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Effect of DCSBD Plasma Treatment on the Mechanical Properties of Polymer Films

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Polymers and their surface modifications are the subject of intensive research due to their wide industrial applications in the fields of food packaging, biomedicine and electronics. The most widely used polymer films include polyethylene (PE), polyethylene terephthalate (PET) and polypropylene (PP), whose surface and mechanical properties can be optimized through physicochemical modifications. Diffuse coplanar surface barrier discharge (DCSBD) represents an effective method for modifying the surface properties of polymers without significantly affecting their structural properties. This study focuses on analyzing the effect of DCSBD plasma discharge on the mechanical behavior of PE, PET and PP films by means of dynamic mechanical analysis (DMA). Experimental samples were exposed to DCSBD plasma discharge and subsequently subjected to DMA over a wide temperature range. The measurement results showed significant changes in the storage modulus (E'), loss modulus (E'') and loss angle (tan δ), while a decrease in material stiffness and a shift in glass transition temperatures (T_g) were identified.

Keywords: Polymer films, Dynamic mechanical analysis (DMA), DCSBD plasma, Surface treatment

1 Introduction

The study of polymers and their surface treatments is gaining increasing attention due to the growing demand for advanced materials in various industrial applications. The most widely used polymer films include polyethylene (PE), polyethylene terephthalate (PET) and polypropylene (PP), which find wide application in the fields of food packaging, biomedicine and electronics. However, improving their surface properties without affecting the mechanical properties is a challenge [1,2]. One of the most effective surface treatment methods is diffuse coplanar surface barrier discharge (DCSBD), which is intensively investigated for its ability to modify the surface of polymers without affecting their structural properties. Šrámková et al. investigated the effect of atmospheric discharge plasma treatment on BOPP films and found a significant improvement in adhesion and surface wettability [3]. Štěpánová et al. demonstrated that DCSBD roll-to-roll plasma treatment significantly improved the adhesion properties of LLDPE/PA tubular films, even when the plasma was applied only to the outer side of the film [4].

The key method for evaluating the mechanical and viscoelastic changes caused by DCSBD plasma treatment is DMA analysis. This method provides important data on the storage modulus (E'), loss modulus (E'') and loss angle (tan δ), which allow characterizing the mechanical behavior of materials under dynamic loading. Janík et al. investigated the effect of plasma treatment on PVC films, noting significant changes in the viscoelastic properties of the material due to plasma-initiated surface changes [5]. Similarly, Cristea et al. analyzed PLA-based renewable materials using DMA, emphasizing the importance of temperature transitions in evaluating their functional properties [6]. In the case of PET films, Choi et al. used DMA to predict the long-term viscoelastic stability, providing important insights into the aging and degradation of polymeric materials [7].

In addition to mechanical properties, plasma treatment is also being investigated for sterilization and safety in the food industry. Mošovská et al. demonstrated that treatment of food surfaces with cold atmospheric plasma effectively inhibited pathogenic bacteria, suggesting its potential use in antimicrobial applications [8]. Bonnaillie et al. investigated the effect of humidity on the mechanical properties of edible casein films using humidity-controlled DMA (DMA-RH), emphasizing the need for thorough mechanical characterization of materials intended for food packaging [9].

Despite significant advances in plasma treatment research, it remains important to further investigate the long-term effects of plasma modification on the degradation and stability of polymers. Understanding these aspects is crucial for optimizing processing parameters and ensuring the long-term functionality of plasma-treated polymer films. This study focuses on the effect of DCSBD plasma treatment on the mechanical behavior of polymer films using DMA analysis. By analyzing the storage modulus (E'), loss modulus (E") and loss angle (tan δ), this work aims to provide a comprehensive view of how plasma surface treatment affects the viscoelastic properties of PE, PET and PP films. The analysis of food grade plastic films compares standard films with films that have been surface modified by diffuse coplanar surface barrier plasma discharge (DCSBD) for sterilization and cleaning purposes.

2 Materials and methods

All measurements were performed on a dynamic-mechanical analyzer DMA Q800 from TA Instruments. The device is designed to measure the viscoelastic properties of materials. The temperature range of the device is from -150 °C to 600 °C, while cooling is provided by liquid nitrogen. The TA Universal Analysis software ver. 4.5A, which is supplied directly with the device, was used to evaluate the measured data. The result of the DMA analysis are curves of the storage modulus (E'), loss modulus (E") and loss angle (tan δ) as a function of temperature, from which the transition temperatures for individual materials were subsequently determined and compared. The transition temperatures from E" and tan δ were determined from the maximum value of the peak (local maximum) corresponding to a given temperature transition. The transition temperatures from E' were determined by the extrapolated value of „OnSet¨ from the slope of the curve. The determination of transition temperatures is described in the ASTM D4065 standard [10].

Three types of plastic films were used for the experiment, namely polyethylene (PE), polyethylene terephthalate (PET) and polypropylene (PP) films. Individual samples for DMA analysis were cut (cut) from the films to the desired shape, with the dimensions of the PE samples being 22 mm x 8 mm x 0.03 mm, the dimensions of the PET samples being 22 mm x 8 mm x 0.25 mm and the dimensions of the PP samples being 22 mm x 8 mm x 0.35 mm. Subsequently, ½ of all samples were surface-modified by DCSBD plasma discharge for the purpose of sterilization and cleaning of their surfaces. Based on this, the surface-modified samples were designated PE+P, PET+P and PP+P.

DCSBD is one of the types of surface dielectric barrier discharges, and this type of plasma discharge was used for double-sided surface treatment of plastic films on a planar ceramic dielectric located on the KPR 200 mm plasma reactor line (Fig. 1).

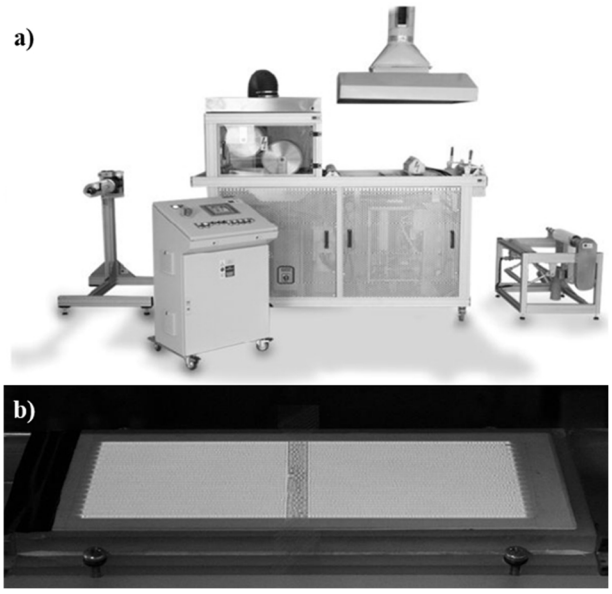

Fig. 1 *a) Plasma reactor KPR 200 mm; b) Planar ceramic dielectric generating plasma discharge*

DCSBD generates a diffusion type plasma in air at atmospheric pressure, while with a plasma power density of up to 100 W.cm^{-3} it allows short exposure times. The maximum power of the device is 400 W. The homogeneity of the generated plasma increases with increasing power density of the plasma discharge. The depth of the generated plasma is maximum up to 0.3 mm. The plasma reactor is suitable for effective hydrophilization and activation of surfaces of various materials (films, textiles, plastics, glass, ceramics, metals, wood) [11-13], for cleaning and sterilization of surfaces, or for processing printed circuit boards and silicon wafers in electrical engineering [14,15].

The modification of the surface of the film samples took place at atmospheric pressure and at laboratory temperature, with a plasma reactor power of 350 W, the exposure time was set at 5 seconds for each side of the film sample. The samples were placed on a glass slide on a flat ceramic dielectric surface to ensure the flatness of the film samples.

The DMA analysis used a tension-film geometry, which is suitable for measuring the viscoelastic properties of thin films. The samples were clamped between two clamps (Fig. 2), one clamp being movable and the other clamp being static.

DMA analysis of PE and PE+P samples was carried out in the temperature range from -70 °C to 110 °C, analysis of PET and PET+P samples was carried out in the temperature range from -70 °C to 170 °C and analysis of PP and PP+P samples was carried out in the temperature range from -70 °C to 110 °C. All tested samples were first cooled to a temperature of

-70 °C and then heated to the final temperature at 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 materials. For all measured material samples, average *E′*, *E″* and *tan δ* curves were created using the OriginPro software ver. 9.1.0., with a minimum of 3 measurements for each sample. The measurement output is therefore the *E′*, *E″* and *tan δ* curves and the measured transition temperature values, which are subsequently compared within individual materials.

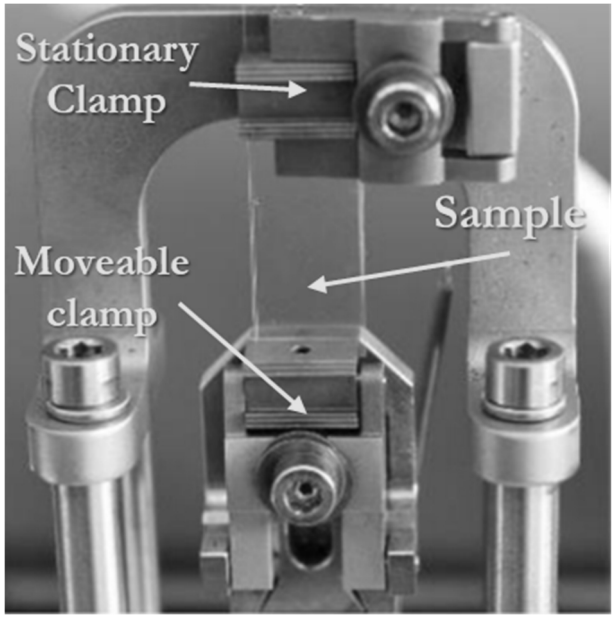

Fig. 2 *Measurement using the tension-film geometry*

3 DMA analysis of PE films

By comparing the curves of the storage modulus of polyethylene samples of PE and PE+P films (Fig. 3) in the temperature range from -70 °C to 110 °C, it can be observed that the E' value decreases for both samples. At a temperature of ≈ 100 °C, both samples degrade, losing their useful properties. The E' values of the PE+P sample compared to PE are significantly lower in the temperature range, which is related to the decrease in the stiffness of the material exposed to DCSBD plasma discharge. The E' curve of the PE sample has a more gradual decreasing tendency compared to the PE+P curve, which is related to the lower content of the crystalline phase in the PE+P sample. The lower content of this phase is related to surface treatment, when the increased temperature (up to 70 °C) causes α-relaxation, which is associated with movement of the main polymer chain and melting of crystallites in the volume of the material.

By comparing the loss modulus curves of polyethylene samples of PE and PE+P films (Fig. 4) in the temperature range from -70 °C to 110 °C, it can be observed that the PE+P sample shows higher loss modulus values at negative temperatures compared to the PE sample. On the contrary, β-relaxation, i.e. the movement of the side branches of the main polymer chain, of the PE+P sample will occur at a lower temperature (-9.62 °C) compared to the PE sample (-0.15 °C) (Fig. 6). At positive temperatures, the PE sample shows higher loss modulus values, up to a temperature of ≈ 100 °C, when both samples degrade. α-relaxation, the movement of the main polymer chain, will occur again earlier in the PE+P sample (50.51 °C) compared to the PE sample (53.57 °C), which can be attributed to the lower content of the crystalline phase in the modified sample (Fig. 6).

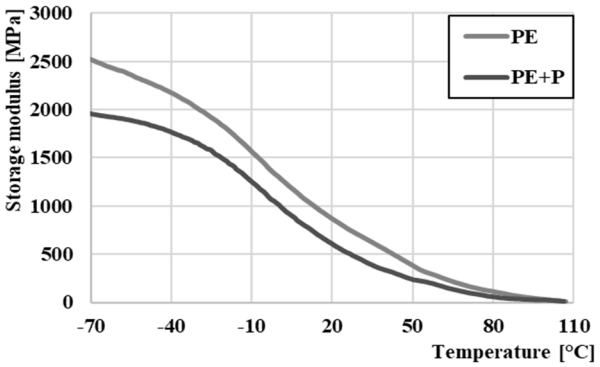

Fig. 3 *Storage modulus curves of PE films*

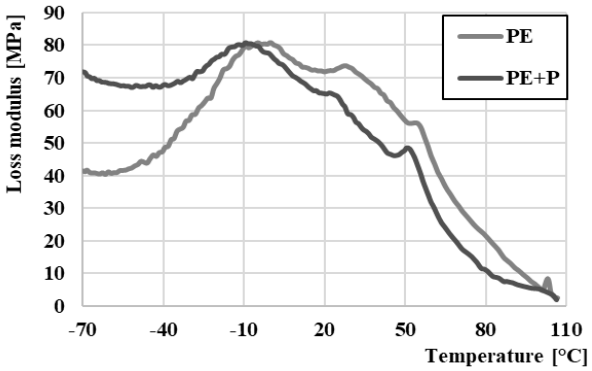

Fig. 4 *Loss modulus curves of PE films*

By comparing the tan δ curves of polyethylene samples of PE and PE+P films (Fig. 5) in the temperature range from -70 °C to 110 °C, it can be stated that the PE+P sample achieves higher tan δ values compared to the standard PE sample. Based on the tan δ curves, it is possible to determine the α-relaxation values for both samples. By measuring the local maximum of tan δ, it was found that the movement of the main polymer chain occurs at a lower temperature again for the PE+P sample (51.69 °C) compared to the PE sample (56.00 °C) (Fig. 6). The pronounced peak on the tan δ curve of the PE sample and the decrease in the tan δ curve of the PE+P sample above 100 °C are associated with the melting of the material, when various anomalies occur on the curves.

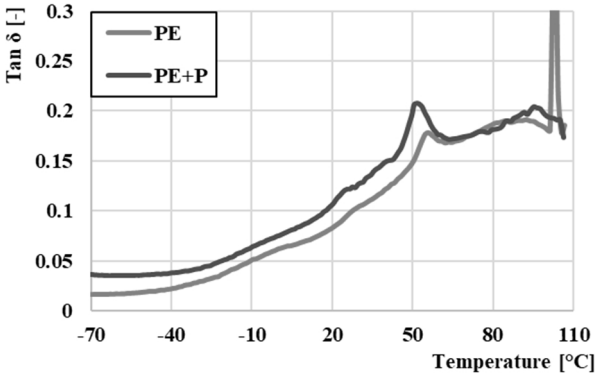

Fig. 5 *Tan δ curves of PE films*

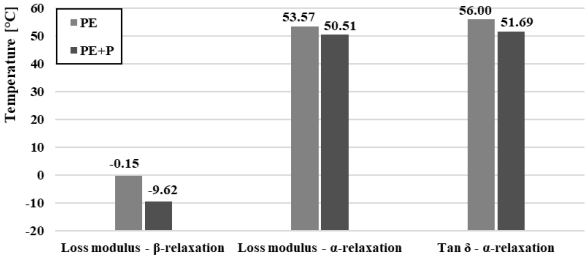

Fig. 6 *Measured a-relaxation and β-relaxation values of PE films*

By DMA analysis of PE plastic films, by comparing the E', E" and tan δ curves and by comparing the measured transition temperatures of both samples, it can be concluded that the DCSBD plasma, by its effect (temperature above the α-relaxation of PE), reduces the stiffness of the material, due to the reduction of the crystalline fraction in the volume of the material. Therefore, there is an assumption that the PE film modified by DCSBD plasma discharge will have lower stiffness compared to the standard film, while maintaining its performance properties under normal conditions.

4 DMA analysis of PET films

By comparing the storage modulus curves of polyethylene terephthalate samples of PET and PET+P films (Fig. 7) in the temperature range from -70 °C to 170 °C, it can be observed that the E' values of the PET+P sample compared to the PET sample are very low in the temperature range, which is related to a significant decrease in the stiffness of the material exposed to DCSBD plasma discharge. By measuring and 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 PET+P sample (55.50 °C) is significantly lower compared to the PET sample (63.48 °C) (Fig. 10). This temperature at E' is related to the mechanical failure of the material, so it can be assumed that the PET film modified by DCSBD plasma discharge will lose its performance properties significantly earlier than the standard film.

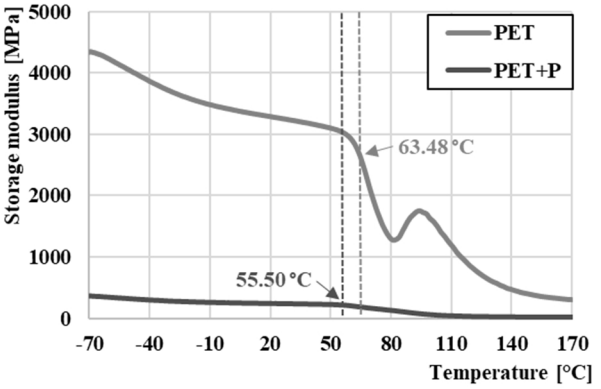

Fig. 7 *Storage modulus curves of PET films*

By comparing the loss modulus curves of polyethylene terephthalate film samples PET and PET+P (Fig. 8) in the temperature range from -70 °C to 170 °C, it can be stated that the PET+P sample again shows significantly lower E" values compared to the PET sample. E" is related to the transferred energy converted into heat during one load cycle of the measured sample. By measuring the glass transition temperature (T_g) from the maximum peak values of both E" curves, it can be summarized that the T_g temperature of the PET+P sample (86.25 °C) is significantly lower compared to the PET sample (103.04 °C) (Fig. 10).

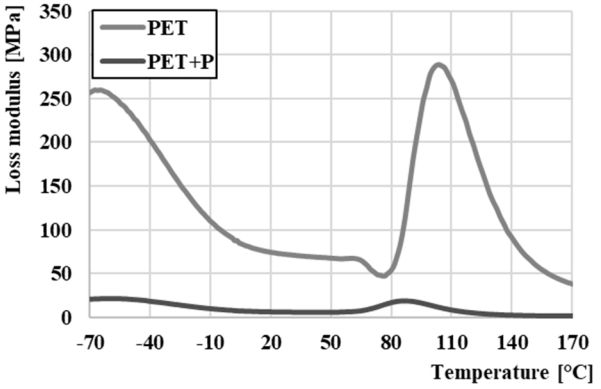

Fig. 8 *Loss modulus curves of PET films*

By comparing the tan δ curves of polyethylene terephthalate PET and PET+P film samples (Fig. 9) in the temperature range from -70 °C to 170 °C, it can be stated that the PET+P sample achieves higher tan δ values up to the T_g temperature, compared to the standard PET sample. Based on the tan δ curves, the T_g temperature values were determined by measuring the local maximum (peak). Tan δ represents the ability of the material to absorb and safely dissipate energy. Tan δ values are also described as the loss factor or damping capacity of the material. The higher the tan δ value, the more effective the damping capacity of the material [16, 17]. As can be seen in Fig. 9, the tan δ value is slightly higher for the PET+P sample, which is related to the reduced stiffness of the sample. By comparing the T_g temperature of both tan δ curves, it

can be concluded that the T_g temperature of the PET+P sample (102.55 °C) is significantly lower compared to the PET sample (117.16 °C) (Fig. 10).

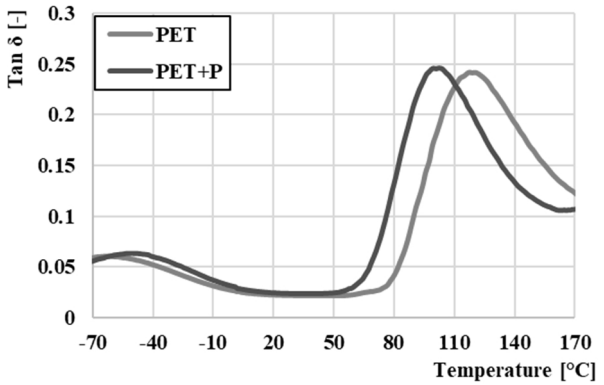

Fig. 9 *Tan δ curves of PET films*

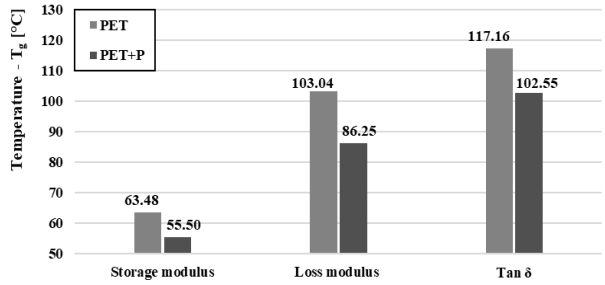

Fig. 10 *Measured T_g values from E', E'' and tan δ of PET films*

By DMA analysis of PET plastic films, comparing the E', E" and tan δ curves and comparing the measured T_g temperatures of both samples, it can be stated that the DCSBD plasma significantly reduced the stiffness of the material and also the T_g temperature. It can be assumed that the modified film loses its useful properties under normal conditions of use.

5 DMA analysis of PP films

By comparing the storage modulus curves of polypropylene samples of PP and PP+P films (Fig. 11) in the temperature range from -70 °C to 110 °C, it can be observed that the E' values of the PP+P sample compared to the PP sample are lower in the temperature range, which is related to the decrease in the stiffness of the material exposed to DCSBD plasma discharge. For the PP+P sample, a more pronounced slope of the curve can be observed in the temperature range from -10 °C to 20 °C compared to the PP sample. 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 PP+P sample (-12.59 °C) is slightly higher compared to the PP sample (-14.29 °C) (Fig. 14). Since PP is prone to embrittlement at negative temperatures, in this case a higher T_g temperature has a rather negative effect. PP material surface-modified by DCSBD plasma discharge will mechanically fail earlier with decreasing temperature of its use compared to standard material.

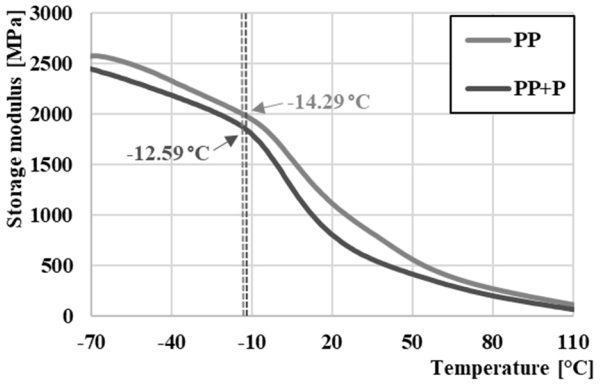

Fig. 11 *Storage modulus curves of PP films*

By comparing the loss modulus curves of polypropylene samples of PP and PP+P films (Fig. 12) in the temperature range from -70 °C to 110 °C, it can be stated that the PP+P sample shows significantly lower E" values compared to the PP sample. Based on the measurement of the glass transition temperature (T_g) from the maximum peak values of both E" curves, it can be stated that the T_g temperature of the PP+P sample (0.19 °C) is significantly lower compared to the PP sample (2.95 °C) (Fig. 14).

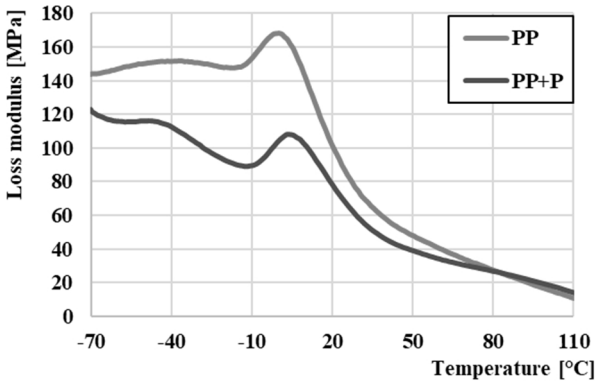

Fig. 12 *Loss modulus curves of PP films*

By comparing the tan δ curves of polypropylene film samples PP and PP+P (Fig. 13) in the temperature range from -70 °C to 110 °C, it can be stated that the PP+P sample achieves lower tan δ values compared to the standard PP sample. Based on the tan δ curves, the T_g temperature values were determined. As can be seen in Fig. 13, the tan δ value is lower for the PP+P sample compared to the PP sample, which leads to a reduced damping capacity. By comparing the T_g temperature of both tan δ curves, it can be stated that the T_g temperature of the PP+P sample (11.49 °C) is slightly lower compared to the PP sample (12.40 °C) (Fig. 14).

By DMA analysis of PP plastic films, comparing the curves E', E" and tan δ and comparing the measured temperatures T_g of both samples, it can be stated

that the DCSBD plasma reduced the stiffness of the material and also the temperature T_g (except E'). It can be assumed that the modified film will retain its useful properties under normal conditions of use. DCSBD plasma modification of PP film reduced the interval of its use in negative temperatures.

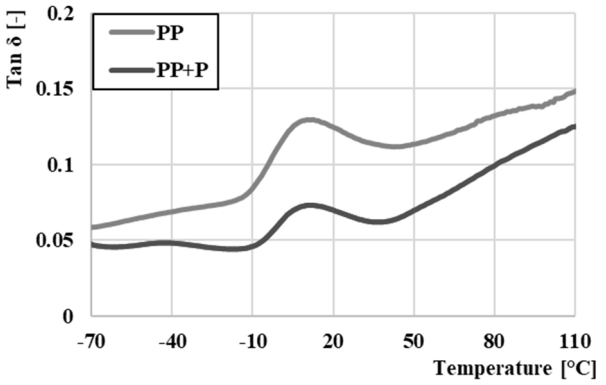

Fig. 13 *Tan δ curves of PP films*

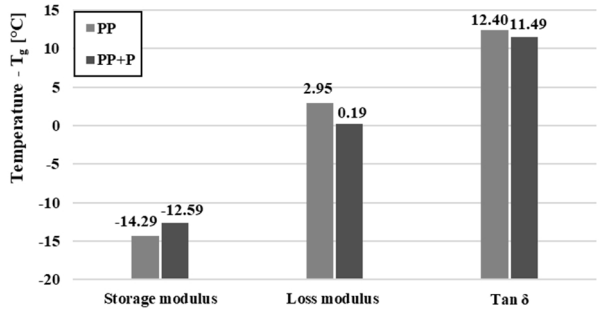

Fig. 14 *Measured T_g values from E', E'' and tan δ of PP films*

6 Discussion

By DMA analysis of PE plastic films, by comparing the E', E" and tan δ curves and by comparing the measured transition temperatures of both PE samples, it can be stated that the DCSBD plasma discharge, by its effect (temperature above the α-relaxation of PE), reduces the stiffness of the PE material, due to the reduction of the crystalline fraction in the volume of the material. There is therefore an assumption that the PE film modified by the DCSBD plasma discharge will have a lower stiffness in practice compared to the standard PE film, while maintaining its useful properties under normal conditions. In addition, it was found that β-relaxations, i.e. the movement of the side branches of the main polymer chain, in the PE+P sample will occur at a temperature of -9.62 °C, which is 9.47 °C lower than in the PE sample (-0.15 °C). α-relaxation, the movement of the main polymer chain, will occur earlier again in the PE+P sample, at a temperature of 50.51 °C, which is 3.06 °C less compared to the PE sample (53.57 °C). This temperature difference can be attributed to the lower content of the crystalline phase in the modified sample. By DMA analysis of PET plastic films, by comparing the E', E" and tan δ curves and by comparing the measured T_g temperatures of both samples, it can be stated that the DCSBD plasma discharge significantly reduced the stiffness of the material and also the T_g temperature of the PET film. By measuring the T_g from the E' curves of both samples, it was found that the temperature T_g = 55.50 °C of the PET+P sample is 7.98 °C lower compared to the PET sample (63.48 °C). By measuring Tg from the E" curves, it was found that the temperature T_g = 103.04 °C of the PET+P sample is 16.79 °C lower compared to the PET sample (86.25 °C). This shift could have been caused by an inappropriately set plasma reactor power, a longer or inappropriately selected exposure time, overheating of the sample on the ceramic dielectric, or a combination of these factors. By measuring T_g from the tan δ curves of both samples, it was found that the temperature T_g = 117.16 °C of the PET+P sample is 14.61 °C lower compared to the PET sample (102.55 °C). It can be assumed that the modified film loses its useful properties under normal conditions of use. By DMA analysis of PP plastic films, by comparing the E', E" and tan δ curves and by comparing the measured T_g temperatures of both samples, it can be stated that the DCSBD plasma reduced the stiffness of the material and also the T_g temperature (except E'). By measuring T_g from E' curves, it was found that the temperature T_g = -12.59 °C of the PP+P sample is 1.70 °C higher compared to the PP sample (-14.29 °C). By measuring Tg from E" curves, it was found that the temperature T_g = 0.19 °C of the PP+P sample is 2.76 °C lower compared to the PP sample (2.95 °C). By measuring T_g from tan δ curves of both samples, it was found that the temperature T_g = 11.49 °C of the PP+P sample is 0.91 °C lower compared to the PP sample (12.40 °C). It can be assumed that the modified film will retain its performance properties under normal conditions of use, while DCSBD plasma modification of the PP film shortened the interval of its use in negative temperatures.

7 Conclusion

Based on the DMA analysis of PE, PET and PP plastic films, it can be concluded that DCSBD plasma discharge significantly affects the properties of the investigated materials. The results of the study can be evaluated in the following:

- PE film has a decrease in stiffness due to a reduction in the crystalline content, while it should retain its performance properties under normal conditions of use.
- PET film after modification shows a significant reduction in both stiffness and glass transition temperature, indicating a potential loss

of performance properties under normal conditions.

- A decrease in stiffness and slight changes in T_g temperature were found for PP film, while the material should remain functional under standard conditions, but with limited resistance to low temperatures.

Overall, it can be said that the impact of DCSBD plasma discharge depends on the type of polymer and its structure, and for some materials it can lead to improved processability, while for others it can limit their applicability under certain conditions.

Acknowledgement

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