DOI: 10.5604/01.3001.0016.2672

Volume 115 • Issue 2 • December 2022

Journal
of Achievements in Materials and Manufacturing Engineering

International Scientific Journal
published monthly by the
World Academy of Materials
and Manufacturing Engineering

Study of the adhesion between TPU and PLA in multi-material 3D printing

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ABSTRACT

Purpose: In the Fused Filament Fabrication (FFF/FDM) technology, the multi-material manufacturing additive method is achieved by a single nozzle or multiple nozzles working simultaneously with different materials. However, the adhesion between different materials at the boundary interface in FDM multi-material printing is a limiting factor. These studies are concerned with improving and study the adhesion between two polymers.

Design/methodology/approach: Due to the numerous applications and possibilities of 3D printed objects, combining different materials has become a subject of interest. PLA is an alternative to the use of petrochemical-based polymers. Thermoplastic Polyurethane is a flexible material that can achieve different characteristics when combined with a rigid filament, such as PLA. To improve the adhesion between PLA and TPU in multi-material FFF/FDM, we propose the comparison of different processes: post-processing with acetone immersion, surface activation during printing with Acetone, surface activation during printing with tetrahydrofuran, post-processing annealing, and connection of printed parts with tetrahydrofuran.

Findings: Modifying the 3D printing process improved the quality of the adhesive bond between the two different polymers. Activation of the surface with THF is the treatment method recommended by the authors due to the low impact on the deformation/degradation of the object.

Research limitations/implications: In the study, adhesion was considered in relation to the circular pattern of surface development. Further analysis should include other surface development patterns and changes in printing parameters, e.g. process temperatures and layer application speed.

Practical implications: 3D printing with multi-materials, such as PLA biopolymer and thermoplastic polyurethane, allows for the creation of flexible connections. The strengthening of the biopolymer broadens the possibilities of using polylactide. Examples of applications include: automotive (elements, where flexible TPU absorbs vibrations and protects PLA from cracking), medicine (prostheses with flexible elements ensuring mobility in the joints).

Originality/value: Multi-material printing is a new trend in 3D printing research, and this research is aimed at promoting the use and expanding the possibilities of using PLA biopolymer.

Keywords: Multi-material printing, PLA, TPU, FDM, Adhesion

Reference to this paper should be given in the following way:

E. Brancewicz-Steinmetz, R. Valverde Vergara, V.H. Buzalski, J. Sawicki, Study of the adhesion between TPU and PLA in multi-material 3D printing, Journal of Achievements in Materials and Manufacturing Engineering 115/2 (2022) 49-56. DOI: https://doi.org/10.5604/01.3001.0016.2672

MANUFACTURING AND PROCESSING

1. Introduction

Fused Filament Fabrication (FFF/FDM) is a rapid prototyping method for three-dimensional objects [1-3]. Because of its numerous application possibilities, FDM is widely used in Additive Manufacturing (AM) [4,5]. This enables the production of complex geometries using a wide range of materials. In FDM, the material is melted and directed through a nozzle. It constructs successive layers under computer control according to a 3D-CAD model previously set [3,4]. This technology has grown significantly over the last ten years. Studies indicate that FDM changes manufacturing practices in different industry sectors and healthcare [5–11]

Due to the numerous applications and possibilities of 3D printed objects, combining different materials has become a subject of interest [12,13]. In the FDM method, the multi-material manufacturing additive method (MMAM) is achieved by one single mixed nozzle or multiple nozzles working simultaneously with different materials [14,15]. Multiple nozzle systems increase the printing speed by making the manufacturing process continuous. Furthermore, compared to other methods, multi-material FDM printing requires a lower cost for small-footprint machines [16]. Using multi-material techniques can create partswith elastic properties and functionalities (e.g., mechanical, physical, electrical, chemical, optical) [12,15,16]. MMAM has been studied and applied in different areas, such as structural applications (mechanical and civil), medical prostheses, the automotive sector, agriculture, and production for robotic systems [11,14, 17-19].

Polylactic acid (PLA) is FDM's most widely used material, including its combination with other polymers [13,20,21]. PLA is an alternative to the use of petrochemical-based polymers. It is derived from renewable sources, and it is known to have both excellent biodegradability and biocompatibility [22-24]. PLA has a relatively low melting temperature (180-220°C) with a glass transition temperature of 60-65°C [13,25]. This material is inexpensive, has high strength, and is known to be stable while printing [23]. However, its use is limited in plastic practices due to its brittleness, low impact resistance, and flexibility. The use of pure PLA in FDM applications can have drawbacks such as poor mechanical properties, wettability, and thermal stability [26]. For industrial applications, the strength properties of PLA might be insufficient [13]. Consequently, the efforts to improve the toughness and mechanical properties of the material by combining it with different polymers are an area of interest for industry and technology [23, 27-29]

Thermoplastic Polyurethane is a flexible material that can achieve different characteristics when combined with a rigid filament, such as PLA [15,30]. Thermoplastic Polyurethane has an exceptional combination of flexibility, toughness, durability, and biocompatibility [22,31]. TPU is a linear elastomeric material composed of soft (mainly polyester and polyether) and hard segments (short chains of diisocyanate). The flexibility and toughness can be adjusted by regulating the ratio and the chain length [22,23]. The high performance and variety of physical property combinations enable various applications [32-34].

The use of the FDM method with PLA and TPU blends has been demonstrated to help design and fabricate personalized orthoses. The production with combined filament was revealed to be more suitable than the production with pure PLA [35]. Szarlej et al. [36] focused on manufacturing TPU/PLA composition added with amikacin sulfate to obtain antimicrobial properties that can be applied in skin generation and fabrication of antibacterial wounds. Luchini et al. [37] studied the possibility of using a PLA/TPU combination for personal protective equipment [13]. Hong et al. [34] investigated the mechanical properties of PLA/TPU blends. The research showed that PLA, with the addition of TPU, transforms from brittle to ductile fracture. Mo et al. [38] developed a graft polymer by adding dicumyl peroxide (DCP) and glycidyl methacrylate (GMA) to blends made of TPU and PLA. The results showed improved compatibility in the modified blend compared to the PLA/TPU blends without adding any other compound.

Additionally, the combination of the TPU and PLA filaments in the multi-material FDM method can result in an element with singular characteristics;. At the same time, PLA absorbs the load, TPU limits cracks. Studies demonstrated that multi-material samples made of PLA and TPU in the sandwich form had their tensile strength and elastic modulus improved [15].

However, the adhesion between different materials at the boundary interface in FDM multi-material printing is a limiting factor. The physical boundary interface and the chemical one should be considered in this scenario [39]. Arifvianto et al. [15] state that the principles of adhesion between PLA and TPU are possibly related to Van der Waals forces and wetting mechanisms. Liu Z. et al. [22] identify the hydrogen bonding between the urethane groups of TPU and ester groups of PLA and the Van der Waals force as the origin of interactions between these two polymers. Despite this compatibility, a drop in Young's modulus and tensile strength in the interface of PLA and TPU was noticed compared to the interface with the same material.

Brancewicz-Steinmetz et al. [33], to strengthen the adhesion between PLA and TPU, studied the influence of printing parameters, such as layer height, infill density, infill pattern, printing temperature, and print speed. Based on the Pearson correlation between the patterns analyzed, they concluded that the circles/circles performed the best results. Furthermore, the annealing without Acetone in post-processing treatment performed an increased shear strength compared to annealing, annealing in acetone vapours, and acetone vapors at room temperature [40]. It is important to mention that the solvent tetrahydrofuran (THF) is used for the research. This chemical has excellent solubility and diffusion properties. THF is widely used as a solvent for vinyl, epoxy adhesives, and special resins [41].

To improve the adhesion between PLA and TPU in multi-material FDM, we propose the comparison of different processes: post-processing with acetone immersion, surface activation during printing with Acetone, surface activation during printing with tetrahydrofuran, post-processing annealing, and connection of printed parts with tetrahydrofuran.

2. Materials and methods

2.1. Materials

The 3D printer used in the research is the Ultimaker 3. For the test, the Ultimaker PLA and Fillamentum Flexfill TPU 98A have a diameter of 2.85 mm. 3D printing with PLA is simple and does not require unique treatments or requirements. However, for printing with TPU, it is necessary to dry the material for 24 hours before its use. For this end, two TPU boxes were used. While one was being used for printing, the other one was left to dry.

2.2. Samples

A cylindrical 3D model was created using Ansys Workbench software with the SpaceClaim module (Ansys Inc, Cannonsburg, PA, USA). The sample geometry consists of two superimposed cylindrical solid figures. The model (Fig. 1 – created in Ansys SpaceClaim) shows the dimensions of the sample.

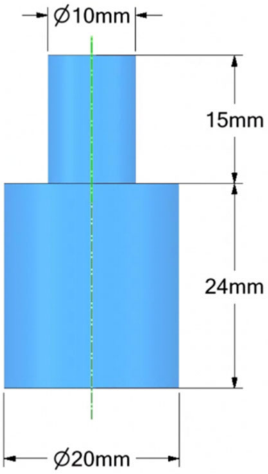


Fig. 1. Sample Model for the shear strength test

The individual files of the two parts were uploaded to Cura Software. The printing parameters were assigned to each part and merged to form one sample. Two different configurations were used to test the adhesion strength. The printing pattern applied for both parts was a concentric one.

For each treatment, a total of 14 samples were made. Half of them correspond to the TPU/PLA configuration, which means that the most oversized cylinder is printed with TPU and the small one with PLA. In comparison, the remaining seven samples correspond to PLA/TPU configuration, which is the opposite arrangement.

2.3. Printing parameters

The printing parameters used in Cura Software are presented in Table 1. These parameters were studied and analyzed in depth in the Brancewicz-Steinmetz et al. [33] articles for each one of the materials.

Table 1.
Printing Parameters (PLA – Polylactic acid, TPU – Thermoplastic Polyurethane)

Print Setting	Extruder 1: PLA	Extruder 2: TPU
Top/Bottom Line Width	0.35 mm	0.35 mm
Layer Height	0.2 mm	0.2 mm
Wall Line Court	4	4
Top/Bottom Layers	6	6
Infill Density	100%	100%
Infill Pattern	Triangles	Triangles
Printing Temperature	200°C	210°C
Print Speed	70 mm/s	30 mm/s
Top/Bottom pattern	Concentric	Concentric

2.4. Adhesion treatments

The following treatments are intended to strengthen the inter-material bond and increase the adhesive strength of the materials. The treatments are divided into two categories: post-printing treatments and mid-printing treatments. The first category encompasses acetone immersion, annealing at 220°C, and glueing two parts with THF. The mid-printing treatments include two tests: surface activation with acetone and THF solvent.

Acetone immersion

The Acetone Immersion treatment involves immersing the printed samples completely in Acetone for two seconds (Fig. 2). After the pieces are submerged, they are left to dry for 24 hours.

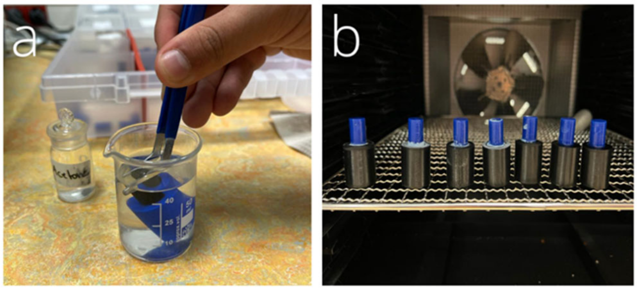


Fig. 2. a) Acetone immersion treatment, b) next step of acetone immersion treatment

Annealing 220°C

Samples were submitted to 220°C in a chamber for 2 minutes. Then, they were exposed to room temperature to cool down. The samples are heated in groups of 7, the first corresponding to TPU/PLA and the second to PLA/TPU.

Surface activation

In this process, 0.1 ml of the solvent is inserted into the upper surface of the large cylinder. The part has just been printed, and the nozzle has been changed to continue with the small cylinder. For this, it is necessary to pause the printing and wait for the building plate to lower until it has the correct elevation to insert the solvent. The process has to be resumed immediately once the plate has lowered. A pipette was used to place the solvent to have an exact measurement. Surface Activation with Acetone and THF:

- 0.1 ml of Acetone is inserted into the upper surface of the large cylinder (Fig. 3);
- 0.1 ml of THF is inserted into the upper surface of the large cylinder.

Fig. 3. Surface activation with solvent (acetone or THF)

Gluing two parts with THF

For this treatment, the small cylinder is wholly immersed in THF (Fig. 4a). The piece remains submerged for 2 seconds. After that, the piece is removed to be placed on the upper surface of the large cylinder (Fig. 4b).

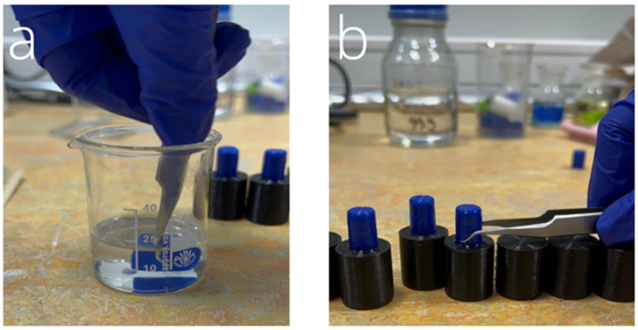
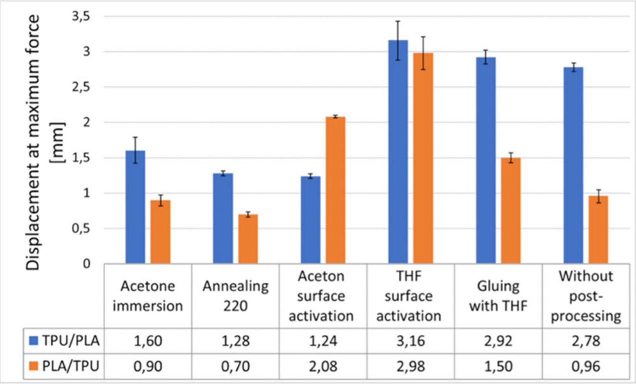


Fig. 4. a) Submerging the small cylinder in THF; b) placing the piece on the upper surface of the big cylinder

3. Shear test and roughness test results

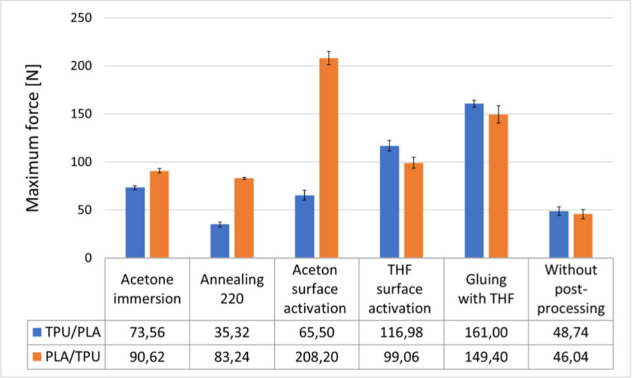
The interaction at the interface of two surfaces of two polymers (PLA-TPU and TPU-PLA) and five treatments (one series without additional treatment) were tested by printing seven replicas for each; a total of twelve combinations were obtained.

A compilation of the arithmetic means obtained from five samples (the results of the extremes were rejected) for each series of samples are shown (Fig. 5 – displacement at maximum force, Fig. 6 – maximum force, Fig. 7 – shear strength).



	Acetone immersion	Annealing 220	Aceton surface activation	THF surface activation	Gluing with THF	Without post-processing
■ TPU/PLA	1,60	1,28	1,24	3,16	2,92	2,78
■ PLA/TPU	0,90	0,70	2,08	2,98	1,50	0,96

Fig. 5. Displacement at maximum force – an overview of displacement at maximum force for all series in two materials configurations

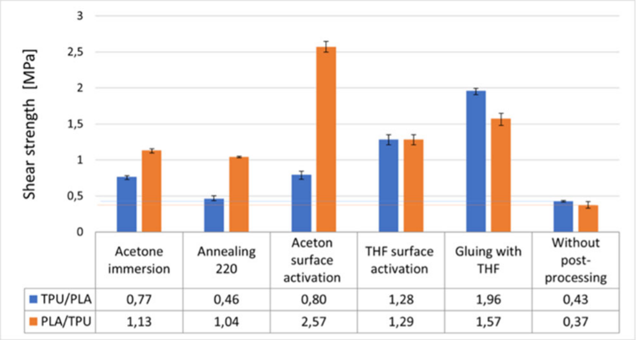


	Acetone immersion	Annealing 220	Aceton surface activation	THF surface activation	Gluing with THF	Without post-processing
■ TPU/PLA	73,56	35,32	65,50	116,98	161,00	48,74
■ PLA/TPU	90,62	83,24	208,20	99,06	149,40	46,04

Fig. 6. Maximum force – an overview of maximum force for all series in two materials configurations

Discussion

Displacement results indicate a decrease for samples subjected to annealing and after immersion in Acetone. The best results for displacement were obtained for activating the sample surface with the THF solvent. When analyzing the results for THF activation, it should be noted that for two material combinations, the results are similar (PLA/TPU, TUP/PLA).



	Acetone immersion	Annealing 220	Aceton surface activation	THF surface activation	Gluing with THF	Without post-processing
■ TPU/PLA	0,77	0,46	0,80	1,28	1,96	0,43
■ PLA/TPU	1,13	1,04	2,57	1,29	1,57	0,37

Fig. 7. Shear strength- an overview of shear strength for all series in two materials configurations (reference lines for untreated samples)

The maximum force applied to the sample (208.2 N) was obtained for PLA/TPU and surface activation with Acetone. The following is the best result for THF bonding (161 N and over 149 N for TPU/PLA and PLA/TPU).

Treatments after or during printing improved the shear strength of the samples compared to untreated ones. Considering the standard deviations and the results for the THF treatment, it should be noted that for this treatment, the result is ‘statistically insignificant, which means that it does not matter in which order the polymers are printed.

Activation of the THF surface will strengthen the bond the same for the two material sequences. Glueing two polymers after printing with THF solvent gave better results than activating the THF surface. Activating the surface, however, has its advantage in that we do not have to determine the geometry ourselves when glueing. The printer does it for us; users only have to stop the printing between layers to instill the solvent.

The best results were achieved for activating the PLA surface with Acetone (strength 2.57 MPa). The TPU polymer printed on PLA achieved better adhesion than PLA applied to the activated TPU surface. As these studies have proven, modification of the 3D printing process from many materials can improve the connection quality at the interface of two polymers.

The best proposal, according to the authors, is to activate the surface with THF – due to its universal application, no effect on the destruction/degradation of the entire sample and no effect on the geometry of the objects (when immersing in Acetone and heating, the dimensions of objects may change). The treatments used in this article are a proposal and encouragement for researchers to modify and improve 3D printing processes to achieve satisfactory parameters and use biodegradable polymers.

4. Conclusions

Modifying the 3D printing process improved the quality of the adhesive bond between the two different polymers, using post- or in-process treatment answers problems with multi-material printing and poor bonding at the interface of materials. The best solution proposed in this study is to use the THF solvent to activate the surface or to glue together elements printed separately.

Activation of the surface with THF is the treatment method recommended by the authors due to the low impact on the deformation/degradation of the sample that may occur during glueing, immersion in Acetone, or annealing.

Acknowledgements

This work was completed while the first author was the Doctoral Candidate in the Interdisciplinary Doctoral School at the Lodz University of Technology, Poland. The authors duly acknowledge Medical University of Lodz; University Laboratory of Material Research, especially Michał Krasowski, for providing infrastructural facilities, and scientific support and sharing his wisdom during the research. The authors would also like to thank Get 3D Ltd. Company for their technical support and consulting.

Additional information

The research was financed by the Foundation of the Lodz University of Technology (Fundacja Politechniki Łódzkiej) and presented during scientific conference Materials Engineering - Materials and Technologies "IMMT 2022", Łódź-Słok, 20-23 November 2022.

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