

Characteristics of biodegradable polymers when subjected to ceramic coatings

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Abstract. This manuscript highlights the behavior of a biodegradable polymer (Arboblend V2 Nature) coated with three distinct ceramic powders, Cr₂O₃-chromium oxide thermal spray powder, ZrO₂ 18TiO₂ 10Y₂O₃-zirconia-titania-yttria composite powder and Cr₂O₃-xSiO₂-yTiO₂-chromia-silica composite powder. The coated was realized on injected samples by using the Atmospheric Plasma Spray (APS) technique. The current investigation will explain the results related to the surface quality, micro-structure, morphology, mechanical, thermal and tribological properties. Thus, from a structural point of view the most uniform deposition was obtained in the case of composite powder based on zirconia. The thermal behavior of the samples coated with ceramic micro layers achieved stability up to temperatures slightly above 200 °C, pyrolysis taking place around 340 °C. The micro indentation, DMA, and scratch analysis responses were significantly influenced by the crystalline structure of the samples and the presence of the Cr₂O₃ compound. Based on the increased characteristics of the coated samples the authors of the present paper consider that from parts made of biodegradable polymers and coated with ceramic micro-particles are appropriate for some applications that required harsh operating conditions.

Keywords: Ceramic coating / biodegradable plastic / ceramic micro particles / structure / thermal behavior / tribology

1 Introduction

In current academia, significant emphasis has been placed on the research and development of biodegradable materials as a viable solution to address the issue of environment pollution. Biodegradable materials are currently generating considerable interest in several technological domains such as food packaging, automotive, medicine and other modern technological applications. Their widespread applicability can be attributed to their environmentally friendly nature, capacity to decompose naturally, compatibility with biological systems, and cost-effectiveness [1]. The utilization of renewable resources in the manufacturing of polymeric materials confers three distinct environmental advantages. Initially, the feedstock utilized have the potential to be substituted, either through inherent ecological processes or through human intervention. Furthermore, the biodegradability of the final products mitigates the potential contamination that would arise from the disposal of a comparable amount of

traditional plastics. Typically, bio polymeric materials are disposed by landfilling or composting once they have reached the end of their functional lifetime. Moreover, taking advantage of this methodology can effectively address the simultaneous consideration of social, environmental, and economic consequences [1,2]. There exists a diverse array of polymers, encompassing both natural and manufactured variants. Nevertheless, the extensive implementation of biodegradable materials still requires further endeavors.

Polymeric surfaces can be coated using many processes, including physical vapor deposition (PVD), thermal spray (TS), electroplating, and chemical vapor deposition (CVD) [3–8]. The utilization of any of these approaches is anticipated to provide both benefits and drawbacks, contingent upon the variables inherent in the coating procedure. Therefore, prior to achieve the coating, is necessary to choose a methodology, to realize a comprehensive analysis in order to determine the controllable variables that significantly impact the quality of the coated parts. Initially, an examination is conducted on the process parameters associated with the material, specifically the substrate and surface conditioning. These parameters take

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in to account the characteristics of the coating powder, such as its chemical composition, particle size, and powder flow rate. Additionally, the properties of the gas, including its composition, temperature, and pressure, are taken into consideration. Lastly, the coating process is evaluated, which involves factors such as the spraying distance, substrate temperature and its oxidation state, energy of particle impacts and temperature of the particles themselves. These controllable factors have impact on the output characteristics such as: porosity, micro hardness, composition, deposition efficiency, adhesion strength, electrical conductivity, surface roughness, and others.

The existing body of technical literature primarily focuses on the investigation of coatings containing ceramic and metallic micro particles, specifically examining the surface properties of the deposited micro layers such as wear resistance, adhesion, corrosion resistance, and hardness [9–12]. Additionally, researchers have also explored the impact of process parameters and their optimization [13].

The thermal coating process is widely utilized in various industries from automotive to aerospace, to enhance the corrosion and wear resistance and also the lifespan of equipment components. For instance, thermal coatings (tungsten, chromium carbide, $\text{Cr}_3\text{C}_2\text{-NiCr}$) are applied to components operating under extreme thermal conditions, such as vanes, turbine blades and fuel parts to extend their lifespan by improving resistance to wear, corrosion heat and oxidation [14–16]. The application of TPS (titanium plasma spraying) on polymers, such as polyethylene and polyamide, demonstrates favorable mechanical properties and increased hardness [17].

Atmospheric Plasma Spray (APS) is a widespread thermal deposition technology utilized in many different fields of activities. The coatings resulting from zirconium and chromium ceramic layer are extensively employed in mechanical contexts [18]. The primary limitations associated with these coatings are attributed to: presence of micro-cracks, inconsistencies in isolation, and residual stresses that arise within the cooling process [19].

Aluminum, nickel, chromium and zirconium are used as powdered elements and exhibit favorable characteristics for applications necessitating increased toughness with modest resistance to corrosion, friction and scratch. Zinc-based coatings are commonly utilized in various applications where resistance to corrosion is compulsory. However, it should be noted that zinc, when not alloyed with other elements like nickel or tungsten carbide, exhibits relatively low mechanical properties, causing delamination heavily influenced by the carbon dioxide's affinity for the polymer matrix (in a humid environment) [20–22].

The high-purity Al_2O_3 powder, also known as aluminum oxide or alumina, is commonly utilized for plasma spray coating of materials. This powder exhibits electrical insulating properties, specifically in relation to thermal conductivity and dielectric characteristics. In addition, the coatings exhibit wear resistance, possess elevated hardness, have enhanced temperature stability, and exhibit chemical inertness. The material's high level of purity guarantees that it will not introduce any impurities to the semiconductor components [23].

The incorporation of ceramic particles and/or metal particles into a bio polymeric substrate has been well recognized for its ability to combine the properties of all three material types. The enhancement of the composite's physical and mechanical qualities may be a potential outcome [24–26].

The main research in the field of polymer matrix coatings refers to the examinations as: structural and morphological analysis [9,10,13,27–37], influence of input factors on structure, mechanical properties and adhesion [38–50], adhesion strength [11,12,51,52]; coating oxidation resistance [12,53], tribological properties [10,54–58], electrical resistance and conductivity [54,59,60], thermal behavior [10,60,61] etc.

This present manuscript presents an analysis conducted by the authors on three distinct types of ceramic micro-powders. The coatings were realized onto Arboblend V2 Nature as substrate materials. The samples to be coated were acquired through the utilization of injection molding, whereas the coating process was atmospheric plasma spraying.

The objective of this article was to obtain a novel material exhibiting enhanced characteristics, so enabling its effective utilization as a replacement for traditional plastics within the automotive sector and not only. In light of the stated purpose, the study team opted for the use of Arboblend V2 Nature, a biodegradable material whose qualities have been previously investigated by the researchers. Subsequently, the application of the APS approach was undertaken to achieve the coating with ceramic micro particles, followed by an examination of its corresponding properties. The current investigation is not observed inside the research literature, so the proposed study, which incorporates both technological advancements and experimental findings, represents a novel contribution.

2 Materials and methods

Arboblend V2 Nature was covered with ceramic micro-particles. The polymer's matrix is lignin, extracted from annual vegetable plants, so it doesn't need wood raw materials that take decades to mature and be used in the forestry and paper industries [62–64]. This is crucial because this substance uses lignin from paper industry waste. The Arboblend V2 Nature structure can also contain natural vegetable fibers, polylactic acid (PLA), cellulose, bio-polyamides (bio-PA) and a small amount of natural additives [26,65–67].

SZ-600H equipment (SHEN ZHOU, Zhangjiagang, China) was used to inject samples for ceramic coating. Samples had $10 \times 50 \times 70 \text{ mm}^3$ and the used injection molding parameters were: 165°C – melting temperature; 80 m/min – injection speed; 100 MPa – injection pressure; 30s – cooling time. The injected samples were coated using atmospheric plasma spray (APS) technology, namely an SPRAYWIZARD-9MCE (Sulzer-Metco, Westbury, New York) with a USA/9MB spraying gun. The parameters utilized, depending on the used powder were: N_2 and H_2

pressure – 3.4–3.7 bar, N₂ gas flow – 39–44 NLPM, H₂ gas flow – 6.6 NLPM, electric DC – 400A; for the powder dispenser: carrier gas flow – 5.1–5.3 (NLPM), air pressure – 1.4 bar; material flow – 126–144 g/min, spray distance 137–145 mm; number of passes – 7.

The rate at which the micro particles were deposited remained constant. The ceramic layer that was deposited had a thickness on the order of micrometers. To control the melting temperature of the samples, a laser pyrometer was utilized during the entire operation.

The coating was made with three distinct ceramic powders: Amdry 6420–chromium oxide thermal spray powder (Cr₂O₃), (10–105) μm; Metco 136F – Chromia–Silica composite powder (Cr₂O₃-xSiO₂-yTiO₂), (9–110) μm; Metco 143–zirconia–titania–yttria composite powder (ZrO₂ 18TiO₂ 10Y₂O₃), (3–40) μm.

Three distinct micro powders were put onto three samples that were injected using Arboblend V2 Nature. In order to achieve a consistent and homogeneous powder deposition, a total of seven passes were conducted on each sample. The ceramic micro-particles were procured from the Oerlikon Metco company (Bella Vista, New South Wales, Australia) [68].

The following equipment was used to characterize the coated samples:

The QUANTA 200 3D electron microscope (FEI Company, Fremont, CA, USA) performed SEM analysis (*Scanning Electron Microscopy*). Key parameters were: microscope chamber pressure – 60 Pa; detector – Large Field Detector for non-conductive samples; tilt angle – 0°; secondary electron acceleration voltage – 20 Kv; working distance – 15 mm; magnification power – 100–1000×

X-ray diffraction analysis (XRD) was done with the X'Pert Pro MRD X-ray diffractometer (PANalytical, Almelo, the Netherlands) with 45 kV voltage and diffraction angle (2θ), changing between 10 and 90 degrees. Two X'Pert Data Collector programs, X'Pert High Score Plus version 3 and X'Pert Data Viewer version 2.2g (Malvern Panalytical, Malvern, UK), were used to process the data and make the graphs.

The CETR UMT-2 microtribometer was used for micro-indentation and scratch tests. Test conditions. For the scratch analysis, a 0.4 mm (radius at the tip) NVIDIA blade was used, the samples were placed on a table, and throughout the test, the samples were compressed with a 10 N vertical force. The table was moved (by translation motion) 10 mm in 60 seconds, and the test speed was 0.167 mm/s. Parameters such as vertical force F_z, horizontal force F_x, duration, and distance travelled in the horizontal direction Y (of the sample's fixed mass) were recorded by the program during the automatic test. Thus, the values obtained for the apparent friction coefficient (A-COF) being determined by the software.

The micro-indentation test involved using a Rockwell type indenter, which is a cone with a diamond tip that has an angle of 120° and a peak radius of 200 microns. The samples were placed on a table and subjected to a vertical force of 10 N as indicated in the study, following specific procedures and timeframes. Hardness and Young's modulus were calculated statistically with the utmost precision using three samples of each powder type. The indenter's

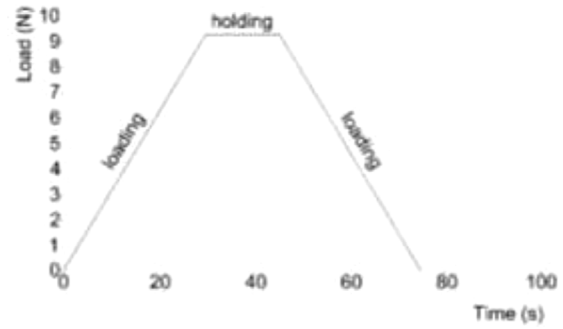


Fig. 1. Variation of loading with time.

vertical travel distance C, time, and vertical force F_z were recorded by the program during the automatic test (with capacitive sensor). Sensor of (0.2–20) N, loading duration of 30 s, holding time of 15 s, and unloading time of 30 s were additional process parameters (see Fig. 1). To ensure experimental repeatability, three samples of each ceramic powder were tested in micro-indentation tests. The standard deviation shows how different a group of integers is when compared to the average value, which was produced by computing the arithmetic average.

The relationship by which the soft hardness is calculated, equation (3), as well as the force-displacement graphi graphical mode are:

$$H_{IT} = \frac{F_{max}}{A_P} \quad (1)$$

where F_{max} is the maximum force and A_P is the projection of the contact area at this force.

The schematic representation of the indenter trace, as well as the typical evolution of the force with displacement, are shown in Figure 2.

Dynamic Mechanical Analysis (DMA): DMA 242 Artemis NETZSCH equipment, standardized samples 25 × 4 × 2 mm. For composite material viscosity testing, three-point bending was used. The test temperature ranged from RT (room temperature) to 373.15 K (100 °C), with a step of three Kelvin degrees, dynamic force of 5 N, strain of 50 μm, and frequency of 1 Hz.

Mettler Toledo TGA/SDTA 851 apparatus determined *TG*, *DTG*, and *DTA thermogravimetric curves*. Thermally decomposed samples weighed was 2.9–3.9 mg. 20 cm³/min air flow was used. The study was conducted at 25–700 °C at 10 °C/min. The beginning (T_{onset}), peak (T_{peak}), and end (T_{end}) temperatures of each thermal deterioration stage were determined. W% loss mass residue was also determined for each stage.

3 Results and discussion

In order to have an accuracy notation of the sample the following notation was established: sample 1 coated with zirconia–titania–yttria composite powder, MetcoTM 143, further noted with P2–143–7 passes; sample 2 coated with chromium oxide powder, Amdry 6420, noted with

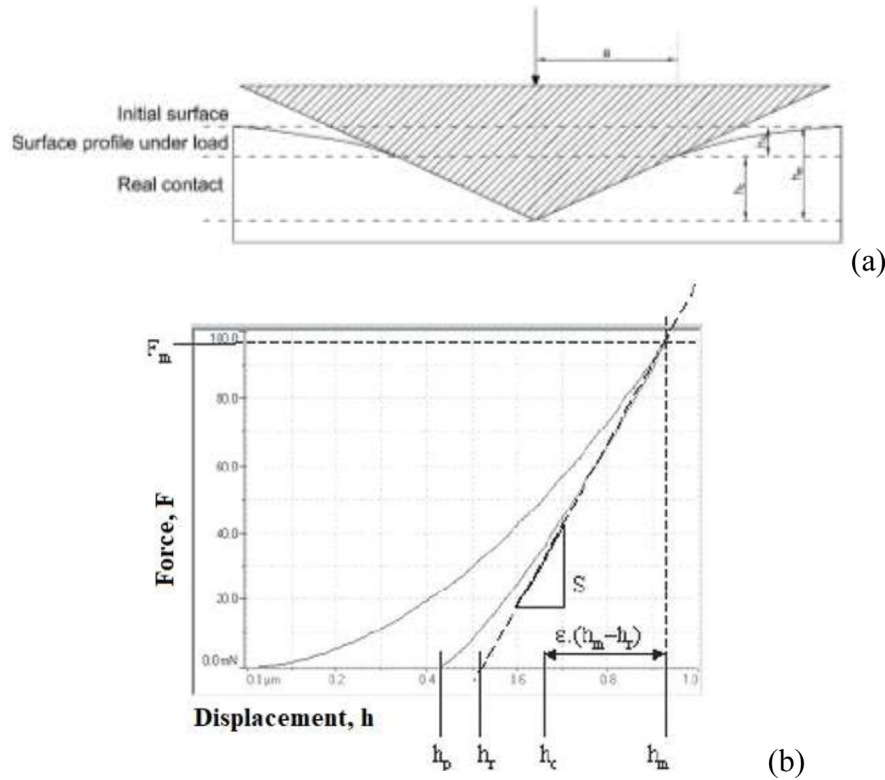


Fig. 2. Schematic of Berkovich indenter showing the indentation projection (a) and surface areas and depth parameters (b) – according to the technical documentation.

P5-6420-7 passes; sample 3 coated with chromia-silica composite powder, Metco 136F, noted with P8-136-7 passes.

3.1 Scanning electron microscopy (SEM) analysis

Figure 3a depicts the morphological characteristics of the Arboblend V2 Nature material, which has been coated with zirconia-titania-yttria composite powder. Inspection reveals a uniform coating of the biopolymer substrate. The coating is composed of spherical particles with diameters ranging from 3 to 40 micrometers. The spherical shape of the particles is maintained as a result of the quick cooling that occurs upon contact with the Arboblend V2 Nature substrate. The traditional flattening mechanism observed in coatings on metal substrates, characterized by the formation of splats, is not observed in this images [69]. The presence of diverse particles in terms of shape and size inside the fundamental matrix, occurring in significant amounts and exhibiting uniform distribution, contributes to the enhancement of mechanical and tribological characteristics. Yttrium oxide exhibits a porous spherical morphology in the shape of a sintered agglomeration.

Figure 3b highlight the polymer matrix containing particles originating from the chromium oxide coating. A portion of these particles exhibit heterogeneous distribution, whereas another portion is incorporated inside the polymeric structure. The dimensions of these objects range from (10-105) μm , exhibiting rectangular

forms that are characteristic of chromium oxide. The formation of spherical micro particles can be ascribed to the inclusion of Fe_2O_3 and SiO_2 inside the Amdry 6420 powder, with maximum concentrations of 0.4% and 0.45% respectively. In contrast to the P2-143-7 passes sample, the material exhibited a reduced level of powder-like visual characteristics, indicating a diminished potential for coating and embedding chromium oxide inside the polymeric substrate.

The presence of a coating composed of chromium oxide, silicon oxide, and titanium oxide ($\text{Cr}_2\text{O}_3\text{-xSiO}_2\text{-yTiO}_2$), as shown in Figure 3c, reveals a non-uniform distribution of micro particles, similar to the coating presented in Figure 3b. The particles exhibit diverse forms, primarily polyhedral in the case of TiO_2 , but also including spherical for SiO_2 and rectangular for Cr_2O_3 . These particles possess diameters ranging from 9 to 110 μm .

The particles are incorporated within the polymer matrix. Both Figures 3b and 3c reflect the presence of chromium oxide, and it is observed that this compound does not exhibit superior adhesion in comparison to the sample coated with P2-143-7 passes (Fig. 3a), which lacks chromium oxide.

3.2 X-ray diffraction analysis

The objective of this investigation was to determine whether the Arboblend V2 Nature samples exhibited any distinctive crystallization phases on their surface that were

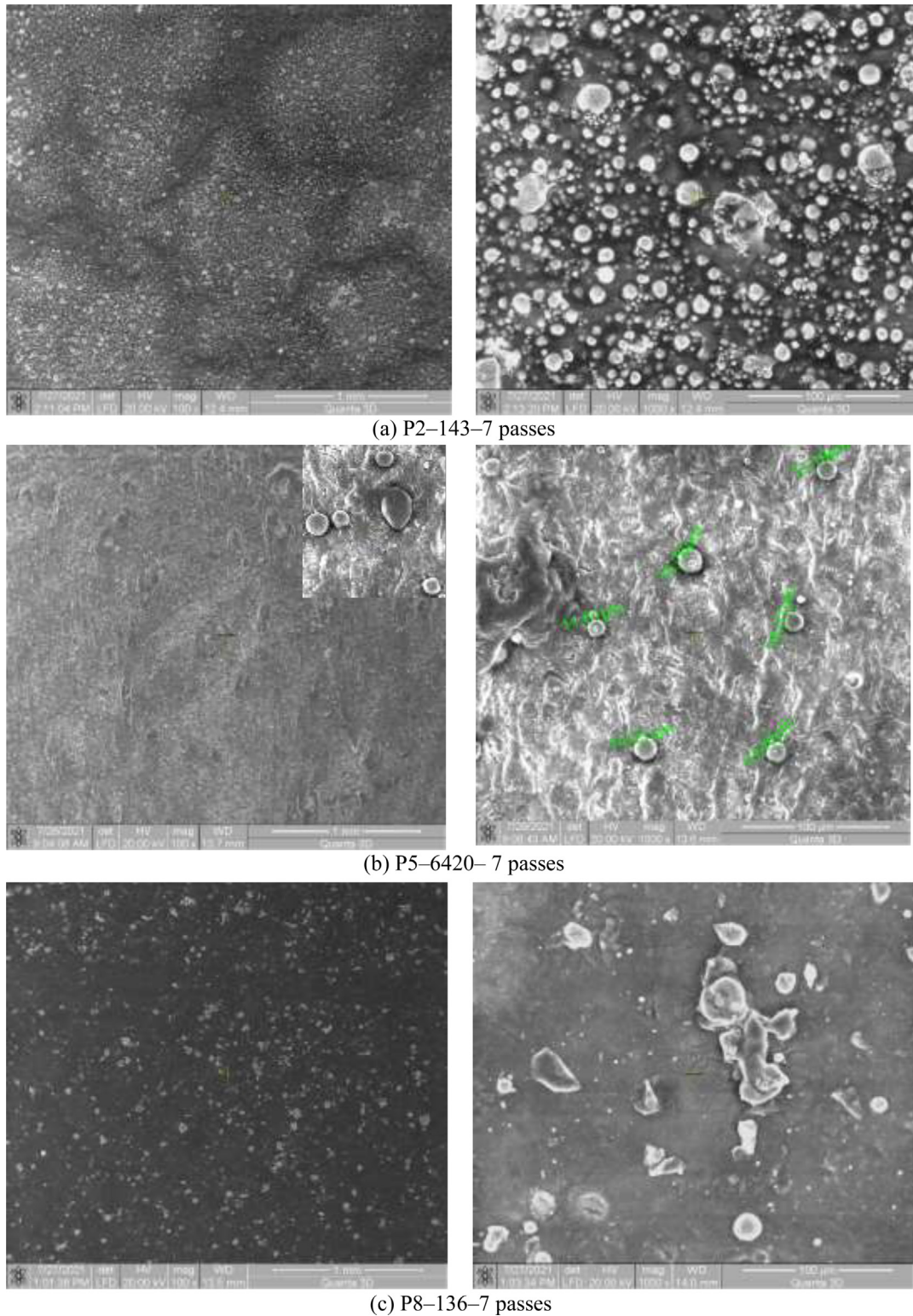


Fig. 3. Ceramic-coated sample – SEM analysis.

specific to the ceramic micro-powders. The identification of crystallization phases was achieved by a process of comparing the gathered data with existing scientific literature.

Figure 4 displays the phase diffractograms for P2-143-7 passes, P5-6420-7 passes, and P8-136-7 passes ceramic coatings. Two of the three samples have a crystalline structure (P2-143-7 passes, Fig. 4a and P8-136-7 passes,

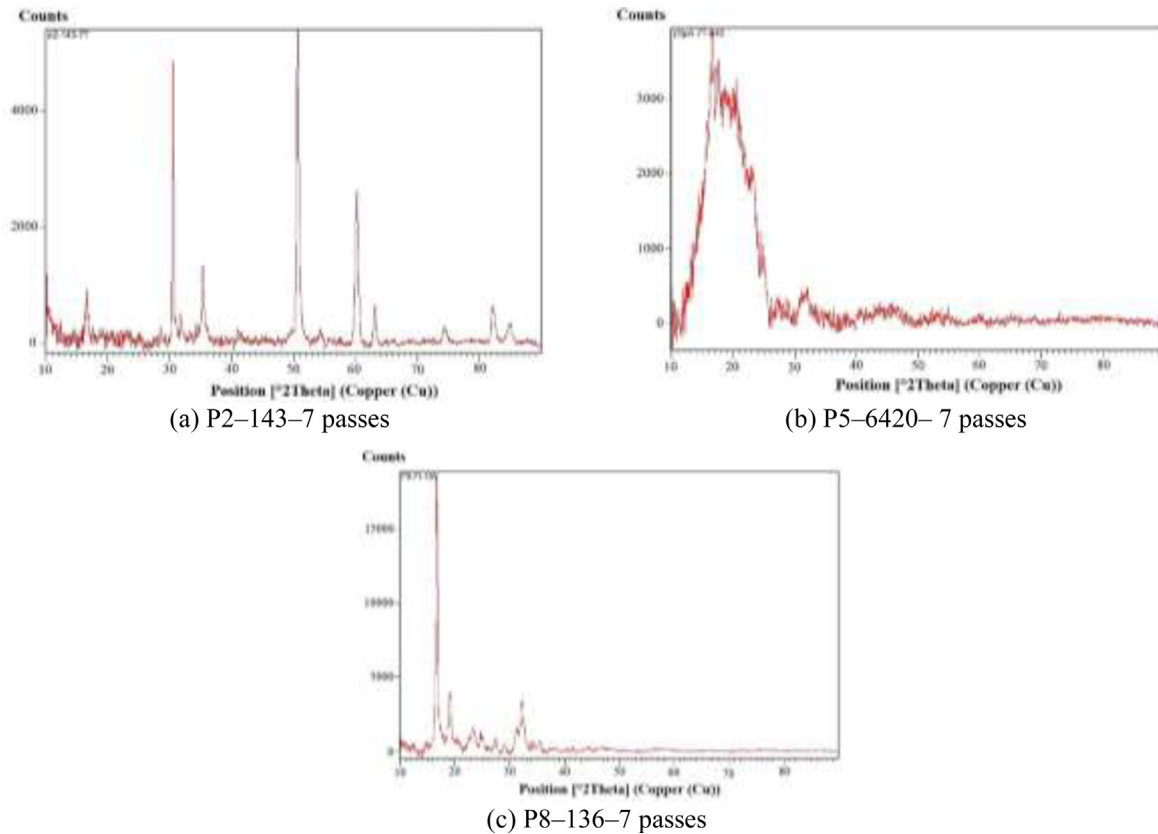


Fig. 4. Ceramic-coated sample XRD analysis.

Fig. 4c) with specific peaks, and the third (P5-6420-7 passes, Fig. 4b) has a semi-crystalline structure with small peaks of chromium oxide at four distinct 2θ angles, 24.25° , 33.39° , 35.88° , and 54.61° , respectively [70–72]. The prominent peak at 16.73° may be due to polylactic acid $((C_3H_4O_2)_n)$ in its chemical composition [73,74]. Because the deposited layer is very thin, the equipment detects one of the fundamental material elements – PLA.

The ceramic powder's high zirconium dioxide content makes the P2-143-7 passes sample crystalline. Thus, ZrO_2 has peaks at $2\theta = 31.14^\circ$, 38.43° , 60° , 82° , and 84.78° [69,74,75]. Titanium dioxide was associated with the peaks from 27.43° , 28.34° , 63.02° , and 74.41° angles [76,77]. Y_2O_3 crystallizes at the low angle from $2\theta = 43^\circ$ [78].

The polymer matrix, which contains polylactic acid (16.73°) and lignin (19.04°), causes diffraction maxima in the P8-136-7 passes sample [79,80]. Ceramic micro powder reflect diffraction angles as: Cr_2O_3 crystallisation maxima at 30.35° , 31.70° , 35.16° , 50.48° , and 54.12° [70–73,81]. SiO_2 microspheres have a peak at $2\theta = 22.5^\circ$ [82]. The compound has no further diffraction peaks. In Metco 136F micro powder, titanium dioxide (TiO_2) diffraction angles are 27.33° and 32.13° [76,77].

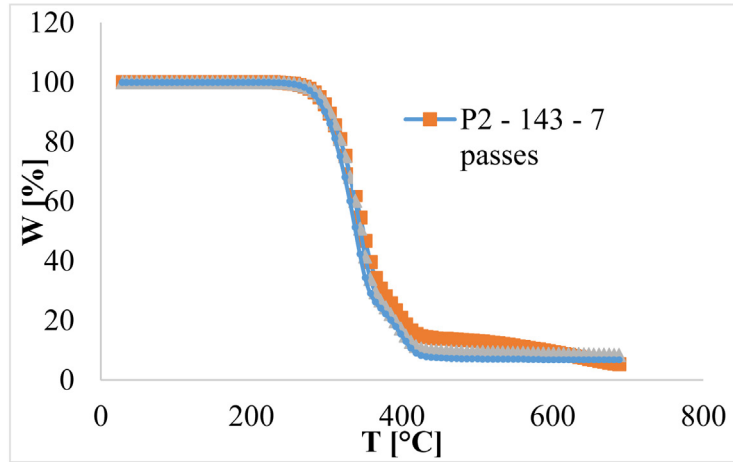
3.3 Thermogravimetric analysis

Evaluating the thermal stability of Arboblend V2 Nature samples that have been coated with ceramic powders is crucial. This knowledge is particularly important for

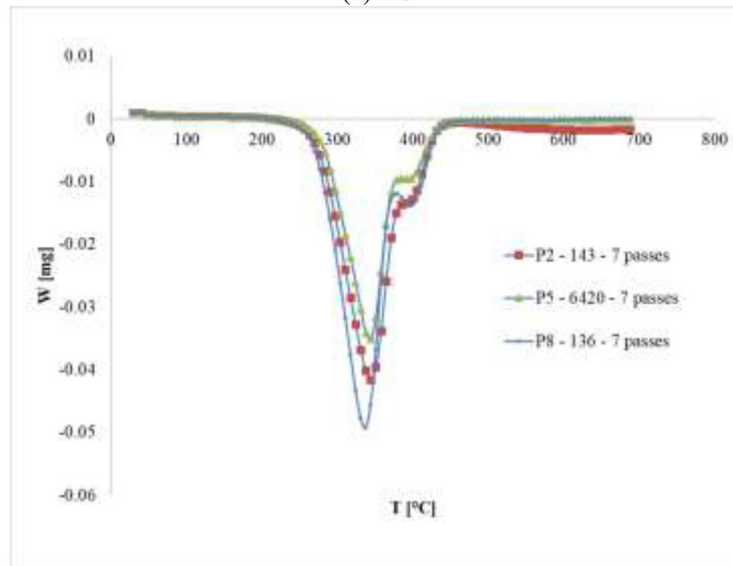
applications that involve operating under harsh conditions, where properties such as wear resistance and thermal resistance are required. Therefore, it is necessary to investigate the thermogravimetric behaviour of these samples. In Figure 5, a comparison is presented between the thermogravimetric (TG), derived thermogravimetric (DTG), and differential thermal (DTA) curves of the three samples that have been coated with ceramic layers produced through a series of seven consecutive passes.

Figure 5 presents the primary thermogravimetric properties of the P2-143-7 passes, P5-6420-7 passes, and P8-136-7 passes coated samples.

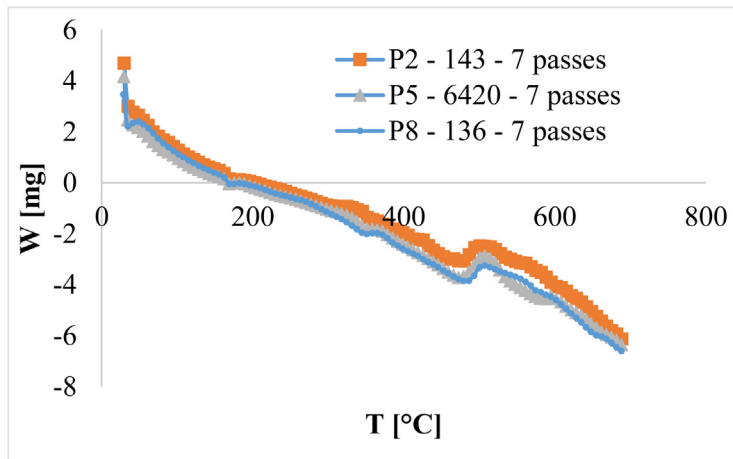
The experimental analysis of the three coated samples, each having distinct ceramic layers, reveals the occurrence of two distinct stages of decomposition. The initial stage, observed at a temperature of approximately $345^\circ C$, exhibits a substantial mass reduction exceeding 85%. This decomposition process is attributed to the structural deterioration of lignin, which serves as the fundamental component of the substrate material. This phase involves the synthesis of aromatic hydrocarbons, specifically guaiacyl-/syringyl-type and hydroxy-phenolic chemicals, among others [83]. The producer [63] has indicated that the analyzed biopolymer contains PLA as additional ingredient, which undergoes significant decomposition within the specified temperature range [83,84]. Based on the findings reported in the literature [85,86], it has been observed that both PLA and pure lignin undergo total degradation when exposed to temperatures up to $500^\circ C$.



(a) TG



(b) DTG



(c) DTA

Fig. 5. Thermogravimetric curves of the coated samples.

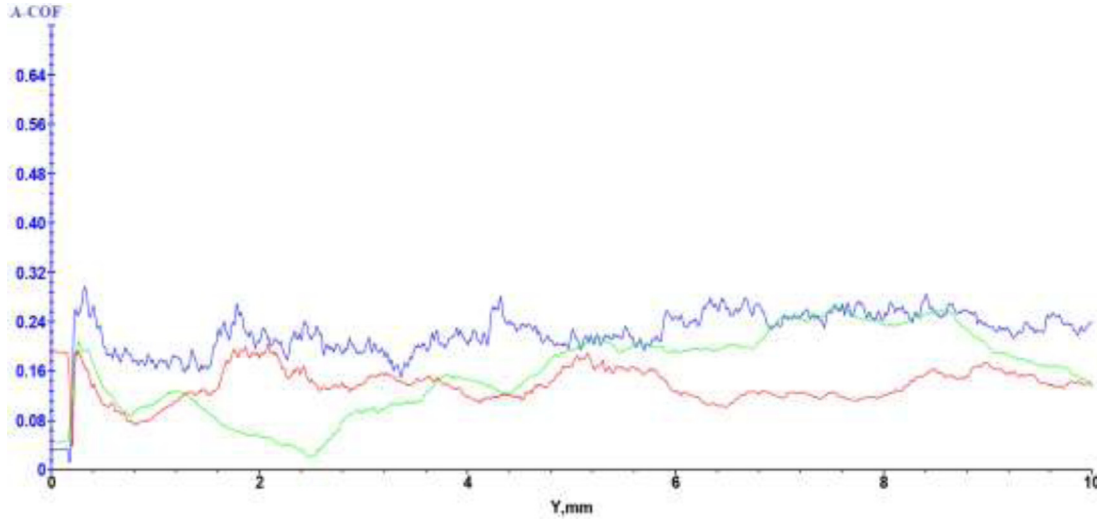


Fig. 6. Scratching behaviour of ceramic-coated samples: blue curve—P2-143-7 passes; green curve—P5-6420-7 passes; red curve—P8-136-7 passes.

The curves obtained from the thermogravimetric (TG) study were overlapped in order to emphasize their distinct behavior. It can be observed from Figure 5a that the thermal stability of the coated samples is comparable.

During the second stage, characterised by a T_{peak} of approximately 425°C, a modest reduction in mass, accounting for less than 10%, is observed. This reduction can be attributed to the thermal oxidation of the carbonic residue resulting from the pyrolysis of lignin and/or PLA, as well as other biodegradable components present in the Arboblend V2 Nature, such as binders introduced by the manufacturer (wax, resin, shellac etc.) [26]. At a temperature of 700°C, the presence of residual mass is seen, which varies depending on the type of ceramic powder utilized.

The sample P5-6420-7 passes have the largest percentage of residue, measuring 6.5%. This can be attributed to the presence of a greater quantity of micro particles compared to the P2-143-7 passes sample, where the deposition of ceramic powder is significantly lower.

The ceramic powders have not yet achieved their melting point, which is around 2500°C, at the end of the analytical temperature. Their working temperature ranges from 540°C (P5-6420-7 passes, P8-136-8 passes) to 980°C (P2-143-7 passes) [68]. Furthermore, it is quite probable that the residual mass includes inorganic chemicals that are present in the composition of the biopolymer [87].

In Figure 5c, the DTA curves reflect the melting temperature of Arboblend V2 Nature, which is observed to be at 169°C. This value closely aligns with the results obtained from calorimetric analysis of the substrate material (Arboblend V2 Nature), [88].

3.4 Scratch analysis

The objective of this study was to assess the adhesion properties of the ceramic coatings applied onto the surface of the Arboblend V2 Nature bio-polymeric material through the implementation of a scratch test.

The apparent coefficient of friction is calculated as, equation (2):

$$COF = \frac{F_f}{F_z} \quad (2)$$

where F_f is given by the horizontal force F_x that opposes the movement of the scraper blade through the material, equation (2):

$$F_f = (F_x^2 + F_y^2)^{0.5} \quad (3)$$

$F_y = 0$ (because the blade is moving in the x direction).

Upon examination of the curves depicted in Figure 6, it is evident that the blue curve—P2-143-7 passes, exhibits a very large number of peaks in comparison with the other 2 samples. This observation suggests that the adhesion between the deposited thin layer and the polymeric material is superior compared to the other two coatings. This enhanced adhesion can be attributed to the uniform and homogeny distribution of the micro-ceramic powder. The remaining two tests exhibit favorable scratching behavior, however the P5-6420-7 passes demonstrate greater A-COF values compared to the P8-136-7 passes.

A higher number of peaks observed in the fluctuation of the apparent friction coefficient indicates a stronger adhesion between the deposited layer and the polymeric substance.

In the case of the P5-6420-7 passes sample (green curve), a significant peak with high amplitude is observed at the end of the test. This occurrence can be attributed to the process of deposition granulation. There is a high likelihood that the cutting tool's tip (pin) has encountered an area of deposited material with a greater granulation.

The behavior of the samples injected from Arboblend V2 Nature and coated with ceramic micro-powders was observed during the 60-second testing period, as depicted in Figure 7.

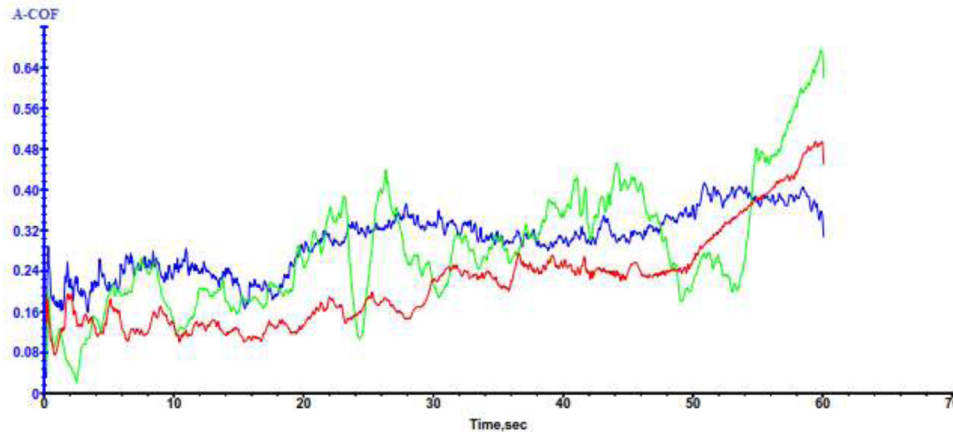


Fig. 7. A-COF fluctuation with test time for ceramic micro-powder-coated samples: (blue) P2-143-7 passes; (green) P5-6420-7 passes; (red) P8-136-7 passes.

In the case of sample P2-143-7 passes, there is a progressive rise in A-COF detected within the testing time but not very much. This increase could be attributed to the fact that a reduced amount of ceramic powder was deposited in front of the blade that performs the scratch. The separation of the micro-particles is to be expected because it is well known that they are brittle structures. But, the almost stabilized value of A-COF indicates good adhesion of the ceramic layer. The average value of A-COF was determined to be 0.30 ± 0.06 with a maximum of 0.41 achieved at second 51.

The P5-6420-7 sample exhibits a distinct behavior, as indicated the green curve, which contrasts significantly with the sample P2-143-7 passes. The observed variations in the measurements can be ascribed to the varying sizes (ranging from 9 to $30 \mu\text{m}$) of the micro-particles that make up the ceramic powder. The observed upward trend observed during the final 10 seconds of the testing period can be attributed to the detachment of ceramic micro-particles from the surface of the sample. This phenomenon resulted in a gradual rise in surface roughness over time. The sample's mean value of A-COF, specifically 0.34 ± 0.16 , with a maximum of 0.76 at the end of the test. So, exhibits the greatest value when compared to the mean values of the other two analyzed samples.

The test conducted for tribological analysis, referred to as P8-136-7 passes, successfully meets the required criteria, similar to the previous test. The test results indicate variations seen during the duration of the test, with the final coefficient of friction (A-COF) reaching a peak value of 0.49. The mean value of A-COF for the given sample is the smallest, namely 0.21 ± 0.09 . The recorded value closely approximates that of the injected samples and is not protected by a ceramic layer [89]. The observed similarity can be attributed to the non-uniform coating of the sample. The adhesion and incorporation of Chromia-Silica composite powder, as evidenced by the SEM images, exhibited a significantly low level. This characteristic was observed in both the P5-6420-7 passes and the P8-136-7 passes. The absence of deposition can be attributed to the

thermal characteristics of chromium oxide, which is present in a significant proportion in both the P5-6420-7 passes and P8-136-7 passes samples.

3.5 Microindentation test

In order to perform the microindentation test, three samples were subjected to testing for each variant of ceramic powder utilized in the coating of the Arboblend V2 Nature biopolymeric material. The purpose of doing many tests was to verify and establish the stability of the experiment. Figure 8 illustrates the progressive changes in force relative to the depth of penetration for the three samples under examination.

Based on the data acquired from micro-indentation analysis, it is shown that the P8-136-7 passes sample, while lacking a homogeneous coating, exhibits the most favorable micro hardness values ($0.16 \pm 0.00 \text{ GPa}$), with a maximum indentation depth of $54.42 \pm 1.18 \mu\text{m}$. In comparison to the zirconium-based ceramic coating (P2-143-7 passes), the chromium oxide coatings exhibit a greater level of hardness (P5-6420-7 passes, P8-136-7 passes) [64]. An additional rationale for the findings can be attributed to the decreased layer thickness resulting from the smaller dimensions of micro particles in P2-143-9 passes, as compared to the other two types of ceramic micro powders.

3.6 Dynamic mechanical analysis

The dynamic mechanical analysis (DMA) diagrams, which are obtained by conducting temperature scans, depict the changes in storage modulus (E') and internal friction (expressed as the ratio of loss modulus to storage modulus, $\tan \delta = E''/E'$), throughout a heating cycle. Figure 9 presents the variations of storage modulus and internal friction during the heating process.

As can be seen, samples P2-143-7 passes and P8-136-7 passes did not record results corresponding to this type of test because this type of analysis is performed on the

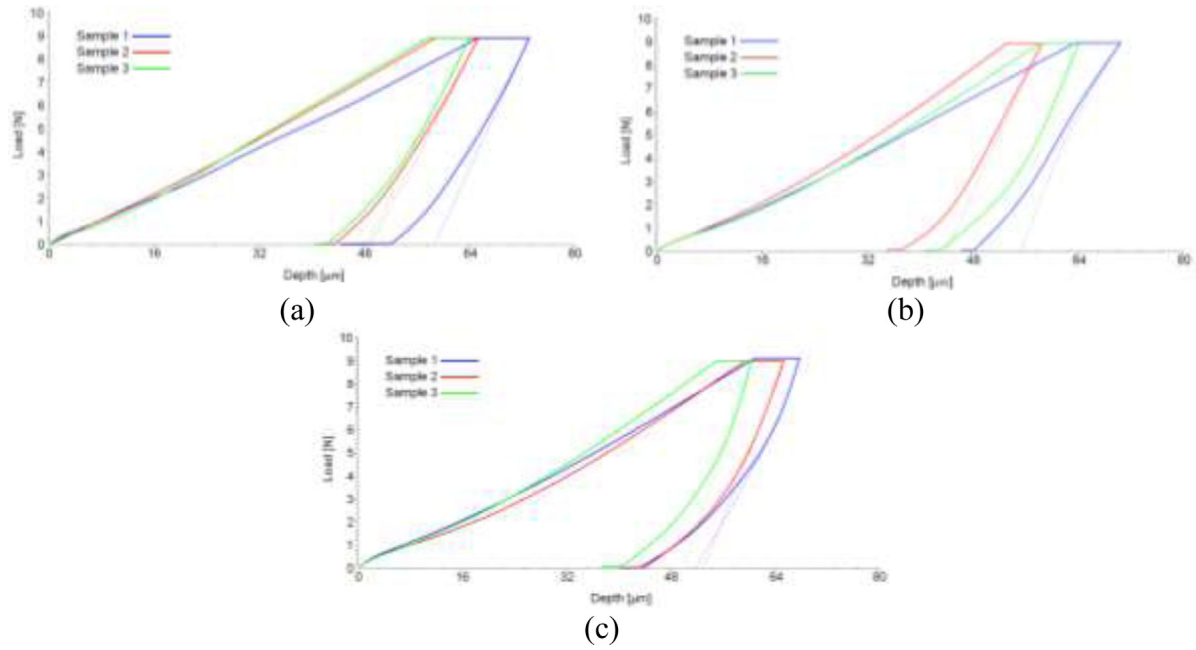


Fig. 8. Microindentation test results for: (a) P2-143-7 passes, (b) P5-6420-7 passes, (c) P8-136-7 passes.

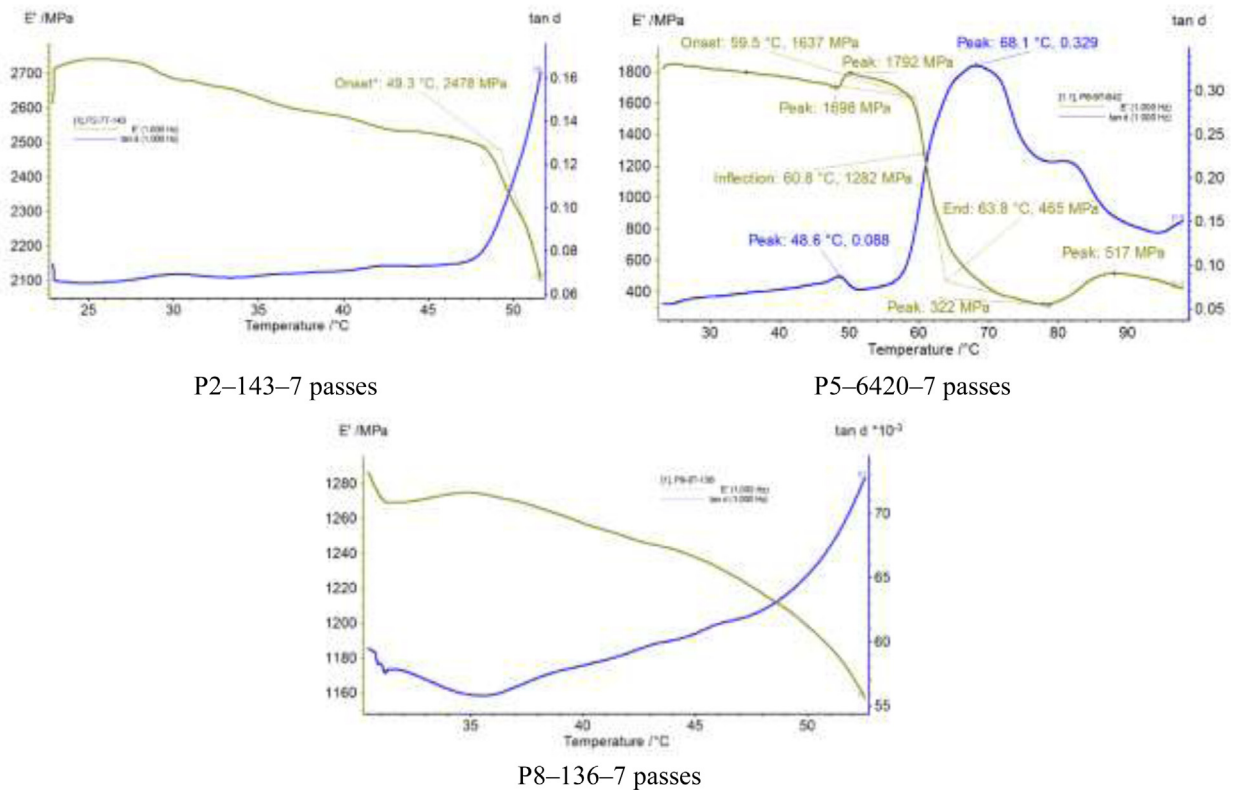


Fig. 9. DMA analysis of samples coated with ceramic powders.

surface of the samples at a micro level. Therefore, due to the hardness but also the fragility of the samples covered with ceramic powders, the mentioned samples yielded in the first seconds of the test (three-point bending method). Therefore, in what follows we will only analyze the graph for sample P5-6420-7 passes. Most likely, the testing on this sample could be done because the deposition of the powder on this sample was not exactly uniform and the result obtained refers more to the characteristics of the substrate and not of the coating.

The elastic response of the P5-6420-7 passes sample was 1792MPa, which was recorded at 51°C. Damping occurred within the 68.1°C temperature range and has a value of 0.329.

Upon examining the shape of the storage modulus and damping curves associated with the P5-6420-7 passes coated sample, it was seen that the sample had a distinct and concentrated peak in the tan curve. This finding suggests that the material displayed homogeneity and behaved as a cohesive entity. The relationship between the shape of damping peaks and the crosslink density of the material was verified through a comprehensive analysis involving thermal and structural assessments. In the case when the crosslink density is high, the material exhibits homogeneity, resulting in a sharp damping peak.

Conversely, when the crosslink density is low, the material displays broad peaks. The aforementioned observation is further supported by the distinctive shape of the E' curve, characterized by a rapid and nearly vertical decline. This indicates that a phase transition took place within a brief time period and temperature range. Nevertheless, it is imperative not to overlook the minor peaks observed on the internal friction curve. These peaks indicate the occurrence of multiple sequential phase transformations, likely attributed to processes such as water evaporation during polymer heating or decomposition of matrix and additives present in the material's chemical composition.

4 Conclusions

Bio-based polymers for coatings are of interest to academics worldwide. The purpose of multilayer coatings, including ceramic, metal, and reinforcements, is to improve the underlying material. This upgrade improves the material's performance in some industrial applications and may allow it to replace metals. Coatings improve samples' wear resistance, hardness, and thermal resilience. These coatings are ideal for harsh operational situations, such as the automotive industry.

The melting point of the base material [35,61] increases from 172°C to 174°C, depending on layer thickness. Ceramic powder thickness depends on micro particle size. Thus, micro particle size positively correlates with coated material thermal resistance.

No significant changes were seen in thermal degradation temperature ranges or mass loss. The observed dissimilarities were related to ceramic micro particle dimensions. SEM surface analysis showed that the zirconium oxide composite powder had a satisfactory level

of homogeneity in micro particle integration. The adhesion between the polymer matrix and the other two ceramic micro powders was found to be insufficient, possibly due to their higher working temperature (2435°C) than the melting point of the polymer matrix (170°C). X-ray diffraction (XRD) revealed tiny ceramic layers. These materials are hard because of their crystalline or semi-crystalline structure, making them ideal for hardness-intensive applications. The micro-indentation test revealed that sample P8-136-7 passes, coated with $\text{Cr}_2\text{O}_3\text{-xSiO}_2\text{-yTiO}_2$, had the maximum hardness value of $0.16 \pm 0.00\text{GPa}$. The deposition was non-uniform based on SEM and scratch studies. This is because chromium oxide, silicon oxide, and titanium oxide microparticles adhere less than the other two. The average apparent friction coefficient (A-COF) was 0.34 ± 0.16 .

The data showed excellent chemical bonding between the thin ceramic layers and Arboblend V2 Nature bio-based polymer. These coated materials are appropriate for use in different industrial applications due to their mechanical behaviour. These can replace non-biodegradable polymeric automotive and electronics parts like phone covers, housings, worm wheels, and wiper systems.

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