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Research paper

Mechanical Properties of Material Extrusion Printed Samples Made of PLA After Treatment in an Acetone Bath

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Abstract. This paper presents a study of the effect of chemical treatment using pure acetone on strength specimens made from PLA polylactide produced by 3D FDM/FFF printing technology. The strength specimens were designed in accordance with the ISO 527 standard. The chemical treatment was carried out using baths at different time intervals. The hardness of the samples was measured, and a tensile test was performed. The results show that with prolonged contact with acetone, the hardness of the material decreases by approximately 45% compared to untreated samples. When analysing the tensile tests, it was noted that the tensile strength decreases with longer chemical bath time. At the same time, the material becomes more ductile, resulting in a high elongation at break in tension of up to 40% relative to untreated samples.
Keywords: 3D printing, PLA, acetone, hardness, tensile strength

1. INTRODUCTION

The Fourth Industrial Revolution 4.0 encompasses many technologies; one of the leading ones being 3D printing [1]. The rapid development of additive technologies has promoted their use in industries such as mechanics, architecture, art, and medicine as functional models or prototypes and as repair tools for existing components [2-4]. The popularity of additive technologies is due to the simple operation of the equipment, access to a wide range of model materials with different properties, intuitive control programs, and, above all, the possibility of producing geometrically advanced models. However, layered parts manufacturing is sometimes responsible for lower dimensional and shape accuracy and surface roughness compared to conventional fabrication methods [5, 6]. Due to the low accuracy of the process, it is essential to use appropriate finishing technology. Finishing can be carried out by machining, abrasion and chemical treatment. In the case of the PLA material under study, the most commonly used finishing treatment is chemical treatment with acetone. Previous studies have shown that samples deform and eventually fail after prolonged contact with the solvent [7]. In these articles, the chemical treatment was performed by immersing the samples in the solvent and by vaporising the sample. Studies on the effect of chemical treatment on the physical and mechanical properties of additively manufactured models, confirm the effect of solvent on weight, hardness and tensile strength [8]. In addition, the results obtained show an evident change in roughness between untreated and acetone-bathed samples [9, 10].

Along with chemical treatment, machining is also a subject of research. Using the article [11] as an example, the effects of cutting speed, cutting tool feed and depth of cut on the surface roughness of samples made of PLA material were investigated. The authors also drew attention to another aspect - regardless of the tool, the technological parameters of machining should be adapted to the material. The authors' prediction was confirmed in the article [12]: in the case of PLA, higher cutting speeds should be used than in the case of ABS material.

Referring to the optimisation of the additive process, the authors of the article [13] proved in their research that the model's direction (equivalent to its position in the working platform) influences the final roughness after machining. In addition to roughness, finishing also affects shape deviations. Article [14] investigated the effect of machining on the cylindricity deviation of models. The cylindricity deviation decreased; however, deformation of the sample occurred in the case of thin-walled parts. An example of other finishing technologies is abrasive machining. The authors of the article [15] proposed finishing through vibratory abrasive machining.

After performing a literature analysis, it was noted that a small number of articles addressed the effect of chemical treatment on material strength properties. The study's primary objective is to determine the impact of the exposure time of the samples in the acetone solution on mechanical properties. The secondary objective is to compare the results obtained with surface roughness measurements to determine the time that provides the greatest decrease in roughness while maintaining good strength properties.

2. MATERIALS AND METHODS

2.1. FFF/FDM Technology

The most common 3D printing technology is FDM/FFF (Fused Deposition Modelling / Fused Filament Fabrication), in which a model is built up in layers by extruding a thermoplastic material through a heated nozzle. Plasticised by elevated temperature, the material is applied in the printer's working space according to the set layer geometry, which cools down after extrusion and bonds with the previously applied layer. The technological parameters in this technology that largely determine the surface properties are layer thickness, filament feed speed and path width [16]. Of the parameters listed, layer thickness significantly impacts the properties of the surface layer and the mechanical properties of the 3D-printed part.

The test samples were made using a MakerBot Sketch machine from MakerBot (merged with Ultimaker company in August 2022).

2.2. PLA (Polylactic Acid) Material

Poly(lactic acid) or Poly(lactide) (PLA) is a biodegradable material derived synthetically from lactic acid or by processing, e.g. maize, sugar cane or starch. Biodegradation of PLA occurs through the action of hydrolysis, a gradual degradation of the material due to the action of water. This process can be accelerated by increasing the temperature or placing the material in an acidic environment [17].

In order to overcome the disadvantages of PLA, such as high stiffness and brittleness at room temperature, thermal degradation, etc., the material can be modified by plasticisation, incorporation of fillers, blending with different polymers, copolymerisation, crosslinking [18].

The chemical composition of the material and selected mechanical properties are shown in Table 1. The data presented are for PLA material, which is produced directly by MakerBot [19, 20]. However, it should be noted that the mechanical properties of the 3D printed part depend on the printing parameters, such as layer height and fill density. The disadvantage of additive technologies is the anisotropy of mechanical properties, meaning that they depend on the part's positioning on the printer's working platform [21].

Table 1. Selected mechanical properties and chemical composition of PLA [19, 20]

Parameter	Value STD*	Value MAX**	Unit
Compressive Strength	17.9	93.8	MPa
Tensile Strength	46.8	65.7	MPa
Flexural Strength	61.8	94.7	MPa
Information on ingredients (>98 %):	1,4-Dioxane-2,5-dione, 3,6-dimethyl-, (3R-cis)-, polymer with (3S-cis)-3,6- dimethyl-1,4-dioxane-2,5-dione and trans-3,6-dimethyl-1,4-dioxane-2,5-dione		

* STD or Standard resolution, standard profile settings,
** MAX or High resolution, 100% infill

2.3. Samples Preparation

The samples were designed in SOLIDWORKS (Dassault Systemes SolidWorks Corp., Waltham, MA, USA). The geometrical dimensions of the sample were in accordance with ISO 527 (Figure 1a). An STL file was then generated with the following parameters: number of triangles 252, deviation tolerance 0.0045 mm and angle 6° (Figure 1b). The STL file was loaded in MakerBot Print software (Figure 1c) and then printed after setting the technological parameters (Figure 1d).

Modified technological parameters relative to those built into the machine software were adopted to produce the samples. The nozzle temperature was 220°C, a linear fill pattern and maximum fill were used, and the layer height was 0.1mm. All other parameters remained unchanged.

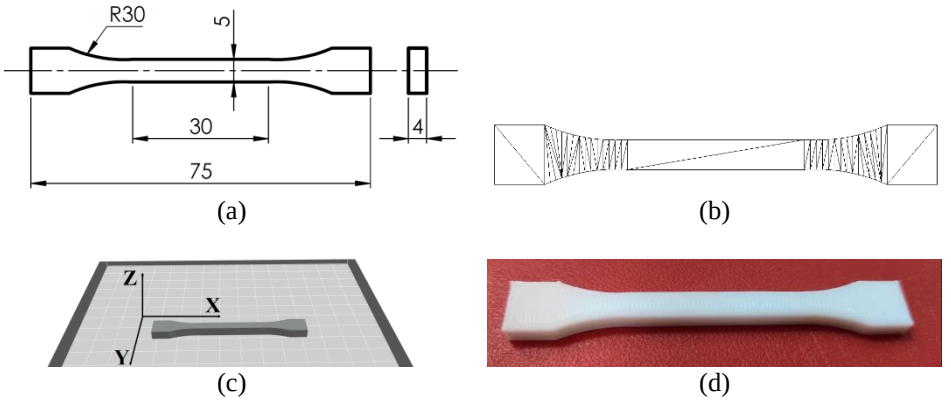


Fig. 1. Samples: (a) dimensions of the sample, (b) triangle mesh of the STL file, (c) virtual printer workbench, (d) produced test sample

The designed samples were printed 'flat' in the 0° (printing direction) without supporting structures, as shown in Figure 1c. The decision for the following arrangement of the samples was made based on our own experimental studies, which clearly show that samples printed in the Z-axis (vertical) show the worst strength properties, while similar values were obtained for samples printed on the edge (Y-axis). However, edge-printed samples, i.e. in the Y-axis, require mechanical cleaning of the surface from the support material, which in turn may result in damage or incomplete removal of the material. Losses or over-removal of material can negatively impact the reliability of the tests.

2.4. Measurement Technologies

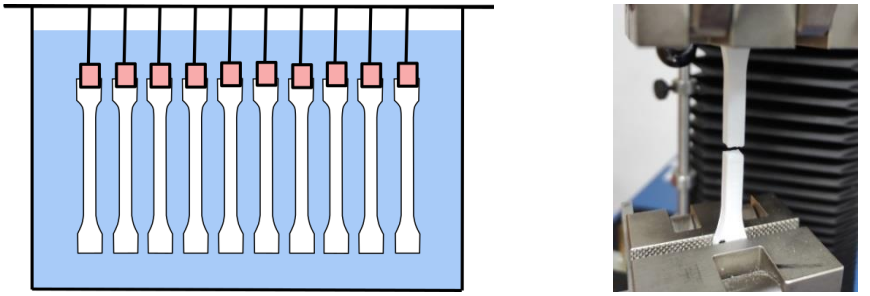
The printed samples were placed in a sealed vat filled with pure acetone (Figure 2a). The bath times were respectively: 1 min, 15 min, 30 min, 45 min and 60 min. Prior to the main test, a Mitutoyo digital micrometer was used in the test to measure the width of the measurement base and the actual cross-section. The device has a resolution of 0.001mm. The hardness of the samples was tested using a Hildebrand Shore D hardness tester. Measurements were taken in accordance with ISO 868. The roughness of the samples was measured using a surface roughness tester (INSIZE CO., Suzhou New District, China). The main tensile tests were carried out on an Inspekt Mini strength testing machine using LabMaster (Hegewald and Peschke, Nossen, Germany). The tests were conducted at a speed of 1 mm/min. Figure 2b shows a sample after rupture. The values of the individual sizes were calculated from the formulae:

$$\overline{x} = \frac{\sum_{i=1}^{n} x_i}{n} \tag{1}$$

where: n – group size; x_i – single test result

$$SD = \sqrt{\frac{1}{(n-1)} \sum_{i=1}^{n} (x_i - \bar{x})^2} \quad (2)$$

where: n – group size; x_i – single test result; $\bar{x}$ – test mean value;



(a) (b)

Fig. 2. Samples: (a) acetone bath visualization, (b) sample after tensile test

3. RESULTS AND DISCUSSION

3.1. Hardness

The hardness was measured using the Shore D scale method, five measurements were taken of each sample, and the mean value and standard deviation were then calculated. The hardness measurement results are shown in Figure 3. The hardness test samples were not reused for the static tensile test.

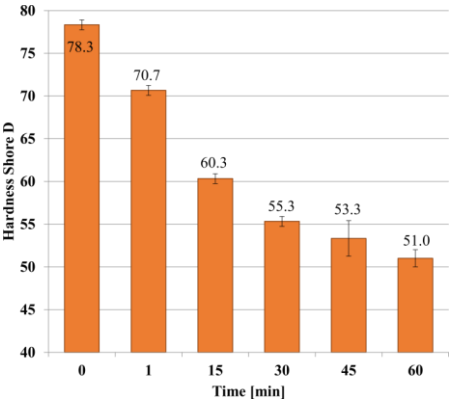


Fig. 3. Shore hardness (D scale)

The surface hardness of the samples decreased as the acetone bath time was increased. After 1 min, the hardness decreased by about 10%, while after 15 min, the hardness decreased by about 23% (compared to the 0 min reference sample).

The difference in hardness between the sample in contact with acetone for 1 min and the sample in contact for 15 min is about 15%. For the 15 min and 30 min samples, the difference was about 8%, while for the 30 min and 40 min samples, it was only 4%. The difference in hardness between samples soaked longer than 15 min is increasingly smaller. This means that the greatest effect of acetone on sample hardness occurs during the first 15 minutes of the bath.

3.2. Roughness

Roughness measurements were made for five random specimens from each of the six groups tested prior to the tensile test. The measurements were made on the upper surface of the specimen (located in the XY plane, as shown in Figure 1c), and each measurement was repeated three times. The results of the roughness measurement are shown in Figure 4. The roughness values given here are defined as follows: R_a is the arithmetic mean deviation of the measured profile from the centerline of the evaluation length, and R_z is the average distance between the highest peak and lowest valley in each sampling area.

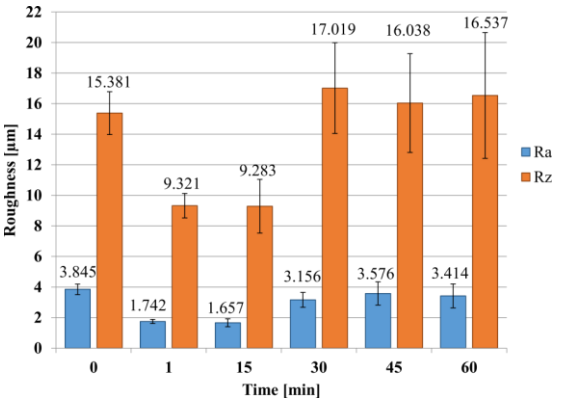


Fig. 4. Roughness

The surface roughness decreased after a bath time of 1 min by 55% for the R_a parameter and 39% for the R_z parameter. In comparison, after 15 min, these parameters decreased by about 57% R_a and 40% R_z (compared to the 0 min reference samples).

Increasing the bath time in acetone caused surface deformation and deterioration of surface roughness, reaching values of the R_a parameter similar to untreated samples and higher values of the R_z parameter with a much higher standard deviation. In the case of samples above the 15 min time, the difference in R_a between the reference samples decreased (compared to the 1 min and 15 min samples), amounting to 18% for 30 min, 7% for 45 min, and 11% for 60 min respectively. However, the parameter R_z, as a result of surface deformation, increased by: 11% for 30 min, 4% for 45 min, 8% for 60 min.

3.3 Tensile Strength

Static tensile tests were performed for six series (A – 0 min, B – 1 min, C – 15 min, D – 30 min, E – 45 min, F – 60 min) of 10 samples each (Figure 6). The first series (Figure 6a) corresponds to the testing of reference samples, i.e. not chemically treated. Prior to testing, each sample was measured using a micrometre to determine its cross-section. Measurements of the thickness and height of the sample were taken at a selection of three points (contained in the base) the same for each sample.

Analysing the results of the tensile strength measurements of the samples, as shown in Figure 6, it can be seen that the tensile strength decreased successively with increasing bath time in pure acetone. The first minute resulted in a decrease in tensile strength R_m of approximately 17%. The difference became increasingly greater for the subsequent measurement series, with respect to the reference series A, and amounted to C 42%, D 50%, E 47%, and F 57%, respectively. The standard deviation of the tensile strength for each measuring series remained at a similar level and was in the range of 0.57–0.86.

In the case of elongation at break, the values increased successively with the bath time of the samples in pure acetone. In relation to reference series A, the C series had a value 17% higher, D 17% higher, E 35% higher, and F 48% higher. The exception is series B, where after 1 min the value remained at a similar level, 1% lower compared to series A.

After the tests, the fracture surfaces of the samples were also analysed. It is noteworthy that during the tests, when the maximum tensile force F_m was reached on the samples of series A, B, C, D, no significant constriction appeared at the point of break, the so-called neck. However, in the case of samples from the E and F series, a neck formation was observed moments before break, and the sample cross-section at the rupture point assumed an irregular, quasi-oval shape (Figure 5).

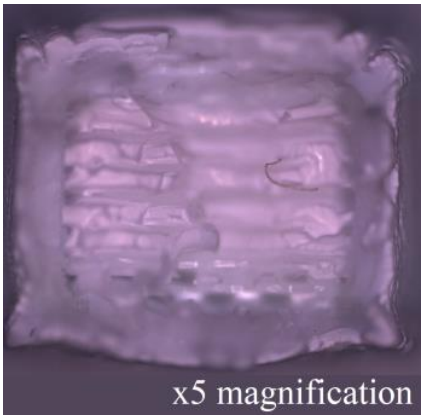


Fig. 5. Macroscopic fracture surface of the E series sample after test

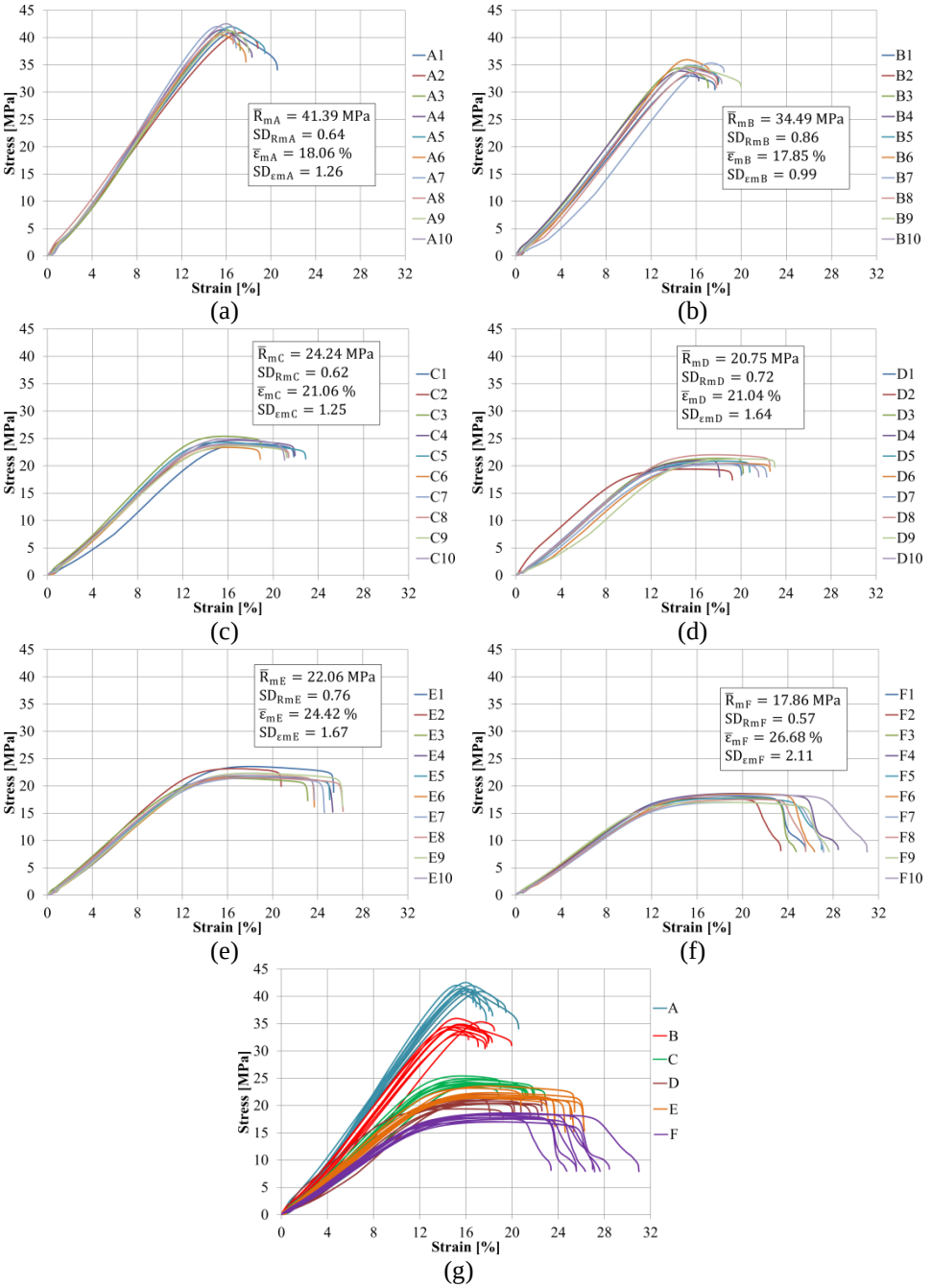


Fig. 6. Results of the tensile test: (a) reference, no contact with acetone, (b) acetone bath for 1 min, (c) acetone bath for 15 min, (d) acetone bath for 30 min, (e) acetone bath for 45 min, (f) acetone bath for 60 min, (g) overall results

4. CONCLUSIONS

The study of the effect of the acetone bath showed that the longer the contact time between the solvent (pure acetone) and the sample (made of PLA), the elongation of the sample increases, while the tensile strength gradually decreases. In other words, the study indicates that contacting PLA samples with pure acetone increases the plasticity of the sample at the expense of its tensile strength with longer contact. In addition, longer acetone bath times result in a gradual decrease in the hardness of the samples tested.

Analysing the results, taking as a criterion the lowest value of surface roughness while maintaining the highest possible mechanical properties (hardness, tensile strength), a soaking time of 1 minute can be specified as suitable for using an acetone bath.

Future research will focus on performing comparative studies of vaporisation treatments and those using other solvents.

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Właściwości mechaniczne próbek wytworzonych technologią wytłaczania warstwowego z materiału PLA po obróbce w kąpieli w acetonie

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Streszczenie. W artykule przedstawiono badanie wpływu obróbki chemicznej przy użyciu czystego acetonu na wytrzymałość próbek wykonanych z polilaktydu PLA w technologii druku 3D FDM/FFF. Próbki wytrzymałościowe zostały zaprojektowane zgodnie z normą ISO 527. Obróbkę chemiczną w roztworze acetonu przeprowadzono w różnych odstępach czasu. Zmierzono twardość próbek i przeprowadzono statyczną próbę rozciągania. Wyniki wskazują, że przy dłuższym kontakcie z acetonem twardość materiału spada o około 45% w porównaniu z próbkami niepoddanymi obróbce. Analizując testy rozciągania, zauważono, że wytrzymałość na rozciąganie zmniejsza się wraz z wydłużeniem czasu trwania kąpieli chemicznej. Jednocześnie materiał staje się bardziej plastyczny, co powoduje, że wydłużenie przy zerwaniu jest o 40% większe w porównaniu do próbek niepoddanych obróbce chemicznej.
Słowa kluczowe: druk 3D, PLA, aceton, twardość, wytrzymałość na rozciąganie