deflection Temperature of Plastics Under Flexural Load in the Edgewise Position [16].

PLA polymer was printed using the Smart 3D printer JCR 600/600 PRO equipment. The time required for printing all the samples was 6 h and the main 3D printing properties were presented in table 2.

Table 2. - 3D printing properties

Default printing speed	2200 mm/min
Outline underspeed	90 %
Solid infill underspeed	100 %
Support structure underspeed	90 %
x/Y Axis movement speed	3600 mm/min
Z Axis movement speed	1002 mm-min
Adjust printing speed for layers below	10 sec

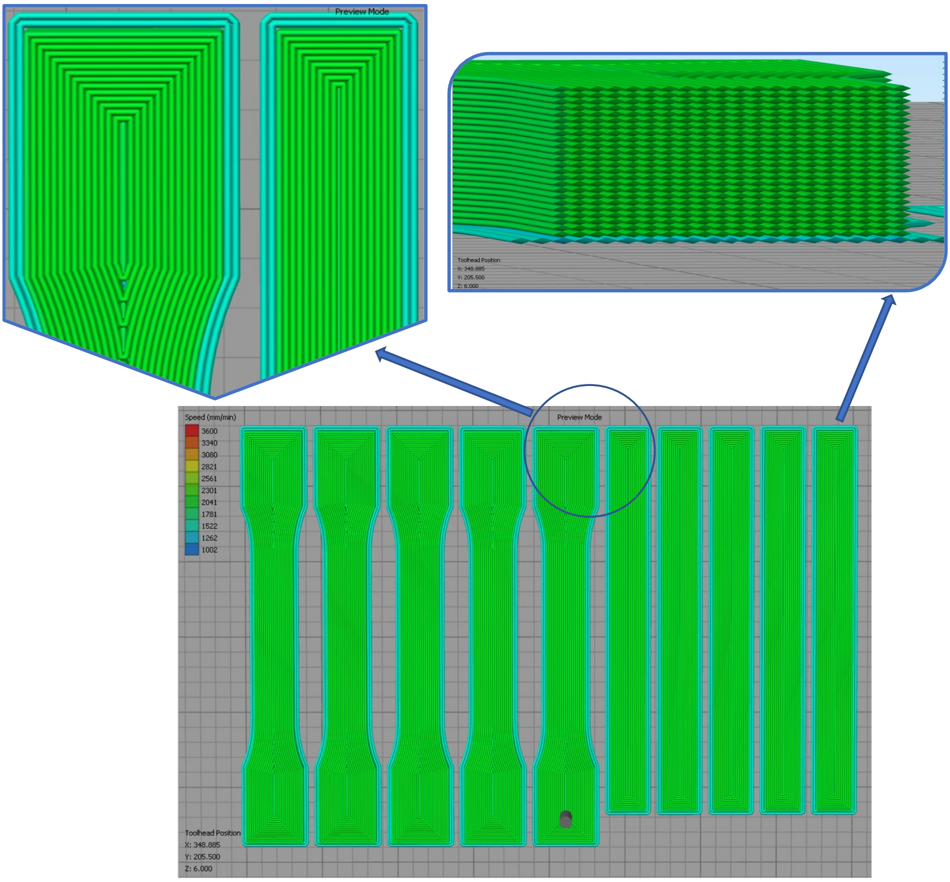


Fig. 1 - Filament orientation during 3D printing

As illustrated in figure 1, 5 samples were printed for each type of investigation carried out, using the same parameters of the printing process. The printing parameters were considered in such a way as to obtain an alignment of printed material along the sample axis and with 0% of infill parameters.

The aria of interest for mechanical testing of the sample can be considered homogenous within 0□ alignment of filaments and almost no voids, as confirmed by the optical microscopy captured after mechanical testing.

3. RESULTS AND DISCUSSIONS

3.1 Mechanical testing

The mechanical tests were performed at various temperature starting from room temperature (about 25°C) to elevated temperature (40°C and 50°C respectively) using INSTRON 5982 mechanical test facility, equipped with a 10 kN force cell and a climatic chamber.

3.1.1 Tensile testing

The tensile testing was performed in accordance with the international standard ASTM D638 Standard Test Method for Tensile Properties of Plastics, with a test speed of 5mm/min [14]. A number of 5 samples were tested for each temperature and the results are illustrated in figures 5 and 9.

Figure 2 shows the stress-strain curves corresponding to the samples tested at room temperature, figure 3 shows the stress-strain curves of samples tested at a temperature of +40°C, and those tested at +50°C are shown in Figure 4. The average value of all results taken into consideration is shown in Table 3.

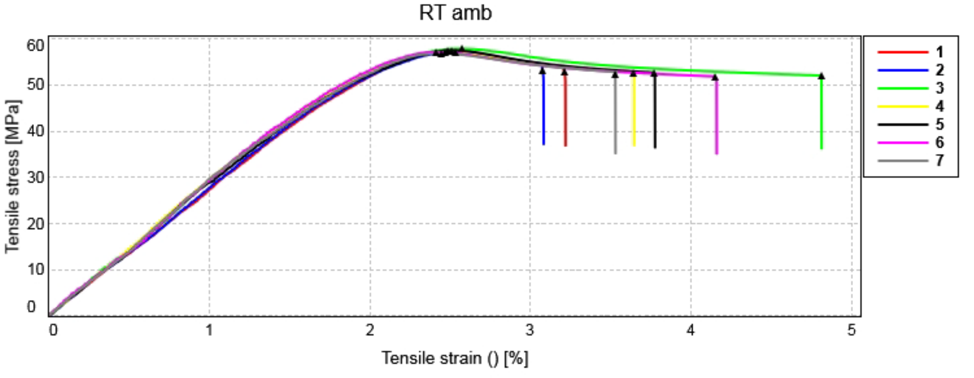


Fig. 2 - Stress-strain curves of 3D printed PLA materials during tensile testing at room temperature

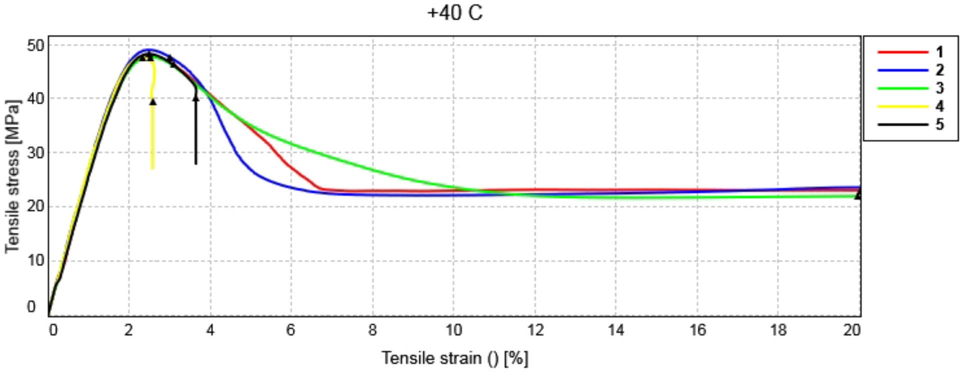


Fig. 3 - Stress-strain curves of 3D printed PLA materials during tensile testing at high temperature (+40 °C)

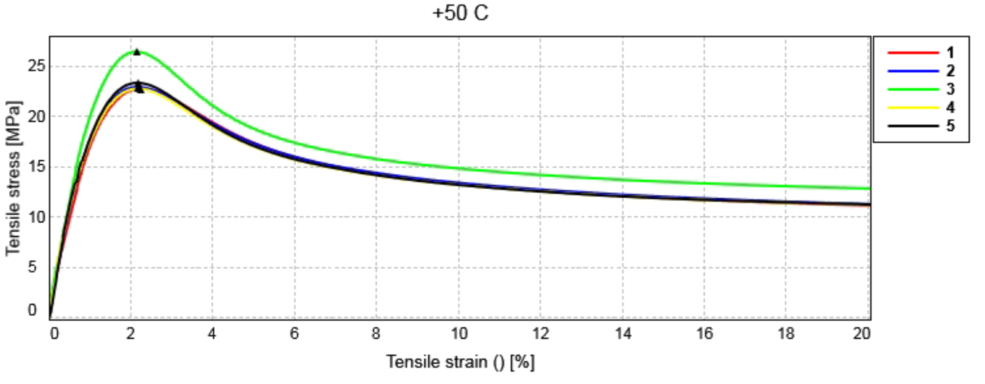


Fig. 4 - Stress-strain curves of 3D printed PLA materials during tensile testing at high temperature (+50 °C)

The macroscopic appearance of the samples before and after the tensile testing is illustrated in figure 5. It can be observed that all specimens tested at room temperature break during tensile loading, while specimens tested at higher temperatures mainly failed by elongation of the narrow area and fracture of the internal layers.

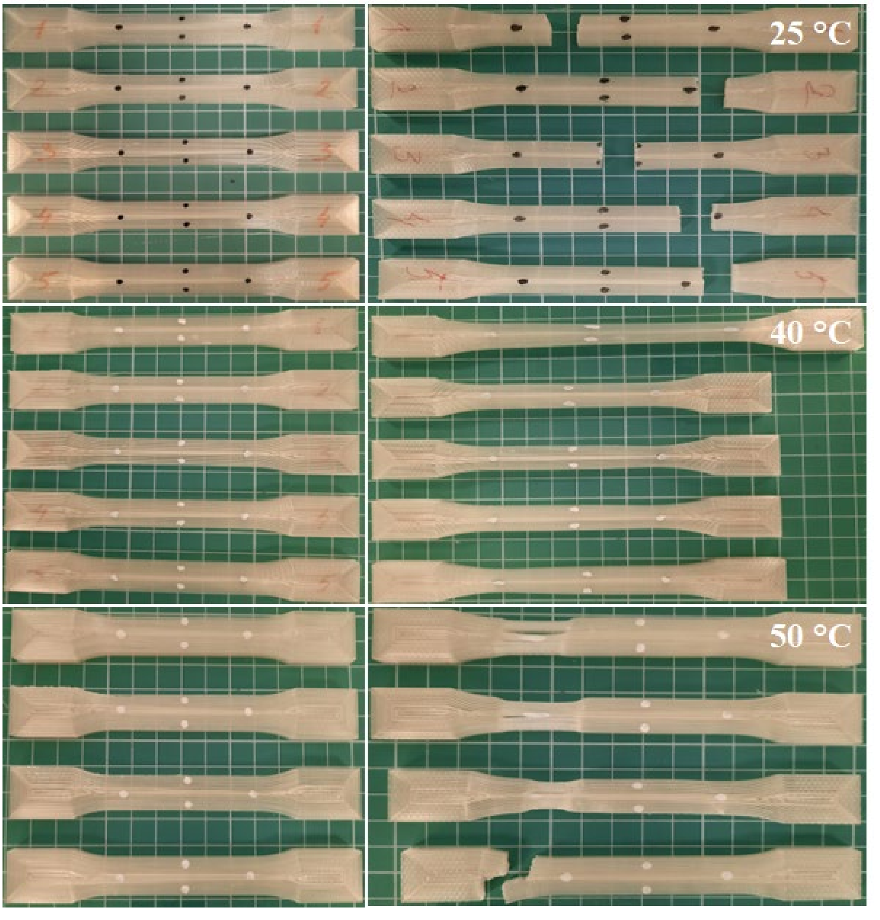


Fig. 5 - 3D printed PLA samples before and after tensile testing

The average results of the Young modulus of elasticity, strength and elongation at tensile load are illustrated in table 3 for 3D printed PLA samples.

Table 3. - Tensile mechanical properties of 3D printed PLA materials

Temp used	Load [kN]	Modulus [GPa]	Strength [MPa]	Elongation [%]
25 °C	4.58	3.001	57.26	2.49
40 °C	3.8	2.889	47.53	2.67
50 °C	1.89	2.443	23.66	2.18

As shown in table 3, the results obtained from the tensile testing of the 3D printed samples indicated that the temperature exposure of the 3D printed PLA material decreases the mechanical properties as the temperature increases.

3.1.2 Three- point bending testing

The mechanical 3-point bending test was performed in accordance with the international standard ASTM D790 Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials, using 10 mm/min speed testing [15]. For the 3-point bending test, 5 specimens were tested for each temperature, the average value of the properties measured during testing are shown in table 4.

Figures 6-8 show the force-displacement curves for all 3 temperatures (room temperature, +40 °C and +50 °C).

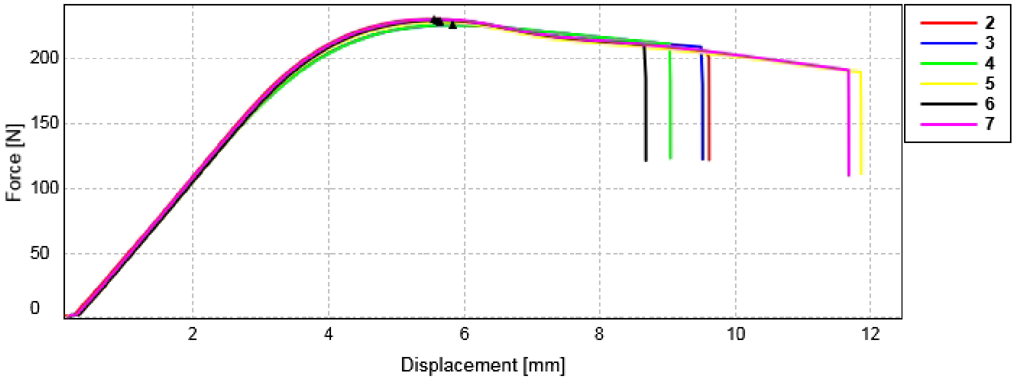


Fig. 6 - Force-displacement curves of 3D printed PLA materials during flexural testing at room temperature

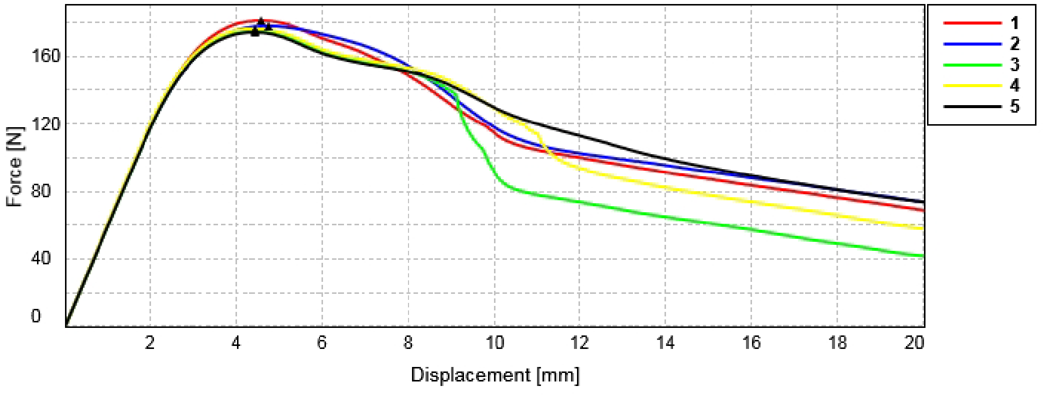


Fig. 7 - Force-displacement curves of 3D printed PLA materials during flexural testing at high temperature (+40 °C)

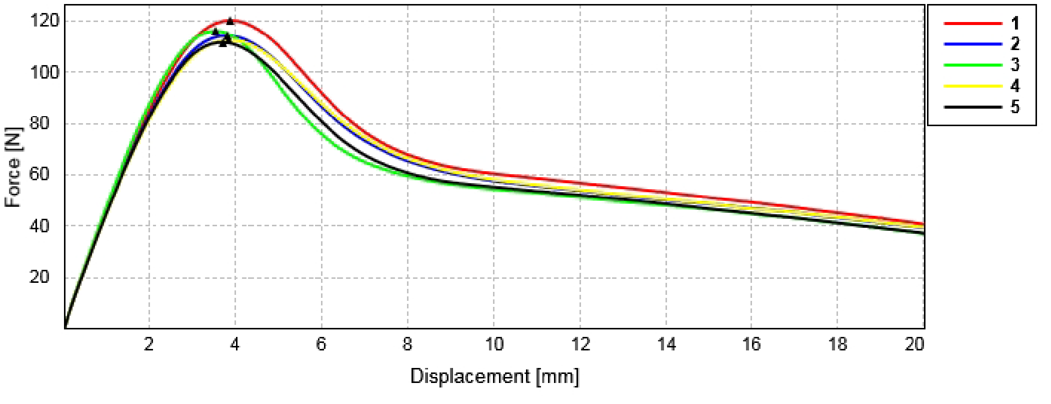


Fig. 8 - Force-displacement curves of 3D printed PLA materials during flexural tests at high temperature (+50 °C)

The images of the samples before and after the 3-point bending test are presented in figure 9.

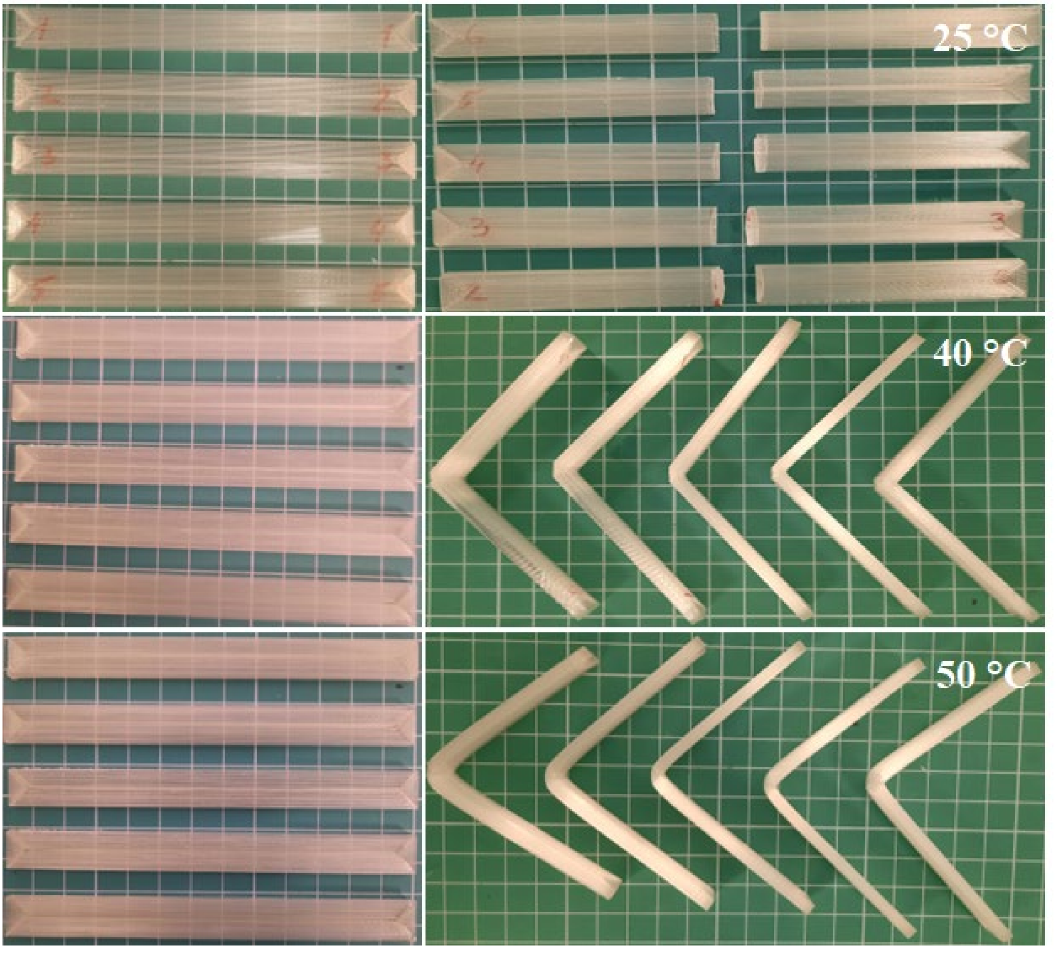


Fig. 9 - 3D printed PLA samples before and after mechanical flexural testing

Table 4. – Mechanical properties at 3-point bending test

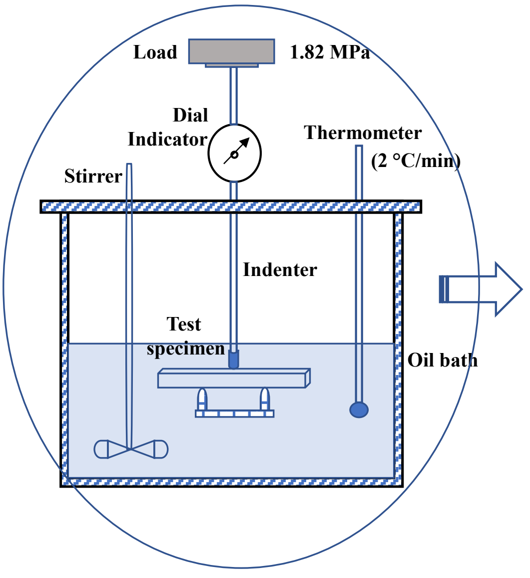
Temp used	Load [N]	Modulus [GPa]	Strength [MPa]	Elongation [%]
25 °C	228	2.89	90.59	5.31
40 °C	176.76	2.47	77	4.14
50 °C	114.87	2.35	49.32	3.45

As expected, according to the values in table 4, the results obtained from the 3-point bending testing of the 3D printed samples indicated that temperature exposure of the PLA material decreases the mechanical properties as the temperature increases.

3.2 Thermo-mechanical testing

The heat deflection temperature (HDT), also known as the deflection temperature under load (DTUL), is an important property of polymers.

It gives an indication of the temperature at which materials start to "soften" when exposed to a fixed load at elevated temperatures. The HDT is defined by ASTM D 648 Standard Test Method for Deflection Temperature of Plastics Under Flexural Load in the Edgewise Position [16], as the temperature at which a sample bar deflects by 0.25 mm under a fixed bending load in a three-point test under a flexural load of 1.82 MPa (Fig. 10). Basically, it tests the stiffness of a material as the temperature increases [16-18].



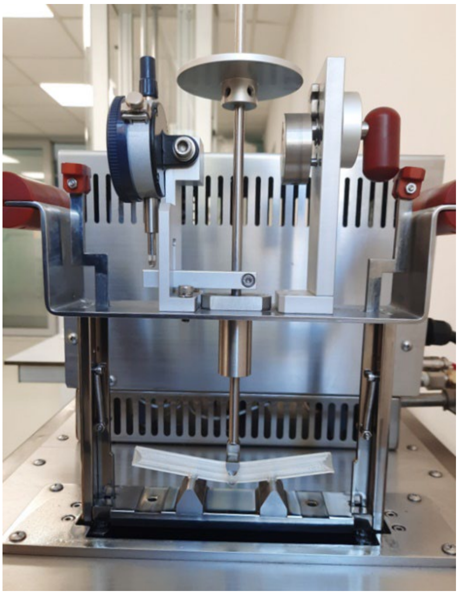


Fig. 10 - HDT test equipment scheme and photo

Figure 11 illustrates the graph of the 5 samples that were tested at HDT and shows the temperature evolution up to a deformation of 0.25 mm. The average of the temperature results of the 5 samples was illustrated with a solid line.

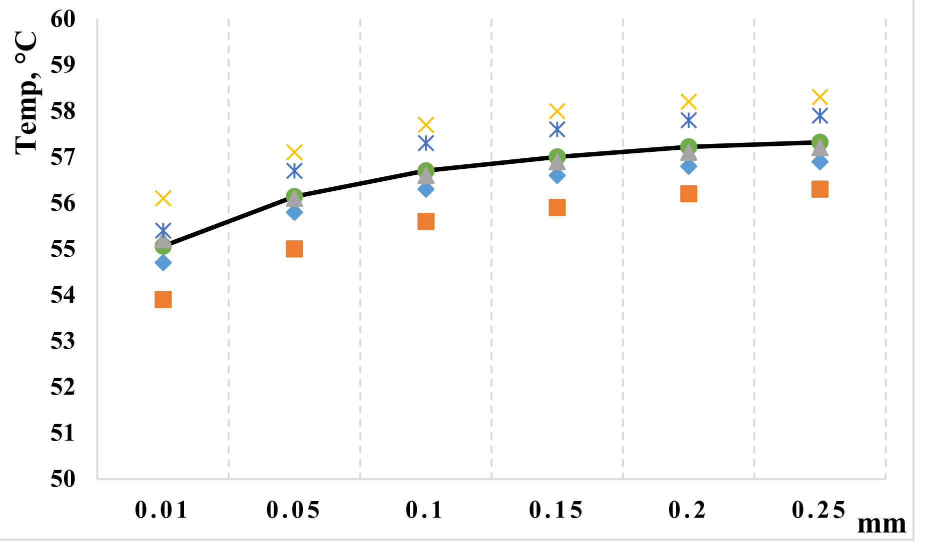


Fig. 11 - Determination of the HDT temperature and its evolution until the final deflection

The graph in figure 11 illustrates that the maximum temperature at which 3D printed PLA cannot be used for structural applications is 57.3°C.

Figure 12 shows the behaviour of the 3D printed PLA samples after being subjected to the thermal stability test under mechanical load.

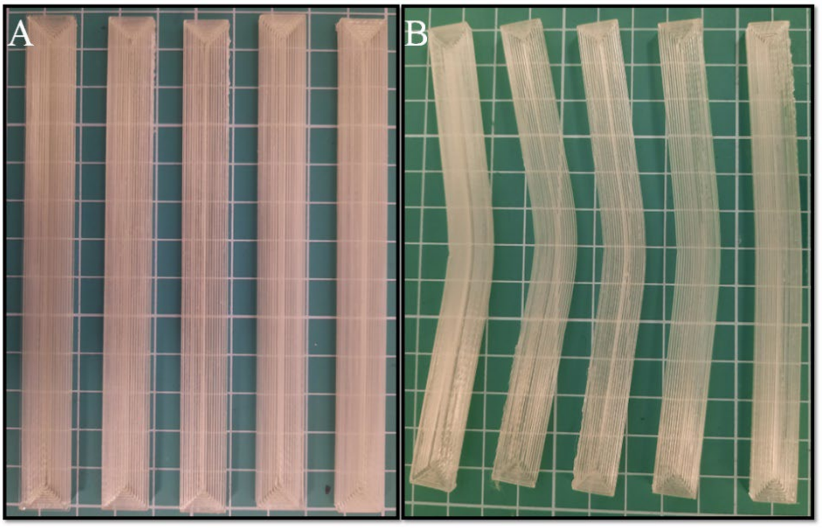


Fig. 12 - 3D printed PLA samples for HDT testing, A- before the test, B- after the test

3.3 Morphostructural analysis

From microscopic point of view, the 3D printed PLA materials were analysed by optical microscopy and scanning electron microscopy (SEM), respectively. In both morphostructural analyses techniques, the images were acquired in the failure area of sample subjected to mechanical tensile and 3-point bending testing. Optical microscopy images were captured on the surface of the fracture area, while SEM images were capture in the fracture cross-section.

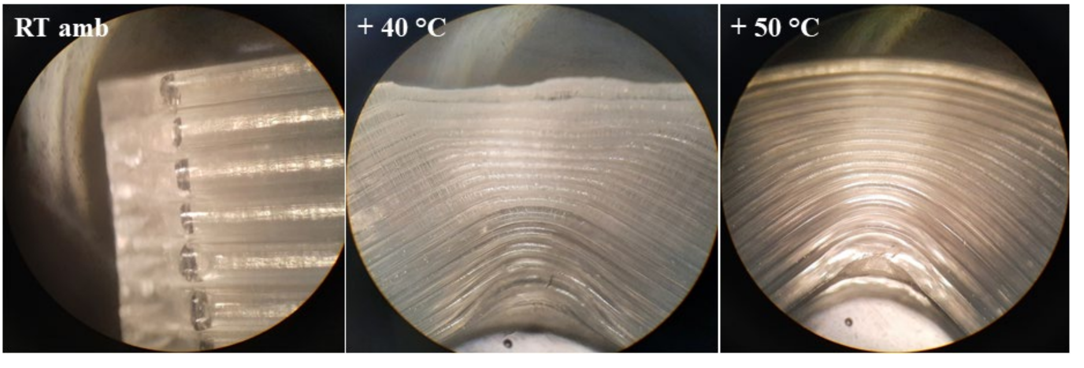


Fig. 13 - Optical micrographs recorded in 3-point bending test failure zone of the 3D printed PLA sample

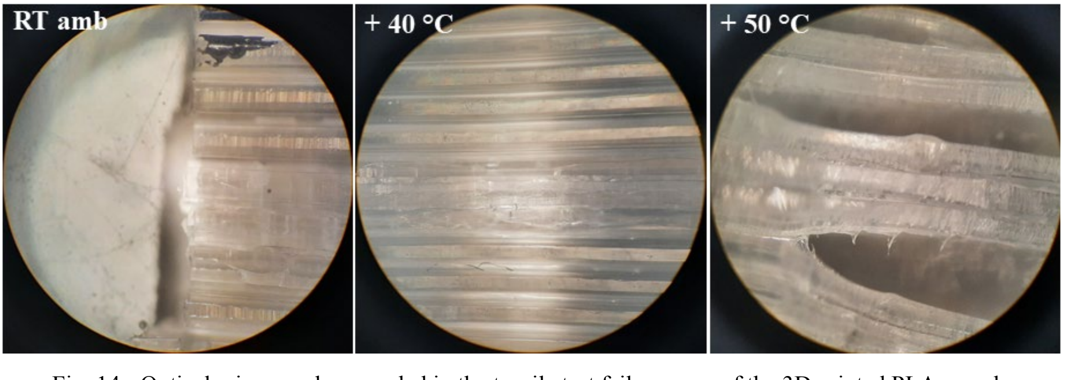


Fig. 14 - Optical micrographs recorded in the tensile test failure zone of the 3D printed PLA sample

Optical micrographs (figure 13 and figure 14) illustrate the fracture appearance of PLA thermoplastic polymer after mechanical testing. It can be observed that during testing at ambient temperature, the PLA filaments were sectioned while, during testing at high temperatures (+ 40 °C and + 50 °C, respectively) the polymer filaments tend to thermally deform, to elongate without the appearance of cracks on the surface.

In the case of tensile tested samples, at a temperature of +50°C, it is possible to observe separation of the filaments, with areas of thermal degradation, representing areas of melted polymer.

Figure 15 illustrates the scanning electron microscopy for the 3-point bending test failure area of the 3D printed PLA sample.

While the samples tested at 25°C broke after the test, it can be seen that the samples tested in the high temperature range show a pronounced plastic deformation, with an increase in the width of the filaments that make up the sample, as an effect of thermo-mechanical deformation of PLA thermoplastic material.

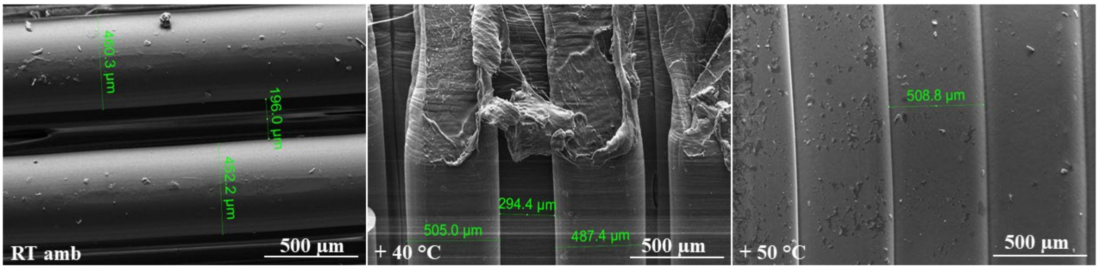


Fig. 15 - SEM micrographs recorded in 3-point bending test failure area of the 3D printed PLA sample

Figure 16 illustrates the scanning electron microscopy for the tensile test failure area of the 3D printed PLA sample.

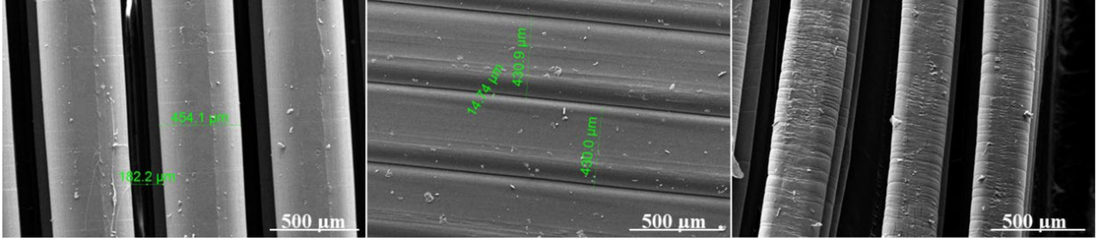


Fig. 16 - SEM micrographs recorded in the in tensile test failure area of the 3D printed PLA sample

Alike the specimens tested by 3-point bending, the specimens tested for tension at ambient temperature also fractured. The samples tested at high temperature, due to the nature of the material, tend to deform thermo-mechanically through thinning and thermal degradation of the filaments that constitute the samples. There are also areas of filament separation with islands of molten polymer.

4. CONCLUSIONS

3D-printed PLA samples with specific shapes were made for mechanical tests (3-point bending and tensile respectively) and thermal stability tests under mechanical loading (HDT).
The mechanical testing was carried out in 3 different temperature ranges (25, 40 and 50°C).
The mechanical results, both in 3-point bending and tensile, of the 3D printed PLA material samples are directly influenced by the temperature range at which the testing is carried out,

with strength and modulus of elasticity values decreasing with an increasing temperature range.

Thermal stability tests under mechanical loading illustrated the temperature limit above which the material cannot be used for structural applications.

The morpho-structural analyses illustrated the type of failure following mechanical tests in tensile and 3-point bending, respectively. The nature of the PLA thermoplastic material indicates areas of thermal deformation with the appearance of regions of thermal degradation and dimensional change of the filaments that constitute the samples following tests in high temperature domains.

Corroborating the mechanical results with the morpho-structural information, it is observed that the nature of the PLA thermoplastic material is directly influenced by the temperature to which it is subjected, the mechanical properties being diminished with the increase of the thermal range.

ACKNOWLEDGEMENT

This work was supported by a grant of the Romanian Ministry of Education and Research, CCCDI UEFISCDI, Project number PN-III-P2-2.1-PED-2021-3408, within PNCD III.

This work was also supported by the POC Project “Platformă de Dezvoltare Tehnologică pentru Tehnologii “Green” în Aviație și Fabricație Ecologică cu Valoare Adăugată Superioară; TGA- Technologies for Green Aviation”, Cod SMIS: 127115.

This article is an extension of the paper presented at *The International Conference of Aerospace Sciences*, “***AEROSPATIAL 2022***”, 13 - 14 October 2022, Bucharest, Romania, *Hybrid Conference*, Section 4. Materials and Structures (*Thermo-mechanical properties of fused filament fabricated PLA at elevated temperatures*, Book of Abstracts - International Conference of Aerospace Sciences, “AEROSPATIAL 2022”, Hybrid conference, Bucharest, Romania, 13-14 October, 2022, ISSN 2067 – 8614, ISSN-L 2067 – 8614, https://aerospatial-2022.incas.ro/files/book_of_abstracts__aerospatial-2022.pdf).

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