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Study of the Effect of Pretreatment of 3D Printed PLA Filament Modified by Plasma Discharge and Changes in its Dynamic-mechanical Properties

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The presented study focuses on the study of polylactide acid (PLA) material, which is a frequently used material in 3D printing. Surface modification using DCSBD plasma discharge is proposed as a way to improve the adhesion between individual layers of material. Adhesion is a critical factor for achieving high-quality print output, as low adhesion can cause individual deposited layers of material to separate and ripple during printing. Dynamic Mechanical Analysis (DMA) is used to determine the glass transition temperature (T_g) of a material. T_g is important because it determines how difficult the material is to print, the closer the T_g is to room temperature, the easier the material is to print. However, after printing a layer of material and subsequently cooling it to room temperature, the material begins to shrink and wave due to the change in material expansion. This can have a negative effect on the adhesion between the layers of the material, which can lead to separation of the layers. The presented study tries to find a way to improve the adhesion of individual layers of material. Surface modification by plasma discharge appears to be a promising method that could improve the adhesion between individual layers of PLA material.

Keywords: PLA, Polymers, Materials, 3D printing, DMA, Surface modification

1 Introduction

The Fused Filament Fabrication (FFF) is one of the most used and researched 3D printing technologies, due to its ease of use, its low cost, and its ability to process traditional thermoplastic polymers. Among the thermoplastics, polylactic acid (PLA) is one of the most used materials in FFF 3D printing, and it is possible to obtain PLA-based polymers with the required properties for a specific application. Coppola et al. studied the influence of printing temperature in the 3D printing process of PLA/clay nanocomposites. They demonstrated that printing temperature should be chosen considering not only melting temperature, but also polymer architecture and/or nanocomposite morphology in the case of nanocomposite systems. The authors stated that potential applications could be found in both with respect to improving mechanical properties if the correct temperature is used [1]. Kanazawa et al. presented a new approach for the design and fabrication of non-thermal plasma reactors used at atmospheric pressure by using 3D printing technology. They verified the feasibility of such an approach and to evaluate a novel plasma reactor prototype prepared using a 3D printer. They found that 3D printing technology has opened new doors for the design and production of a non-thermal plasma reactor prototype and such technology to offer a variable, fast and cost-effective manufacturing process for the development of plasma reactor prototypes [2]. Fodor investigated the fatigue properties of plastic materials produced by rapid prototyping using the DMA method. He carried out the planning of tests for the injection of standard technical plastic samples by injection molding and further implemented the experience gained in this way when designing tests of equal-sized parts created by rapid prototyping. Fodor designed these tests so that they could help designers in the field of military, in the development of security technology and improvement of recovery [3]. Cristea et al. in their review offers a perspective of the main phenomena that can be revealed in a DMA experiment and systematizes the information that can be obtained for each region (glassy, glass transition, rubbery, cold-crystallization and melting) using PLA material. The review intends to offer indices that one should pay attention to in the interpretation of a DMA experiment, even if the investigator has only basic skills with DMA investigations [4]. Doddamani performed Dynamic Mechanical Analysis (DMA) on a 3D printed eco-friendly fly ash cenosphere/lightweight HDPE composite. Cenospheres with different volume percentages were mixed with HDPE, filaments were extruded and fed into an FDM-based 3D printer. He found that HDPE with 60% vol. cenosphere recorded higher values, indicating the potential of an environmentally friendly 3D printed lightweight composite for use in weight-sensitive structures [5]. Kariž et al. in the study investigated and compared 3D printed fused deposition (FDM) parts made of polylactic acid (PLA), polylactic acid with wood flour additive (Wood-PLA), and acrylonitrile-butadiene-styrene (ABS) polymers bonded to wood using polyvinyl acetate (PVAc) glue. Specimen surfaces were bonded as either untreated, sandblasted, plasma treated, or sandblasted and plasma treated to evaluate the effect of each surface preparation on the bonding ability of 3D printed surfaces. The results show that suitable surface preparation is essential in achieving sufficient bond strength. Sanding significantly increased the bond strength, but the combination of sanding and plasma treatment gave the best results [6]. Spoerk et al. presented study on the effect of the printing bed temperature on the adhesion of parts printed by fused filament fabrication (FFF) by means of an in-house developed adhesion measuring device. It was observed exemplarily for PLA and ABS that the optimal adhesion of the printed sample to the printing bed can be achieved by heating the printing bed at temperatures slightly above the T_g of the filament material. Increasing the temperature above the filament's T_g leads to a reduction of the surface tension between the printing bed and the printing material and to a larger contact area that ultimately causes better adhesion between the bed and the filament [7]. Abourayana et al. evaluated the use of a barrel atmospheric plasma system for the treatment of acrylonitrile butadiene styrene and polylactic acid polymer particles. Treatments were carried out in a helium discharge with either oxygen or nitrogen addition. The plasma activated polymer particles were then used to prepare filaments, which in-turn were then used to fabricate parts by additive manufacturing. The resulting polymer parts exhibited up to a 22% increase in tensile strength, compared to parts fabricated using inactivated polymer particles [8]. Kumar et al. studied 3D printing of PLA hybrid composite matrix (having magnetic characteristics) to investigate the flexural and pull-out properties. The photo micro-graphic analysis and Shore D hardness has been performed on the printed samples and multifactor optimization tool has been used for optimizing the printing conditions [9]. Chiulan et al. in their review paper provided an overview of the main 3D-printing technologies currently employed in the case of poly (lactic acid) (PLA) and polyhydroxyalkanoates (PHA), two of the most important classes of thermoplastic aliphatic polyesters. Moreover, a short presentation of the main 3D-printing methods is briefly discussed. The processability of PLA and PHB blends and composites fabricated through different 3D-printing techniques, their final characteristics and targeted applications were thoroughly reviewed [10]. Galeja et al. in their research work aims to evaluate different raster angles (45°, 55°, 55’°, 60° and 90°) on the static, as well as rarely investigated, dynamic mechanical properties of 3D printed acrylonitrile butadiene styrene (ABS) materials. Configuration named 55° was based on the optimal winding angle in filament-wound pipes, which provides them exceptional mechanical performance and durability. Also, in the case of 3D printed samples, it resulted in the best impact strength, comparing to other raster angles, despite relatively weaker tensile performance. All 3D printed samples showed high values of impact strength considering their calculated brittleness, which provides new insights into understanding the mechanical performance of 3D printed structures [11]. Beucher et al. in their study treated the PLA and PHB biopolymers with variable deposition geometry in a plasma-enhanced chemical vapor deposition (RF-PECVD) process with O_2 plasma. The sample surfaces were analyzed for topography with AFM and for their chemical composition by synchrotron supported XPS. Likewise, the barrier property was determined by WVTR and the SFE and CAH by contact angle analysis [12]. The cited articles prove that despite extensive research work related to 3D printing and plasma surface treatment technology, much research is still needed to properly understand these processes.

2 Materials and methods

In the presented article, samples of biodegradable PLA material were measured and compared. The samples were printed using a 3D printer using FFF technology. A total of 9 samples were printed in dimensions: 65 mm x 12.8 mm x 3.2 mm. Plates printed in this way were measured 3 in an unmodified state (reference sample - PLA), 3 in a modified state (plasma-chemically treated by DCSBD plasma discharge; PLA + DCSBD) and 3 plates printed from a filament that was pre-treated by DCSBD plasma discharge (filament PLA + DCSBD) (Fig. 3). The printed PLA + DCSBD test sample (Fig. 1) and PLA filament + DCSBD (Fig. 2) were placed above the ceramic dielectric at a distance of 0.26 mm.

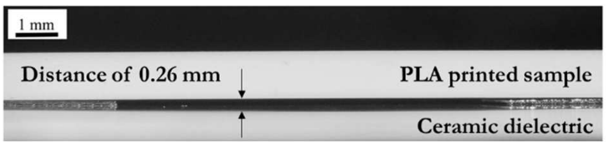

Fig. 1 *Test sample from PLA printed by a 3D printer and its placement over a ceramic dielectric (PLA + DCSBD)*

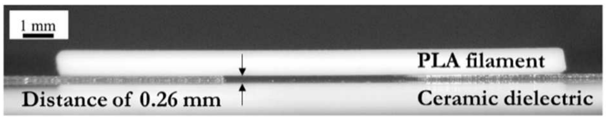

Fig. 2 *PLA filament and its placement above the ceramic dielectric (PLA filament + DCSBD)*

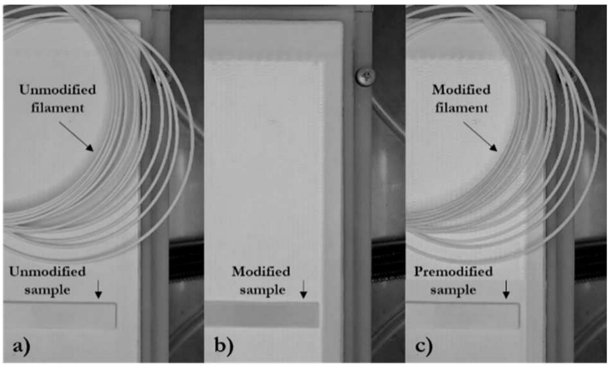

Fig. 3 *Test samples: (a) PLA in unmodified state (PLA); (b) PLA sample plasma-chemically modified by DCSBD plasma discharge (PLA + DCSBD); (c) PLA filament pre-treated by DCSBD plasma discharge (PLA filament + DCSBD)*

The power of the reactor of the line for the surface modification of polymeric materials (KPR 200 mm, VÚCHV a.s., Svit) was set to 350 W. At this power, a homogeneous layer of DCSBD plasma is generated. The plasma exposure time for the filament was 5 s and for the printed test bodies 30 s for each side. Thus, a double system of operation of the KPR 200mm plasma reactor was used: a system with dynamic unwinding/winding of the material (PLA filament) and a static positioning system when the test bodies were stably positioned on the ceramic dielectric. In both cases, the plasma was generated on a planar electrode placed across the line frame.

Thermograms were recorded during the plasma modification process with a TESTO 868 thermal camera. The SuperResolution function was activated, and the images were processed in the computer application IRSoft 4.7. They were recorded from the operator's position, above the ceramic dielectric. The emissivity was set using the Testo ε-Marker. With the Testo ε-Marker it is possible to determine the emissivity (ε) and reflected temperature (RTC) of the measurement object automatically. For better temperature identification, temperature profiles were made – horizontal and vertical (or rectangular histogram shape).

DMA analysis was performed using a DMA Q800 from TA Instruments on printed samples with the above sample dimensions using the Dual Cantilever geometry, in the temperature range 40 °C – 100 °C, at a heating rate of 3 °C, at frequencies of 10 Hz, amplitude of 15 μm, under atmospheric pressure. The measured results were processed using TA Universal analysis V4.5A software. During DMA analysis, the glass transition temperature (T_g) was measured and determined for Storage modulus, Loss Modulus and tan δ.

3 Results and discussion

3.1 DMA analysis

During the dynamic-mechanical analysis, DMA records of storage modulus (E'), loss modulus (E”) and loss factor tan δ were evaluated, while the glass transition temperature (T_g) was measured for individual PLA samples. To determine T_g for E', it was necessary to find out the extrapolated "OnSet" value of each curve. When determining T_g for E” and tan δ, it was based on the maximum value of the peaks of the individual curves. Measured and average T_g temperatures and tan δ values for standard PLA material (without plasma modification). PLA test samples modified with plasma (PLA + DCSBD), and for PLA filament modified with plasma (PLA filament + DCSBD) are shown in Tab. 1.

Tab. 1 *Measured and averaged glass transition temperatures and tan δ values for standard PLA material (without plasma), plasma modified PLA test bodies (PLA + DCSBD), and for PLA filament modified by plasma (PLA filament + DCSBD)*

Sample name	Storage Modulus [°C]	Loss Modulus [°C]	tan δ [°C]	tan δ value
PLA (1)	59.60	64.25	70.66	2.041
PLA (2)	60.29	64.59	71.21	1.992
PLA (3)	59.07	63.51	69.72	1.989
PLA average	59.65	64.11	70.53	2.007
PLA + DCSBD (1)	56.02	61.81	69.43	1.990
PLA + DCSBD (2)	57.44	63.32	71.19	2.062
PLA + DCSBD (3)	56.85	62.40	69.76	2.042
PLA + DCSBD average	56.77	62.51	70.13	2.031
PLA filament + DCSBD (1)	57.63	62.63	69.42	1.687
PLA filament + DCSBD (2)	57.31	62.32	69.10	1.625
PLA filament + DCSBD (3)	57.41	62.06	68.68	1.604
PLA filament + DCSBD average	57.45	62.34	69.07	1.639

Based on the comparison of the average T_g values of E', it can be stated that the highest T_g temperature was measured for the standard sample (PLA = 59.65 °C). In plasma-treated samples, compared to the standard PLA sample, the T_g decreased, while the average value for PLA + DCSBD samples was lower by 2.88 °C (PLA + DCSBD = 59.65 °C) and for PLA filament + DCSBD sample was lower by 2.2 °C (PLA filament + DCSBD = 57.45 °C). Since the T_g for the storage modulus is related to the mechanical failure of the material, from the obtained results it can be concluded that mechanical failure with increasing temperature and cyclic stress will occur earlier in the plasma treated samples than in the standard printed sample. In contrast, as can be seen in Fig. 4 compared to the standard sample, the plasma-treated samples showed a higher value of the elastic modulus in units of MPa in the area below T_g, which can lead to tighter deposition of the individual layers of the material during its printing and better adhesion between the individual layers.

The loss modulus E" is related to the transferred energy converted into heat during one loading cycle of the measured PLA sample. By comparing the average T_g values of the E" (Table 1 and Fig. 5), it can be concluded that the highest T_g temperature was measured again with the standard sample (PLA = 64.11 °C). In plasma-treated samples, compared to the standard PLA sample, the T_g decreased, while the average value for PLA + DCSBD samples was lower by 1.6 °C (PLA + DCSBD = 62.51 °C) and for PLA filament + DCSBD samples was lower by 1.77 °C (PLA filament + DCSBD = 62.34 °C).

Based on the comparison of the average T_g values of tan δ, it can be concluded that the highest T_g temperature was measured for the standard sample (PLA = 70.53 °C). In the case of plasma-treated samples, there was a slight decrease in the T_g temperature compared to the standard PLA sample, while the average value for PLA + DCSBD samples was lower by 0.4 °C (PLA + DCSBD = 70.13 °C) and for PLA filament + DCSBD sample was lower by 1.46 °C (PLA filament + DCSBD = 69.07 °C).

The tan δ value determines the ability of the PLA material to absorb and dissipate energy. The value of tan δ thus indicates the damping ability of the material, from which it follows that the higher the stated value of tan δ, the more effective the material's damping ability is. Based on the values of tan δ (Table 1) and the curves obtained by DMA analysis (Fig. 6), it can be stated that, on average, there was a slight increase in the value of the plasma-treated sample PLA + DCSBD, on the contrary, there was a more significant decrease in the value of the pre-treated sample PLA filament + DCSBD. The results of the DMA analysis after plasma chemical treatment of the polymer surface agree and correspond with previously published results [13-21].

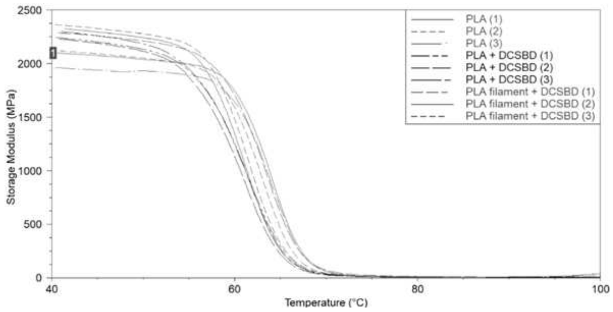

Fig. 4 *Storage modulus curves of individual samples measured by DMA analysis*

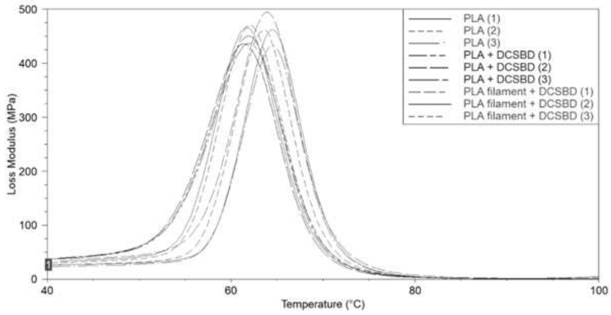

Fig. 5 *Loss modulus curves of individual samples measured by DMA analysis*

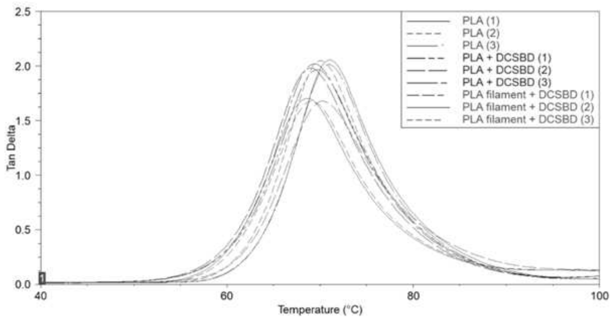

Fig. 6 *Tan δ curves of individual samples measured by DMA analysis*

3.2 Infrared thermography applied to the process of plasma-chemical modification – PLA + DCSBD

The average temperature calculated from the surface of the ceramic dielectric (obtained from the rectangle-shaped histogram) at the power of the experiment (350 W) and without the stored PLA test samples was ~ 53 °C. The temperature interval observed during the PLA plasma-chemical modification process was obtained from the vertical and horizontal profiles (marked as P1, P2, P3 in Fig. 7a and Fig. 7b. By using vertical profiles (Fig. 8a, it was possible to determine the temperatures of the ceramic dielectric in the vicinity of the samples and the surface temperatures of the PLA samples. In profile P1 in Fig. 8a, high temperature peaks can be observed – the temperature of the ceramic dielectric between the samples. From the vertical temperature profiles P1 – P3, a temperature interval was recorded for:

- ceramic dielectric: 63 – 63.8 °C.
- surface of PLA test samples: 32.5 – 34.3 °C.

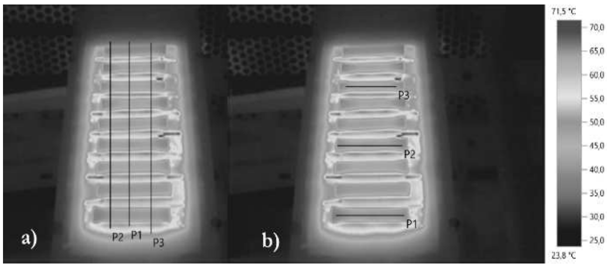

Fig. 7 *Thermograms of PLA test samples placed over a ceramic dielectric during plasma modification; (a) vertical profiles, (b) horizontal profiles*

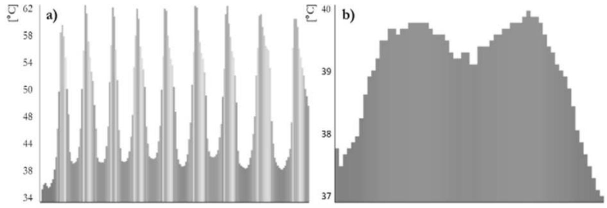

Fig. 8 *(a) Display of the vertical temperature profile (P1) and (b) display of the horizontal temperature profile (P2) during modification of PLA test bodies with DCSBD plasma. The temperature profiles shown were selected due to their central location on the ceramic dielectric*

Using the horizontal profiles P1 – P3, Fig. 8 (b), more accurate (average) temperatures on the surfaces of the PLA test samples were obtained = 38.6 °C – 40.3 °C. The temperature of the ceramic dielectric is therefore close to the monitored glass transition temperature of PLA. It can be assumed, that PLA surfaces only 0.26 mm above the ceramic dielectric reached a similar temperature.

3.3 Infrared thermography applied to the process of plasma-chemical modification – filament PLA + DCSBD

Surface temperature, which the PLA filament reaches during DCSBD plasma modification could be determined from the temperature profiles of P1, P2, P3 (vertically intersecting PLA filament – Fig. 9). After being exposed to heat and plasma, the PLA filament changed its shape: from the original straight shape, it became bent.

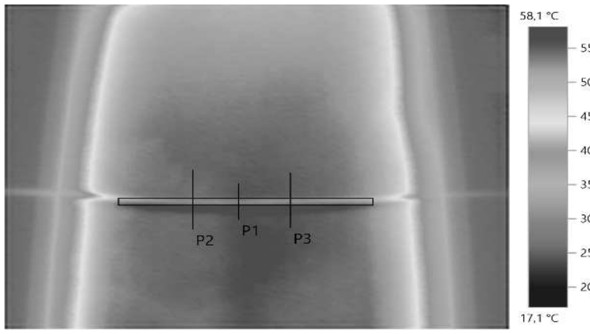

Fig. 9 *Vertically intersecting PLA filament*

The temperature of the PLA filament on the active ceramic dielectric generating the DCSBD plasma can be determined from the temperature profiles as the lowest temperature (the highest temperature is reached by the ceramic dielectric in its close vicinity from the left and right – Fig. 10a). The temperatures of the PLA filament were thus recorded (P1 – P3) in the interval 51.8 °C – 52.4 °C. This is also confirmed by the temperature histogram in the shape of a rectangle in a tight fit around the PLA filament, when the most numerous percentage representation of temperatures is approximately exactly in this interval (Fig. 10b).

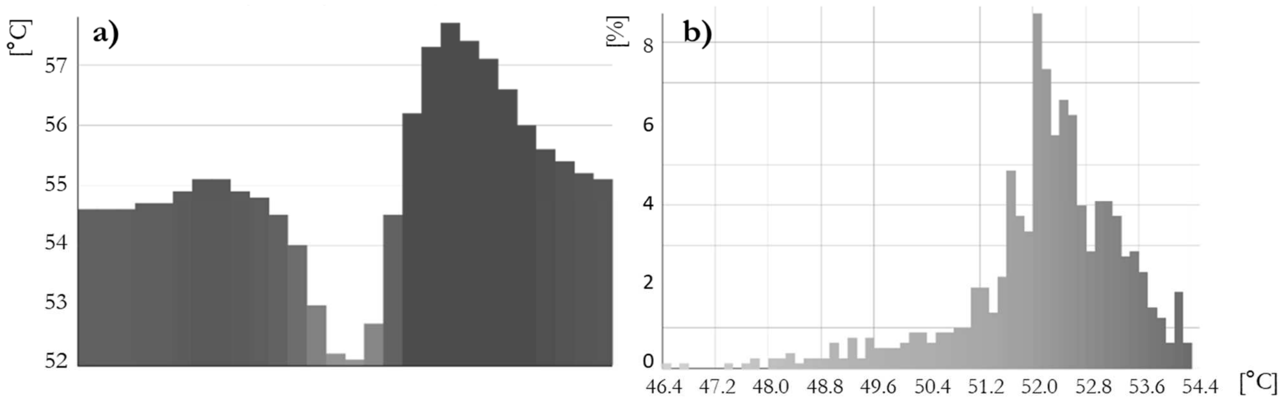

Fig. 10 *(a) Vertical temperature profile P1 across PLA filament during plasma-chemical modification, (b) Histogram of the temperature of the filament and its immediate surroundings during the plasma-chemical modification*

4 Conclusions

The presented work compares PLA test samples printed by a 3D printer: a) samples that were plasma modified after 3D printing; b) samples whose filament was plasma modified before 3D printing. When comparing the properties of such products with respect to PLA products without any of the mentioned plasma modifications, differences were noted. These were evident when applying the thermal DMA method, while decrease in glass transition temperature were observed. Thus, DCSBD plasma chemical modification can be helpful in the extrusion, storage, and mutual adhesion of individual print layers - PLA layers can be joined more firmly even at a lower temperature. This is also evidenced by the higher values of the storage modulus when the plasma-modified test samples showed higher MPa values. The plasma-modified test samples exhibited higher elasticity – the ability to resist irreversible, plastic damage under cyclic loading. The above-mentioned changes are evaluated in the context of observation and recording of plasma chemical modification using a thermal camera - namely constructed temperature profiles and histograms. The obtained results can be partly influenced by the atmospheric conditions during 3D printing and the inhomogeneity of the surface modification by plasma - especially in the case of PLA filament (deviations in maintaining the distance from the ceramic dielectric and changes in the winding speed of the modified filament).

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