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PAPER

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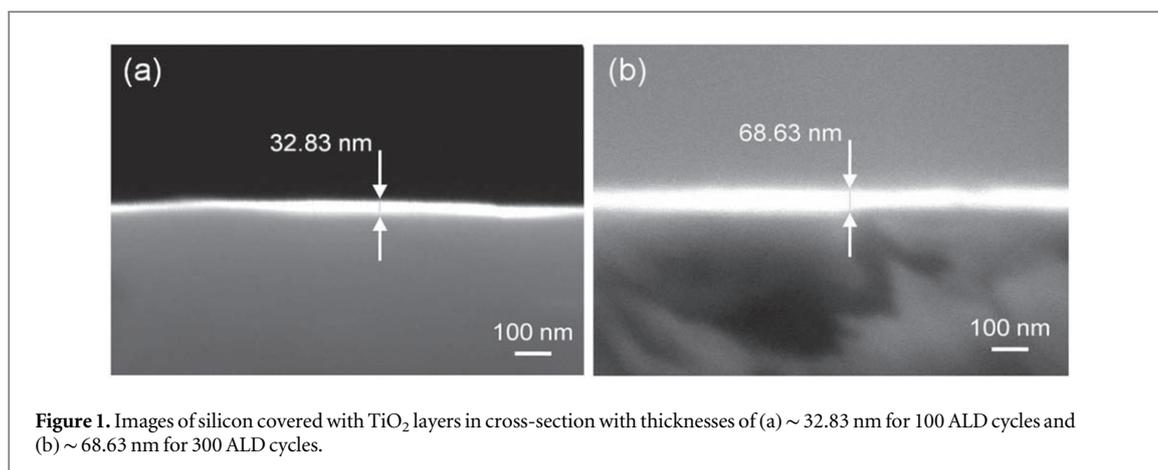
Abstract

Atomic layer deposition (ALD) was used to coat a polyethylene terephthalate (PET) polymer substrate with TiO₂ film. The TiO₂ was grown onto the surface with better film coverage by using thicker ALD deposition. Further evaluation on the coated substrates indicated that the reactive sites of –C=O that existed on PET surface played a significant contribution to facilitating the initial ALD growth of the TiO₂ thin film. The chemical composition of the coated substrates was characterised using energy dispersive X-ray spectroscopy, which showed that increasing the TiO₂ film thickness increased the Ti element content. Two growth mechanisms, namely, diffusion growth and direct coordination through the precursor coordination with surface reactive sites of –C=O with product release occurred simultaneously in the initial growth of TiO₂ coating onto PET polymer by ALD. The surface alteration of the coated ALD was characterised by Fourier transform infrared spectroscopy, which showed that the hydroxyl –OH groups emerged in the TiO₂ ALD film.

1. Introduction

ALD can produce highly uniform and conformal thin films on non-uniform high aspect ratio and 3D porous surfaces at low processing temperatures. The ALD process has become popular in a variety of fields, including semiconductors, displays, flexible electronics and catalysis [1–4]. ALD is a chemical deposition process for gaseous component adsorption on solid substrates in which two complementary self-limiting first- and second-half reactions are performed successively to generate a monolayer [5–7]. The ability of a thin film to grow at low temperature is one of the most important issues in the deposition on soft materials such as polyethylene terephthalate (PET) polymer. Some characteristics of polymers, such as lightness, resilience, flexibility and transparency, are advantageous in various applications, including packaging, microfabricated devices and flexible electronics [8, 9]. Brittleness is a common property of inorganic materials. However, the diffusion growth of inorganic materials on polymers enables the maintenance of polymer flexibility without cracking [10, 11]. Film growth on polymers varies depending on temperatures, the presence of polar groups, the growth of materials and the dosing condition of precursors.

The TiO₂-coated PET substrates are potentially employed as the clinical implanted materials in improving the biocompatibility of the polymer materials [12], UV-screening agent as its ability to absorb in the UV spectrum [13], optical coating [14], flexible solar cell [15–17], moisture barrier layer [18], self-cleaning surface [19], and flexible sensors [20]. Additionally, in terms of actual use in the future, the TiO₂ ALD films on soft material substrates, particularly polymer surface, would be crucial for their widespread use in industrial products. This study investigated the growth ability of TiO₂ film on PET polymer surface through the ALD process. The reactive surface groups of –C=O promoted the initial growth. Meanwhile, as the polymer structure



consisted of polymer chains, the diffusion growth mechanism also occurred in the first several ALD cycles. Results indicated that TiO₂ film growth onto the PET polymer surface by ALD occurred via two mechanisms.

2. Experimental method

Silicon (Si) and PET films were used as substrates. The PET was cleaned in an ultrasonic machine (20 min) at a temperature and power of 35 °C and 80 W, and then dried in a vacuum oven (1 h) at 45 °C. Afterwards, TiO₂ was deposited onto the substrates by ALD with various thickness. The tetrakis (dimethylamido) titanium (TDMAT) and H₂O precursors were exposed for 20 and 30 milliseconds, with purge durations of 15 and 18 s, respectively. The deposition temperature was set at 120 °C, and the thickness values were in the range of 100–600 ALD cycles. The carrier and purge gases were both argon gas at a flow rate of 20 sccm.

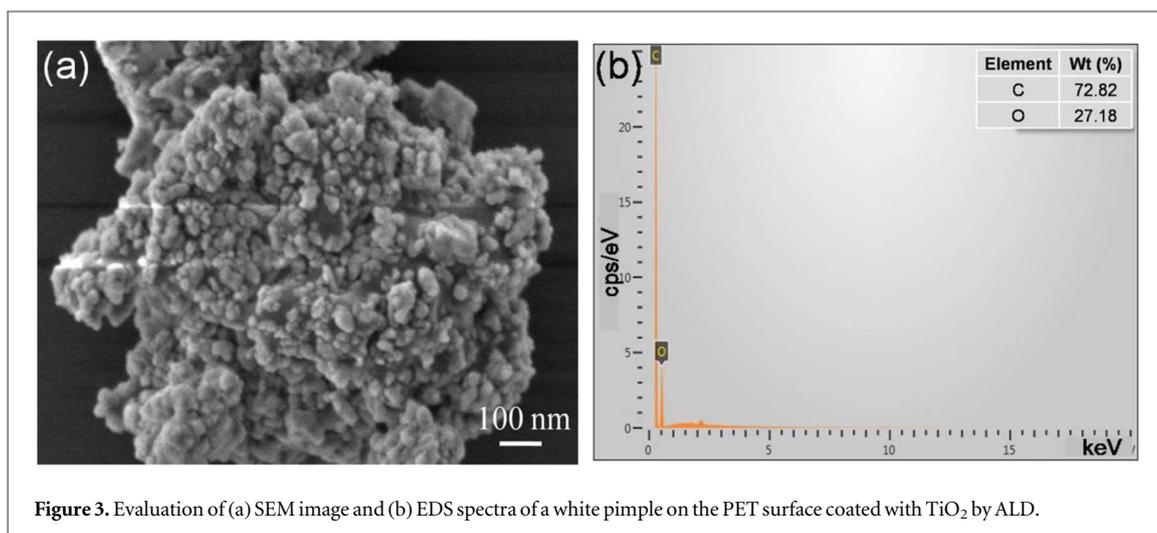
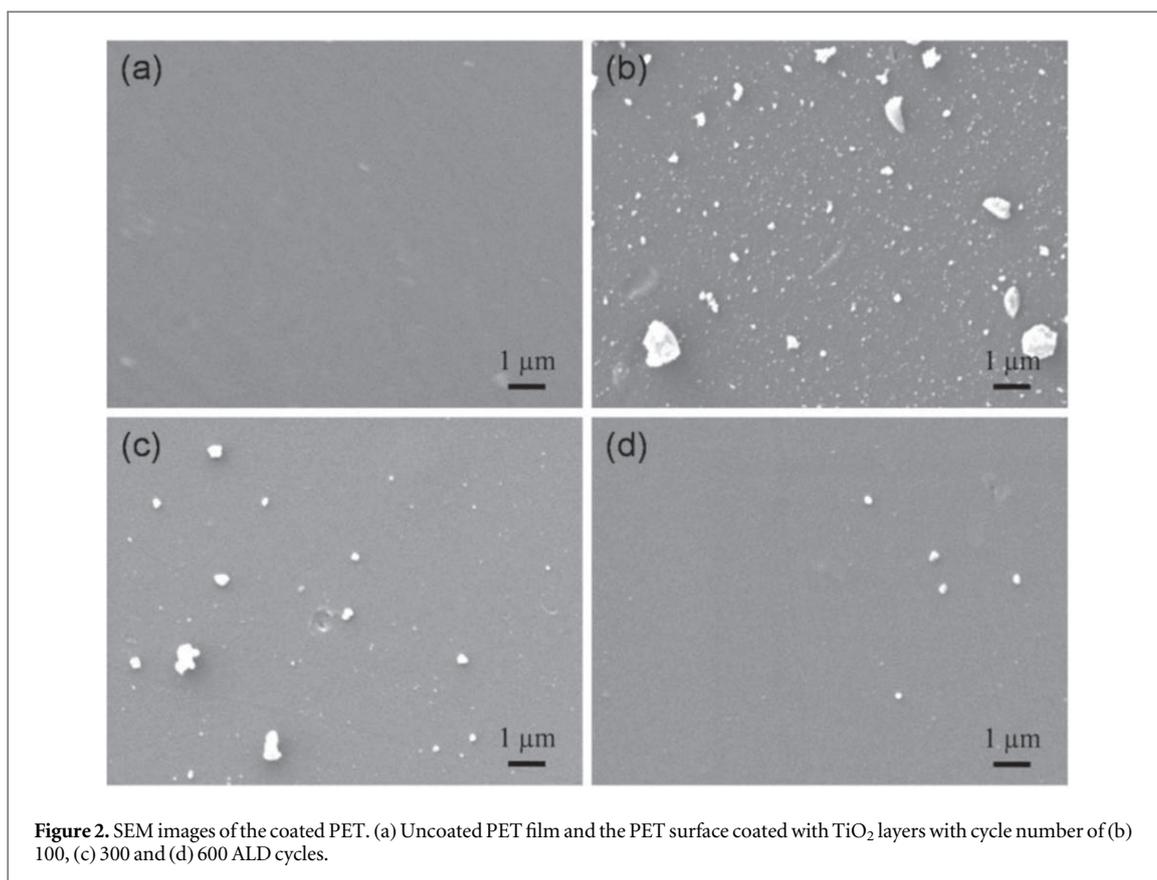
Using a high-resolution scanning electron microscope, a cross-sectional image of the coated silicon and a surface view of the coated PET were produced (JEOL, JSM-IT300). Energy dispersive X-ray spectroscopy (EDS) was used to characterise the coated substrate's elements. Thermo Nicolet Nexus 670 (Thermo Scientific, Waltham, MA, USA) and Smart iTR (diamond ATR, Thermo Scientific) spectral range of 600–4,000 cm⁻¹ with a resolution of 3.81 cm⁻¹ was used to measure the films' Fourier transform infrared (FTIR) spectra.

3. Results and Discussion

After 100 and 300 cycles, cross-sectional SEM pictures of an ALD-produced Si substrate coated with TiO₂ films are shown in figure 1. The images for 100 and 300 ALD cycles showed smooth surfaces with thicknesses of ~ 32.83 and ~ 68.63 nm, respectively. It indicates that the ALD process parameters can be used to deposit TiO₂ layer onto PET and Si surfaces as both substrates are coated simultaneously at the same time and process in the ALD reactor.

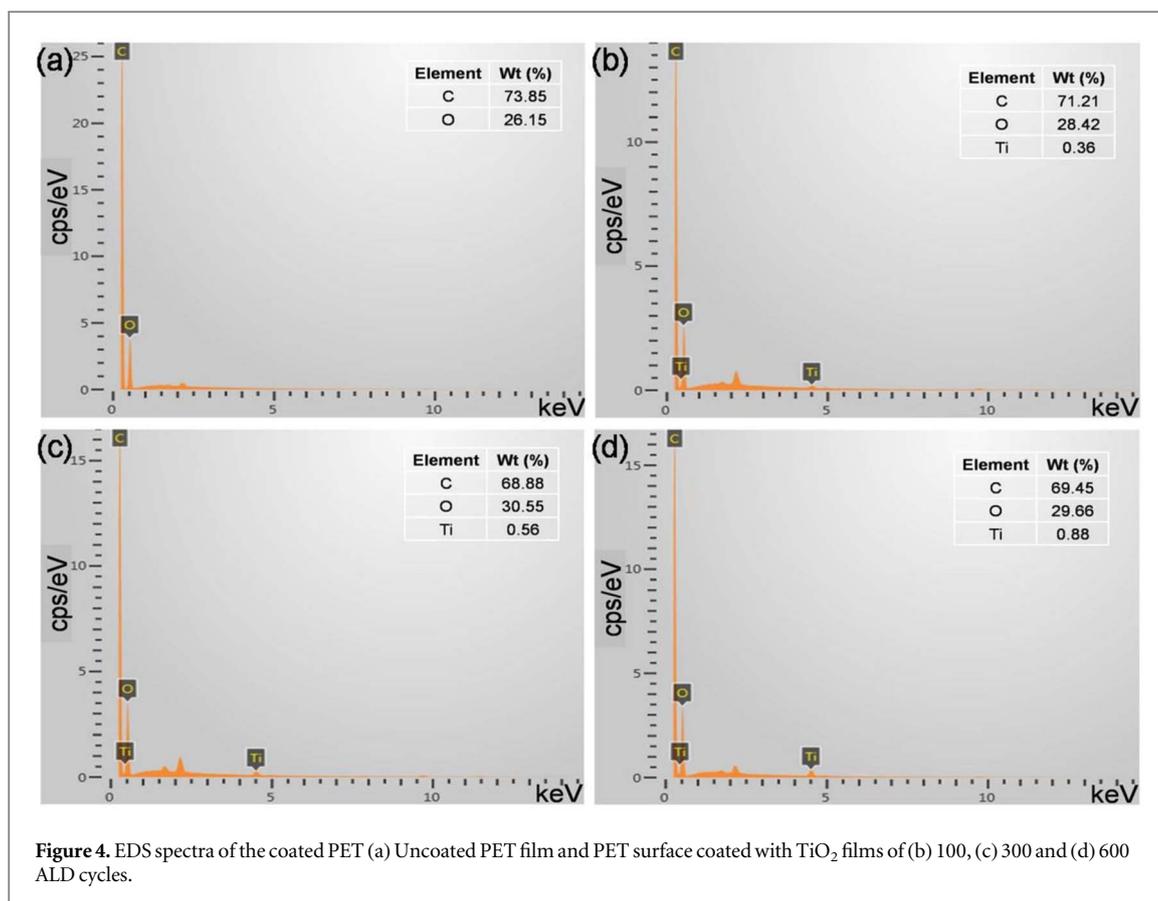
Figures 2(a)–(d) show SEM images of untreated PET and PET coated with TiO₂ films by ALD at 100–600 ALD cycles. Figure 2(a) shows that the PET surface without the ALD film has a smooth morphology. The presence of ALD layer led to the emergence of pimples on PET surface coated with TiO₂ film with a thickness range of 100–600 cycles by ALD, as shown in figures 2(b)–(d). The number of pimples decreased with increasing TiO₂ thickness (figures 2(b)–(d)). This result indicated that the increase of the number of ALD cycles improved the ALD layer with better film coverage, as shown by the reduction of pimple formation and the increased smoothness of the surface. The PET coated with TiO₂ film with the highest coverage had a coating thickness of 600 ALD cycles (figure 2(d)). These findings are consistent with our previous research, in which the presence of ALD film reduced surface roughness. Surface roughness dropped significantly on ALD-deposited surfaces with better film coverage [21].

A polymer is a huge molecule or macromolecule constructed of numerous subunits, while a macromolecule consists of a structure made up of multiple repeating units [22]. Polymers, like PET, are composed of many polymer chains with microporous structures that contain space between the polymer chains. The emergence of pimples is most likely due to the influence of the early growth in the first few cycles via the diffusion growth process, which can break the chain of polymer backbone. As the pimples grow and expand to the surface's edge, the gap between the polymer chains in the pimples becomes looser than on a regular PET surface. These phenomena may explain why the thinner ALD film obtained with fewer ALD cycles still lacks a coverage layer, and why the low number of ALD cycles are insufficient to fill the space between polymer chains, resulting in a



relatively higher surface roughness. Increasing the number of ALD cycles to generate a thicker ALD film is required to improve film coverage and reduce surface roughness. The TiO₂ nucleation clusters will fill the space between polymer chains as the cycle number increases, finally coalescing and closing the space to be continuous. Meanwhile, the presence of C=O groups on the PET surface contributes to the facilitation of ALD growth during the first few ALD cycles in order to create the initial closed ALD layer. After the space between the polymer chains is completely closed, the TDMAT and H₂O exposures enable to grow the TiO₂ films normally since the functional -OH groups on the TiO₂ surface have been created.

The pimples that emerged on PET polymer coated with TiO₂ ALD layer are shown in figures 2(b)–(d). The chemical composition evaluation was conducted by using EDS, as shown in figure 3(b). Figure 3 shows the SEM image (figure 3(a)) and the EDS-spectra (figure 3(b)) and focuses on the pimples on the PET polymer coated with TiO₂ film by ALD. A white pimple was shown at high magnification. It emerged on the PET surface during the

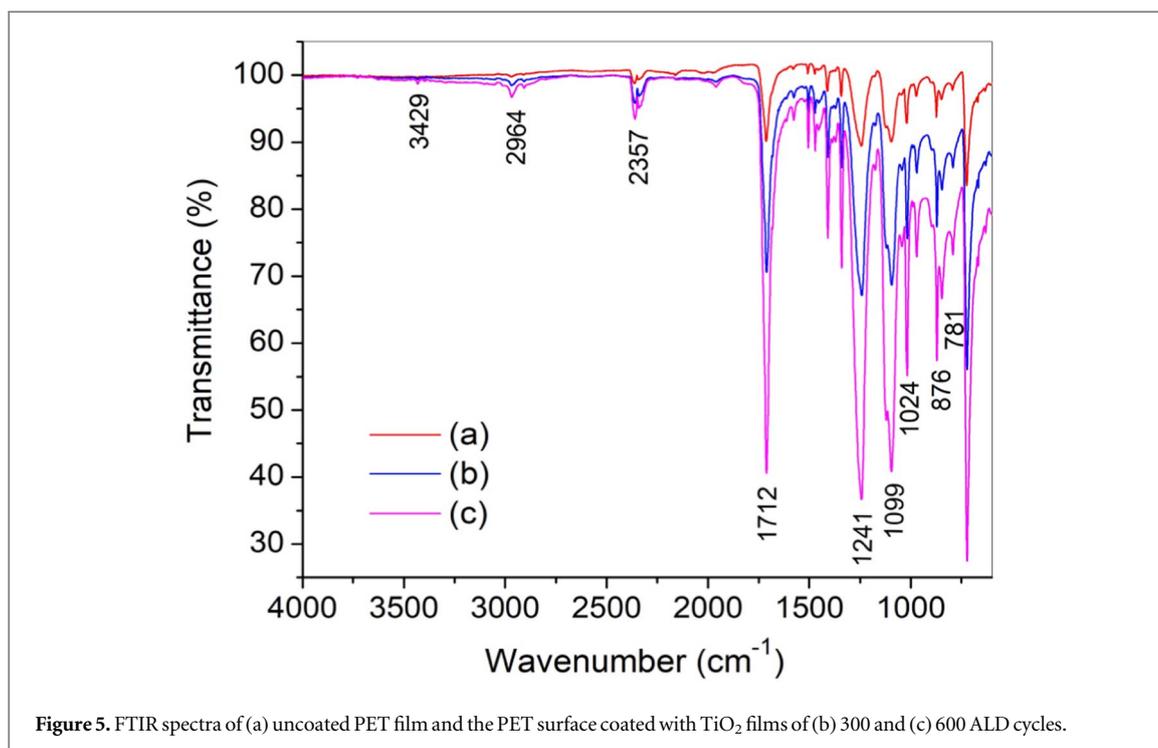


ALD deposition process (figure 3(a)). The chemical composition of the pimple (figure 3(a)) included carbon and oxygen at quantities of 72.82% and 27.18%, respectively, as shown in the EDS spectra.

PET is a semi-crystalline polymer having surface groups that are responsive to ALD nucleation of C=O polar groups [23, 24], allowing the polymer backbone to show Lewis base properties and making the polymer surface reactive during precursor exposure, resulting in a strong Lewis acid [25–27]. By exposing the carbonyl site of C=O, coordinating with the precursor (i.e. TDMAT; Lewis acid to form an oxygen-titanium-(N(CH₃)₂)₃) becomes possible [28–31]. A reaction that leads to the formation of a covalent titanium-oxygen bond can occur in the ALD growth of TiO₂ onto the PET surface via N(CH₃)₂ migration from TDMAT to the electrophilic carbon site of the PET polymer [26, 32–34].

The TDMAT reaction with the carbonyl forming acetal groups and the transfer of N(CH₃)₂ to the carbon of the polymer backbone eliminates the carbonyl stretch of C=O [26, 35]. After H₂O exposure, the acetal unit of N(CH₃)₂ likely reacted with water, forming –OH and releasing NH(CH₃)₂. During the reaction, the generated functional –OH groups can be filled by the subsequent precursor exposure. Using this growth method, the carbonyl site of C=O can be replaced by related components of the TiO₂ ALD material. The C=O groups are most commonly found in the polymer subsurface. The interaction of the precursor and the carbonyl site can break hydrogen bonding, causing the polymer chain framework to open, allowing the precursor molecule to diffuse further into the subsurface region and affecting the smoothness of the coated surface [21, 36–39]. During the diffusion process, molecular weight at the near surface of the PET substrate may decrease because of the breaking of the polymer chains. This abovementioned phenomenon may be indicated by the emergence of the pimples on the PET surface coated with TiO₂ film by ALD. The decrease in molecular weight enabled the further promotion of the initial growth due to the enhancement of the number of reactive sites on the PET polymer surface in which the first coordination of the carbonyl site of the PET polymer with precursor (i.e., tetrakis (dimethylamido) titanium) can benefit from the new polar groups emerging that came from the breaking of polymer chains. Surface morphology before and after TiO₂ ALD coating can reveal these typical ALD growth mechanisms.

The EDS spectra of PET without coating (figure 4(a)) and PET polymer coated with TiO₂ film by ALD with thicknesses of 100–600 ALD cycles (figures 4(b)–(d)) are shown in figure 4. The spectrum of bare PET was found to be dominated by C and O elements, with 73.85% and 26.15 percent of the total weight, respectively. The presence of ALD coating with various thicknesses onto the PET polymer surface led to the reduction of the C elements to 71.21%, 68.88% and 69.45% for the PET polymer coated TiO₂ films with thicknesses of 100, 300 and



600 ALD cycles, respectively. This finding implied that polymer chains formed from C element were broken down and reacted with the ALD precursors during the ALD reaction mechanism process. Moreover, the ALD growth mechanism of TiO₂ onto the PET surface started with the diffusion growth mechanism. The presence of ALD coating increased the O quantities; O content increased to 28.42%, 30.55% and 29.66% in weight for PET polymer coated with TiO₂ films with thicknesses of 100, 300 and 600 ALD cycles, respectively. This finding indicated that the increase of the end chain with O atoms due to the broken chain of polymer backbone was obtained during the diffusion growth mechanism. The emergence of a new end chain with O atoms resulted in the reaction with Ti of TDMAT, which previously coordinated with the carbonyl site of C=O by transferring a N(CH₂)₂ to the polymer backbone. Some of the achieved end chains with O atoms acted as reactive sites, which in turn reacted with the subsequent TDMAT exposure. The oxygen concentration on the PET surface changed after the TiO₂ film was deposited using ALD, and this is ascribed to the fact that the oxygen density of the O in the Ti-O bonds on the uppermost surface of the TiO₂ ALD layer is larger than the oxygen density of the O in the C=O groups on the uncoated PET surface. This phenomenon is consistent with our previous studies employing ALD on PET that was covered with oxide thin films, which demonstrates that the oxygen concentration rises as a result of these films presence [21, 40]. Figures 4(b)–(d) clearly show that the Ti element content improved with increasing thickness of the TiO₂ ALD coating. Ti element contents were 0.36%, 0.56% and 0.88% for the PET polymers coated with TiO₂ with thickness values of 100, 300 and 600 ALD cycles, respectively.

Figure 5 shows the infrared spectra of PET surface with and without TiO₂ ALD coating within the range from 600 to 4000 cm⁻¹. Chemical structure alteration of PET surface due to the presence of TiO₂ ALD layer can be observed through the peak change of infrared band in the region. Low-molecular-weight compounds are produced when the ALD growth is via diffusion mechanism; the formed nucleation from the precursor reaction may collide with the near surface polymer structure to cause the scission of the polymer chains [41]. The deposited TiO₂ ALD film was responsible for the alteration of the FTIR spectra (figure 5). These spectral changes were clearly visible in the bands of 781, 876, 1024, 1099, 1241, 1712, 2357, 2964 and 3429 cm⁻¹. Among these bands, the absorption peak at 3429 cm⁻¹ corresponded with the hydroxyl group (–OH) [40, 42, 43], which played an important role in the ALD growth after the surface was covered by the coalescence of the TiO₂ ALD nucleation. The amplitude of the transmittance peak at 3429 cm⁻¹ was slightly increased by presence the TiO₂ ALD film, and it was further enhanced with increasing film thickness. This finding suggested the elevated density of hydroxyl–OH groups on the surface of the TiO₂ film with a thickness of 600 ALD cycles.

4. Conclusions

ALD was used to successfully deposit TiO₂ coating onto the PET polymer substrate, as indicated by the alteration of the surface appearance and chemical composition. By increasing the TiO₂ film thickness, a deposited ALD

layer with better film coverage was achieved, as shown by the reduction of the number of pimples. In general, the presence of the TiO₂ ALD layer on the PET substrate led to the increase in oxygen element quantity. The content was 26.15% on the uncoated PET. The content increased to 28.42%, 30.55% and 29.66% on the PET substrate coated with TiO₂ film with 100, 300 and 600 ALD cycles, respectively. Meanwhile, the Ti element content increased with increasing thickness of the TiO₂ ALD layer, i.e., 0.36%, 0.56% and 0.88% for the PET substrate coated with TiO₂ with thickness values of 100, 300 and 600 ALD cycles, respectively. The presence of TiO₂ ALD led to the formation of hydroxyl –OH groups, which increased in quantity on the PET surface coated with thicker TiO₂ film by ALD. Study of the ALD growth mechanisms on PET polymer surfaces with microporous characteristics, owing to the fact that the substance was created from a network of polymer chains with spaces between them, with typical reactive sites of –C=O groups can encourage the exploration of many different ALD applications with a variety of ALD materials on both flat and complex substrates due to the ability of the ALD process to grow thin films conformally onto the substrates of non-uniform and three-dimensionally porous structure surfaces like polyurethane and polyimide porous sponges.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Declarations

Edy Riyanto is the main contributor, the other authors are co-author. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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