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Surface characterization of oxide films formed on aluminium alloy with the incorporation of functionalized carbon nanotube

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KEYWORDS

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Anodic aluminium oxide
Hardness
Surface properties
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ABSTRACT

Aluminium oxide film incorporated with functionalized CNTs were fabricated by an anodizing method using 20% diluted sulphuric acid. This study aims to examine the influence of different acid molarity during the CNTs functionalization toward the surface characterization of aluminium oxide composite film. The functional group that is attached at the surface of CNTs were analyzed using FTIR while the surface properties were analyzed using x-ray diffraction (XRD), scanning electron microscopy (SEM), 3D optical profiler and Vickers microhardness tester. FTIR result shows that the presence of hydroxyl group at 1650cm^{-1} proves that the CNTs were successfully functionalized. The results obtained from the SEM analysis indicate that the incorporation of functionalized CNTs contributes to a reduction in the porosity of the composite film by 72%. The result of 3D profiler revealed that the functionalized CNTs in the composite film reduce the average surface roughness to 65%. Besides that, the microhardness was enhanced by 94%.

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1.0 INTRODUCTION

Aluminum alloy (AI) is a metallic material composed primarily of aluminum and other foreign elements. Nowadays aluminium alloy is widely used in many different industries such as aerospace industry due to its excellent strength-to-weight ratio (Zhu et al., 2023). However aluminium alloys generally have lower hardness compared to other metals such as steel or iron. To increase the hardness, the surface of the aluminium alloy needs to be modified. Surface modification of aluminium alloys can enhance the surface properties by increasing the hardness of aluminium alloy (Novák et al., 2023). Anodizing is one of the surface modifications on the aluminium alloy that creates a thick, durable, and corrosion-resistant oxide layer, namely anodic aluminium oxide (AAO) (Yu et al., 2023). However, AAO contains many small nanopores that can potentially give rise to the formation of cracks. The formation of cracks will cause stress at the surface of AAO which can eventually reduce the overall hardness of the material evermore (Zhang et al., 2023).

Currently there has been a significant focus on investigating the strengthening properties of composite oxide films in various research studies. The incorporation of particles into the oxide film can have a substantial impact on impeding the onset and propagation of fatigue cracks, hence mitigating surface defects. A study on microstructure and mechanical properties of fly ash particulate reinforced AA6061 aluminum alloy composites shows that an increase of 132% of microhardness compared to unreinforced AA6061 alloy (David Raja Selvam et al., 2013). The resistance to fracture propagation during tensile loading is attributed to the presence of these fly ash particles. Beside that there is a study on the effect of boron carbide reinforcement particles on mechanical properties of aluminum alloys which show a maximum of 24% increase in hardness compared to without the present of boron carbide (Padmavathi & Balu Naik, 2023). Boron carbide possesses the capability to function as a nucleation site, facilitating the creation of smaller aluminum grains during the process of solidification and hence increase aluminium hardness. Moreover, there has been a study on Enhancement of tribological properties of AA7075 aluminum alloy using nano-silicon carbide reinforcement which prove that the incorporation of SiC into the AA7075 matrix leads to a notable enhancement in hardness and wear resistance (Phaneendra et al., 2023).

Incorporation of carbon nanotubes (CNTs) into aluminium alloys is one of the composite oxide films that help improve hardness, strength, and overall mechanical properties. When integrated into the pores of the aluminium alloys, CNTs act as strengthening agents which alter the fundamental material behavior at the nanoscale level. The high aspect ratio and exceptional strength of CNTs enable them to form load-bearing networks, effectively hindering dislocation motion and enhancing its hardness (Guo et al., 2017). However, mostly all commercialized CNTs are in the form of heavy entangled in nature which result in poor dispersion in any solvent or aqueous solution. These commercialized CNTs are also known as unfunctionalized CNTs or pristine CNTs. Unfunctionalized CNTs are raw CNTs that have not undergone any chemical modification on their surfaces. This means that unfunctionalized CNTs tend to agglomerate in nature due to high van deer walls forces between each individual CNTs since it does not have any additional functional group to help interact with any solvent or aqueous solution (Ibrahim, 2013). This agglomeration will minimize the probability of CNTs to be located inside the pores due to size difference. To reduce the agglomeration of the CNTs and improve its dispersion it needs to undergo a process known as functionalization of CNTs. Functionalization of CNTs refers to the process of modifying the surface of the CNTs by introducing functional groups onto their sidewalls that is produced by a strong oxidizing agents like sulphuric acid with the help of an ultrasonic

bath. Microscale shockwaves are created by the strong local heating and pressure produced by an ultrasonic bath. Reaction rates will be accelerated by this wave's ability to increase the kinetic energy of the particles in the solution. Carbon-carbon bonds at the surface of carbon nanotubes will physically break due to the high-energy conditions produced by this wave (Liang et al., 2016). Beside from the help of the shockwave the CNTs surface also develops defects because of the strong oxidizing chemicals. These defects cause the nanotubes' sp^2 carbon-carbon bond to break, exposing reactive sites that allow functional groups containing oxygen to bond. The present of these functional groups can improve CNTs dispersion since it has hydrophilic properties which mean that it can react with any solvent or aqueous solution compared to unfunctionalized CNTs that is hydrophobic in nature (Dubey et al., 2021).

Research in this field aims to improve the mechanical, thermal, and electrical properties of materials based on aluminium by utilizing the unique properties of carbon nanotubes (CNTs) into aluminium alloys. This research is novel because it may lead to composite materials that perform better than conventional aluminium alloys. Carbon nanotubes are well-known for their remarkable stiffness and strength. They can strengthen aluminium alloys when added, giving the material better mechanical qualities such greater hardness, strength, and resistance to wear. This is crucial in sectors like the aerospace and automotive industries where it's preferred to use lightweight materials with high strength for better fuel efficiency (Thirugnanasambantham et al., 2021). Besides that, the oxide layer that is formed by anodizing will help to improve corrosion resistance of the aluminium alloy. This can be beneficial for marine industry as this oxide layer helps protect aluminium from slat water environment which is the main cause of corrosion of marine structures. This is because metals submerged in or exposed to the environment can create a galvanic pair in the presence of seawater. Two different metals or alloys in electrical contact form a galvanic pair. Anodic metal corrodes because of electrons moving from the anode to the cathode (Nejneru et al., 2019).

In this work the surface of aluminium alloy was modified using anodization with the incorporation of CNTs. Before anodization the CNTs were functionalized with different molarity of sulphuric acid (H_2SO_4) to improve interfacial interaction with aluminium alloy. The dispersion of the CNTs is examined using FTIR analysis. The structural and surface mechanical properties of aluminium surface were analyzed and discussed using SEM and 3D profiler analysis while the mechanical properties were studied using micro hardness test. This study is expected to pave a new way in CNTs modified aluminium surface for a better mechanical property.

2.0 MATERIALS AND METHODS

2.1 Raw Materials

Aluminium alloy AA2017-T4 disk with 25 mm diameter (Misumi Malaysia Sdn. Bhd), MWCNTs (≥98% carbon, Sigma-Aldrich), sulphuric acid (95-98%) and copper sheet (99.9% pure solid copper with 200 mm × 200 mm × 0.5 mm) were purchased.

2.2 Materials Preparation

Aluminium alloy with diameter of 25 mm is used in this experiment. Aluminium alloy is then cut in which the thickness of each small rod is 4 mm. The surface of the aluminium alloy is then polish using sandpaper from 800 grit to 2000 grit to reduce surface roughness (Hu et al., 2023). The sample is then cleaned using an ultrasonic bath for 10 minutes to remove any impurities.

The as-purchased CNTs was functionalized with H_2SO_4 solution using different molarity of acid, which is 5M, 8M, and 10M. The CNTs are then sonicated for 2 hours at 60 °C. After 2 hours, the sample are then filtered using vacuum and then left to dry for overnight. Different molarity of acid can be used during the functionalization process of CNTs because different molarity of acid will produce different amount of functional group that will be attach to the wall of the CNTs. Hence the sample 5M, 8M, and 10M refers to different molarity of sulphuric acid that is used during CNTs functionalization process that is 5 molarity of sulphuric acid, 8 molarity of sulphuric acid and 10 molarity of sulphuric acid respectively.

2.3 Anodizing Setup

Based on figure 1 the aluminum alloy is immersed in an electrolyte containing 2.55 M of sulphuric acid (H_2SO_4) and connected to the positive terminal of a DC power source. Sulphuric acid with the concentration of 2.55M is the most optimum concentration as any higher concentration will cause crack due to strong reaction occurring in the electrolyte. Copper plate is also placed in the electrolyte and connected to the negative terminal. FCNTs were then added to the electrolyte at the concentration of 1g/L, and it was stirred at 250 rpm to ensure that the CNTs were well dispersed. When an electric current is applied, an oxide layer begins to form on the aluminum surface. The anodizing is run for 60 minutes under the room temperature and voltage of 15V. 60 minutes of anodizing time is enough to produce a thick and dense oxide layer (Mohamad et al., 2020). Once the desired anodizing and post-anodizing treatments are complete, the aluminum alloy is thoroughly rinsed with distilled. This is to ensure that no remaining acids at the aluminium surface can potentially lead to a continued chemical reaction which could have an impact toward the properties of the anodic layer. After rinsing, the sample AAO, AAO-CNT, AAO-5M, AAO-8M and AAO-10M is dried to complete the process.

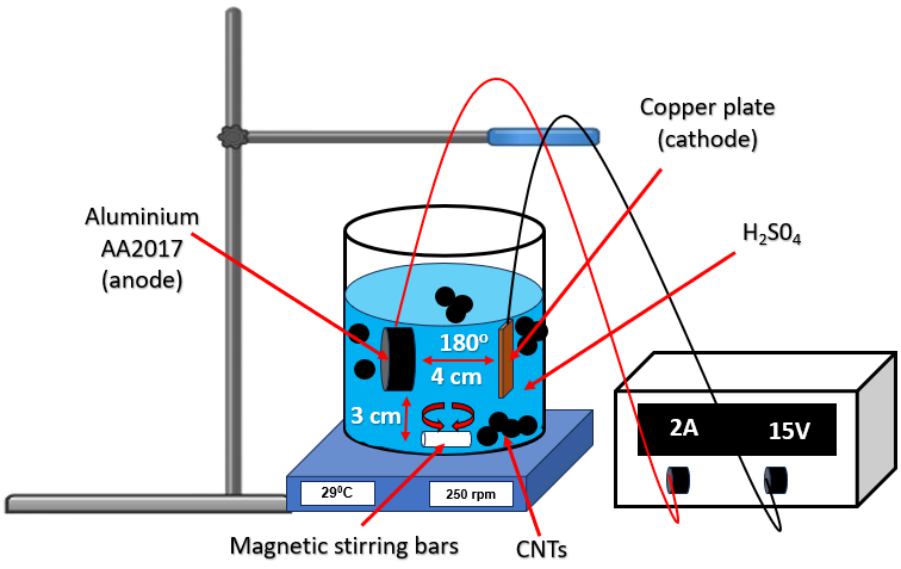

Figure 1: Schematic diagram of anodizing set-up.

Table 1: Anodizing parameters.

Constant variable	
Cathode	Copper plate
Anode	AA2017
Voltage (V)	15
Temperature	Room temperature
Electrolyte	20% H_2SO_4
Anodizing time (min)	60
Current (A)	2

Table 2: Sample abbreviation for CNTs/aluminium oxide composite film.

Sample	Remark	Label
Anodic aluminium oxide	Without CNTs addition	AAO
Anodic aluminium oxide-	As-receive CNTs	AAO-CNTs
Carbon nanotube composite	5 M functionalized CNTs	AAO-5M
	8 M functionalized CNTs	AAO-8M
	10 M functionalized CNTs	AAO-10M

2.4 Surface Characterization

The investigation of CNTs functionalization is conducted by a Fourier Transform Infrared Spectrometer (Perkin Elmer, spectrum 100). The phase study of anodic aluminum oxide (AAO) and AAO-carbon nanotubes (AAO-CNTs) was conducted using an X-ray Diffractometer (XRD, model PW 3040/60 MPD X'pert Pro, manufactured by Panalytical, Philips, Netherlands). The surface morphology and elemental composition were analyzed using Scanning Electron Microscopy with Energy Dispersive X-ray Spectroscopy (SEM-EDX) technique, specifically employing the JEOL JSM IT-100 instrument located in the United States. The analysis of surface topography was conducted utilizing a Digital Microscope (HIROXKh-8700, USA). The microhardness of AAO was assessed using the Micro Vickers Hardness method, specifically the HM-G30 instrument manufactured by Shimadzu Corporation in Japan. The sample was subjected to a load of 0.1 HV (980.7 mN) for a duration of 10 seconds.

3.0 RESULTS AND DISCUSSION

Figure 2 shows the picture of dispersion of CNTs after left for 1 day in 15 ml of H_2SO_4 acid. Determining whether CNTs are well-dispersed using only naked eyes can be challenging due to their extremely small size. A well-dispersed CNTs solution in a liquid medium may appear more transparent or have a consistent color throughout. If CNTs are poorly dispersed and agglomerated, the solution may appear dark, have visible clumps, or show non-uniform coloration. Based on figure 2 unfunctionalized CNTs have the lowest dispersion since the solution looks darker compared to all the functionalized CNTs.

FTIR can be used to analyses the functionalization of CNTs by detecting the presence or absence of different functional groups at the surface of CNTs. Carboxyl groups (-COOH) are functional groups that can be detected at the surface of CNTs through chemical functionalization processes. A carbonyl group which is composed of carbon atom double bonded to an oxygen atom can only be in the spectral region of 1700 – 1800 cm^{-1}. Based on figure 3 there is a strong

absorption band in the range of 1700 cm^{-1} to 1765 cm^{-1}. This band corresponds to the stretching vibration of the carbonyl group (C=O bond). Beside that there is a broad and strong absorption band in the range of 2500 to 3300 cm^{-1} which corresponds to the stretching vibration of the hydroxyl group (O-H bond) that remains the same after functionalized of CNTs. The presence of both the C=O bond and O-H bond in the IR spectrum strongly indicates the presence of a carboxyl group (Wang et al., 2020).

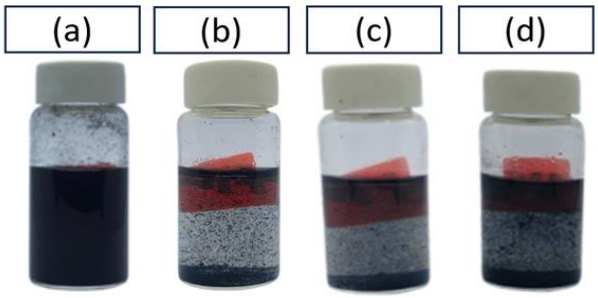

Figure 2: Dispersion of CNT in 1 days: (a) Unfunctionalized CNTs, (b) FCNTs (5M), (c) FCNTs (8M), (d) FCNTs (10M).

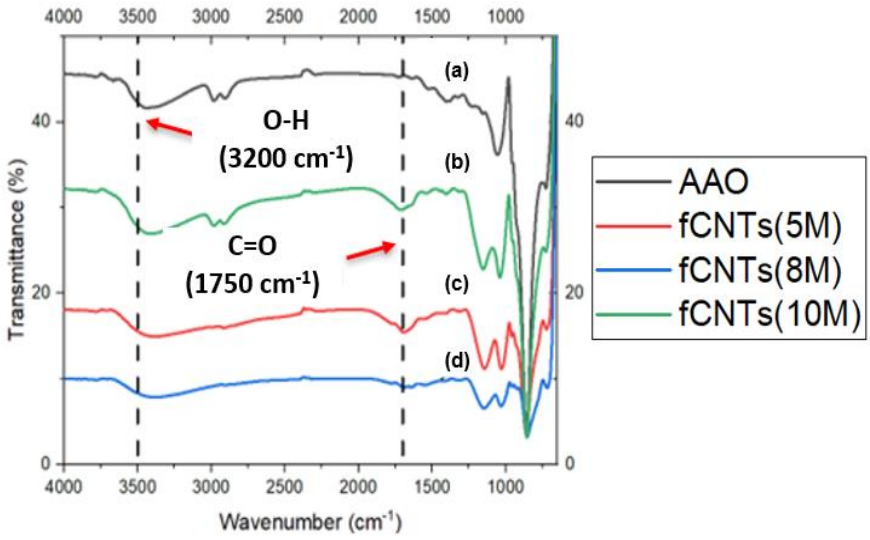

Figure 3: FTIR result for different molarity of CNTs in acid: (a) Unfunctionalized CNT, (b) FCNTs (5M), (c) FCNTs (8M), (d) FCNTs (10M).

3.1 Voltage and Temperature Change During Anodization Process

Figure 4(a) shows graph for temperature against time while figure 4(b) show graph for voltage against time. The initial temperature of the electrolyte was around 31^0C. As the anodizing time started to increase, the temperature of the electrolyte also started to increase rapidly. The main reason for the temperature increase is Joule heating, which occurs due to the electrical resistance of the electrolyte and the anode (Chowdhury et al., 2014). As the direct current (DC) passes through the electrolyte bath and the anode, electrical resistance causes the conversion of

electrical energy into heat. This heat is then transferred to the surrounding electrolyte, leading to an increase in its temperature. However, it is observed that the temperature of the electrolyte starts to decrease rapidly when CNTs are added into the electrolyte. The reduction in temperature is due to CNTs properties that have good thermal conductivity that help to control and maintain the temperature of the electrolyte. The incorporation of CNTs into the electrolyte has been found to facilitate the dissipation of heat that is produced during the anodizing process. By using this measure, the occurrence of localized hotspots is effectively mitigated, thereby promoting a more uniform temperature distribution throughout the aluminium alloy. Consequently, the likelihood of overheating or thermal damage to the material is significantly reduced.

A greater voltage is usually applied at the start of the anodizing process to accelerate the oxide layer's growth. Usually, this is called the "initiation phase." As anodizing time starts to increase, the voltage will gradually start to decrease (voltage drop) due to several factors. One of the factors that contribute to the voltage drop is that the oxide layer on the metal surface progressively grows and thickens during the anodizing process. The electrolyte and the metal surface are separated by the growing oxide layer, which causes resistance to increase. As an insulator, the oxide layer that forms on the metal surface prevents electricity from flowing through it and elevates resistance. Resistance

increases when the oxide layer thickens since the current must travel through the insulating oxide over a longer distance which causes the voltage to decrease. Besides that, byproducts like aluminium hydroxide are produced during the anodizing process. These byproducts will increase resistance and cause a voltage drop if they build up in the electrolyte or on the surface of the metal being anodized (Marquardt et al., 2008).

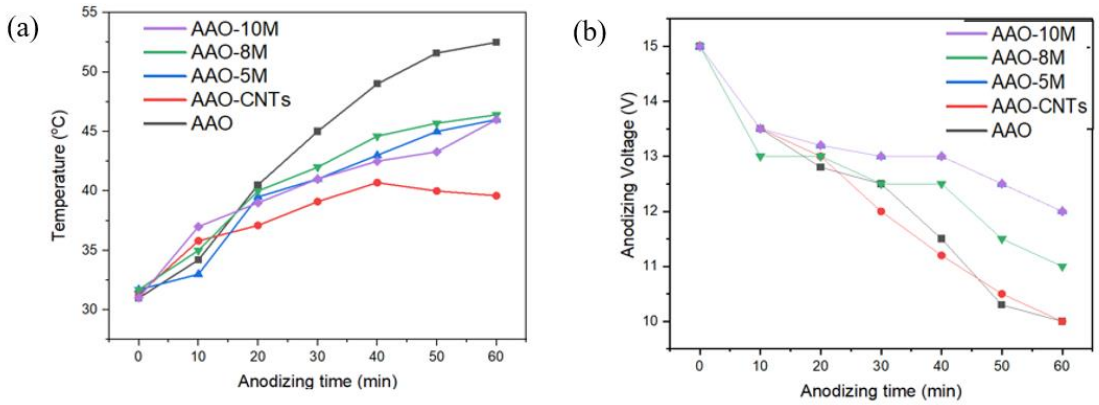

Figure 4. (a) Anodizing voltage against anodizing time (b) Electrolyte temperature against anodizing time.

3.2 Microstructure Properties

Different peaks in an X-ray diffraction (XRD) pattern provide valuable information about the crystallographic structure and composition of the material being analyzed. Each peak corresponds to specific diffraction angles, and the positions and intensities of these peaks reveal important characteristics about the material. Peaks corresponding to the orthorhombic Al_2O_3 (2 1 1) and Al_2O_3 (0 2 1) crystallographic planes can be seen in the XRD patterns. It was discovered that the coating created on the aluminum substrate is made of aluminum oxide with a unique crystal structure.

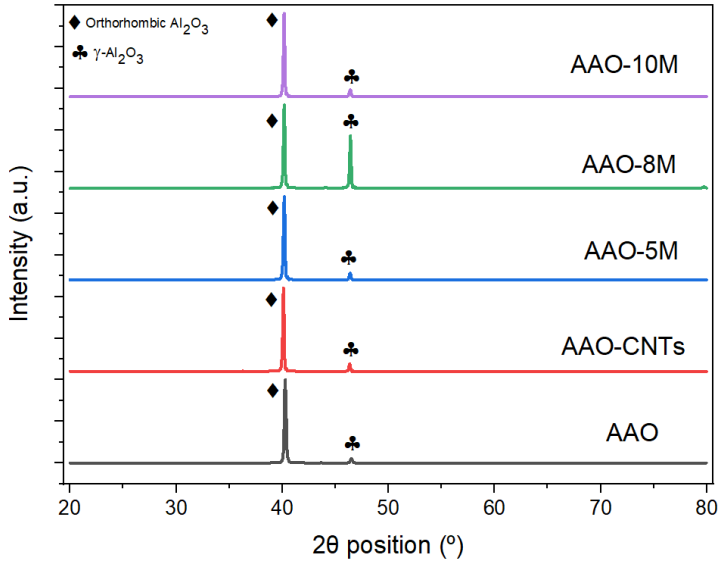

Figure 5: XRD pattern of anodizing voltage with different molarity of acid.

3.2.1 Scanning Electron Microscope (SEM)

Figure 6 illustrates the surface morphologies of composite oxide layers that were developed on AA2017 in two different conditions: without reinforcement and reinforced with CNTs. Statistical measurement was conducted using the software Image J to show that the average area of pores for AAO is around 492.73 µm. The average pores area of AAO-CNTs is reduced to 294.68 µm, that is by 40%. The average area is further reduced when functionalized CNTs are added to the electrolyte in which 5M, 8M and 10M is reduced to 265.01 µm (-46%), 134.95 µm (-72%), and 224.51 µm (-54%) respectively. CNTs have excellent mechanical properties, including high tensile strength and stiffness. When CNTs are incorporated into an aluminum alloy, they act as effective reinforcing agents. The strong interfacial bonding between CNTs and the aluminum matrix improves the overall mechanical properties of the alloy, making it more resistant to deformation and reducing the likelihood of porosity formation (Lee et al., 2011).

Throughout the duration of the anodizing process, composite oxide films contain the components Al, O, S, and C. The detection of Al and O components provides evidence for the initiation of oxide layer formation on the surface. Moreover, it has been proven that the film surface was dispersed with CNTs particles due to the existence of a small quantity of carbon element (5 to 10 wt%) during the anodizing process. Based on the table there can be seen an increase percentage of carbon distribution when for all 3 sample that is AAO-5M, AAO-8M, and AAO-10M. The increase in percentage of carbon is due to the present of functional group that is form at the surface of CNTs during functionalization process which help improve its dispersion. The better the dispersion of CNTs in the electrolyte the higher the chance that more CNTs will be detected.

The complete formation of the oxide layer can be proved by comparing the change in the element distribution of oxygen element before anodizing and after 60 minutes of anodizing. Before the anodization aluminium alloy has the lowest oxygen element of about 3.02 wt.% only (Mohamad et al., 2020b). When aluminium alloy undergoes anodization for 60 minutes the oxygen element increases rapidly up to 52 wt.%. The change in this oxygen element clearly proves

the complete formation of the oxide layer since the oxide layer is primary made up of oxygen element. Besides that, based on previous research 60 min anodizing time is the optimum time for the completion of oxide layer since it has the highest thickness of the composite oxide film (Mohamad et al., 2020b). Moreover, the substrate that is used which is AA2017 which is the same as this research supports this result since different aluminium alloy series have different properties which will cause different outcomes.

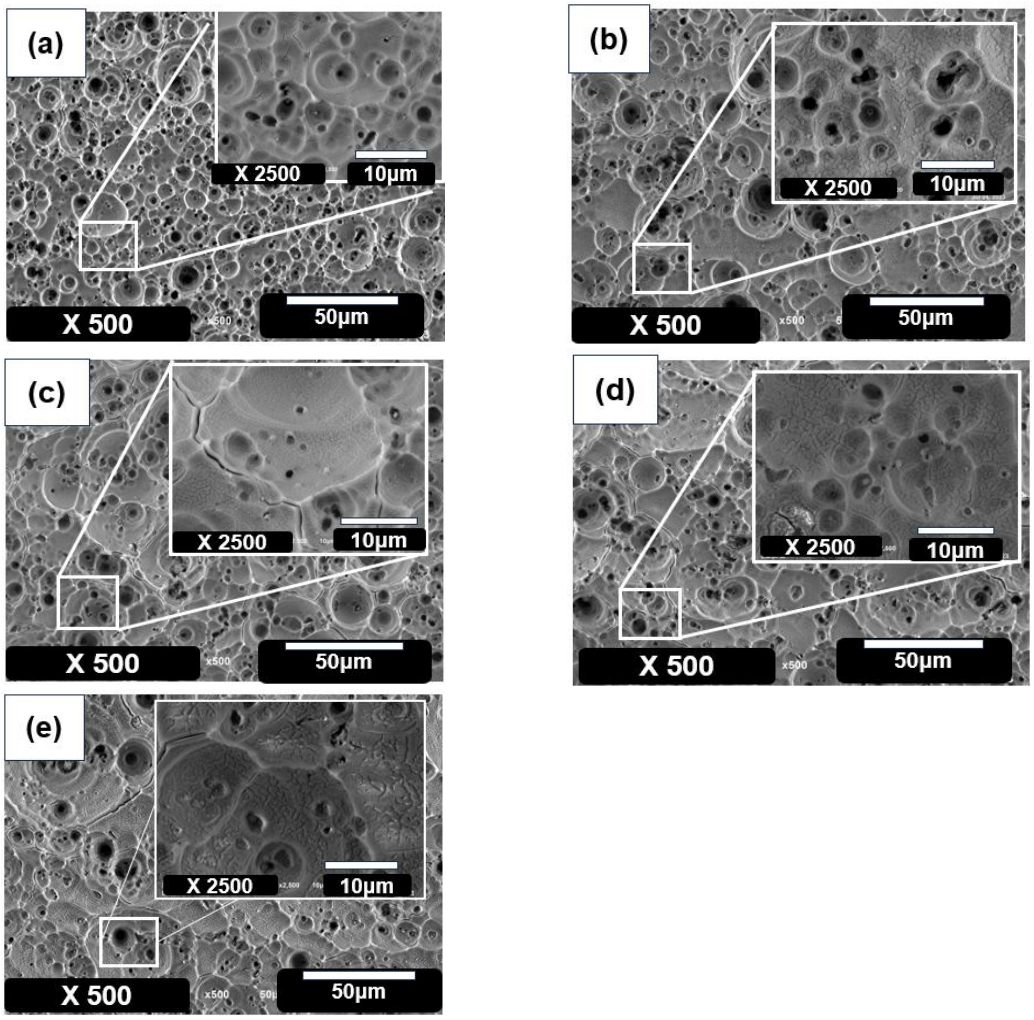

Figure 6. Surface morphologies of composite oxide films: (a) AAO (b) AAO-CNT (c) AAO-5M (d) AAO-8M (e) AAO-10M.

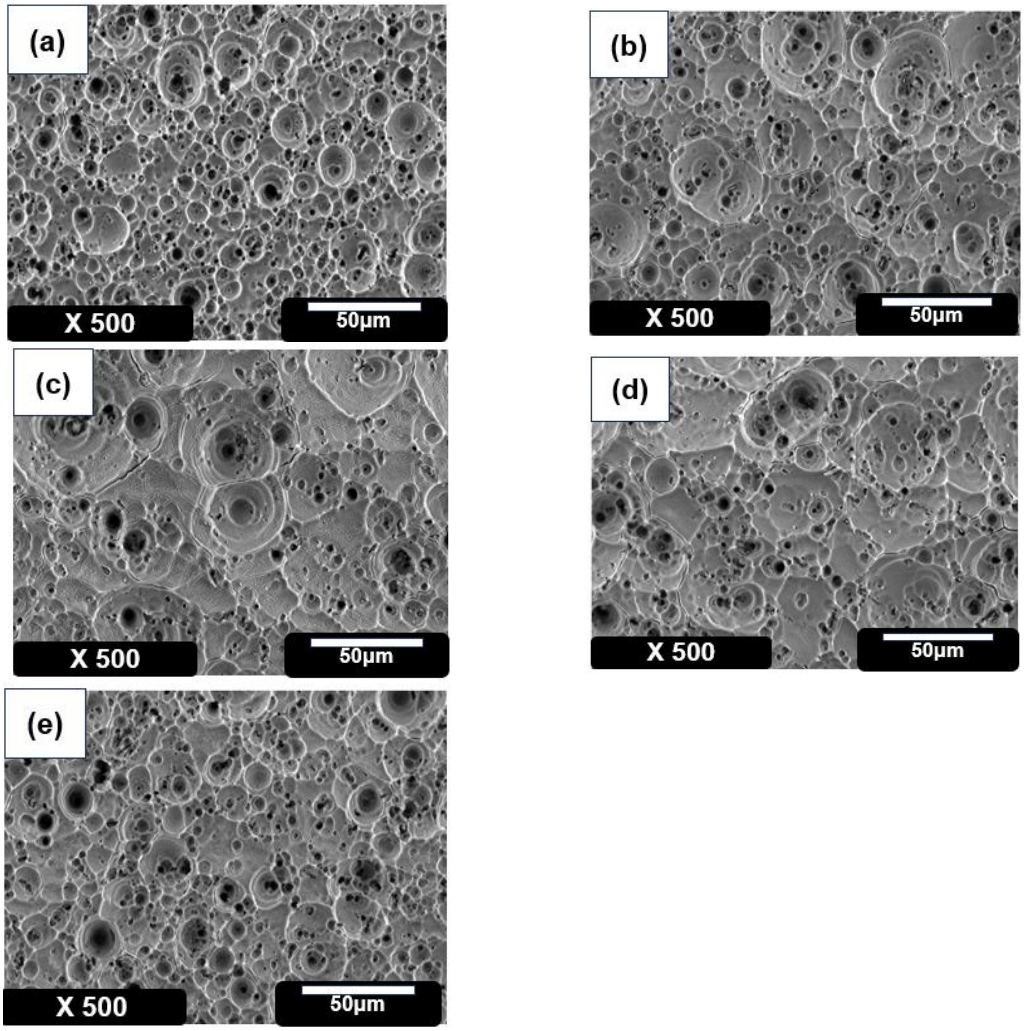

Figure 7: Surface morphologies of composite oxide films with EDX: (a) AAO (b) AAO-CNT (c) AAO-5M (d) AAO-8M (e) AAO-10M.

Table 3: Chemical compositions of the composite oxide films.

Sample label	Element distribution (wt.%)			
	AI	O	C	Other elements
Sample AAO	44.53	45.68	2.91	6.88
Sample AAO-CNTs	39.52	52.03	2.62	5.83
Sample AAO-5M	44.48	46.60	3.82	5.10
Sample AAO-8M	39.13	47.51	7.11	6.25
Sample AAO-10M	43.81	47.02	3.49	5.68

3.2.3 Surface Topographies of Composite Oxide Films

The analysis of surface characteristics, including pore dimension (width and depth) and surface roughness, was conducted using a 3D surface profiler. Figure 8 displays the three-dimensional profiles of AA2017 alloy and composite oxide films. According to the chart presented in Figure 9, AAO sample displayed the highest surface roughness and a non-uniform surface characterized by numerous crests and troughs in which the surface roughness value of around 5.90 ± 0.47 μm, as measured by Ra. AAO sample has the highest Ra value because of its high porosity at the oxide layer (Dong et al., 2022). Sample AAO-CNTs show a 32% decrease in surface roughness to 4.10 ± 0.52 μm. AAO-5M, AAO-8M, and AAO-10M show a decrease in surface roughness to 2.71 ± 0.32 μm (54%), 1.43 ± 0.21 μm (65%), and 2.40 ± 0.37μm (59%) respectively. The decrease in surface roughness is because CNTs act as fillers within the coating matrix, helping to fill in surface imperfections and irregularities. The presence of CNTs can enhance the overall smoothness of the coating and reduce the effective surface roughness of the aluminum alloy (Sarand et al., 2022).The trend for the pore dimension is the same as the roughness in which AAO sample has the highest value which is 46.67 ± 0.14μm for height and 48.37 ± 0.21μm for width. The pores dimension of sample AAO-CNTs start to decrease to 33.45 ± 0.52μm (-28%) for height and 45.32 ± 0.17 (-7%) μm for width. All the functionalized samples that is AAO-5M, AAO-8M, and AAO-10M show a decrease in the height of the pores to 17 ± 0.21 μm (64%), 14.53± 0.33 μm (70%), and 23.34 ± 0.37μm (49%) respectively.

8 molarity of sulphuric acid that is used during the functionalization process of CNTs is the most optimum molarity for the best dispersion since it has the highest reduction of surface roughness. Improved dispersion increases the capacity of CNTs to fill pores by reducing agglomeration and ensuring more equal distribution of individual particles (Chandran et al., 2014). This also can be proved by comparing the Element distribution (wt.%) of carbon for AA0-8M has the highest value that is 7.11 wt.%. The roughness and the pores dimension of the sample AAO-10M started to increase due to the wall of the CNTs that was damaged during the functionalization process. The wall of the Carbon nanotube tends to break or damage when exposed to extremely high concentration of strong acid. Sulfuric acid is regarded as one of the strongest acids. An acid is considered strong if it totally dissociates (ionizes) in a solution and generates a large amount of hydronium ions (H_3O^+). When increasing the molarity of acid, the pH of the solution will decrease. Since there are more acid molecules accessible to participate in the reaction, a rise in acid concentration can speed up the rate of reaction. This can potentially damage the wall of the carbon nanotube (Ma et al., 2010). The structural integrity of the nanotubes may be compromised when the walls of the CNTs are damaged. Defects on the CNT surface like vacancies or dangling bonds may become visible because of the damage. These flaws may lead to higher reactivity and enhance the likelihood that the CNTs may interact with other materials or with one another in a way that encourages agglomeration (Wulan et al., 2018). This is because the interaction between CNTs is significantly influenced by van der Waals forces. These forces can be amplified by exposed defects and damaged walls, which strengthens the attractions between neighboring CNTs and encourages agglomeration. This can also be seen in figure 2(d) in which the sample FCNTs (10M) start to agglomerate as there is more precipitate at the top and bottom of the sample and that the solution is becoming less transparent. CNTs that agglomerates, have the potential to produce uneven surface elevations if they are not dispersed equally throughout the oxide layer. The distribution that is not uniform will cause the pore dimension (width and depth) to increase causing surface roughness to increase.

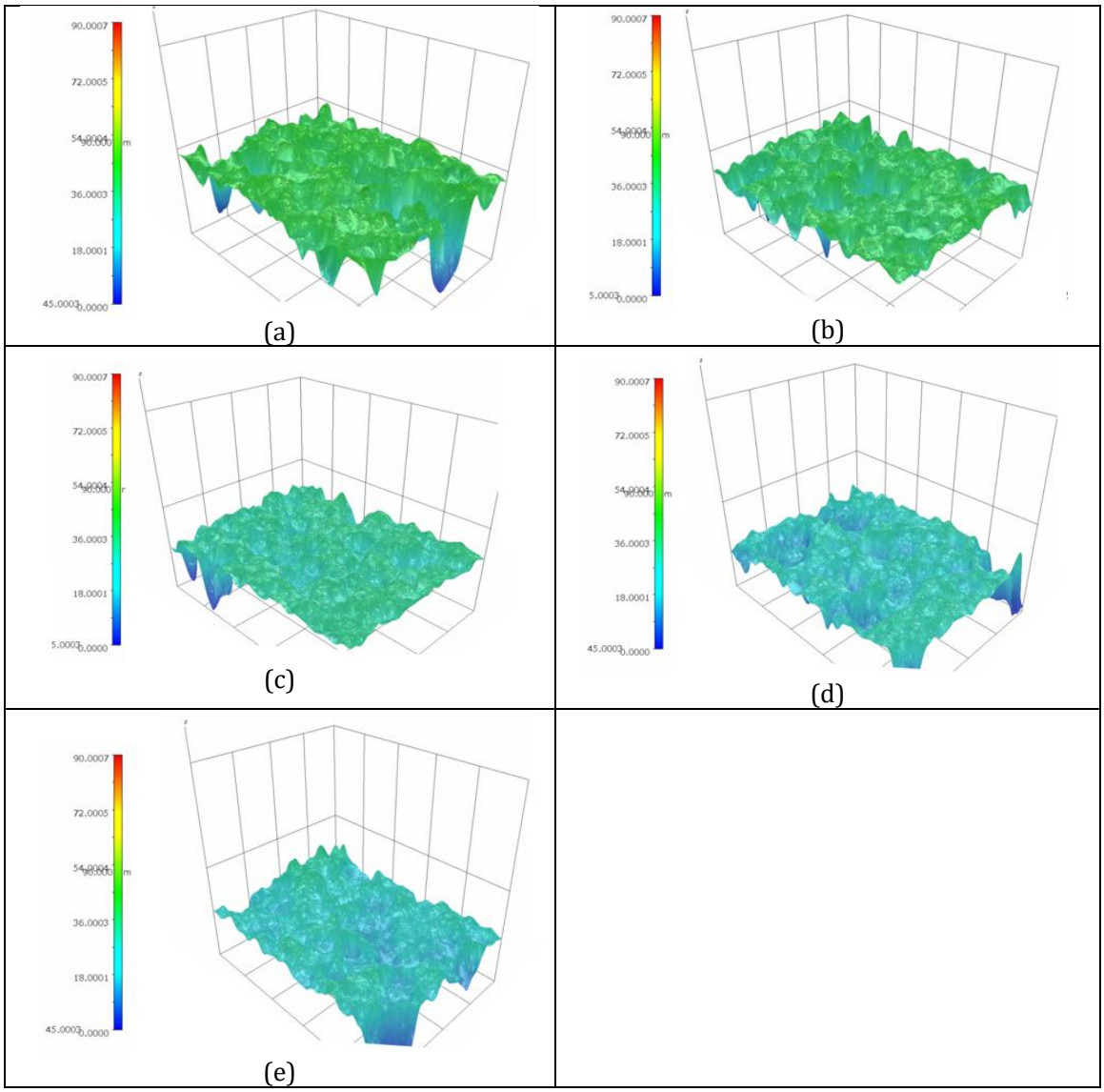

Figure 8: 3D surface profiles of AA2017 alloy and composite oxide films with different molarity: (a) AAO, (b) AAO-CNTs, (c) AAO-5 M, (d) AAO-8M, (e) AAO-10 M.

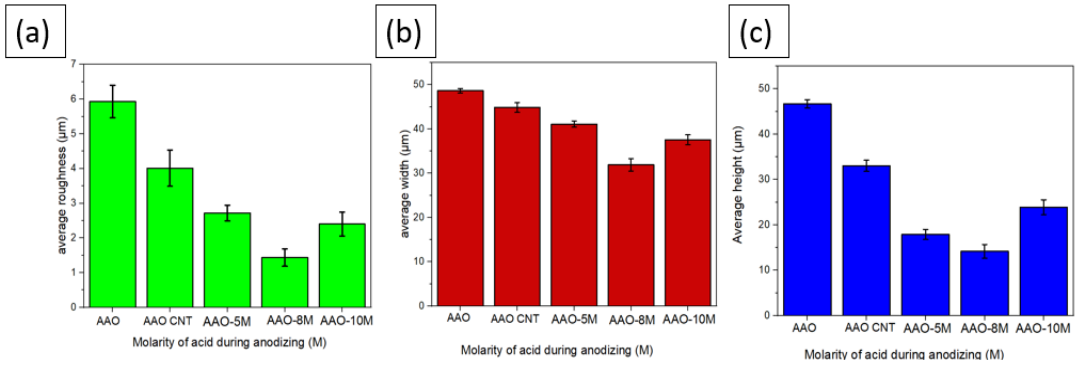

Figure 9: The analysis of surface topography of a) Average surface roughness, b) Average width, and c) Average depth.

3.2.4 Growth Mechanism of Composite Oxide Layer

Aluminum ions with a charge of Al^{3+} are generated at the contact between the metal and oxide. These ions then disperse inside the oxide layer near the interface between the oxide and metal.

$$Al \rightarrow Al^{3+} + 3e$$

The process of electrolyzing water takes place around the pore base, namely near the interface between the electrolyte and oxide.

$$2H_2O \rightarrow 2O^{2-} + 4H^{+}$$

As a result of the presence of an electric field, the O^{2-} ions undergo migration within the barrier layer, moving from the interface between the electrolyte and oxide to the contact between the oxide and metal. At the oxide/metal interface, these ions engage in a reaction with the Al^{3+} ions, resulting in the formation of Al_2O_3.

$$2Al^{3+} + 3O^{2=} \rightarrow Al_2O_3$$

Based on figure 10a when functionalized carbon nanotubes (fCNTs) are introduced into the oxide layer, there is an increased likelihood that these CNTs will occupy the pores. fCNTs possess functional groups that modify the characteristics of CNTs, hence facilitating their enhanced dispersion. The presence of carbon nanotubes (CNTs) within the pores of a material has been observed to contribute to a reduction in porosity.

Based on figure 10b in the case of unfunctionalized carbon nanotubes a lower quantity of CNTs will be present within the pores. The reason for the difficulty of unfunctionalized carbon nanotubes occupying the pores is attributed to their extensively entangled structure, rendering them too large. Therefore, there will be a greater number of pores in comparison to functionalized carbon nanotubes (CNTs).

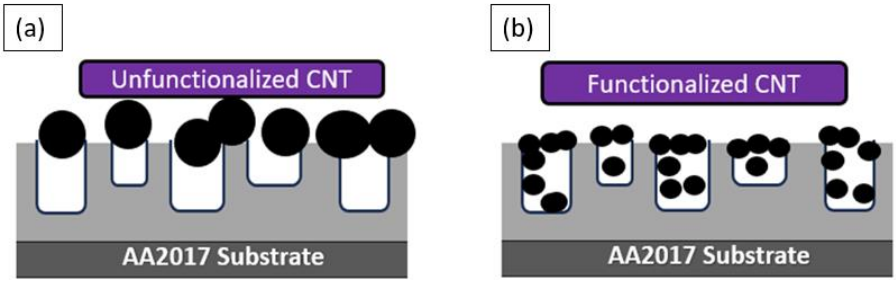

Figure 10: Schematic diagram of growth mechanism of functionalized and unfunctionalized CNTs at the oxide layers.

3.3 Vickers Hardness

The mechanical properties of CNTs composite oxide film were examined by using Vickers microhardness tests on the surface of the samples with the aim of examining the influence of acid molarity during the CNTs functionalization. Figure 11 shows an analysis of the microhardness values of the sample. Based on figure 11 the hardness of the as-receive AA2017 alloy has the lowest microhardness, that is 135 Hv. The hardness of AAO-CNTs shows an increase in hardness to 198 Hv (+47%). Meanwhile the hardness of all the AA2017 with the additional of functionalized CNTs shows an increase in hardness in which AAO-5M, AAO-8M and AAO-10M increase to 212.3 Hv (+57%), 243.9 Hv (+80%) and 226 Hv (+67%) respectively as compared to as-receive AA2017. Among all the sample AAO-8M shows the highest improvement in term of hardness since 8 molarity of sulphuric acid give the highest dispersion of CNTs. This can be proved by the highest reduction of surface roughness as shown in figure 9(a) and size of the pores. Increased dispersion can reduce the amount of cavities, porosity, and flaws that emerge in the material. Defects and voids may serve as sites of stress concentration, causing an early collapse. The material's overall strength and hardness are increased by minimizing these flaws. Beside that carbon nanotubes (CNTs) have the potential to enhance the mechanical properties of composite materials by facilitating effective load transmission when integrated into aluminium alloy matrices. The transmission of mechanical loads from the aluminium matrix to the CNTs, which are known for their high tensile strength and stiffness constitute the mechanism of load transfer. The efficient transfer of load during mechanical stress is made possible by the interaction between CNTs and the AAO matrix. A large amount of the load is carried by the CNTs when external forces are applied, which stops or delays the start and spread of cracks in the AAO matrix (Zhou et al., 2023).

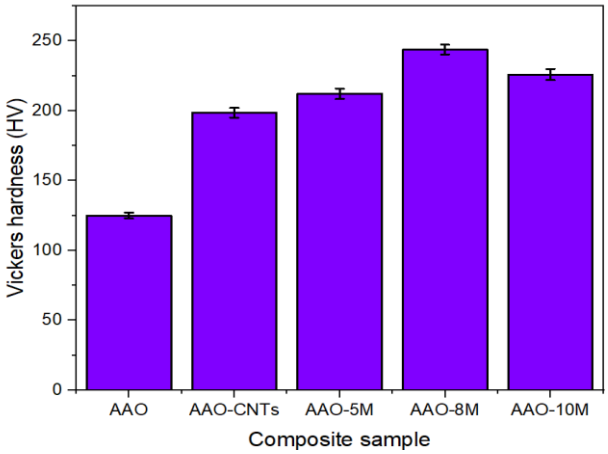

Figure 11. Vickers hardness result: AAO, AAO-CNTs, AAO-5 M, AAO-8M, AAO-10 M.

CONCLUSIONS

In conclusion the oxide layer was successfully formed at the surface of the aluminium alloy after 60 minutes of anodizing time. The influence of different molarity of acid during anodizing was studied. The temperature of the electrolyte increases up to 52.5°C as the anodizing time increases to 60 minutes due to the joule heating that is produced during the anodizing. A strong absorption band that can be observed in the range of 1700 cm-1 to 1765 cm-1 prove the present of carbonyl group (C=O bond) while a broad and strong absorption band in the range of 2500 to 3300 cm-1 prove the present of hydroxyl group (O-H bond) which clearly show that the CNTs was not damage during the functionalization process. AAO-8M has the highest reduction of pores up to 134.95 µm (-72%). The carbon element of AAO-8M has the highest carbon element distribution of up to 7.11 wt.% which proves the present of the CNTs. The pores dimension of AA0-8M has the lowest, that is 14.53± 0.33 µm (-70%). The highest hardness that was achieved was 243.9 Hv from AA0-8M.

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