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Dynamic Mechanical Analysis of PLA Produced by FFF Additive Manufacturing Technology after DCSBD Plasma Treatment

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Dynamic mechanical analysis (DMA) is an important method for evaluating the viscoelastic properties of polymeric materials, especially when investigating their mechanical response to various manufacturing parameters and surface treatments. In recent years, DMA analysis has been intensively used, among others, for the analysis of polylactide (PLA) produced by the fused filament fabrication (FFF) additive technology. The present study focuses on the effect of DCSBD plasma treatment on the dynamic-mechanical properties of PLA samples with different infill geometries (Line, Rectilinear and Concentric). In the study, experimental PLA samples were subjected to DMA analysis in the temperature range of 40 °C to 90 °C in order to analyze the changes in their viscoelastic properties after plasma discharge surface treatment. The results showed a decrease in the glass transition temperature (T_g) for all tested samples, while the extent of the decrease depended on the infill geometry used. The most significant changes were observed in samples with Rectilinear infill, which showed the best mechanical stability after plasma treatment. The study shows that plasma treatment can influence the mechanical properties of PLA products, opening new possibilities for optimizing their processing, reuse and application in technical areas requiring controlled mechanical response.

Keywords: Polylactic acid (PLA), Dynamic mechanical analysis (DMA), DCSBD surface treatment, Additive manufacturing, Fused filament fabrication (FFF), Glass transition temperature (T_g)

1 Introduction

Dynamic mechanical analysis (DMA) is one of the key experimental methods used to evaluate the viscoelastic behavior of polymeric materials under various temperature and mechanical conditions. In recent years, this technique has been widely used to study the mechanical properties of polylactide (PLA) produced by additive technologies, especially by the fused filament fabrication (FFF) method [1]. Its importance lies in the ability to quantify the influence of various manufacturing parameters and surface treatments on the mechanical and dynamic-mechanical behavior of PLA [2,3].

One of the basic factors affecting the mechanical properties of PLA is its surface modification. Kohutiar et al. investigated the effect of plasma discharge on the dynamic-mechanical properties of PLA and demonstrated that this modification changes the mechanical behavior of the material, in particular the glass transition temperature, storage and loss modulus and *tan δ* [4]. Similar conclusions were also reached by Baran and Erbil, who analyzed the surface modification of 3D printed PLA samples using different technological processes and emphasized their importance for improving adhesive and mechanical properties [5]. Tümer and Erbil, in their study, focused on various applications of PLA composites produced by extrusion 3D printing and showed that process parameters and additives can fundamentally affect the mechanical response of PLA [6]. Cristea et al. dealt with the dynamic mechanical analysis of renewable PLA materials and emphasized the importance of DMA analysis in determining critical temperature transitions and long-term mechanical stability of PLA products [7]. Similar research was also carried out by Struz et al., who compared different testing methodologies for PLA samples produced by the FDM method and pointed out the correlation between the internal geometry of the filler and mechanical properties [8]. Plamadiala et al. also focused on the analysis of the parameters of internal fillers, concluding that the right choice of filler structure can significantly affect the mechanical response of PLA products [9].

The mechanical and dynamic-mechanical properties of PLA can also be influenced by the addition of fillers, as demonstrated by the study by Pavona et al., where the effect of $CaCO_3$ fillers on the strength and thermal stability of PLA samples produced by the FDM method was investigated [10]. Liao et al. analyzed the effect of porosity and crystallinity on the mechanical properties of 3D printed PLA and showed that structural characteristics can significantly affect the final mechanical properties of the material [11]. Hedayati et al. focused on the degradation of PLA over time and its impact on the mechanical response, emphasizing that changes in the structure can lead to a gradual decrease in the strength and stability of the material [12].

The presented study describes the analysis of PLA samples produced by the additive technology fused filament fabrication (FFF) with different geometries of their infill. The study compares PLA samples made from standard filament with PLA samples made from filament surface-modified by DCSBD plasma discharge. PLA samples were surface-modified by plasma discharge in order to accelerate the degradation of PLA material in case it is used for the production of test models.

2 Materials and methods

In the experiment, PLA samples produced by the fused filament fabrication (FFF) additive technology with different geometries of their internal filling (infill) and an infill density of 70% were analyzed. Using DMA analysis, PLA samples made from standard filament were measured and compared with PLA samples made from filament surface-modified by DCSBD plasma discharge. Three infill geometries were selected for the PLA samples, namely Line (PLA 1), Rectilinear (PLA 2) and Concentric (PLA 3), which are shown in Fig. 1. The plasma-modified samples were given the corresponding designation PLA 1-3P, according to the geometry of the infill. The modification of the PLA filament was carried out at atmospheric pressure and at laboratory temperature, with a plasma reactor power of 350 W. The filament passed three times through a set of support rollers, with which it was possible to unwind it onto two concave plasma dielectrics to ensure modification of its entire surface [13,14].

A Bambu Lab X1 Carbon 3D printer was used to produce test samples. PLA filament (Bambu Lab PLA Basic) with a diameter of 1.75 mm was used as the material. The PLA filament passed through a nozzle with a diameter of 0.4 mm, which was heated to 220 °C and then deposited on a printing substrate in the form of layers with a thickness of 0.2 mm. The dimensions of the samples were chosen according to the Dual Cantiliver geometry used in DMA analysis of plastic samples (Fig. 2). For this geometry, it is necessary to use sample dimensions of 60 mm x 12.8 mm x 3.2 mm. The printed samples were then conditioned at room temperature for 8 hours before measurement.

The measurements were performed using a dynamic-mechanical analyzer DMA Q800 from TA Instruments, which is designed to analyze the viscoelastic properties of materials. The TA Universal Analysis software ver. 4.5A was used to process the measured data, and the output of the DMA analysis is the curves of the storage modulus (E'), loss modulus (E'') and loss angle (*tan δ*) as a function of temperature, on the basis of which the transition temperatures of individual materials were determined and compared according to the ASTM D4065 standard [15].

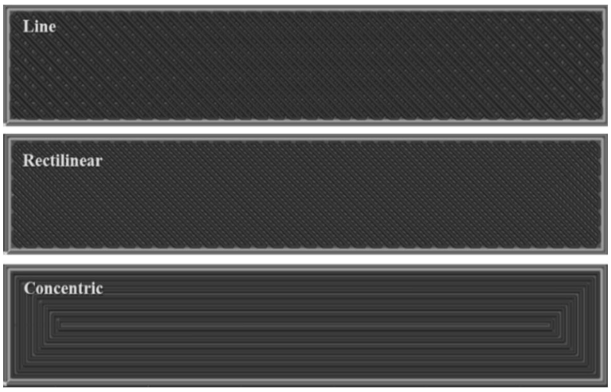

Fig. 1 *Schematic representation of three types of infill geometry*

DMA analysis of PLA 1-3 and PLA 1-3P samples was carried out in the temperature range from 40 °C to 90 °C, with a heating rate of 3 $°C.min^{-1}$, at a frequency of 10 Hz and an amplitude of 15 μm. The result of the experiment is a comparison of the measured data of the samples in the basic form and the surface-modified form within the same and different infill of the PLA samples. The output is the curves E', E'' and *tan δ* and the measured values of the temperatures T_g, which are subsequently compared within the individual groups of PLA samples.

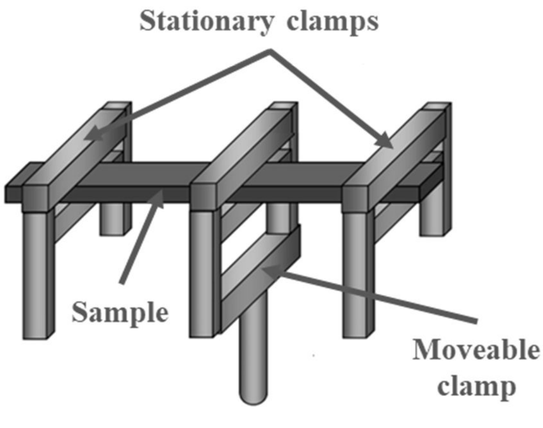

Fig. 2 *Dual Cantiliver geometry used in DMA analysis*

3 Results

By comparing the storage modulus curves of samples PLA 1 and PLA 1P (Fig. 3) in the temperature range from 40 °C to 90 °C, it can be observed that the

E' values of the PLA 1P sample are slightly higher compared to the PLA 1 sample, gradually decreasing after ≈ 56 °C, when there is a sharp decrease to a temperature of ≈ 70 °C. Above this temperature, the material is in a rubbery state. The E' values of the PLA 1 sample gradually decrease after ≈ 59.5 °C, when, as in the modified sample, there is a sharp decrease to a temperature of ≈ 70 °C. By comparing the glass transition temperature (T_g) from the E' curves of both samples, it can be stated that the T_g temperature of the PLA 1 sample (59.60 °C) is higher compared to the PLA 1P sample (56.02 °C) (Fig. 6). Based on a comparison of the T_g temperatures of both samples, it can be concluded that the mechanical failure of the PLA 1P sample occurs at a lower temperature compared to the PLA 1 sample, while it retains its performance properties after a temperature of ≈ 56 °C.

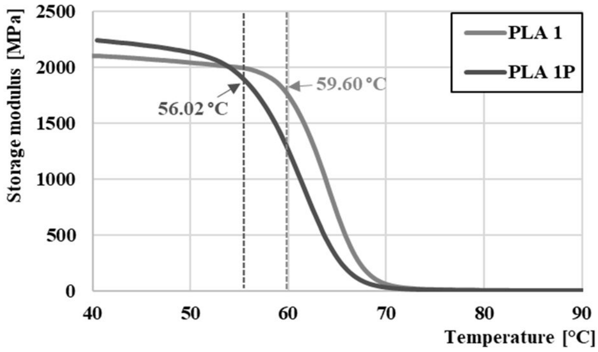

Fig. 3 *Storage modulus curves of PLA 1 and PLA 1P samples*

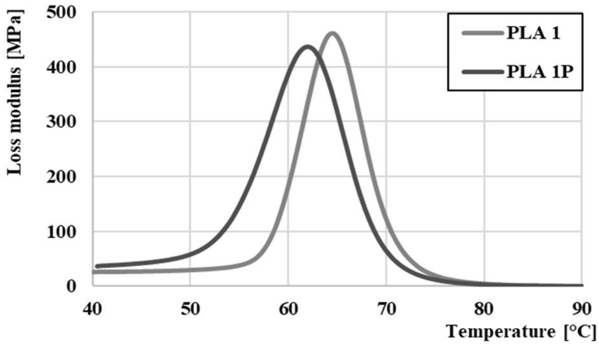

Fig. 4 *Loss modulus curves of PLA 1 and PLA 1P samples*

By comparing the loss modulus curves of samples PLA 1 and PLA 1P (Fig. 4) in the temperature range from 40 °C to 90 °C, it can be stated that the PLA 1P sample shows slightly different E'' values compared to the PLA 1 sample. In the case of sample PLA 1, a sharp increase in E'' values occurs at a temperature of ≈ 57 °C, while in the case of PLA 1P this increase is not so sharp and begins at a temperature of ≈ 49 °C. The glass transition itself based on E'' has a slower course in the PLA 1P sample compared to the PLA 1 sample. Based on the measurement of T_g from the maximum peak values of both E'' curves, it can be stated that the T_g temperature of the PLA 1P sample (61.81 °C) is lower compared to the PLA 1 sample (64.25 °C) (Fig. 6).

By comparing the *tan δ* curves of PLA 1 and PLA 1P samples (Fig. 5) in the temperature range from 40 °C to 90 °C, it can be stated that the PLA 1P sample achieves almost the same *tan δ* value compared to PLA 1 sample, so the damping ability will be approximately the same for both samples. By comparing the T_g temperature of both *tan δ* curves, it can be stated that the T_g temperature of PLA 1P sample (69.43 °C) is slightly lower compared to PLA 1 sample (70.66 °C) (Fig. 6).

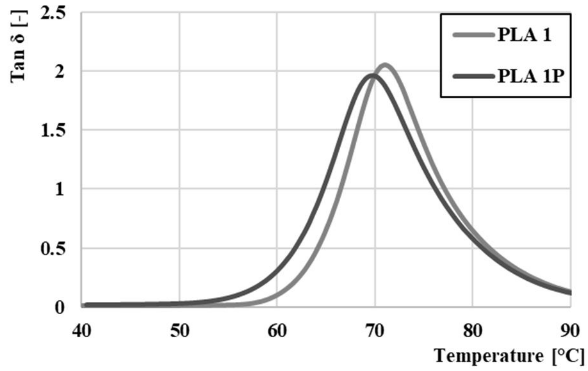

Fig. 5 *Tan δ curves of PLA 1 and PLA 1P samples*

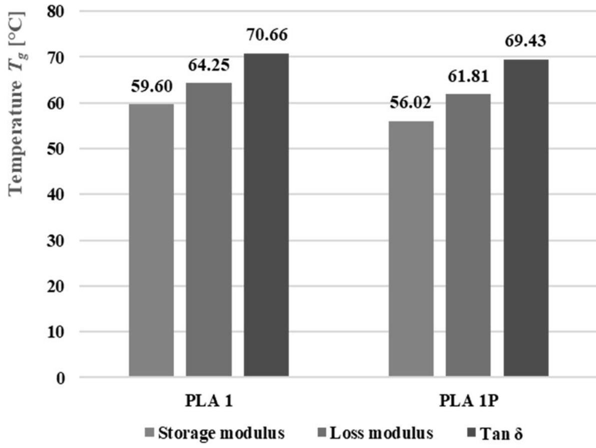

Fig. 6 *Measured T_g values of PLA 1 and PLA 1P samples*

By analyzing the storage modulus curves of the PLA 2 and PLA 2P samples (Fig. 7) in the temperature range from 40 °C to 90 °C, it can be observed that the E' values of the PLA 2P sample are higher compared to the PLA 2 sample, gradually decreasing after ≈ 57 °C, when there is a sharp decrease to a temperature of ≈ 70 °C. The E' values of the PLA 2 sample gradually decrease after ≈ 60 °C, when, as with the PLA 2P sample, there is a sharp decrease to a temperature of ≈ 70 °C. By comparing the T_g temperature from the E' curves of both samples, it can be stated that the T_g temperature of the PLA 2 sample (60.29 °C) is higher compared to the PLA 2P sample (57.31 °C) (Fig. 10). Based on a comparison of the T_g temperatures of both samples, it can be concluded that the mechanical failure of the PLA 2P sample occurs at a lower temperature compared to the PLA 2 sample, while it retains its performance properties up to a temperature of ≈ 57 °C.

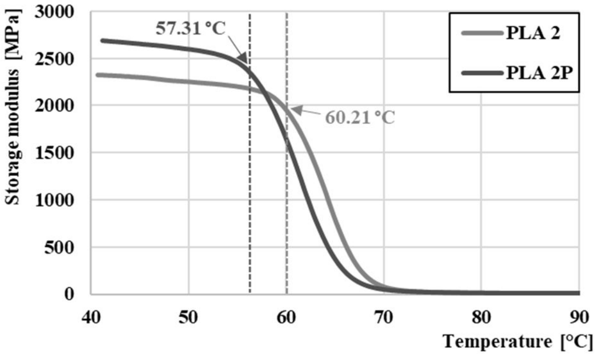

Fig. 7 *Storage modulus curves of PLA 2 and PLA 2P samples*

By measuring and comparing the loss modulus curves of the PLA 2 and PLA 2P samples (Fig. 8) in the temperature range from 40 °C to 90 °C, it can be stated that the PLA 2P sample shows slightly different E'' values compared to the PLA 2 sample. In the case of the PLA 2 sample, a sharp increase in E'' values occurs at a temperature of ≈ 56 °C, while in the case of PLA 2P this increase begins at a temperature of ≈ 53 °C. The glass transition itself based on E'' has almost the same course in the PLA 2P sample compared to the PLA 2 sample. Based on the measurement of T_g from the maximum peak values of both E'' curves, it can be stated that the T_g temperature of the PLA 2P sample (62.32 °C) is lower compared to the PLA 2 sample (64.25 °C) (Fig. 10).

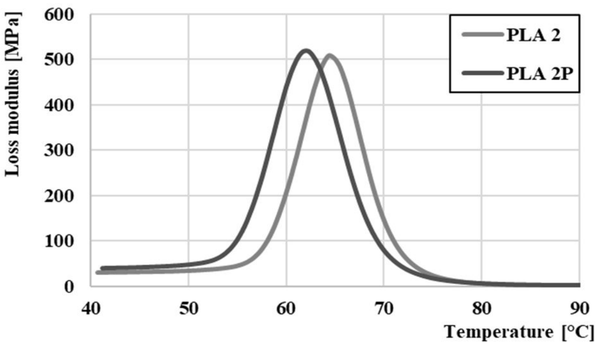

Fig. 8 *Loss modulus curves of PLA 2 and PLA 2P samples*

By comparing the *tan δ* curves of PLA 2 and PLA 2P samples (Fig. 9) in the temperature range from 40 °C to 90 °C, it can be stated that the PLA 2P sample achieves a lower *tan δ* value compared to the PLA 2 sample, so the damping ability will be lower for the modified sample. By comparing the T_g temperature of both *tan δ* curves, it can be stated that the T_g temperature of the PLA 2P sample (69.10 °C) is lower compared to the PLA 2 sample (71.21 °C) (Fig. 10).

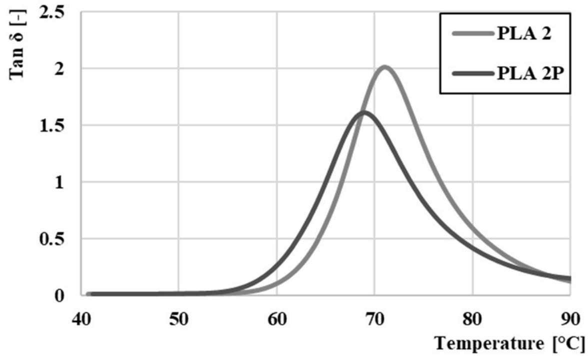

Fig. 9 *Tan δ curves of PLA 2 and PLA 2P samples*

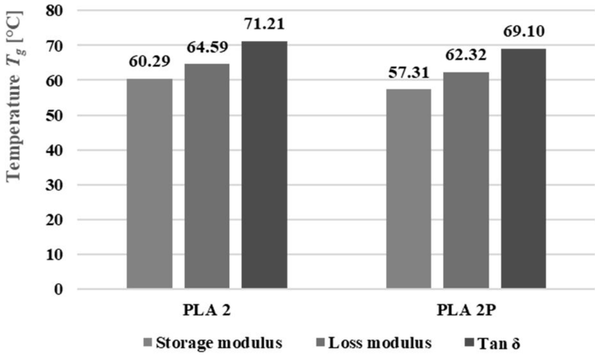

Fig. 10 *Measured T_g values of PLA 2 and PLA 2P samples*

By analyzing the storage modulus curves of the PLA 3 and PLA 3P samples (Fig. 11) in the temperature range from 40 °C to 90 °C, it can be observed that the E' values of the PLA 3P sample are higher compared to the PLA 3 sample, gradually decreasing after ≈ 55 °C, when there is a sharp decrease to a temperature of ≈ 70 °C. The E' values of the PLA 3 sample gradually decrease after ≈ 58 °C, when, as with the PLA 3P sample, there is a sharp decrease to a temperature of ≈ 70 °C. By comparing the T_g temperature from the E' curves of both samples, it can be stated that the T_g temperature of the PLA 3 sample (59.07 °C) is higher compared to the PLA 3P sample (57.41 °C) (Fig. 14). Based on a comparison of the T_g temperatures of both samples, it can be concluded that the mechanical failure of the PLA 3P sample occurs at a lower temperature compared to the PLA 3 sample, while it retains its performance properties up to a temperature of ≈ 57 °C.

By comparing the loss modulus curves of PLA 3 and PLA 3P samples (Fig. 12) in the temperature range from 40 °C to 90 °C, it can be stated that the PLA 3P sample shows almost the same E'' values compared to the PLA 3 sample. In the case of the PLA 3 sample, a sharp increase in E'' values occurs at a temperature of ≈ 56 °C, in the case of PLA 3P this increase begins at a temperature of ≈ 54 °C. The glass transition itself based on E'' has almost the same course in the PLA 3P sample compared to the PLA 3 sample. Based on the measurement of T_g from the maximum peak values of both E'' curves, it can be stated that the T_g temperature of the PLA 3P sample (62.06 °C) is lower compared to the PLA 3 sample (63.51 °C) (Fig. 14).

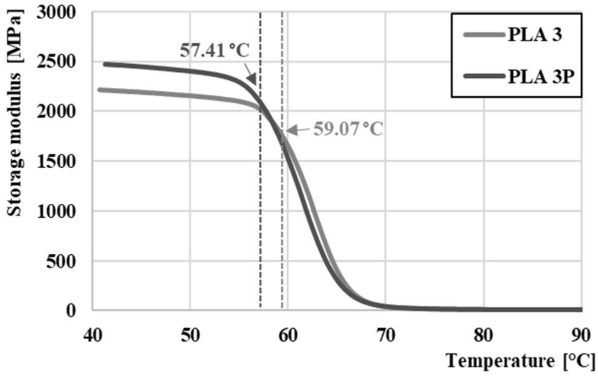

Fig. 11 *Storage modulus curves of PLA 3 and PLA 3P samples*

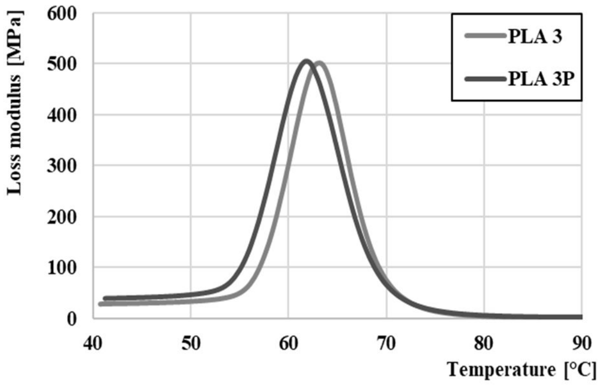

Fig. 12 *Loss modulus curves of PLA 3 and PLA 3P samples*

By comparing the *tan δ* curves of the PLA 3 and PLA 3P samples (Fig. 13) in the temperature range from 40 °C to 90 °C, it can be stated that the PLA 3P sample achieves a lower *tan δ* value compared to the PLA 3 sample, meaning that the damping ability of the modified sample will be lower. By comparing the T_g temperature of both *tan δ* curves, it can be stated that the T_g temperature of the PLA 3P sample (68.68 °C) is slightly lower compared to the PLA 3 sample (69.72 °C) (Fig. 14).

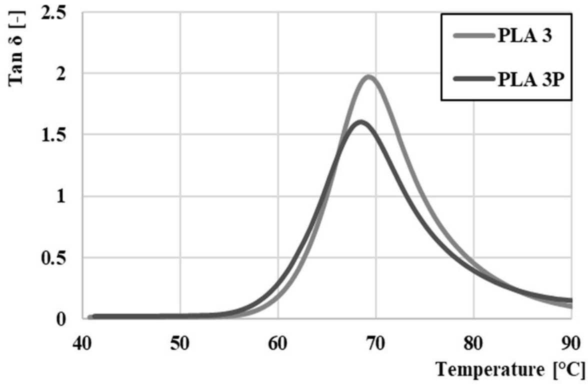

Fig. 13 *Tan δ curves of PLA 3 and PLA 3P samples*

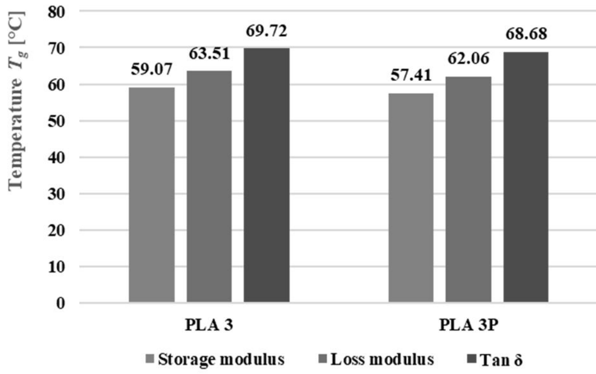

Fig. 14 *Measured T_g values of PLA 3 and PLA 3P samples*

By DMA analysis of PLA samples, comparison of E', E'' and *tan δ* curves and comparison of measured T_g temperatures of individual groups of samples, it can be stated that modification of DCSBD by plasma discharge reduced the T_g temperature of all three types of PLA samples. It can be assumed that the modified samples will retain their performance properties under normal conditions of use with a slight reduction in the use interval at elevated temperatures.

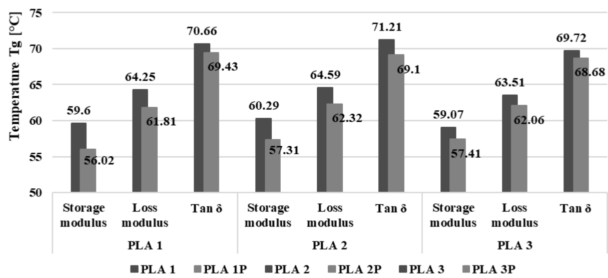

Fig. 15 *Comparison of measured T_g values from E', E'' and tan δ curves of PLA 1-3 and PLA 1-3P samples*

Fig. 15 shows the measured T_g values from the E', E'' and *tan δ* curves for all groups of samples. Based on the measured T_g temperatures, it can be stated that in addition to plasma modification, differences were also recorded within the geometry of the internal filler used while maintaining the same filler density. The most suitable internal filler used in the PLA 2 (Rectilinear) sample appears to be PLA 1 (Line) and the last one is PLA 3 (Concentric). When evaluating the internal filler, it is necessary to consider the orientation of the print and the direction of the load application with respect to the arrangement of the individual layers in the sample volume, while in this experiment the load always acted perpendicularly to the material layers [16, 17].

4 Conclusion

The primary objective of this research was to treat the surface of PLA with DCSBD plasma discharge in order to accelerate its degradation by disrupting the integrity of the surface structure and at the same time verify whether the treated samples retain their mechanical properties. The results of this study are particularly relevant in the field of mechanical engineering, where the surface treatment of PLA material with plasma discharge allows for faster degradation, thus facilitating recycling and reuse in 3D printing. This fact is especially advantageous for the recycling of test models and temporary structural elements.

Based on the DMA analysis of PLA samples produced by additive technology, by comparing the resulting curves E', E'' and *tan* δ and by comparing the measured T_g temperatures of individual groups of samples, it can be stated that the modification of DCSBD by plasma discharge reduced the T_g temperature for all three infill types of PLA samples. The T_g value for E' decreased by an average of 2.74 °C within the three types of infills due to the effect of plasma discharge. The T_g value for E'' decreased by an average of 2.05 °C within the three infill types due to the effect of plasma discharge. The T_g value for *tan* δ decreased by an average of 1.46 °C within the three infill types due to the effect of plasma discharge. It can be assumed that the plasma-modified samples will retain their performance properties under normal conditions of use with a slight shortening of the use interval at elevated temperatures.

Based on the measured T_g temperatures, it can be stated that in addition to DCSBD plasma modification, differences in T_g were also recorded within the geometry of the internal filler used while maintaining the same filler density. The most suitable internal filler used in the PLA 2 (Rectilinear) sample appears to be PLA 1 (Line) and the last one is PLA 3 (Concentric).

When evaluating the infill, it is necessary to consider the orientation of the print as well as the direction of the load in relation to the arrangement of the individual layers in the volume of the samples, because these factors are key to the resulting values of the material's mechanical properties. The correct orientation of the layers can significantly affect the strength, stiffness and resistance of the samples to external loads, while inappropriate arrangement can lead to premature failure of the structure.

Acknowledgement

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References

[1] PERNICA, J., SUSTR, M., DOSTAL, P., BRABEC, M. & DOBROCKY, D. (2021). Tensile Testing of 3D Printed Materials Made by Different Temperature. In: *Manufacturing Technology*, Vol. 21, pp. 398-404.

[2] ARUNPRASATH, K., VIJAYAKUMAR, M., RAMARAO, M., ARUL, T. G., PAULDOSS, S. P., SELWIN, M., RADHAKRISHNAN, B. & MANIKANDAN, V. (2022). Dynamic Mechanical Analysis Performance of Pure 3D Printed Polylactic Acid (PLA) and Acrylonitrile Butadiene Styrene (ABS). In: *Materials Today: Proceedings*, Vol. 50, Part 5, pp. 1559-1562. https://doi.org/10.1016/j.matpr.2021.09.113.

[3] JIN, F., LU, W., AN, X., ZHU, H. & WANG, J. (2024). Mechanical Properties and Compression Performance of 3D Printed HIPS Polymer Lattice Structure. In: *Manufacturing Technology*, Vol. 24, pp. 378-392.

[4] KOHUTIAR, M., JANÍK, R., KRBATA, M., BARTOSOVA, L., JUS, M. & TIMÁROVÁ, Ľ. (2023). Study of the Effect of Pretreatment of 3D Printed PLA Filament Modified by Plasma Discharge and Changes in Its Dynamic-Mechanical Properties. In: *Manufacturing Technology*, Vol. 23, No. 4, pp. 461-467. https://doi.org/10.21062/mft.2023.050.

[5] BARAN, E. H. & ERBIL, H. Y. (2019). Surface Modification of 3D Printed PLA Objects by Fused Deposition Modeling: A Review. In: *Colloids and Interfaces*, Vol. 3, No. 2, p. 43. https://doi.org/10.3390/colloids3020043.

[6] TÜMER, E. H. & ERBIL, H. Y. (2021). Extrusion-Based 3D Printing Applications of PLA Composites: A Review. In: *Coatings*, Vol. 11, No. 4, p. 390. https://doi.org/10.3390/coatings11040390.

[7] CRISTEA, M., IONITA, D. & IFTIME, M. M. (2020). Dynamic Mechanical Analysis Investigations of PLA-Based Renewable Materials: How Are They Useful? In: *Materials*, Vol. 13, No. 22, p. 5302. https://doi.org/10.3390/ma13225302.

[8] STRUZ, J., TROCHTA, M., HRUZIK, L., PISTACEK, D., STAWARZ, S., KUCHARCZYK, W. & RUCKI, M. (2024). Wear and Dynamic Mechanical Analysis (DMA) of Samples Produced via Fused Deposition Modelling (FDM) 3D Printing Method. In: *Polymers*, Vol. 16, No. 21, p. 3018. https://doi.org/10.3390/polym16213018.

[9] PLAMADIALA, I., CROITORU, C., POP, M. A. & ROATA, I. C. (2025). Enhancing Polylactic Acid (PLA) Performance: A Review of Additives in Fused Deposition Modelling

(FDM) Filaments. In: *Polymers*, Vol. 17, No. 2, p. 191. https://doi.org/10.3390/polym17020191.

[10] PAVON, C., ALDAS, M., SAMPER, M. D., MOTOC, D. L., FERRANDIZ, S. & LÓPEZ-MARTÍNEZ, J. (2022). Mechanical, Dynamic-Mechanical, Thermal and Decomposition Behavior of 3D-Printed PLA Reinforced with $CaCO_3$ Fillers from Natural Resources. In: *Polymers*, Vol. 14, No. 13, p. 2646. https://doi.org/10.3390/polym14132646.

[11] LIAO, Y., LIU, C., COPPOLA, B., BARRA, G., DI MAIO, L., INCARNATO, L. & LAFDI, K. (2019). Effect of Porosity and Crystallinity on 3D Printed PLA Properties. In: *Polymers*, Vol. 11, No. 9, p. 1452. https://doi.org/10.3390/polym11091452.

[12] HEDAYATI, R., ALAVI, M. & SADIGHI, M. (2024). Effect of Degradation of Polylactic Acid (PLA) on Dynamic Mechanical Response of 3D Printed Lattice Structures. In: *Materials*, Vol. 17, No. 15, p. 3674. https://doi.org/10.3390/ma17153674.

[13] JANÍK, R., KOHUTIAR, M., DUBEC, A., ECKERT, M., MORICOVÁ, K., PAJTÁŠOVÁ, M., ONDRUŠOVÁ, D. & KRBATA, M. (2022). DMA Analysis of Plasma Modified PVC Films and the Nature of Initiated Surface Changes. In: *Materials*, Vol. 15, No. 13, p. 4658. https://doi.org/10.3390/ma15134658.

[14] JANÍK, R., KOHUTIAR, M., SKALKOVÁ, P., PAJTÁŠOVÁ, M., LABAJ, I., ŠULCOVÁ, J. & DOBROVSKÁ, J. (2023). Modification Process of Selected Thermoplastics with Cold Plasma and Evaluation of Changes in Their Properties. In: *Materialwissenschaft und Werkstofftechnik*, Vol. 54, No. 4, pp. 401-414.

[15] ASTM D4065; American Society for Testing Materials. (2020). Standard Practice for Plastics: Dynamic Mechanical Properties: Determination and Report of Procedures. ASTM International, West Conshohocken, PA, USA.

[16] HANON, M. M., MARCZIS, R. & ZSIDAI, L. (2021). Influence of the 3D Printing Process Settings on Tensile Strength of PLA and HT-PLA. In: *Periodica Polytechnica Mechanical Engineering*, Vol. 65, No. 1, pp. 38-46.

[17] BECHNÝ, V., MATUŠ, M., JOCH, R., DRBÚL, M., CZÁN, A., ŠAJGALÍK, M. & NOVÝ, F. (2024). Influence of the Orientation of Parts Produced by Additive Manufacturing on Mechanical Properties. In: *Manufacturing Technology*, Vol. 24, pp. 2-8.