INVESTIGATION ON IMPROVING MECHANICAL PROPERTIES OF ALUMINIUM ALLOY SHEET 5052-H32 FOR AUTOMOTIVE APPLICATION

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FACULTY OF ENGINEERING

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ABSTRACT

The extensively used of Aluminum alloys are for numerous purposes because of their interesting compound of excellent particular strength, easy to shape, great manufacturing method, high electrical conductivity and availability in various applications, especially for automotive purpose. Aluminium Alloy Sheet 5052-H32 is studied in this research because it is one of the characters of material which is regularly applied for manufacturing automotive application. Two methods are used in this research, Anodization and Plasma Electrolyte Oxidation (PEO). The aims are to build the oxide layer on the surface of Al alloy sheet 5052-H32, also to analyze the microstructure and mechanical properties of deposited coatings as well as to compare the performance of anodization and PEO coating in Al alloy sheet 5052-H32. The oxide layer coating is seen by conducting surface characterization. The improvement of surface hardness by microhardness test from 35.3 HV to 65.8 HV for Anodization coating, and PEO coating is in the average of 67.9 HV. The adhesion test to determine the adhesion strength was achieved by the failure load was around 2023.9 mN for Anodization coating, and PEO coating slightly lower was around 2005.4 mN. The mechanical properties for the surface hardness and adhesion strength of aluminum oxide coated of Al alloy sheet 5052-H32 improved.

Keywords: Aluminium alloy, Anodization, PEO, SEM, Microhardness Test

ABSTRAK

Aluminium aloi digunakan secara meluas untuk pelbagai tujuan kerana kompaun yang menarik, mereka mempunyai kekuatan khusus yang tinggi, kekonduksian elektrik yang tinggi, kebolehbentukan yang besar, proses pembuatan yang hebat, dan ketersediaan dalam banyak aplikasi, terutamanya untuk aplikasi automotif. Lembaran Aluminium Aloi 5052-H32 diamalkan dalam penyelidikan ini kerana ia merupakan salah satu jenis bahan yang biasa digunakan untuk pembuatan aplikasi automotif. Dua kaedah digunakan dalam eksperimen ini, Anodization dan Plasma Elektrolit Oksidasi (PEO). Objektifnya adalah untuk mengarang lapisan oksida pada permukaan lembaran Al aloi 5052-H32, juga untuk menganalisis struktur mikro dan sifat-sifat mekanik salutan yang disimpan dan juga membandingkan prestasi anodalis dan salutan PEO dalam aloi aloi 5052-H32. Pelapisan lapisan oksida jelas dilihat dengan melakukan pengenalan permukaan. Peningkatan kekerasan permukaan oleh ujian mikrohardness dari 35.3 HV kepada 65.8 HV untuk lapisan Anodization dan lapisan PEO adalah dalam purata 67.9 HV. Ujian lekatan untuk menentukan kekuatan lekatan diperolehi oleh beban kegagalan sekitar 2023.9 mN untuk lapisan Anodization, dan lapisan PEO sedikit lebih rendah adalah sekitar 2005.4 mN. Sifat mekanikal untuk kekerasan permukaan dan kekuatan lekatan aluminium oksida yang dilapisi lembaran Al aloi 5052-H32 bertambah baik.

Kata kunci: Aluminium Aloi, Anodization, PEO, SEM, Ujian Microhardness

ACKNOWLEDGMENT

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LIST OF SYMBOLS AND ABBREVIATIONS

PEO   : Plasma Electrolyte Oxidation

SEM   : Scanning Electron Microscopy

FESEM : Field Emission Scanning Electron Microscopy

H32   : Tempers (¼ Hard)

Al   : Aluminium

Ti   : Titanium

Mn   : Manganese

Fe   : Iron

Cu   : Copper

Mg   : Magnesium

Si   : Silicon

Cr   : Chromium

Zn   : Zinc

Gpa   : Gigapascal

Mpa   : Megapascal

HB   : Brinell Hardness

HV   : Vicker Hardness

μm   : Micrometer

A   : Ampere

V   : Volt

^{0}C   : Degree Celcius

dm   : Decimeter

s   : Secon

DC   : Direct Current

AC	:	Alternating Current
mA	:	mili Ampere
g/L	:	Gram per liter
mL	:	mili liter
Wt%	:	Weight Percent
At%	:	Atomic Percent

CHAPTER 1: INTRODUCTION

1.1 Introduction

The extensively used of Aluminum alloys are for numerous purposes because of their interesting compound of excellent particular strength, easy to shape, great manufacturing method, high electrical conductivity and availability in various applications, especially for automotive purpose. In recent years, aluminium alloy has been of growing concern for automotive applications due to the global need in weight-saving for a further decline in fuel consumption. Especially sheet applications for new lightweight structural part and body construction are gaining interest and major efforts have been given by all major producers of semi-finished products of aluminium alloys to meet the main demands.

In recent years, materials and energy are consumed by companies which have performed general attention regarding the need to use them more efficiently. The demand for sustainable growth has considerably improved environmental conditions to enhance the representation of support utilization and to reduce waste generation and polluting radiations. To meet the requirements for environmental sustainability, several approaches are adopted in diverse applications and production practices. For illustration in the transportation sector, which provides 19% to the global greenhouse gas emissions, improving the fuel market and lowering the emissions effected by transportations are significant circumstances in the automotive industry.

The aluminum due to its outstanding qualities has taken an essential place in engineering purposes, making it the most produced non-ferrous metal in the metallurgical industry. Aluminium Alloy Sheet 5052-H32 will be studied in this research because it is one of the types of material which is commonly used for manufacturing automotive application. Two methods will be used in this experiment, Anodization and Plasma Electrolyte Oxidation (PEO). The goals are to fabricate oxide layer on the surface of Al alloy sheet

5052-H32, also to analyze microstructure and mechanical properties of deposited coatings as well as to compare the performance of anodization and PEO coating in Al alloy sheet 5052-H32. The samples are characterized by scanning electron microscopy (SEM), and the hardness will be determined using microhardness test and scratch test. After comparing both of results in the experiment that have been done, the outputs will be applied in the real problem.

1.2 Problem Statement of Research

The level of hardness and corrosion protection in many aluminum alloys limits the factors for general use. In recent years, the most universally accepted and costly answer to this obstacle is to coat aluminum using anodization. Lately, plasma electrolyte oxidation (PEO) has been come to be an attractive option. Notwithstanding significant development in PEO coating for aluminum in recent years, very few studies have regularly compared the performance of anodization and PEO coatings. Hence this research will be conducted in these methods.

1.3 Objective of Research

The objectives of reseach are stated below:

a. To fabricate oxide layer on surface of Al alloy sheet 5052-H32 using Anodization coating and PEO coating.

b. To analyze microstructure and mechanical properties of deposited coatings.

c. To compare the performance of anodization and PEO coating in Al alloy sheet 5052-H32.

1.4 Scope of the Research

This research project is limited only for investigating the sample of Al alloy sheet 5052-H32 as the substrate which will be treated in two methods (Plasma Electrolyte Oxidation and Anodization), some parameters for both methods are determined.

1.5 Report Organization

This paper is split into 5 chapters. Chapter one presents the projects by giving brief information about what is demanded in the coming chapters. The objectives and scope of the project are listed in this chapter. In chapter two, a literature review of previous works that are directly and indirectly related to this project is presented. The methodology used is described in chapter three. Chapter four comprises the experiments conducted and the results obtained. The conclusion of the project and recommendations for future works are given all in chapter five.

CHAPTER 2: LITERATURE RIVIEW

2.1 Introduction

This part presents a review of other's work on the related subject. It highlights the purposes of Al alloy 5052-H32 in different work and all other relevant topics that are related to this project.

2.2 Aluminium alloy and their uses

Aluminum is one of the most sufficient metals in the world and is the third most prevalent element, containing 8% of the earth's coat. The adjustable type of aluminum presents it the most extensively employed metal after steel. Aluminum mixtures themselves have been practiced more than thousands of years ago (Zhang, Zhao, Zuo, & Xiong, 2008). However, new aluminum metal was first manufactured about 170 years ago. During the first 100 years of aluminum metal production, the industry in this field grew rapidly. One of the benefits of aluminum that makes it generally used in the industry is the ease of mixing it with other ingredients. Several advantages of aluminum such as its resilient nature, affordable cost, great endurance, and its obvious to be connected with numerous materials make it frequently applied in various kinds of applications (Zalnezhad, Sarhan, & Hamdi, 2013).

Figure 2.1: Aluminium for Automotive application

Aluminium alloy has great special static force making the alloy very engaging to the aircraft and automotive, continuously in the exploration of developments in the energy performance of transport vehicles. By dispersion of hardening into ageing heat and solvent treatment, great static mechanical properties are usually supported. On the other hand, aluminium alloy displays moderately poor wear and fatigue characteristics, in contrast to more familiar metallic materials like steels. The fatigue durability of a material in the high cycle is around a quarter of its tensile strength. Therefore, surface treatments are employed to aluminium alloy (Schlienger & Teufel, 2003).

Materials such as steel or even titanium have been employed as an application before, yet aluminium alloy has frequently being employed in a type of recent applications. The most advanced found in the application of aluminium alloy require to reducing its weight without endangering overall strength and long life achievement (Canyook, Seubsom, Sang-Ngean, Trirujirapapong, & Taweesup, 2018). This is an area of expansion which is an occasion for developing aluminium alloy mechanical properties. The researchers that are some variety of disciplines are willing to the held results from the broad spectrum of the forms drawing on experience with a variety of methods. In aerospace and automotive applications, where most parts are usually presented to repetitive charging or are in connection with each other. the materials are usually employed with cold working to develop their fatigue display (Peter et al., 2012).

2.3 Aluminium alloy 5052-H32

Aluminium Alloy 5052 is a non-heat-treatable 2½% magnesium, 0.25% chromium alloy generally accessible in flat-rolled coil, sheet and plate from a broad variety of manufacturing mills. Like all the 5000-series high magnesium alloys 5052 has fairly great strength and is hardenable to an important degree by cold performance, allowing a series

of "H" tempers. Alloy 5052 is also created as a treadplate (also identified as chequer plate) with largely industrial purposes. Stucco finish sheet is designed typically in 1.0 – 1.5mm thickness. The alloy is also constructed as illustrated seamless tube, wire and bar, and foil, accessible on indent from Atlas (Aalco, 2009).

2.3.1 Corrosion Resistance

Great in an extensive variety of atmospheric circumstances, in food and architectural purposes and it is also adequate in various marine environments. The magnesium content is moderate enough that it does not allow from the stress corrosion cracking that can influence alloys with more than approximately 3½% Mg, such as 5083 (Austral Wright Metals, 2005).

2.3.2 Heat Treatment

Aluminium Alloy 5052 is not hardenable by heat method. It can be significantly strengthened by cold activity and various "H" tempers are given – most usually H32 (¼ Hard) and H34 (½ Hard) – as well as the light strengthened Temper 0 condition. The alloy automatically age-softens at room temperature shortly following cold work but will ultimately attain a steady circumstance: all flat-rolled mill products are supplied with stable properties. This is usually achieved by a stabilization thermal method, either low-temperature thermal processing or as a consequence of heat transported while working, which appears in the H3x tempers. H2x tempers are more firmly strain hardened and then partially annealed, again to immediately approach the needed constant temper properties. To soften Alloy 5052 it can be strengthened by heating to 345°C, hold until consistent temperature then cool: the flow of cooling is not necessary (Zhang et al., 2008).

2.3.3 Welding

Great weldability by all regular methods: gas, electric and resistance welding. GMAW and GTAW are favored and extensively used to produce structural welds. Filler alloys are

normally 5356 although other features alloys are reasonable. Welding of strain deposited tempers will decrease strengths in the heat-affected zones.

2.3.4 Machining

Machinability of the softer tempers 0 and H32 is weak, with the tougher tempers such as H34 being slightly more apparent to machine.

2.3.5 Typical Applications

Automotive components, small marine craft, food processing equipment, cabinets, and in treadplate form is used for manufacturing and marine flooring, trailers and toolboxes. Stucco finish sheet is practiced in construction paneling and similar purposes (Yard, 2019).

Table 2.1: Composition Specification of Aluminium Alloy 5052

Chemical Element	% Persent
Manganese (Mn)	0.0 - 0.10
Iron (Fe)	0.0 - 0.40
Copper (Cu)	0.0 - 0.10
Magnesium (Mg)	2.20 - 2.80
Silicon (Si)	0.0 - 0.25
Zinc (Zn)	0.0 - 0.10
Chromium (Cr)	0.15 - 0.35
Others (Total)	0.0 - 0.15
Other (Each)	0.0 - 0.05
Aluminium (Al)	Balance

Table 2.2: Physical property of Aluminium Alloy 5052

Physical Property	Value
Density	2.68 g/cm³
Melting Point	605 °C
Thermal Expansion	23.7 x10^-6 /K
Modulus of Elasticity	70 GPa
Thermal Conductivity	138 W/m.K
Electrical Resistivity	0.0495 x10^-6 Ω .m

Table 2.3: Mechanical property of Aluminium Alloy 5052

Mechanical Property	Value
Tensile Strength	210 - 260 MPa
Proof Stress	130 Min MPa
Hardness Brinell	61 HB

2.4 Anodization

Anodization is a method for creating decorative and protective films on articles produced from alumnium and its alloys. It is basically a method where a thick film of aluminium oxide is formed upon the outside of the aluminium over the application of a direct current electrical supply (Canyook et al., 2018).
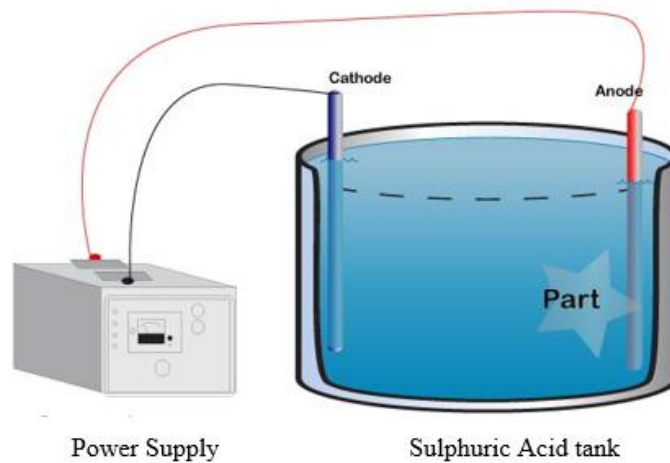
Figure 2.2: Basic of Anodization

When the current is running in the cell the subsequent series of effects is considered to happen. Sulphuric acid starts to decompose, the hydrogen ions going to the cathode where they are degraded to hydrogen gas:

$$2H^+ + 2e{-} \rightarrow H_2(g) \tag{1}$$

Together, negatively energized anions, i.e. hydroxide, sulphate and possibly oxide ions go to the anode. The electrical charge in the circuit creates positively charged aluminium ions (Al^{3+}) to be produced in the anode and turn to move to the cathode. At the anode surface, they respond besides the oxide/hydroxide ions to create aluminium oxide (in the fact of the hydroxide ion, hydrogen ions are delivered toward the solution).

Equations of the anode responses

$$Al \rightarrow Al^{3+} + 3e{-} \tag{2}$$

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

$$2Al^{3+} + 3OH^- \rightarrow Al_2O_3 + 3H^+ \tag{4}$$

For which the overall process is:

$$2Al + 3H_2O \rightarrow Al_2O_3 + 6H^+ + 6e^- \tag{5}$$
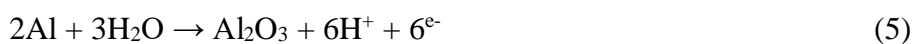
The sulphate ions also act unusual part as the oxide coating contains 12 - 15% sulphate ions. It is recommended that the sulphate ions help the flow of hydrogen ions decreasing the cell voltages needed (Abd-Elnaiem, Mebed, Gaber, & Abdel-Rahim, 2013).

2.4.1 The Development Of The Aluminium Oxide Layer

Pure aluminium responds quickly with oxygen to create aluminium oxide. Once completed the oxide continues strongly bonded to the surface forming an impenetrable layer. Consequently, further effect ceases. The film is extremely thin (0.01µm), and notwithstanding its tenacity, it can be removed by abrasion and chemical corrosion. In such instances, the aluminium is subject to wear or the surface will sign or grow pitted at the site of corrosion (Hirsch, 2009). Anodizing creates much more abundant coatings (12 - 25 µm) which, if suitably sealed, can increase the life of the surface appreciably. Current research in New Zealand has proved that pitting of the surface can be degraded by up to 90% with a 12 µm coating, and by up to 93% with a 25 µm coating. In the beginning stages of anodizing the oxide layer produced is solid and of even flexibility. It gives the most prominent protection to wear and corrosion and consequently is designated the barrier layer. The extension of this layer ceases when the large electrical protection of the oxide reduces the potential of the used voltage in the electrolytic cell (Canyook et al., 2018). The depth of the coating at this step is about 0.08µm. The following growth is so slow and struggles with the acid response:

$$Al_2O_3 + 6H^+ - 2Al^{3+}(aq) + 3H_2O \qquad\qquad (6)$$

which delivers Al^{3+} ions into the solution. Note that the H^+ can be at high concentration near the oxide layer due to one of the anode reactions above. See equation (5).

At base implemented voltages hardly the barrier layer designs. Notwithstanding, the gradual production of Al^{3+} ions tends to smooth out the underlying metal surface and present a brightening result to the particle. Objects such as wheel trims and bumper bars are commonly employed in this method.

At greater voltages, the completion of the layer extends exceeding the barrier layer. Unlike the primary barrier layer this secondary layer, although constitutionally the equal,

has an open pore-like structure: an outcome of the competing anodizing and acid solution methods (Zalnezhad et al., 2013).
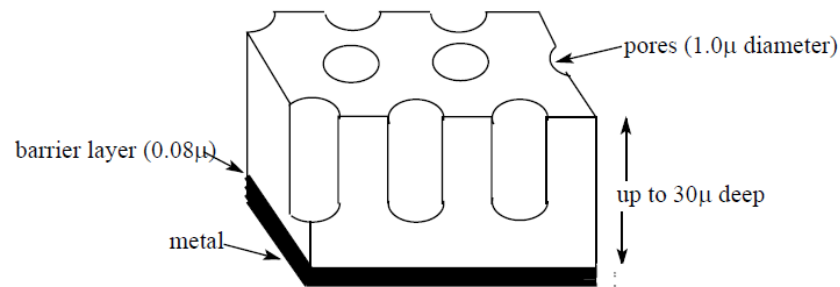
Figure 2.3: Structure of anode layer

The forms needed to create coatings diversify according to the concentration and characteristics of the electrolyte, the voltage-current density implemented, the alloy being anodized and the temperature of the bath. In the bulk of electrolytic plants articles are anodized at a potential of 15 - 20 V and a current density around 1.6 A d-1 m-2: the electrolyte is 3.5 mol L-1 sulfuric acid controlled at temperatures between 20 and 23ºC. Under these circumstances, the quality of the coating is satisfying for most purposes (Zhang et al., 2008). At more eminent electrolyte concentrations and temperatures, also lower voltages or current densities, the acid solution method transpires earlier in the improvement of creating thin, open oxide coatings. Conversely, hard solid coatings are performed at moderate temperatures and large current densities. The conditions installed in each plant are managed by the kind of utilization (Holmberg & Kenneth, 2009).

Table 2.4: Applications of anodised articles and conditions required

Application	Film type	Thickness / μm	Electrolyte
Capacitors - very thin layers of aluminium oxide coating	Barrier	0.01 - 0.5	boric acid, ammonium tartrate
Architectural (silver look)	Sealed, porous	10 - 25	sulfuric acid
Architectural (coloured)	Electrolytically coloured porous coat, sealed	10 - 20	sulfuric acid
Very durable engineering applications	Hard anodising	50 - 120	sulfuric acid, high current density

2.4.2 Sulphuric anodizing

Sulfuric Anodizing is a method that implements a change on aluminum which improves the surface of the material to a normally happening aluminum oxide. The oxide builds up exchanges the surface of the aluminum which then provides greater abrasion defense as well as improved corrosion shelter. The ending from sulfuric anodizing will not only build up the aluminum oxide on the surface but will also comprehend the material the same result. Other than a bright anodize this coating may also be colored in numerous colors. The common application colors incorporate black, clear, green, gold, red, and blue. One other fascinating part of the aluminium oxide is that it is a comprehensive electrical insulator (Abd-Elnaiem et al., 2013).

2.4.3 Change in film thickness during anodization

Anodic film thickness improves with processing time. Nevertheless, various factors such as current density, type of electrolyte, and treatment time, are the thickness extension scale dependant. Originally, there are accelerated and steady improvements in the original thickness, accompanied by anodizing for a considerable period of time, by a small but increasing contraction in thickness in spite of continuous current form. While anodizing, a constant thickness growth of the anodic film and dissolution by chemical intervention

take place. The original thickness is the general thickness minus the thickness of the dissolved oxide. Notwithstanding, the theoretical value is equivalent to processing time when anodizing at continuous current density acid it depends upon faraday's law, which declares that the oxide produced equivalent to the electric charge transferred (ampere-seconds) into the anode (Hirsch, 2009).

2.4.4 Influence of electrolyte concentration

The impact of the development of electrolyte temperature is a proportional increase in the dissolution rate of anodic films produced in thinner, more porous and softer films. For hard coating, low temperatures are used and usually blend with high current densities and strong excitement. Temperatures that vary from 15-250C are usually used in decorative and protective anodization. Maximum thickness is due to higher dissolution energy than electrolytes (Zalnezhad et al., 2013).

2.4.5 Influence of current density

Variations in the current density of 1-2A / dm2 are practiced in ordinary anodization, for some purposes up to 3A / dm2. Current density under this variation makes the film soft, porous and thin. When density increases, films appear more instantaneously with less electrolyte dissolution. As a result, films are more difficult and flow is more limited in areas with excessive heat at very large current densities. Certain conditions are usually managed together with a medium current density of 1A / dm2 which tends to increase image clarity when image reflectivity and accuracy are very important. (Holmberg, Kenneth, 2009).

2.5. PEO Coating Process

2.5.1. Plasma Electrolytic Oxidation

Plasma Electrolytic Oxidation (PEO) is a high voltage electrochemical process which produces hard, dense and wear-resistant oxide coatings on light metals through plasma discharges in the electrolyte composing coatings' required elements.

2.5.2. Characteristics Features of PEO

PEO is an electrochemical surface treatment for producing ceramic coating on light metal substrate such as aluminium, zirconium, magnesium and titanium (Xu, Xia & Li, 2009). This process occurs in a benign electrolyte solution. PEO process is also recognised as micro-arc oxidation, anodic spark deposition, micro-plasma discharging and electrolytic plasma processing as mentioned in the work of (Darband et al, 2017).

Typically, the working electrode (anode) and the counter electrode (cathode) are immersed in a suitable electrolyte of interest. The electrode together with the electrolyte are connected together to a power supply unit that employs high voltage which is up to 800V at current densities of 500 to 2000 mAcm-2 between 2 to 120 minutes in order to allow the formation of plasma discharges at the surface of the metal that lead to the modification of the surface coating. PEO combines the principles of high energy plasma discharges and conventional low voltage. Adekunle (2018) illustrated the basic equipment layout as shown in Figure 2.1.
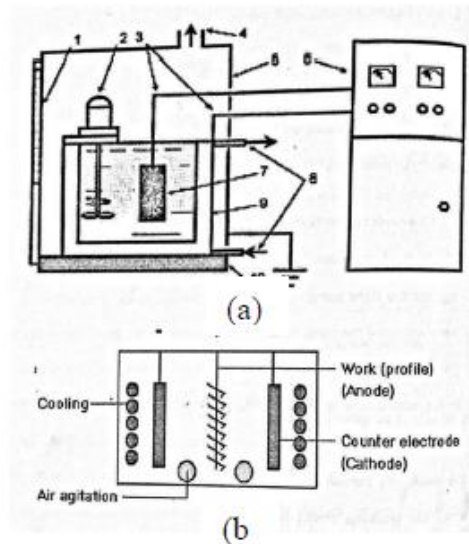
Figure 2.4: Schematic diagram of PEO treatment equipment (Adekunle, 2018)

Based on Figure 2.4 above, (a) represents the arrangement of the PEO treatment

equipment while (b) represents the view of the electrolyte bath;

(a) Arrangement of the PEO treatment equipment

1. Window, 2. Stirrer, 3. Connecting wires, 4. Exhaust/ Ventilation system. 5.

Grounded case, 6. Power supply unit, 7. Workpiece, 8. Cooling system, 9. Bath,

10. Insulating plate

(b) Electrolyte bath

Electrolysis process (dissociation of electrolyte compound into anions and cations) occurs

at a sufficiently high potential difference of a few hundred volts. Anions are attracted

towards the anode while cations towards the cathode. At the anode surface, insulating

layer is formed due to the oxidation process which leads to a slight drop in the applied

current (Li, Liang & Wang, 2013). With a further increase in the applied voltage, the

intense electric field between the electrodes causes crystallization which induces the

formation of micro and nano-crystals on the oxide layer at the anode. The electrolyte is

typically maintained at a temperature in the range of 20 to 70 °C via an external heat

exchanger.

2.5.3. Structures of PEO Coating

The PEO oxide layer generally consists of a porous top layer, compact intermediate layer and thin inner layer which are dense and bonded well to the substrate (Walsh et al., 2009; Wirtz, Brown & Kriven, 1991). The surface morphology is described with many micropores, microcracks and dimples (Curran & Clyne, 2006). During PEO treatment process, micro-arc discharges occur at the anode creating gas bubbles and melting the substrate and oxide layer which leads to the formation of micropores. The microcracks can be attributed to the thermal stress due to the rapid solidification of the molten oxide in the relatively cold electrolyte (Duan, Yan & Wang, 2007).

The high magnification image (Figure 2.5) proves that the coating comprises of a network of fine, surface connected pores which are induced by oxygen. Based on a study conducted by Curran and Clyne in 2006, they stated that high pressures and temperatures are likely to result in notable concentrations of dissolved oxygen in the molten oxides. The evolving oxygen tend to be trapped in the molten substrate as discharges are limited to 10 µm in duration only. Eventually, the trapped oxygen will then create very fine scale interconnected porosity by gaining enough energy from the high temperature of the electrolyte while the melt is rapidly cooled in order to escape. These high density of pores or large voids will then cause poor coating adhesion and reduced strength of the coating layer. Hence, it is crucial to have a uniform, compact and lower porosity of coating to ensure greater hardness and adhesive strength.
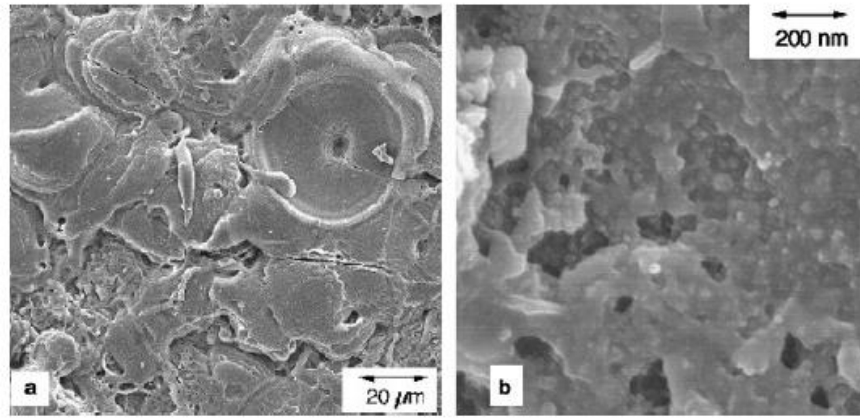
Figure 2.5: (a) Low-magnification and (b) high-magnification image of PEO Coating under Scanning Electron Microscope (SEM) on aluminium alloy substrates. (Curran and Clyne, 2006)

2.5.4. Effects of temperature on PEO Coating

One of the factors that can influence the quality of PEO coating is the electrolyte temperature. Increase in temperature can cause increase in the resulting coating thickness of the specimen as claimed by Michal et al. (2016). Increase in electrolyte temperature increases the conductivity of the electrolyte thus accelerates the chemical reactions at the anode and the cathode. When the conductivity of the electrolyte is high, the energy required to split the reacting molecules is reduced resulting in higher number of free oxygen atoms produced. One of the products of the reacting molecules is oxygen atom which is the primary substance in making up the oxide coating. So, these free oxygen atoms will take part in the chemical reaction on the surface of the anode creating oxide of the anode element. There are desirable range of electrolyte temperature. The electrolyte temperature needs to be maintained consistently at those range for uniform coating thickness. If the temperature is too high, dissolution of the electrolyte will be amplified resulting in low coating thickness and hardness and the speed at which layer dissolves due to effects of electrolyte is greater than the speed at which the oxide layer develops. On the other hand, if the temperature is too low, the oxidation process will become weak (Li, Liang & Wang, 2013; Michal et al., 2016). However, based on a study

conducted by Habazaki et al. (2012), specimen in a higher temperature of electrolyte tends to build thicker coating but lower wear resistance due to higher porosity and lower uniformity compared with the specimen in the lower temperature of electrolyte. This study involves the use of Ti alloy as the specimen which is being processed in the electrolyte of K2Al2O4 (0.15mol/ L), Na3PO4 (0.02mol/ L) and NaOH (0.015mol/ L) at dissimilar electrolyte temperature between 5℃ *to* 40℃. Results demonstrate that the PEO coating composed of higher concentration of α-Al2O3 phase in addition to the Al2TiO5 major phase, showing lower porosity, better uniformity and density at lower temperature of 5℃. However, the thickness variation in the coatings is larger at higher temperatures of 30℃ and 40℃ but with increased porosity and reduced density which is caused by locally concentrated discharges. As a conclusion, temperature of the electrolyte needs to be controlled at a desirable range for uniform coating process. The desirable range of temperature thus need to be discovered through the PEO coating under the new set-up in order to achieve coating with the greatest properties.

2.5.5. Effects of carbon electrode on PEO coating

An electrode is a metal that connects a non-metallic part of a circuit for example electrolyte to ensure continuous flow of current. There are two types of electrode: cathode is where the current leaves acting as the positive extremity of the current system, releasing cations for coating purpose in anodization process; anode is where the current enters acting as the negative extremity of the current system while attracting the cations (Faraday, 1834). There are a few types of materials used as cathode in anodization and PEO process such as stainless steel (current material used), carbon, aluminium and iron. Based on a study conducted by Sreekantan et al. (2011), different types of cathode produce different aspect ratio which means the surface to volume ratio of nanoparticle. Higher aspect ratio exhibits higher surface energy or higher activity of the nanoparticle. Nanotubes formed using stainless steel cathode had a lower aspect ratio (7.00) compared

to the nanotubes formed using carbon (20.00), aluminium (20.00) and iron (21.33) cathodes. Figure 2.6 displays the FESEM images of TiO2 obtained through the article by Sreekantan et al. Based on the images, carbon cathode produced high aspect ratio of nanotubes with a diameter tube of 100 mm and tube length of 2.0μm while stainless steel cathode produced short and non-uniform wall thickness of TiO2 nanotube arrays. Aluminium and iron cathodes produced high aspect ratios of nanotubes, however they are less stable compared to those produced by using carbon cathode as claimed by Allam & Grimes (2008). Iron tends to exhibit greatest rate of mass loss (0.0467 mg/cm2/h), followed by aluminium (0.006 mg/cm2/h) and carbon (0.0015 mg/cm2/h). Hence carbon cathode is the most promising cathode material to produce thicker and uniform PEO coating.
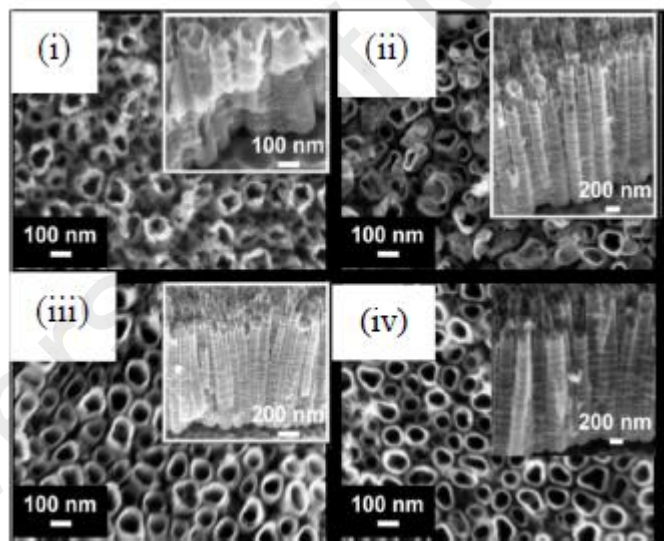
Figure 2.6: FESEM images of TiO2 nanotube arrays manifested using (i) stainless steel,(ii) aluminium, (iii) carbon, and (iv) iron cathodes in glycerol 6 wt% ethylene glycol (EG) and 5 wt% ammonium fluoride (NH4F) at 30 V for 1 hour (Sreekantan et al., 2011)

2.5.3. Effects of electrode distance on PEO coating

PEO process involves two types of electrodes which are cathode, the positive terminal of the current system and anode, the negative terminal. The positions of anode and cathode

in the electrolyte of interest are commonly next to each other. Recently, Ma et al. (2016) stated that the formation of PEO coating is influenced by the distance between anode and cathode. Figure 2.4 displays a graph obtained through Ma et al. (2016) study which indicates decreasing value of average current density as the electrode distance increases. Current flows through the front side is greater compared to the back side of the substrates due to shelter effect (Wei et al., 2007).
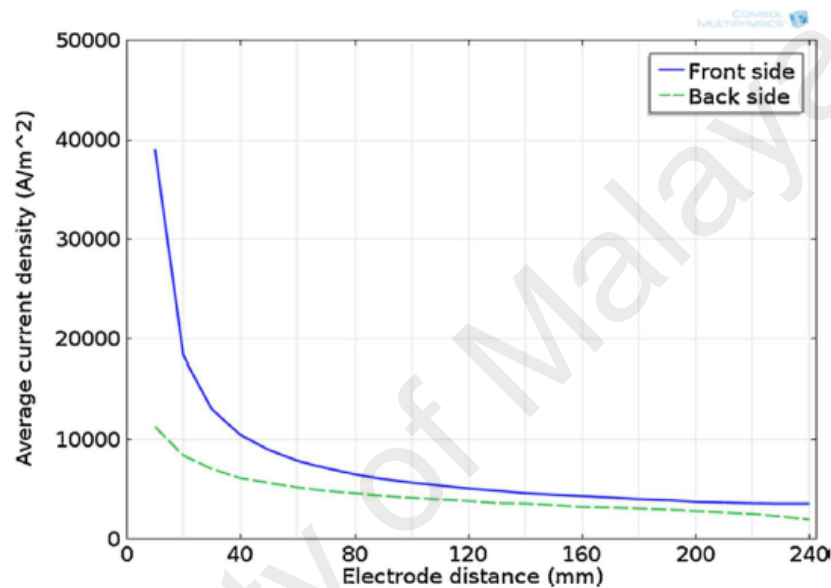

Figure 2.7: Graph of average current density acting on the front and back surfaces of the substrates against electrode distance (Ma et al., 2016)

Based on the Figure 2.7, the average current density declines due to the decreasing electric field strength between anode and cathode. The electric field strength between the electrodes is consistent when the electrode distance is near, thus has a greater effect in the anode current. Hence, the front anode current decomposes reciprocally with the distance between anode and cathode. The peak current which is obtained when the voltage ramps to the set value might decrease when the distance between the anode and the cathode rises. As a result, plasma discharges produced on the surface of the substrate due to the difference between current density and total anode current density decrease (Ma, 2018). Figure 2.5 below which is extracted from the same study shows that the surface of the substrate tends to exhibit uniform coating at the middle range which is 60 – 80 mm. When

the electrode distance is too close, the surface tends to be rough due to thicker deposits formed on it. When the distance is too far, the coating becomes non-uniform again due to less current in the discharges. Finally, graph of dependence of the average coating thickness on the front side and the back side of the substrates against the electrode distance as displayed in Figure 2.9 shows that the thickness of the PEO coating developed on the substrates decreases as the electrode distance increases. This is related to the anode current distribution on the substrates' surface which drops as the distance increases. Therefore, it can be concluded that initial current of anode plays a big role in developing PEO coating. By reducing the distance between anode and cathode helps to minimize the current loss. Thus, higher current density will then produce thicker coating.
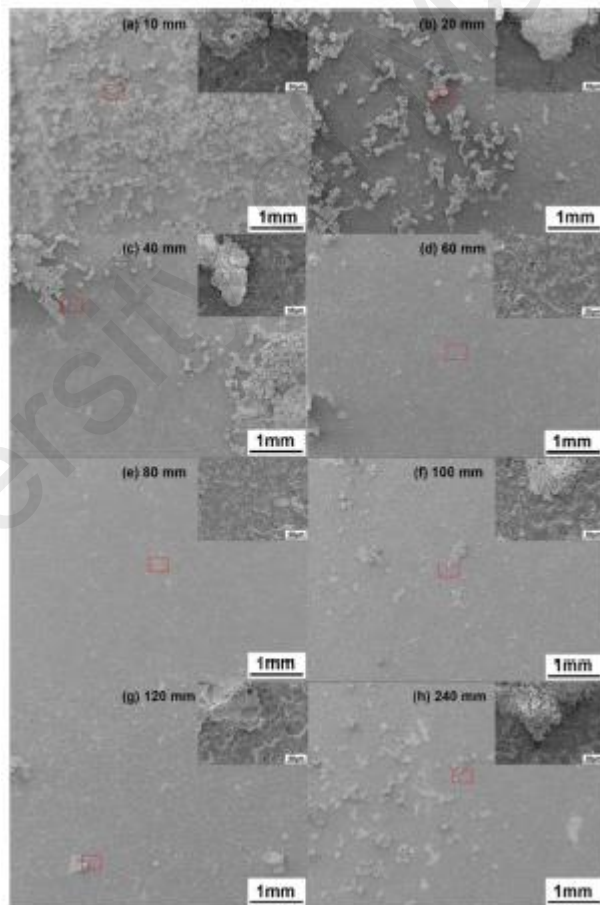

Figure 2.9: Surface structure of PEO coatings developed at different electrode distances (Ma et al., 2016)

CHAPTER 3: METHODOLOGY

3.1 Introduction

This chapter presents the procedure that was undertaken in completing this project. Figure

3.1 below is a flowchart showing the sequence of how the project was conducted.
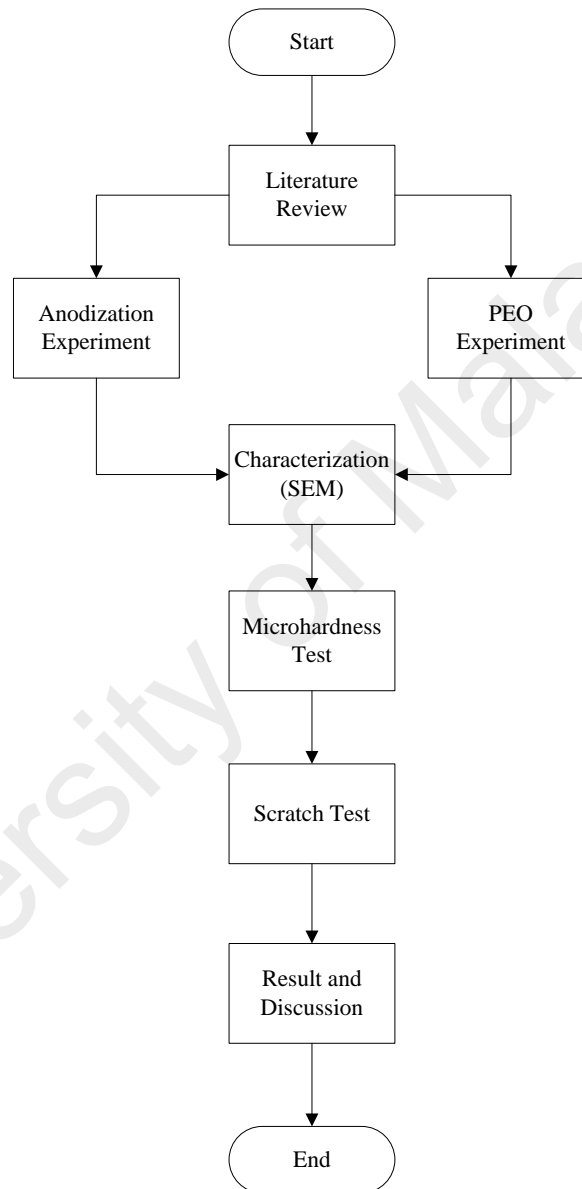
Figure 3.1: Flowchart of the Project

3.2 Material sample

Al alloy sheet 5052-H32 as the substrate which will be treated in two methods (Plasma Electrolyte Oxidation and Anodization).

Figure 3.2: Sample of experiment before treatment

3.3 Sample Preparation

Before beginning with the pretreatment, the piece of aluminum alloy sheet 5052-H32 is smoothed using a polishing machine. To obtain a great result in the polishing process, we require to do in one direction. For managing process of polishing, we can practice kinds of polisher (sandpapers) in different varieties which are started from the number of 800 to the number of 1000. Figure 3.3 shows the polishing machine.

Figure 3.3: Polishing Machine

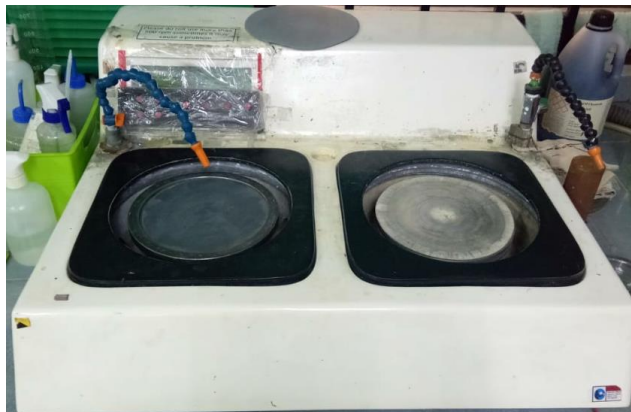After that, place the specimen on the glass (which is filled with the acetone) to immerse the specimen on the ultrasonic bath, for decreasing the cost, we do not need to place acetone on the aluminium bath, it is because we have entered the piece of specimens on the glass. Hence we can apply water and this step is designated cleaning method. It is approximately 10 minutes for the cleaning process applying the ultrasonic bath. Figure 3.4 shows the cleaning process by ultarsonic cleaning bath.

Figure 3.4: Ultrasonic Cleaning Bath

3.4 Anodization Experiment

Serve a handle made out of the corresponding aluminum, it will limit the contamination of the specimen in the anodizing method, so no need to touch it. This holder will later also work as the electrical connection in the anodizing process. DC power supply, connecting wire, carbon graphite, 75 ml beaker, plastic basin are used to conduct the coating process. 7.5 ml of sulphuric acid is diluted with 75ml of distilled water to make 10% concentration of sulphuric acid and it is cooled down to almost 0°C. Figure 3.5 illustrates the scheme of anodization process.
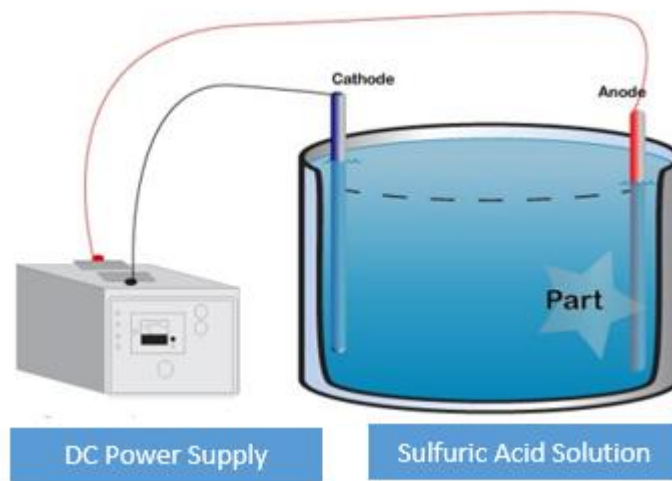
Figure 3.5: Anodization Scheme

For anodizing, the two electrodes setup is used, where the anodize aluminum piece is the anode. The aluminum object is connected to the positive lead. The negative lead is united to the cathode which can be created from carbon graphite. Both electrodes need to be divided and parallel to each other. The anodize substrate requires to be entirely soaked into the electrolyte before beginning the process. The porosity and thickness of this oxide layer depends on the electrical parameters, type of electrolyte H2SO4 (sulfuric acid), its temperature and anodizing time. Figure 3.6 shows the anodization process.
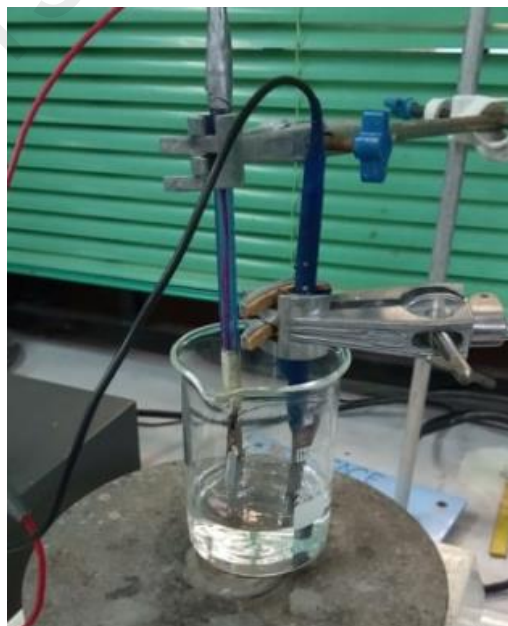
Figuer 3.6: Anodization Process

For limiting from the corrosion, the pores require to be perfectly sealed. There are various methods to perform it, one of the methods is hydrothermal sealing, retaining the anodized specimen into boiling water. Serve the pan and water for boiling the part and it is conducted approximately 20 minutes for boiling. figure 3.7 shows the hydrothermal sealing process.

Figuer 3.7: Hydrothermal sealing

Table 3.1 represents the parameters that used for anodization process. There are some parameters used in anodization process for this experiment (Refdi, 2014).

Table 3.1: Parameters used for anodization process

NO	Parameters	Value
1	Concentration of sulphuric acid	10%
2	Voltage	25 V
3	Current	400mA
4	Temperature	4ºC
5	Time	25 minutes
6	Cathode	Carbon Graphite

3.5 Plasma Elextrolite Oxide (PEO) Experiment

PEO coating is formed in electrolyte containing constant parameters, they are 2 g/L of potassium hydroxide and 9 g/L of sodium silicate and 800 mL distilled water, stainless steel as the cathode. The parameters changed are Voltage under DC power supply and deposition of time. Table 3.2 shows the constant parameters used for peo process and followed by table 3.3 modified parameters used for PEO coating.

Table 3.2: Constant Parameters used for PEO process

NO	Parameters	Value
1	Potassium hydroxide	2 g/L
2	Sodium silicate	9 g/L
3	Cathode	Stainless steel
4	Distilled water	800 mL

Table 3.3: Modified Parameters used for PEO process

	Voltage (V)	Time (Minutes)
Substrate 1	200	15
Substrate 2	200	20
Substrate 3	200	30
Substrate 4	250	15
Substrate 5	250	20
Substrate 6	250	30

3.6 FESEM Characterization
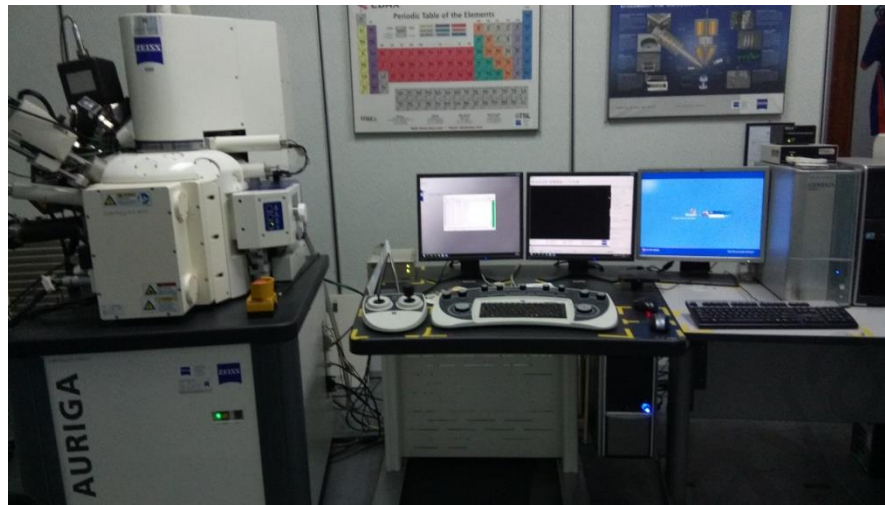
The SEM shows in Figure 3.8.

Figure 3.8 FESEM Equipment

Electrons are released from a field emission source and quickened in a high electrical field gradient. In the high vacuum column, these so-called fundamental electrons are concentrated and diverged by electronic lenses to create a narrow scan beam that bombards the object. As a result, secondary electrons are transmitted from each spot on the target. The angle and velocity of these secondary electrons correlate to the surface structure of the object. A detector catches the secondary electrons and generates an electronic sign. This signal is amplified and converted into a video scan-image that can be observed on a monitor or to a digital picture that can be accumulated and treated further.

3.7 Microhardness Testing

Hardness test systems practice an indenter examination that is relocated into an outside under a particular load. The indentation typically has a designated dwell time. In common mechanical experiment, the size or depth of indentation is measured to define the hardness. Figure 3.9 shows microhardness test.

Figure 3.9: Microhardness Test

3.8 Scracth Test

Figure 3.10 shows Scracth Test.
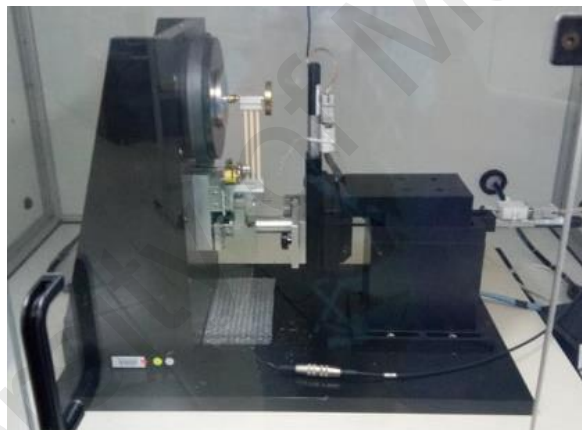
Figure 3.10: Scracth Test

Scratch hardness tests are practiced to ascertain the hardness of a material to scratches and abrasion. Microhardness test is utilized on thin materials with applied load of 1000N and below by using microhardness tester. It is used to calculate the hardness of a material or the material's resistance to penetration. An area of indentation which is made by pressing down a diamond indenter into the depth of the material's hardness is measured to determine the hardness.

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Introduction

In this chapter, the test results of Alluminium alloy sheet 5052-H32 were studied. Moreover, the characterization of samples and some microstructures tests have been conducted.

4.2 The output of Anodization and PEO experiment

These are the output of Anodization and PEO coating, some distinctions from the result is that it depends on the parameters used. Figure 4.1 shows clearly the sample coated differently.
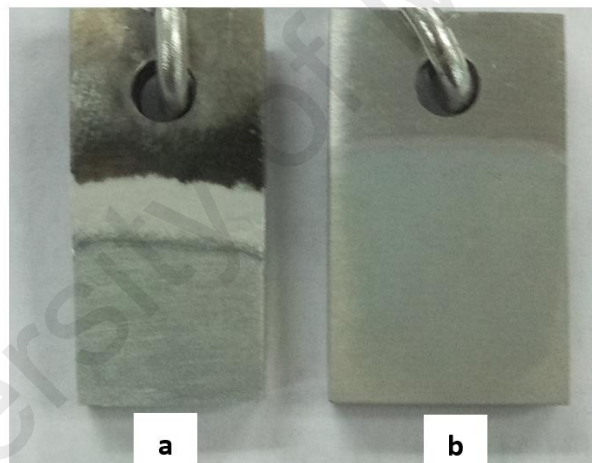

Figure 4.1: coated sample by (a) Anodization, (b) PEO coating methods

4.3 Anodization Coating

4.3.1 Morphological And Elemental Analysis Of The Anodization Coating

Figure 4.2 shows the plan view of the anodization coating. The image is obtained at 500 x magnifications through SEM.
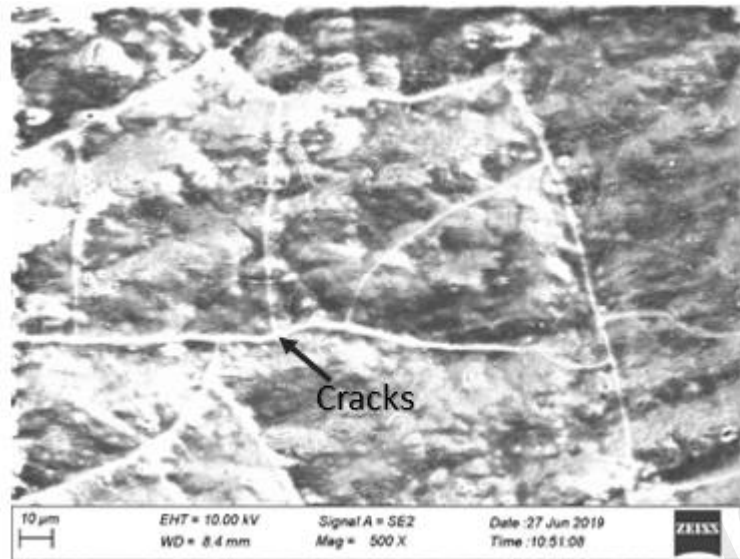
Figure 4.2: Surface Morphology of The Anodization Coating

The surface morphology of anodization coating is presented in Figure 4.2. The aluminium responds quickly with oxygen to create aluminium oxide. Once completed the oxide continues strongly bonded to the surface forming an impenetrable layer. The display of the morphology shows some cracks in the oxide layer. This anodization coating is the combination of 10% concentration of sulphuric acid, 25 V power DC, 400mA, 4°C of temperature, 25 minutes for time deposition, and carbon graphite as the cathode.

4.3.2 The Thickness Of The Anodization Coating

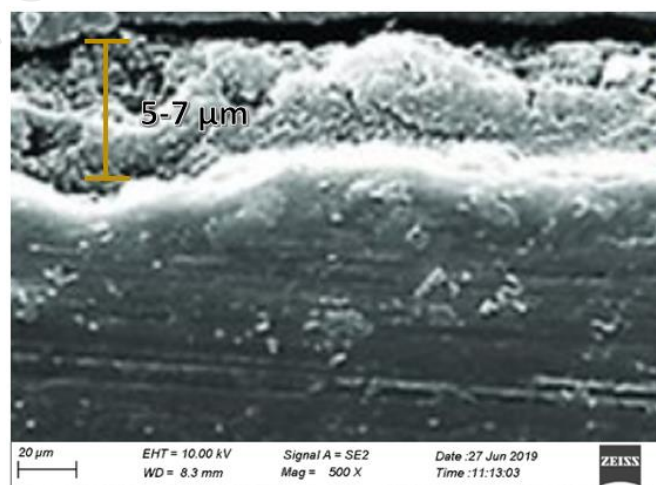Figure 4.3 illustrates the thickness of the anodization coating.

Figure 4.3: Thickness of the anodization coating

Figure 4.3 shows the thickness of anodic film increases with processing time. however, various factors such as current density, electrolyte type, and treatment time also affect thickness (Zalnezhad, Sarhan, & Hamdi, 2013). Initially, there is an increase that is accelerated and steady in the original thickness, then accompanied by anodization for a sufficiently long period of time by a small contraction but an increase in thickness in the form of continuous current. During the anodization process, constant growth of the anodic film thickness and also dissolution with chemical intervention occurs. The original thickness is the general thickness minus the dissolved oxide thickness. Even so, the theoretical value is equivalent to processing time. The continuous current density depends on the faraday law, which states that the oxide produced is equivalent to the electrical charge transferred.

The elemental chemical composition results indicate that the coating is composed of S, O, Zn, Al, C as shown in Figure 4.4. Some elements are from the electrolytes itself, however, there is another element (O) which appears in this result.
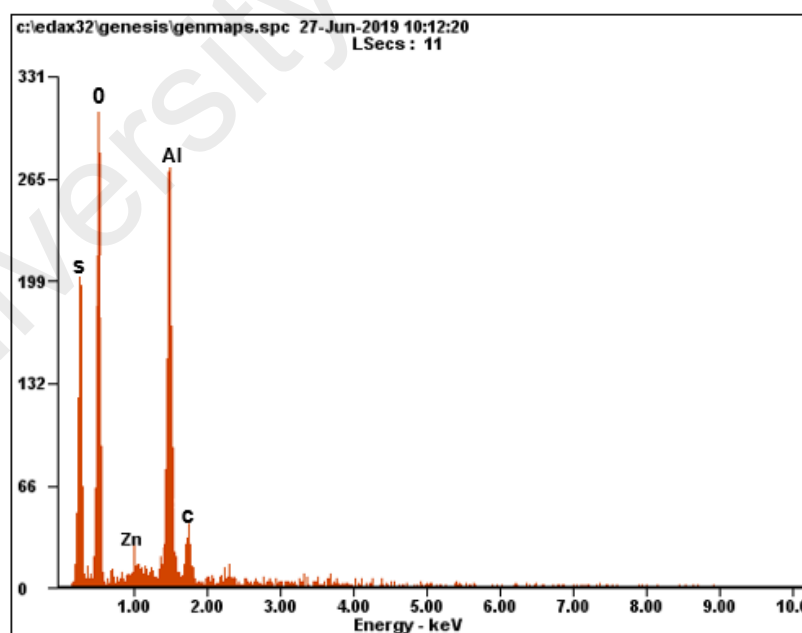

Figure 4.4: Representative EDS spectrum of the Anodization coating

Table 4.1 shows the atomic and weight concentrations of the anodization coating. Wt% is defined as the weight percentage of an element measured divided by the weight of all

elements in the sample multiplied by 100. Weight percentage, wt% gives the concentration of the element in terms of mass fraction of that element in the sample. While atomic % is defined as the number of atoms of that element, at its weight percentage, divided by the total number of atoms in the sample multiplied by 100. S and O are the highest peaks at 35.29 wt% and 34.37 wt% respectively.

Table 4.1: Elemental chemical compositions of the anodization coating

Element	Wt%	At%
S	35,29	47,67
O	34,37	34,86
Zn	1,73	0,43
Al	24,25	14,58
C	3,55	2,05
Matrix	Correction	ZAF

4.3.3 Microhardness Of The Anodization Coating

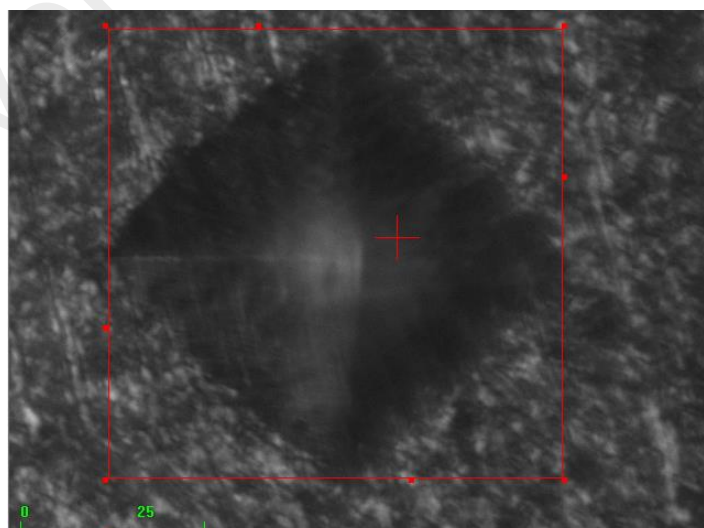Figure 4.5 shows the original sample which is under microhardness vicker test

Figure 4.5: Microhardness Test (Before Coating)

It can be seen from the table 4.2, the constant force used is 980.7 mN, the duration of time is 5 minutes. In order to do this, the result gained is 35.3 HV for the hardness of this material.

Table 4.2: microhardness vicker test result in original material sample

TEST CONDITION	
Test Force (mN)	980.7
Duration Time (Sec)	5
TEST RESULT	
H Length (µm)	61.72
V length (µm)	83.25
Average length (µm)	72.49
Hardness (HV)	35.3

Figure 4.6 shows anodized sample that had been carried out microhardness test
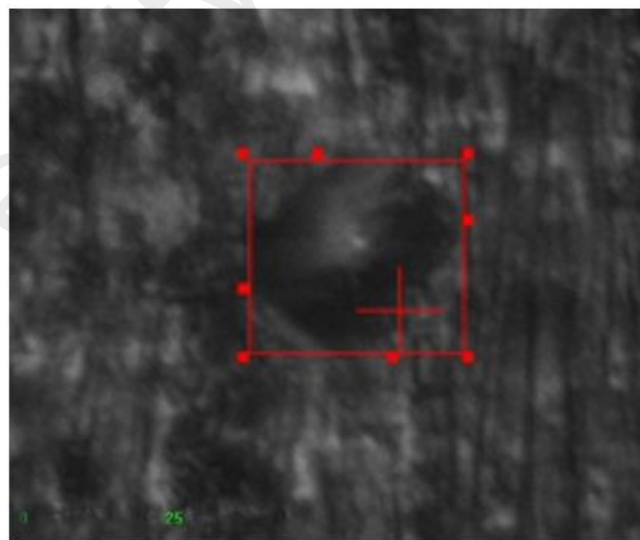

**Figure 4.6: Anodized sample after conducted microhardness test**

It can be seen from the table 4.2, the constant force used is 980.7 mN, the duration of time is 5 minutes. The result increased becoming 65.8 HV.

**Table 4.2: microhardness vicker test result in anodization coating**

TEST CONDITION	
Test Force (mN)	980.7
Duration Time (Sec)	5
TEST RESULT	
H Length (μm)	43.4
V length (μm)	62.75
Average length (μm)	53.08
Hardness (HV)	65.8

4.3.4 Scratch Adhesion Strength Of Anodization Coating

The adhesion strength was graded quantitatively applying a scratch checker. A diamond indenter of 25 μm radius implemented an original load zero toward a sample. The sliding velocity was 5 μm/s. The pressure was improved gradually by 9.2 mN/s. The scratch's length during the scratch test was 1771 μm. In the scratch test, the crucial load could be practiced to determine the adhesion strength. To get the magnitude of the critical load, acoustic signal, friction curve, and microscope observation were utilized. The acoustic signal generated by the delamination of the film could be used to characterize critical load. Scratch adhesion experimentation was conducted on a coated sample to measure critical load Scratch force (adhesion) test on a coated sample and the critical load accompanies with their force and depth versus distance graphs are presented in Figure 4.7.
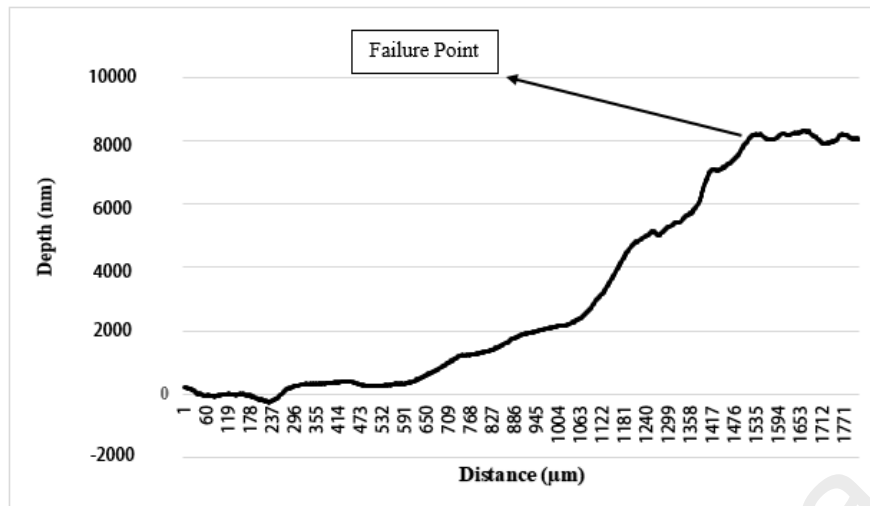
(a)
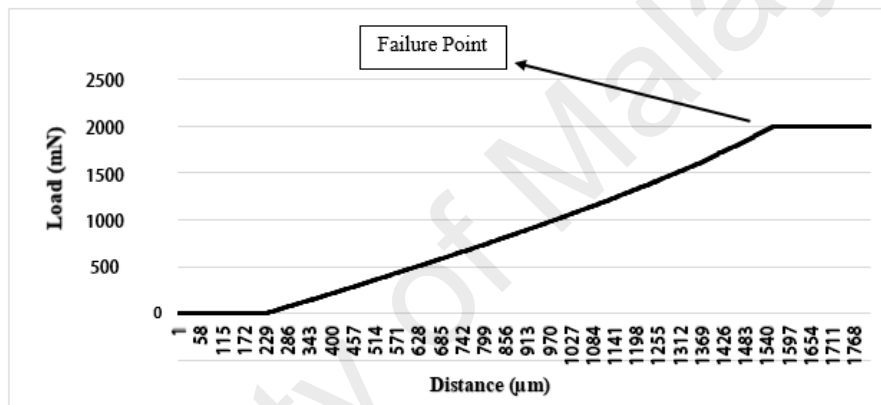
(b)

Figure 4.7: a) Failure point of the scratch force with its depth versus distance graph, b) failure point of the scratch force with its load versus distance graph

4.4 Plasma Electrolytic Oxidation (PEO) Coating

4.4.1 Morphological And Elemental Analysis Of The PEO Coating

The voltage causes a critical value in the growing of oxide film in PEO process, the categorization voltage, and dielectric analysis happens in weak sites across the oxide film. Moreover, sparks are representative of the PEO method and perform a crucial role in the development of the coating. Surface Morphology PEO coating can be seen in figure 4.8.
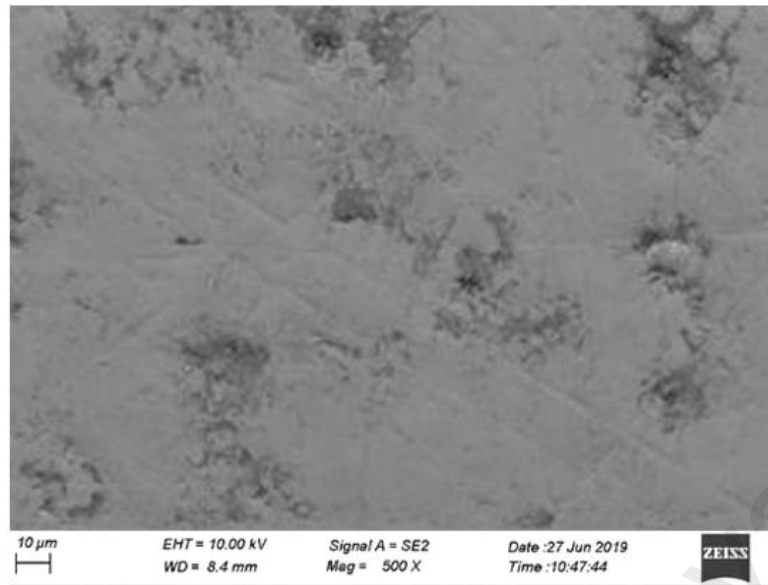
Figure 4.8: Surface Morphology of PEO coating

It seems clearly that the surface morphology of subtrate under PEO coating is smoother and brighter than the substrate under anodization coating. However, some cracks are appeared in the surface. This PEO coating is the combination of 2 g/L of potassium hydroxide, 9 g/L of sodium silicate and 800 mL distilled water and for modifying parameters are DC supply power at 250 V, 30 minutes deposition of time.

4.4.2 Thickness of the PEO coating

The PEO coating can be seen through Figure 4.9.

Figure 4.9: Crosssectional image of PEO coatings

Figure 4.9 illustrates the thickness of PEO coating is influenced by the high electrolyte temperature during the PEO process. The higher temperature of electrolyte generates thicker co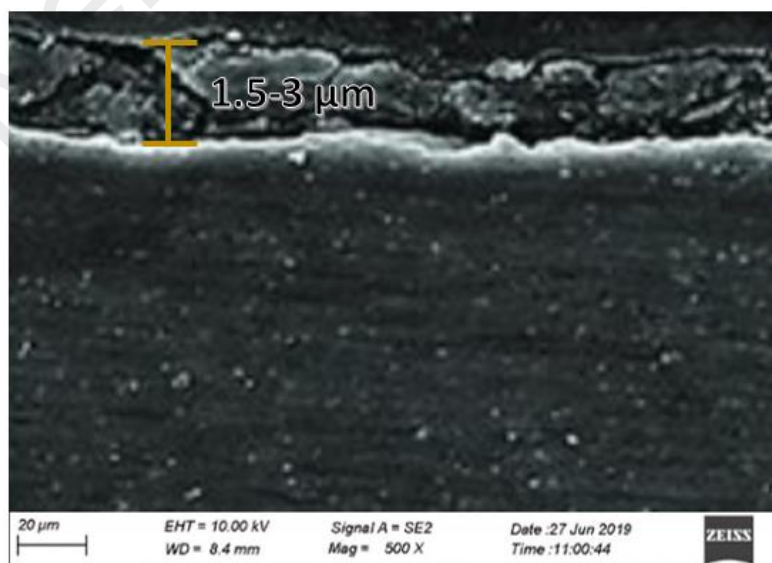ating. Having high temperature during the process influenced the generation of the coating but dissolution of the electrolyte was also amplified. Hence, some areas of the substrate tended to have thinner coating.

The elemental chemical composition results indicate that the coating is composed of C, K, O, Al, Si, Na. It is shown in Figure 4.10. Some elements are from the electrolytes itself, however, there is another element (O) which appears in this result, it indicates that there is oxidation of substrate during PEO process.
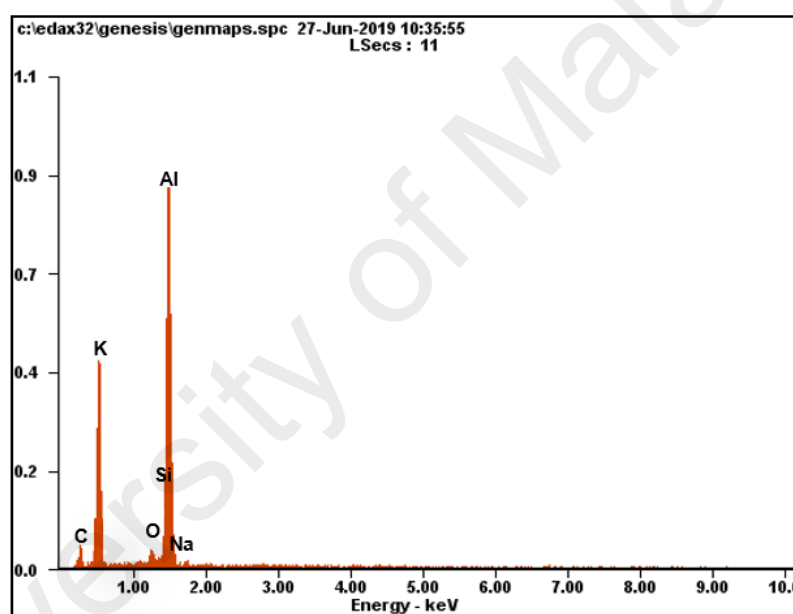
Figure 4.10: Representative EDS spectrum of the PEO coating

Table 4.3 shows the atomic and weight concentrations of the PEO coating. Wt% is defined as the weight percentage of an element measured divided by the weight of all elements in the sample multiplied by 100. Weight percentage, wt% gives the concentration of the element in terms of mass fraction of that element in the sample. While atomic % is defined as the number of atoms of that element, at its weight percentage, divided by the total number of atoms in the sample multiplied by 100. Al is the highest number at 56.89 wt%, while the lowest number is O at 00.51 wt%.

Table 4.3: Elemental chemical compositions of the PEO coating

Element	Wt%	At%
C	08.90	14.97
K	32.74	41.32
O	00.51	0,45
Al	56.89	42.58
Si	00.97	00.69
Na	1,73	0,43
Matrix	Correction	ZAF

4.4.3 Microhardness Vicker Test PEO Coating

The values of hardness are tabulated in Table 4.4 and Figure 4.5 displays the standard deviation of hardness of all the substrates that are deposited via PEO method at different time and voltage. PEO coatings in this experiment is formed in electrolyte containing constant parameters, they are 2 g/L of potassium hydroxide and 9 g/L of sodium silicate and 800 mL distilled water, stainless steel as the cathode. The parameters changed are Voltage under DC power supply and deposition of time.

Table 4.4: Modified Parameters used for PEO process

	Voltage (V)	Time (Minutes)
Substrate 1	200	15
Substrate 2	200	20
Substrate 3	200	30
Substrate 4	250	15
Substrate 5	250	20
Substrate 6	250	30

Table 4.5 PEO coated samples that had been carried out vicker microhardness test

Substrate	1	2	3	4	5	6
Average hardness (HV)	49.2	50.0	63.4	50.2	63.6	67.9
Standard Deviation (σ)	2.6	2.5	3.5	3.7	4.4	7.4

Based on the table 4.5 Substrate 6 (in hydroxide 2g/l, sodium silicate 9 g/l, voltage 250 v, time 30 minutes, distilled water 800 ml, cathode stainless steel) shows the highest hardness at average of 67.9 HV which is in the condition of test force at 980.7 mN and the duration time is 5 secons. Coatings produced with higher porosity have lower hardness, that is why the coating hardness of a sample strongly depending on the coating compactness. The more compact of the coating, the higher the hardness. Therefore, porosity levels is important to be controlled to improve the hardness coating of a sample. These can be done by manipulating the process parameters such as voltage and deposition time. Other factors that affecting the hardness of a coating is the crystalline and amorphous phases which are resulted from the high temperatures that the coating reached locally (Xu, Xia & Li, 2009). These phases can give rise to coatings of high hardness and wear resistance. Standard deviation is used to show how much variation from the mean exists. A low standard deviation is defined as the data points that is close to the mean value. While a high standard deviation is defined as the data points that are spread out over a large range of values. Standard deviation of coating hardness correlate with the structural homogeneity of PEO layers. From the data obtained in Figure 4.5, Substrates (1,2 and 3) have the similar value standard deviation, while Substrate 6 displays the highest standard deviation which is might be due to the homogeneity of PEO coating on the surface.
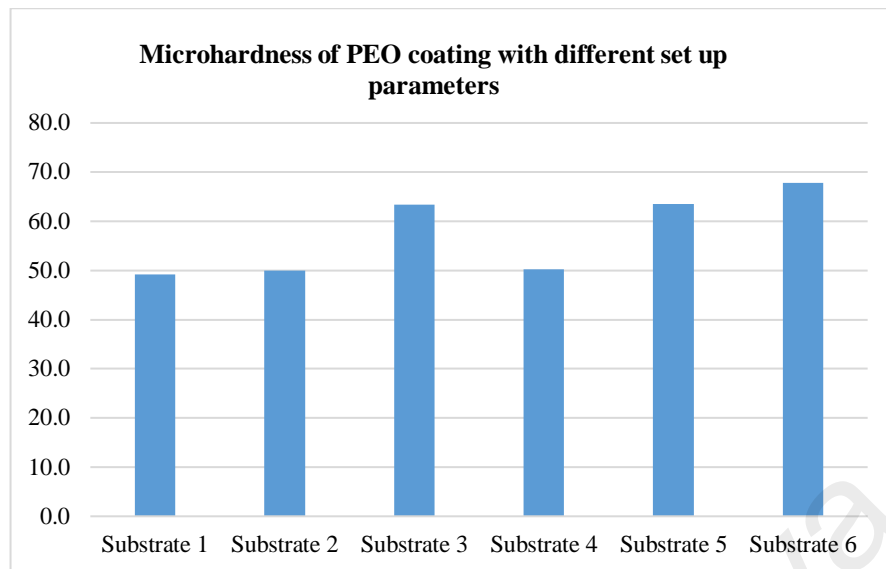
**Figure 4.11: Microhardness of PEO coating with different set up parameters**

As depicted in figure 4.10 the highest number of the hardness of PEO coating is substrate number 6 at 67,9 HV which is the combination of 2 g/L of potassium hydroxide, 9 g/L of sodium silicate and 800 mL distilled water and for modifying parameters are DC supply power at 250 V, 30 minutes deposition of time, respectively. While, the similar second highest is substrate number 3 (63.4 HV) and substrate number 5 (63.6 HV). The three similar lowest hardness value in the PEO coating is substrate number 1, number 2, and number 4 at around 50 HV. There is correlation with the result of hardness, the value shows that the larger value of standard deviation illustrates the higher value of hardness.

4.4.4 Scratch Test (PEO)

It is similar to the anodization coating sample treatment, the film-to-substrate adhesion strength was measured quantitatively using a scratch tester. A diamond indenter of 25 μm radius implemented an original load zero toward a sample. The sliding velocity was 5 μm/s. The pressure was improved gradually by 9.2 mN/s. The scratch's length during the scratch test was 1743 μm. Scratch force (adhesion) test on a coated sample and the critical load accompanies with their force and depth versus distance graphs are depicted in Figure 4.12.
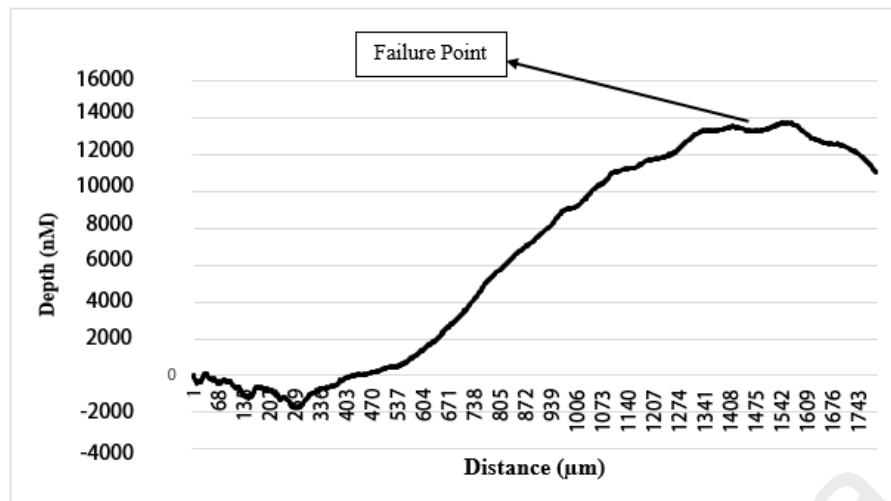
(a)
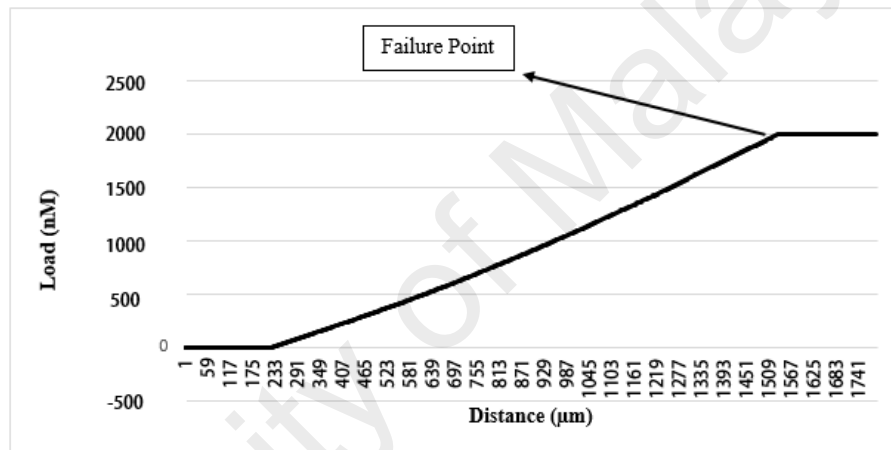
(b)

Figure 4.12: a) Failure point of the scratch force with its depth versus distance graph, b) failure point of the scratch force with its load versus distance graph

CHAPTER 5: CONCLUSIONS

5.1 Summary

In this research project, Anodization coating and Plasma Elextrolite Oxidation (PEO) were used to do surface treatment to improve the performance of Al alloy sheet 5052-H32 for automotive application. Based on the experimental results, the following conclusion can be drawn:

a) The oxide layer coating clearly seen by conducting surface characterization. The value of thickness for Anodiztion coating is in the range of 5-7 μm, while the PEO coating thickness is in the range of 1.5-3 μm.

b) The improvement of surface hardness by microhardness test from 35.3 HV to 65.8 HV for Anodization coating, and PEO coating is in the average of 67.9 HV.

c) The adhesion test to determine the adhesion strength was achieved by the failure load was around 2023.9 mN for Anodization coating, and PEO coating slightly lower was around 2005.4 mN.

d) According to the result of experiment, the mechanical properties for the surface hardness and adhesion strength of aluminium oxide coated of Al alloy sheet 5052-H32 increased.

5.2 Future Work

The following points are suggested for future reserach work

a) Tribological properties have to investigate for Anodization coating and PEO coating of Al alloy sheet 5052-H32.

b) Using different parameters to gain the best coated layer.

c) Applying different methods to improving the mechanical properties of Al alloy sheet 5052-H32.

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