MALAYSIAN JOURNAL OF MICROSCOPY

Journal homepage: https://malaysianjournalofmicroscopy.org

RESEARCH ARTICLE

GROWTH MECHANISM OF DIAMOND-LIKE CARBON FLAKES REINFORCED OXIDE COATING ON AA2017 ALUMINIUM ALLOY

Nur Adilah Mohd Rawian[1], Shahira Liza[1,*], Noor Ayuma Mat Tahir[1], Kanao Fukuda[1], Yazid Yaakob[2], Hiroki Akasaka[3]

[1]Tribology and Precision Machining i-Kohza, Malaysia-Japan Institute of Technology, Universiti Teknologi Malaysia, Kuala Lumpur, Malaysia

[2]Halal Products Research Institute, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia

[3]Department of Mechanical Engineering, School of Engineering, Institute of Science Tokyo, 2-12-1, O-okayama, Meguro-ku, Tokyo 152-8552, Japan

Abstract. The incorporation of diamond-like carbon (DLC) flakes in the oxide coating significantly reduced the surface defect and enhanced the microhardness of the composite oxide coating. However, the growth mechanism for type III disordered porous oxide coating and the crack reduction mechanism when incorporating DLC flakes in the oxide coating remains unclear. The study aims to clarify the growth mechanism by examining the oxide coating with and without incorporating DLC flakes at different anodizing times. Anodizing of the aluminium substrate (AA2017-T4) in 20 wt.% sulphuric acid (H_2SO_4) with a constant current of 2 A and 15 V initial applied voltage at the anodizing time of 2, 4, 6, 8, 10, 20, 30, and 60 minutes was performed. The result revealed that the disordered pores formed at 20 minutes of anodizing time due to fluctuating voltage when anodizing time increased. An initial increase in surface roughness was observed, reaching up to 7 µm at 8 minutes, followed by a sharp decrease to 1.8 µm at 10 minutes. Subsequently, the surface roughness increased again between 20 and 60 minutes of anodizing, ranging from 2.5 µm to 6.5 µm. The incorporation of DLC flakes in the oxide coating significantly reduced the formation of cracks and prevented them from propagating by occupying the pores. In addition, the DLC flakes also act as a physical barrier that hinders the dissolution of Al^{3+} into the electrolyte, which can cause further weakness in the oxide coating if the size of the pores increases. This study managed to reduce the knowledge gaps for the crack reduction mechanism of disordered porous oxide coating.

Keywords: Diamond like carbon, anodizing, surface analysis.

Article Info

Received 5 March 2025
Accepted 5 May 2025
Published 2 June 2025

***Corresponding author: shahiraliza@utm.my**

ISSN: 1823-7010, eISSN: 2600-7444

1. INTRODUCTION

Anodizing is an electrochemical process used to grow and thicken the oxide coatings on the surface of aluminium or other metals. The oxide coatings grow on the aluminium anode when immersed in the electrolyte and connected to the power source. Consequently, the aluminium anode is gradually consumed as the oxide continues to grow. The anodizing process on an aluminium alloy substrate can fulfil several needs, such as corrosion and wear resistance, and enhance its visual appeal, which depends on the working conditions and the purpose. This procedure can be classified into two distinct categories: mild anodizing and hard anodizing. Each category possesses distinctive properties that are tailored to meet specific application requirements. The mild anodizing process forms a self-ordered nanoporous structure with a coating thickness ranging from 8 to 10 µm [1]. Hard anodizing or also known as Type III anodic oxide coating, in contrast, results in thickness ranging from 25 to 125 µm, characterized by a micro-size pore structure [1,2].

Hard anodizing is used for protective or wear applications because it produces hard and thick oxide coating compared to mild anodizing. However, a few issues must be considered regarding the hard anodizing process of heat-treatable aluminium alloy (2XXX, 6XXX, and/or 7XXX series). These types of aluminium alloys often consist of magnesium (Mg) combined with one or more elements, such as copper (Cu), silicon (Si), iron (Fe), and/or manganese (Mn), to create intermetallic phases like Al_2CuMg, Al_7CuFe, and AlCuFeMnSi [3]. The existence of intermetallic phases on aluminium alloy hindered the growth of oxide coating and resulted in uneven surface topography with the formation of microcracks [4,5]. Additionally, the anodizing process induces oxygen evolution activity, which aids the formation of a porous structure in the oxide coating [6]. These notable surface defects and uneven oxide coatings with microcracks and micropores account for poor mechanical and tribological performance when used in sliding applications.

Over the past several years, various types of research on composite oxide coatings have been conducted on anodized aluminium oxide (Al_2O_3) to improve its mechanical and tribological performance. The incorporation of reinforcement particles into the oxide coating has a considerable impact on the reduction of pore dimensions. This is because the particles fill the porous structure, which in turn delays the initiation of microcracks [7].

However, the growth mechanism for Type III anodic oxide coating and the crack reduction mechanism when incorporating the particles remain areas with significant knowledge gaps. Previous studies found that incorporating reinforcing particles inside the Al_2O_3 coating helps improve surface defect and wear resistance. Many researchers have attempted to propose the crack mechanism and crack reduction mechanism of composite self-ordered Al_2O_3 coating; however, the crack reduction mechanism when incorporating the reinforcement particles inside the disordered pores of Al_2O_3 coating remains unclear. Thus, this research attempts to study the crack reduction mechanism of Al_2O_3 coating with disordering pores when incorporated with diamond like carbon (DLC) flakes. Our previous study utilized DLC-deposited on copper (Cu) particles added in H_2SO_4 electrolyte to produce composite Al_2O_3 coating [8,9]. Given the DLC often exists in the form of thin film, it was deposited onto the Cu particles, serving as a carrier or medium that aids in the anodizing process. It was found that incorporating DLC flakes inside the Al_2O_3 coating helps improve surface defect and wear resistance. The ball-on disk test found that DLC functioned as a solid lubricant and decreased the wear rate and friction of the Al_2O_3 coating.

It is crucial to address these gaps to optimize the performance of the oxide coatings. In this study, the growth mechanism for Al_2O_3 coating with and without the DLC reinforcement was investigated, with a particular focus on understanding the influence of reinforcement particles on the growth mechanism and crack reduction for Type III disordered-porous oxide coating.

2. MATERIALS AND METHODS

A disk with a diameter of 25 mm of aluminium AA2017-T4 alloy was served as the anode for this study supplied by Misumi Malaysia Sdn. Bhd. In accordance with the information presented in Table 1, Cu is the primary alloying element for the aluminium alloy AA2017 series.

Table 1: The chemical composition of aluminium alloy AA2017-T4

Element	Al	O	Cu
Weight percentage (%)	91.79	3.02	5.19

Before the anodizing procedure, the aluminium alloy substrate was ground using silicon carbide (SiC) abrasive paper with grain sizes ranging from 600 to 2000 grit, utilizing the rotating polishing machine BAINPOL-VTl to achieve a surface roughness, Ra < 0.1 µm. Afterwards, the sample was cleaned using an ultrasonic bath with deionized water for 20 minutes, followed by ethanol for 20 minutes and acetone for 20 minutes. This procedure was conducted to eliminate foreign contamination, such as dirt and debris.

The reinforcement particle used in this study was DLC-deposited on Cu particles, as can be seen in Figure 1. The particle was acquired from Tokyo Institute of Technology, Japan. The DLC was deposited on Cu particles with a mean diameter of 20 µm (Cu-HWQ-20: Fukuda Metal Foil & Powder Co., Ltd) via chemical vapor deposition (CVD) under an applied voltage of -3.5 kV for a deposition time of 30 minutes. According to SEM and EDS analysis, the DLC film covers approximately 3.74 % of the Cu particle surface with the presence of an oxygen (O) element [8,9]. The DLC film was not deposited to the entire Cu particle surface. It is also commonly recognized that DLC has poor adhesion to Cu due to the inability of carbide to develop.

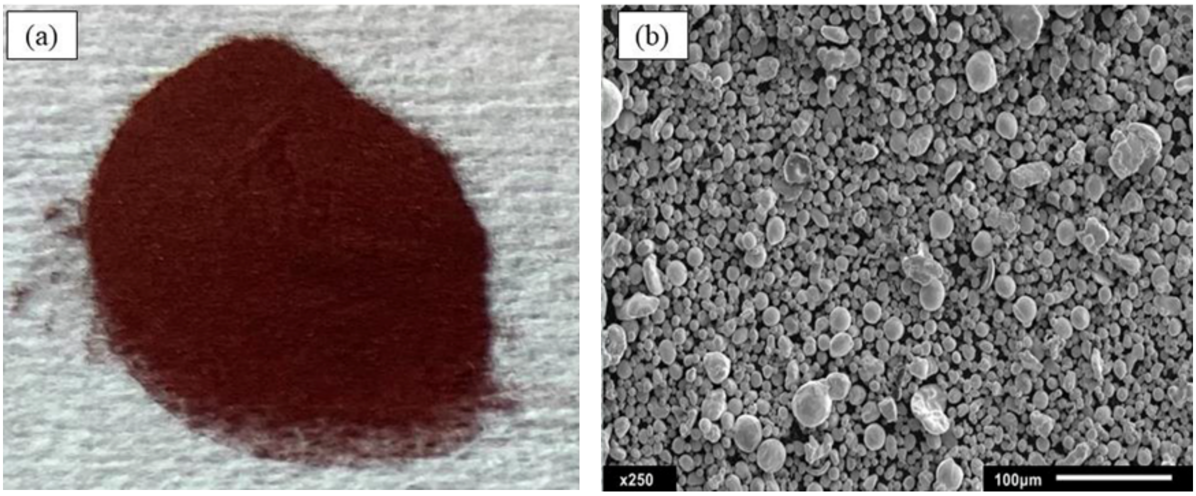

Figure 1: (a) DLC-deposited on Cu particle and (b) SEM image of the particle

The anodizing process was carried out using a constant current of 2 A, an initial voltage of 15 V, and 20 wt.% of H_2SO_4 electrolyte with a relatively 26 °C initial temperature. Throughout the anodizing process, the solution was stirred at 200 rpm to keep uniform ion concentration, aid the dispersion of reinforcement particles, and minimize the accumulation of localized heat. The sample was anodized without reinforcement particles and with 1 gL^{-1} of DLC-deposited Cu particles, respectively. In order to study and compare the growth mechanism of the Type III anodizing process, the anodizing duration was adjusted to 2, 4, 6, 8, 10, 20, 30, and 60 minutes, respectively. The growth of oxide coating in terms of surface morphology and surface topography was then analyzed using Zygo Optical Profiler and scanning electron microscope (SEM).

3. RESULTS AND DISCUSSION

3.1 Effect of Anodizing Time on Surface Morphologies, Surface Roughness, and Cross-section of Al_2O_3 Coating

The surface morphologies of the Al_2O_3 coatings obtained by SEM analysis are displayed in Figure 2. Based on the figure, some pits formed on the surface of Al_2O_3 coating and Al_2O_3 coating with DLC flakes when the anodizing time was from 2 minutes to 4 minutes. Pitting generally forms due to the presence of surface defects, such as flaws in the oxide. Previous study has reported that pit formation on the oxide/metal interface results from the dissolution of intermetallic phases during the anodizing process [10]. Another study mentions that the pitting is caused by the aggressive anions penetrating the anodic Al_2O_3 coating, which is discussed more in a later section [11]. Some DLC flakes were identified on the Al_2O_3 coating after 4 minutes of anodizing, but the flakes were not trapped within the oxide coating. When reacting with an acid electrolyte, the DLC film peeled off from Cu particles and the DLC in the form of flakes embedded into the oxide coating while some of the Cu particles dissolved in the electrolyte. Therefore, the reinforcement particles will be addressed as '*DLC-deposited Cu particles'* before undergoing the anodizing process. In contrast, after being incorporated in the oxide coating, they will be addressed as '*DLC flakes*.'

Figure 2 shows the surface morphologies of Al2O3 coating and Al2O3 coating with DLC flakes at different anodizing times. From Figure 2, the surface of the Al_2O_3 coating changes significantly from 4 minutes to 6 minutes of anodizing. The surface changes can be attributed to the constant growth and evolution of oxide coating. In the beginning, between 2 to 4 minutes, the oxide coating was relatively thin with defects, such as pits and irregularities. At 6 minutes, the Al_2O_3 coating continues to grow on the substrate with a coarser surface roughness, refer to Figure 3. According to Figure 2, the texture surfaces of Al_2O_3 coating and Al_2O_3 coating reinforced with DLC flakes at 6, 8, and 10 minutes are nearly identical. Given the time-dependent nature of the Al_2O_3 coating development, the Al_2O_3 coating may still be in its early growth phases at these minutes, with the textures still evolving and not merging into a continuous coating. A few DLC flakes were also found on the surface of the Al_2O_3 coating at 8 and 10 minutes of anodizing.

After 20 minutes, the Al_2O_3 coating has reached a relatively moderate thickness. The thickness has increased considerably compared to the initial stages, although it has not reached its maximum thickness. The surface texture has become smoother compared to 10 minutes of anodizing. However, it exhibits some roughness because of continuous pore development and oxide growth. At this stage, the pores are in the process of developing; hence, they are uneven and disordered. Based on prior research, pore formation occurs due to cations' outward migration and anions' inward migration [12]. The aluminium ion ($Al^{3+)}$ passed the oxide/electrolyte interface and dissolved in the electrolyte. The dissolution of oxide in the electrolyte forms a porous Al_2O_3 coating. In addition, some DLC flakes were identified inside the pores of the Al_2O_3 coating.

Next, the thickness of the Al_2O_3 coating is considerably greater after 30 minutes of anodizing compared to 20 minutes. Based on Figure 2, at 30 minutes of anodizing, the pores are more pronounced compared to the 20-minute mark, and some degree of disordered pore formation can be observed. After 60 minutes, the thickness of the Al_2O_3 coating has grown significantly thicker. According to the SEM image, the pores are increased by exhibiting variations in size and distribution. Based on previous research, the disordered pores from the early stages might continue developing.

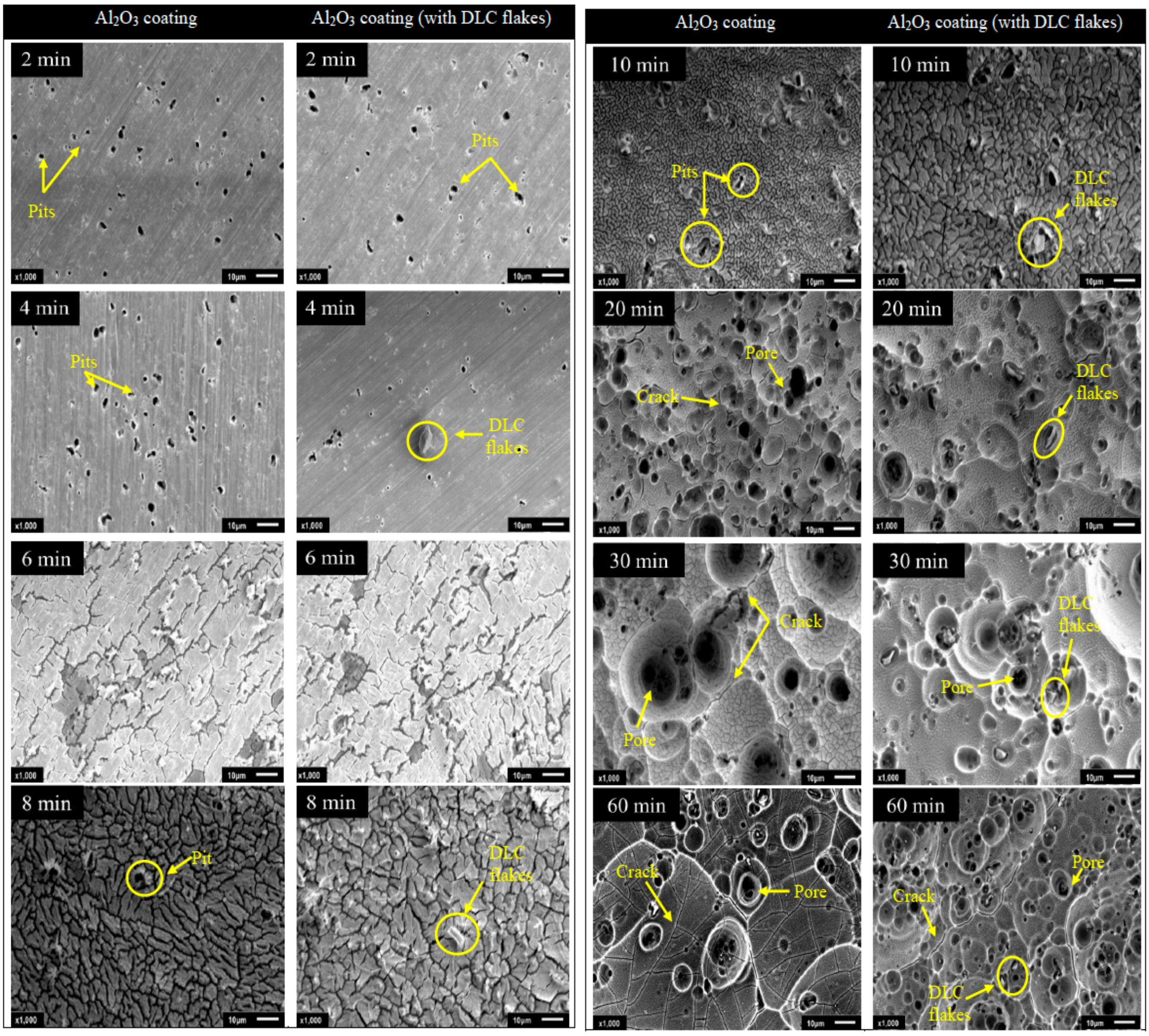

Figure 2: The surface morphologies of Al_2O_3 coating and Al_2O_3 coating with DLC flakes at different anodizing times

The average surface roughness for Al_2O_3 coating and Al_2O_3 coating with the addition of DLC flakes is presented in Figure 3. It is observed that our findings are in agreement with previous studies reporting an initial increase followed by a decrease in surface roughness during the formation of oxide coatings [13]. From 2 to 10 minutes of anodizing, the surface roughness increased due to the non-uniform and irregular growth of the oxide layer during the early stages (Figure 2). A significant reduction in surface roughness was observed between 10 and 20 minutes, likely resulting from the development of a more uniform and continuous oxide layer that fully covers the aluminium substrate. Although the surface roughness increased again between 20 and 60 minutes of anodizing, it remained lower than the roughness observed during the initial 2 to 10 minutes.

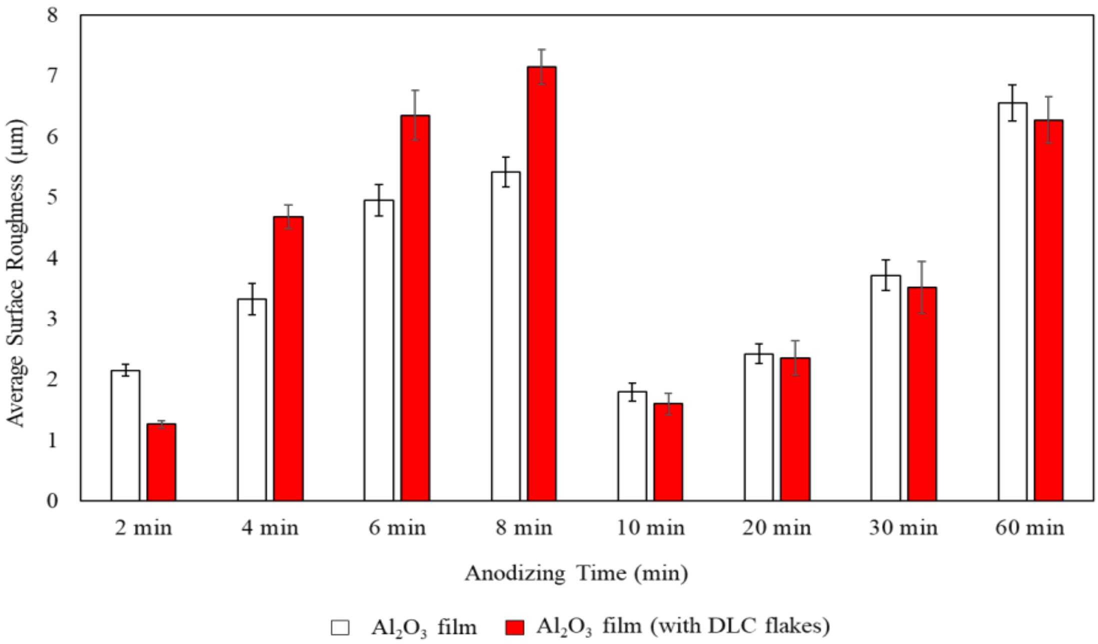

Figure 3: Average surface roughness of Al_2O_3 coating at different anodizing times

3.2 Growth Mechanism of Al_2O_3 Coatings with and without Reinforcement Particles during Anodizing

This section will explain the growth mechanism of Al_2O_3 coating with and without the addition of reinforcement particles. The illustration of the growth process is presented in Figure 4. At 2 to 4 minutes of anodizing, a very thin oxide coating grows at the surface of the AA2017-T4 Al alloy with the existing pits of various sizes for Al_2O_3 coating with and without DLC flakes, as shown in Figure 2. Several mechanisms explain the emergence of pits in the oxide coating, including the dissolution of intermetallic particles or Cu-rich particles on AA2017-T4 Al alloy [4]. The intermetallic particles dispersed within the substrate may have different compositions and electrochemical properties that may dissolve preferentially, forming pits on the surface. Other than that, the initiation of pits on the initial oxide coating can be due to the incorporation of aggressive ions in theelectrolyte. These ions are small and highly mobile, which allows them to reach the oxide/metal interface through defects in the anodic coating like microcracks, inclusions, and voids [11]. These pits can also serve as localized weak points on the Al_2O_3 coating and are more prone to dissolution, initiating pores later during anodizing.

After 6 to 10 minutes of anodizing, the oxide coating starts to grow thicker than in the initial phases. The oxide coating is formed by the migration of aluminium ions (Al^{3+}) that are formed at the substrate and OH^- and O^{2-} ions from the diluted sulphuric acid electrolyte under a high electric field. The negatively charged ions (O^{2-} and OH^-) are pulled towards the metal/oxide interface, resulting in the oxidation of aluminium [14]. The oxide coating becomes thicker but fails to cover the pits formed in the previous stage. These pits remain visible on the oxide layer, with some DLC flakes partially trapped between the pits and the growing oxide coating after 10 minutes, as shown in Figure 2. The effect of DLC on surface roughness is noticeable after 4 minutes of anodizing

At 20 minutes, the voltage started to decrease rapidly, corresponding to the increased resistance. The fluctuation and reduction in voltage value resulted in disordered pore development and irregular coating surface, as shown in Figure 2 and illustrated in Figure 4. The pore formed is due to the field-assisted oxide dissolution. Hoar and Mott's model proposed that oxide at the oxide/electrolyte interface is decomposed to Al^{3+} and O^{2-} ions. The Al^{3+} ions are ejected to the electrolyte, while the O^{2-} ions are in contact with the acid electrolyte and move through the oxide interface to form new oxide. Also, the dissolution happens when some of the generated oxide coating is chemically dissolved in the electrolyte. At this minute, the DLC flakes were incorporated in the pores, as shown in Figure 2. During the anodizing process, the dispersion of the DLC flakes was helped by the magnetic stirrer stirring at 200 rpm. When the DLC flakes moved along the electrolytic region and reached the oxide coating interface by the influence of kinetic movement, they were trapped inside the pores. Some of the DLC flakes were partially trapped in the oxide coating. At 60 minutes of anodizing, the pores are bigger compared to those produced at 30 minutes due to the increased electrolyte temperatures. The thickness of the Al_2O_3 coating formed after 60 minutes can be found in our previous publication by Syazwani et al. [4] for the case without DLC flakes, and in Rawian et al. [8] for the case with DLC flakes.

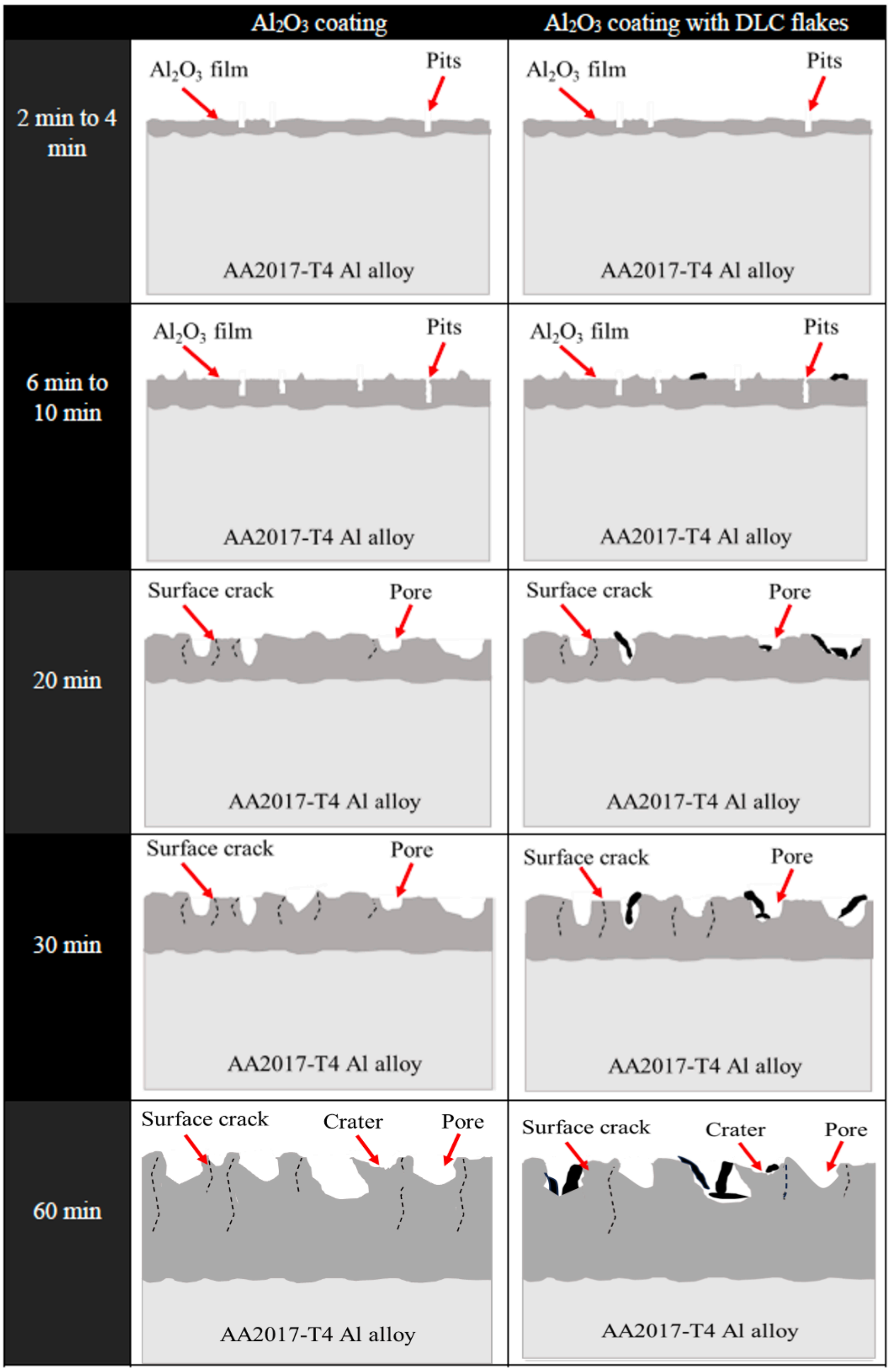

Figure 4: Growth process of Al_2O_3 coating formation with and without DLC flakes

3.3 Effect of DLC Flakes in Crack Reduction

Plenty of research has discussed the mechanism for crack reduction when incorporating reinforcement particles included in the oxide coating. However, the crack formation and reduction mechanism of disordered porous Al_2O_3 coating is still unclear because anodizing is a complex process influenced by various factors, such as differences in anodizing parameters. During anodizing, aluminium is converted to Al_2O_3 coating which resulting in significant volume expansion. In most cases, the volume of aluminium oxide is around 1.2 to 2 times that of the original aluminium, depending on the anodizing parameters [15]. By definition, the volume expansion factor is the ratio of anodized aluminium to the volume of consumed aluminium. In accordance with the Pilling-Bedworth ratio (PBR), which is the ratio between the molar volume of the grown oxide to the molar volume of the metal consumed, the passivating oxide is subjected to tensile stress and easily cracks if the PBR < 1. If the 1 <PBR<2, the oxide coating covers the metal evenly or uniformly. Lastly, the passivating oxide is subjected to an excessive amount of compressive stress and easily crumbles if the PBR >2. Nevertheless, the precise estimation of PBR is rather complicated.

Based on previous research, the porous oxide coating formed by acid electrolytes (i.e., H_2SO_4, $H_2C_2O_4$, and H_3PO_4), a wide range of volume expansion factors (kv) between 0.86 and 1.90 have been reported [16]. For anodization of aluminium in H_2SO_4, previous studies reported that the optimal conditions for the self-ordering of pores are accompanied by moderate volume expansion (i.e., kv = 1.22), while contradiction or excessive volume expansion can cause disordering of pores [17,18]. The volume expansion for the self-ordered porous oxide coating has been reported by several studies, and it normally ranges between 1 and 2. On the other hand, the volume expansion factors for disordered porous oxide coating is less commonly reported in the literature. Since the Al_2O_3 coating produced in this study has disordered pores, it is expected that the oxide coating will undergo significant volume expansion.

At the beginning of anodizing, around 10 minutes, the surface of the aluminium substrate reacts with the electrolyte to form an Al_2O_3 coating. At this stage, the oxide coating is thin. As anodizing continues for 20 minutes, the oxide layer grows and thickens and starts developing disordering pores of various sizes. As mentioned previously, the formation of Al_2O_3 coating involves volume expansion, and the volumetric expansion of Al_2O_3 coating during anodizing can develop stress. The difference in volume expansion between the oxide coating and aluminium substrate or underlying substrate can lead to significant internal stress. The aluminium substrate may constrain the expansion of oxide coating since the oxide occupies more volume than its original aluminium. This causes compressive stress to occur within or near the oxide/metal interface and tensile stress at the oxide/electrolyte interface. Then, since this type of anodizing produced disordering pores and non-uniform oxide coating, these pores can be sites of stress concentration and potential crack initiation, where localized tensile stress can lead to crack initiation. At the same time, dissolution of Al^{3+} ions into the electrolyte during the anodizing can further weakens the oxide coating by increase the pore size that intensifies the development of cracks. Based on the surface morphology examined using SEM analysis shown in Figure 2, crack formation occurred at 20 minutes of anodizing near the structure of pores.

After 60 minutes of anodizing, the crack for Al_2O_3 coating becomes prominent and propagates across the surface, as shown in Figure 4, following the initiation of the cracks previously. Based on previous research, the difference in electrolyte temperature can also contribute to thermal expansion between oxide coating and aluminium substrate and lead to crack formation [19]. However, since the temperature difference in this experiment is less than 40 °C, the effect of electrolyte temperature is not that significant in initiating a crack. Still, it might contribute to propagating the existing crack.

However, with the addition of DLC flakes into the electrolyte, the surface crack on composite Al_2O_3 coating reduced tremendously, as presented in Figure 2 and Figure 5. Based on previous studies, some of the reinforcement is embedded and trapped within the oxide coating itself due to the rapid growth of the oxide [20]. Incorporating DLC flakes within the pores helps distribute tensile stress across the oxide coating, reducing significant stress build-up. Occupying the space within the pores prevents the pores from experiencing localized tensile stress, and as a result, the potential for crack initiation and propagation is reduced. In addition, the DLC flakes in the oxide coating also help to manage the volume change more effectively. The DLC flakes can serve as a physical barrier, preventing the Al^{3+} ions from dissolving into the electrolyte and inhibiting the pore size from increasing. Figure 6 illustrates the crack reduction mechanism for composite oxide coating. To summarize, the dispersion of DLC flakes into the oxide coating results in a reduction in the porosity, which in turn leads to a coating that is less prone to defects. Some studies also suggest that particles with high thermal conductivity in the electrolyte can help dissipate heat more effectively by transferring the heat away from the growing oxide coating. Minimizing thermal gradients within oxide coating makes it less prone to thermal stress and cracking.

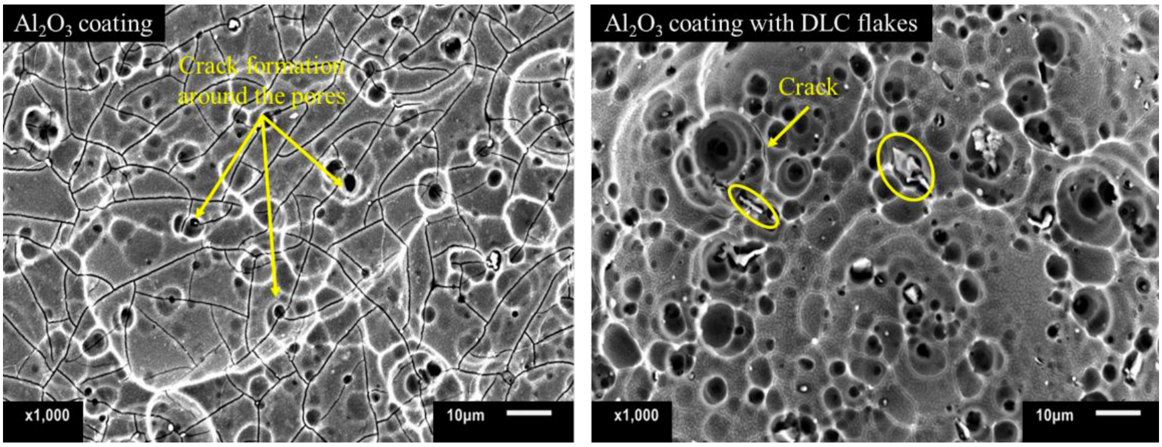

Figure 5: The crack formation for Al_2O_3 coating and Al_2O_3 coating with DLC flakes

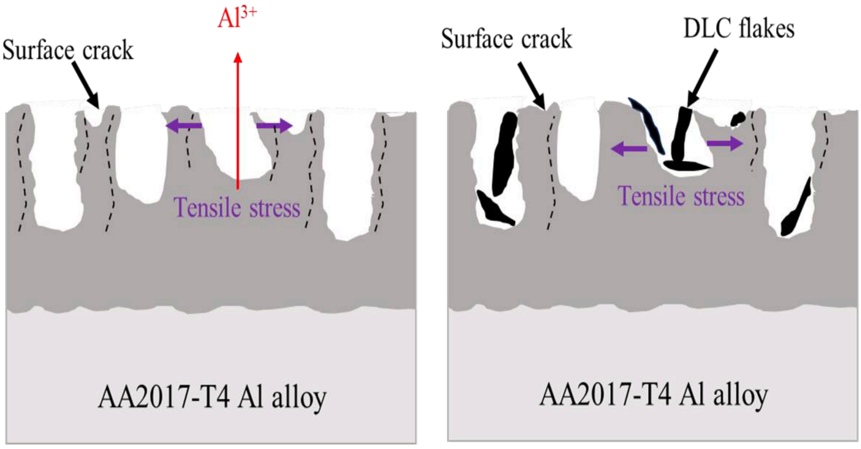

Figure 6: The beneficial impact of DLC flakes on reducing the formation of the crack

4. CONCLUSIONS

The conclusions are stated as follows:

- Based on the growth studies conducted at different anodizing times, the oxide coatings start to form at 2 minutes with a formation of pits for Al_2O_3 coating with and without DLC flakes. At 20 minutes, the voltage fluctuates, forming disordered pores on the oxide coating. At 60 minutes, the pores are bigger compared to those produced at 30 minutes.

- An initial increase in surface roughness was observed, reaching up to 7 µm at 8 minutes, followed by a sharp decrease to 1.8 µm at 10 minutes. Subsequently, the surface roughness increased again between 20 and 60 minutes of anodizing, ranging from 2.5 µm to 6.5 µm.

- The incorporation of DLC flakes in the oxide coating significantly reduced the formation of cracks and prevented them from propagating by occupying the pores. The presence of DLC flakes in the pores helps prevent them from experiencing localized tensile stress, which reduces the potential for crack initiation at the pore sites. In addition, the DLC flakes also act as a physical barrier that hinders the dissolution of Al^{3+} into the electrolyte, which can cause further weakness in the oxide coating if the size of the pores increases.

Acknowledgements

This research is supported by the grant from the Ministry of Higher Education, Malaysia under Fundamental Research Grant Scheme (FRGS/1/2023/TK10/UTM/02/11).

Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare

Compliance with Ethical Standards

The work is compliant with ethical standards

References

[1] Adyono, N., Lestari, W. D. & Endahwati, L. (2021). Military Type III Anodizing: The optimal limit within hardening process of aluminium alloy in a near zero temperature. In Proceedings of the 2nd Borobudur International Symposium on Science and Technology (BIS-STE 2020), 203, 148–153.

[2] Kim, H. S., Kim, D. H., Lee, W., Cho, S. J., Hahn, J. H. & Ahn, H. S. (2010). Tribological properties of nanoporous anodic aluminum oxide film. *Surface and Coatings Technology*. 205(5), 1431–1437.

[3] Kumar, N. S., Pramod, G. K., Samrat, P. & Sadashiva, M. (2022). A critical review on heat treatment of aluminium alloys. *Materials Today: Proceedings*. 58, 71–79.

[4] Mohamad, S., Liza, S. & Yaakob, Y. (2020). Strengthening of the mechanical and tribological properties of composite oxide film formed on aluminum alloy with the addition of graphite. *Surface and Coatings Technology*. 403, 126435.

[5] Main, B., Jiang, L., Marceau, R. K. W. & Barter, S. (2024). Material characterisation of anodising effects on small fatigue crack nucleation in AA7XXX alloys. *Materialia*. 33, 101997.

[6] Cao, J., Gao, Z., Wang, C., Muzammal, H. M., Wang, W., Gu, Q., Dong, C., Ma, H. & Wang, Y. (2020). Morphology evolution of the anodized tin oxide film during early formation stages at relatively high constant potential. *Surface and Coatings Technology*. 388, 3–10.

[7] Lu, C., Feng, X., Yang, J., Jia, J., Yi, G., Xie, E. & Sun, Y. (2019). Influence of surface microstructure on tribological properties of PEO-PTFE coating formed on aluminum alloy. *Surface and Coatings Technology*. 364, 127–134.

[8] Rawian, N. A. M., Akasaka, H., Liza, S., Fukuda, K., Zulkifli, N. A., Tahir, N. A. M. & Yaakob, Y. (2023). Surface and tribological characterization of anodic aluminum oxide coating containing diamond like carbon flakes. *Diamond and Related Materials*. 132, 109674.

[9] Zulkifli, N. A., Liza, S., Akasaka, H., Fukuda, K., Mohd Rawian, N. A., Md Ghazazi, N. A., Mat Tahir, N. A. & Yaakob, Y. (2023). Surface and tribology characterization of diamond-like carbon flakes reinforced oxide film by pulse anodizing. *Ceramics International*. 49(21), 34205–34222.

[10] Wu, H., Ma, Y., Huang, W., Zhou, X., Li, K., Liao, Y., Wang, Z., Liang, Z. & Liu, L. (2018). Effect of iron-containing intermetallic particles on film structure and corrosion resistance of anodized AA2099 alloy. *Journal of The Electrochemical Society*. 165(9), C573–C581.

[11] Cirik, E. & Genel, K. (2008). Effect of anodic oxidation on fatigue performance of 7075-T6 alloy. *Surface and Coatings Technology*. 202(21), 5190–5201.

[12] Schneider, M. & Fürbeth, W. (2022). Anodizing—The pore makes the difference. *Materials and Corrosion*. 73(11), 1752–1765.

[13] Aerts, T., Dimogerontakis, T., De Graeve, I., Fransaer, J. & Terryn, H. (2007). Influence of the anodizing temperature on the porosity and the mechanical properties of the porous anodic oxide film. *Surface and Coatings Technology*. 201(16–17), 7310–7317.

[14] Brudzisz, A. M., Giziński, D. & Stępniowski, W. J. (2021). Incorporation of ions into nanostructured anodic oxides—mechanism and functionalities. *Molecules*. 26(21), 6378.

[15] Vrublevsky, I., Parkoun, V., Sokol, V., Schreckenbach, J. & Marx, G. (2004). The study of the volume expansion of aluminum during porous oxide formation at galvanostatic regime. *Applied Surface Science*, 222(1–4), 215–225.

[16] Vrublevsky, I., Parkoun, V., Schreckenbach, J. & Marx, G. (2003). Effect of the current density on the volume expansion of the deposited thin films of aluminum during porous oxide formation. *Applied Surface Science*, 220(1–4), 51–59.

[17] Jessensky, O., Muller, F. & Gosele, U. (1998). Self-organized formation of hexagonal nanopore arrays in anodic alumina. *Applied Physics Letters*. 72(10), 1173–1175.

[18] Abd-Elnaiem, A. M., Mebed, A. M., Gaber, A. & Abdel-Rahim, M. A. (2013). Effect of the anodization parameters on the volume expansion of anodized aluminum films. *International Journal of Electrochemical Science*. 8(8), 10515–10525.

[19] Liu, W., Zuo, Y., Tang, Y. & Zhao, X. (2008). The cracking behavior of anodic films on cast aluminum alloy after heating in the temperature range up to 300 °C. *Surface and Coatings Technology*. 202(17), 4183–4188.

[20] Arun, S., Hariprasad, S., Saikiran, A., Ravisankar, B., Parfenov, E.V., Mukaeva, V. R. & Rameshbabu, N. (2019). The effect of graphite particle size on the corrosion and wear behaviour of the PEO-EPD coating fabricated on commercially pure zirconium. *Surface and Coatings Technology*. 363, 301–313.