

**PRELIMINARY STUDY ON EFFECT OF TEMPERATURE ON HYDROPHOBIC COATING ON
METAL**

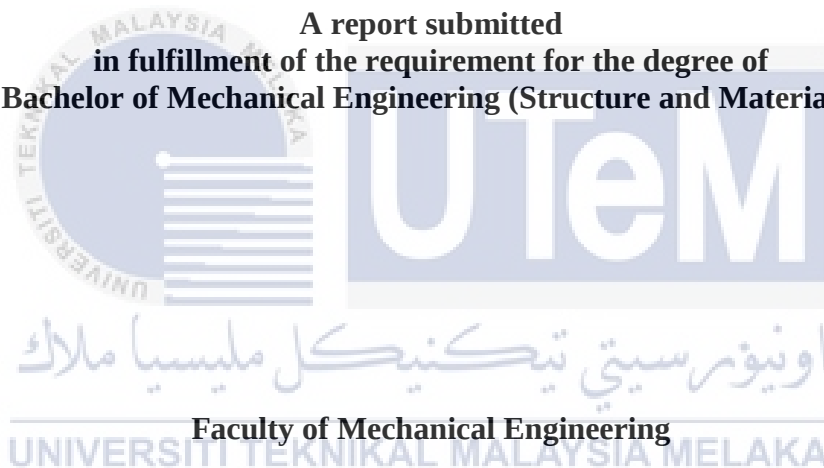


UNIVERSITI TEKNIKAL MALAYSIA MELAKA

**PRELIMINARY STUDY ON EFFECT OF TEMPERATURE ON HYDROPHOBIC
COATING ON METAL**

NUR IZZATI BINTI KUSNAN

A report submitted
in fulfillment of the requirement for the degree of
Bachelor of Mechanical Engineering (Structure and Materials)



Faculty of Mechanical Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2017

DECLARATION

I declare that this project report entitled “Preliminary study on effect of temperature on hydrophobic coating on metal” is the result of my own work except as cited in the references

Signature :

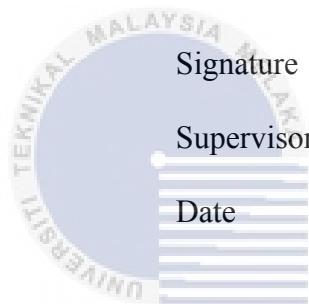
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APPROVAL

I hereby declare that I have read this project and in my opinion this report is sufficient in terms of scope and quality for the award of degree of Bachelor of Mechanical Engineering (Structure & Materials)



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Date

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اونيورسيتي تيكنيكل مليسيا ملاك

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

DEDICATION

To my beloved mother and father



ABSTRACT

Hydrophobic coating has become an important approach in coating technology because it can prevent the surface from harmful effect of water and moisture in the environment. It inhibits high water repellency compared to other coatings. In this study, aluminum substrate has been used to analyze the effect of temperature on the hydrophobic coating to solve the problem in automotive industry, where aluminum that used as a vehicle frame and body is leads to the corrosion and oxides when it contact with water and direct sun even it was coated using polymer based paint to improve it mechanical strength. Therefore, the objectives of this study are to study the effect of temperature for polymer surface on hydrophobic properties at different temperature and time, to analyze the effect of temperature on surface morphology and surface roughness and to perform correlation of these microstructure properties with hydrophobicity of polymer surface. There are two different experiment were conducted which are heat treatment with varies the temperature and heat treatment with varies the times to analyze the effect of temperature on hydrophobic coating by measuring the contact angle, surface roughness, surface morphology and correlation of these microstructural properties with hydrophobicity of the surface. The measurement of the contact angle was done using MSP-3020 Digital Microscope Software, where the digital microscope is connecting to the computer and the analysis on the surface morphology and surface roughness were done using Alicona Infinite Focus *G⁴*. The result indicates that, to produce hydrophobic surface, the temperature of the heat treatment must until reach optimum value with shorter time of exposure to create high surface roughness with complex surface morphology which is at temperature 175°C at times is 5 minutes to 10 minutes. This is because at high temperature, the analysis of the surface morphology shows the surface roughness significantly increases the microstructure change and increases the value of the contact angle, it contributes in hydrophobicity. Before and after this condition, where the temperature of the heat treatment is lower and the longer duration of time exposure to the heat treatment the contact angle starts decreasing and no longer remains as a hydrophobic surface.

ABSTRAK

Lapisan hidrofobik telah menjadi satu pendekatan yang penting dalam teknologi salutan kerana ia dapat menghalang permukaan daripada kesan berbahaya air dan kelembapan dalam persekitaran. Ia dapat menghalang kelekatan air yang tinggi berbanding dengan salutan yang lain. Dalam kajian ini, aluminium substrat telah digunakan untuk menganalisis kesan suhu ke atas lapisan hidrofobik untuk menyelesaikan masalah dalam industri automotif, di mana aluminium yang digunakan sebagai bingkai dan badan kenderaan mengalami kakisan dan oksida apabila ia terdedah secara langsung kepada air dan matahari walaupun ia telah disalut menggunakan cat berasaskan polymer untuk memperbaiki kekuatan mekanikalnya. Oleh itu, objektif kajian ini adalah untuk mengkaji kesan suhu kepada permukaan polimer ke atas sifat-sifat hidrofobik pada suhu dan masa yang berbeza, untuk menganalisa kesan suhu ke atas permukaan morfologi dan kekasaran permukaan dan untuk menghasilkan perkaitan antara ciri-ciri mikrostruktur ini dengan sifat hidrofobik permukaan polimer. Terdapat dua eksperimen yang berbeza telah dijalankan iaitu rawatan haba dengan berbeza suhu dan rawatan hada dengan pilihan masa untuk menganalisis kesan suhu ke atas lapisan hidrofobik dengan mengukur sudut sentuh, kekasaran permukaan, permukaan morfologi dan hubungan kait antara ciri-ciri mikrostruktur ini dengan sifat hidrofobik permukaan. Pengukuran sudut sentuh telah dilakukan dengan menggunakan Perisian MSP-3080 Mikroskop Digital, di mana mikroskop digital telah disambung ke komputer dan analisis mengenai permukaan morfologi dan permukaan kekasaran dilakukan menggunakan Alicona Infinite Focus G⁴. Hasil keputusan menunjukkan bahawa, untuk menghasilkan permukaan hidrofobik, suhu rawatan haba mesti mencapai suhu yang optimum dengan masa pendedahan yang lebih singkat untuk menghasilkan permukaan yang kasar yang lebih tinggi dengan permukaan morfologi yang kompleks iaitu pada suhu 175°C dan pada waktu 5 minit ke 10 minit. Ini kerana, pada suhu yang tinggi, analisis permukaan morfologi menunjukkan kekasaran permukaan meningkat dengan ketara, perubahan mikrostruktur dan meningkatkan nilai sudut sentuh, ia menyumbang dalam sifat hidrofobik. Sebelum dan selepas keadaan ini, di mana suhu rawatan haba yang rendah dan masa pendedahan pada rawatan haba yang lama, sudut sentuh mula berkurang dan permukaan tersebut tidak lagi berada dalam keadaan hidrofobik.

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In the name of Allah the Most Gracious Most Merciful

Alhamdulillah for giving me the courage and the determination with strength to complete this research project. Also, with His guidance I able to finish up this research, despite all difficulties.

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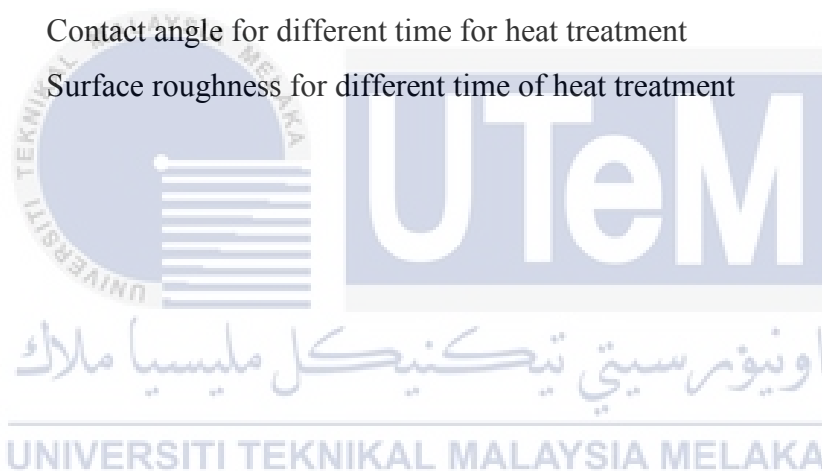
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LIST OF ABBREVIATIONS

USB = Universal Serial Bus



LIST OF SYMBOL

A = sliding angle

H = Hysteresis

R_A = Surface Roughness



CHAPTER 1

INTRODUCTION

1.1 Background

Superhydrophobic coating is inspired by the unique structure of the Lotus leaf refers to self-cleaning mechanism, where the image of water droplet on the Lotus leaf is as shown in Figure 1.1. This is known as “lotus effect”, which is occurred only due to low energy surface topography with hierarchical morphology. A hierarchical morphology allows the formation of air pockets that reduced the contact area between an applied water droplet and the surface (Satish et al, 2013). A superhydrophobic surface are water repellent with large water static contact angle, (CA) above 150° and low sliding angle lower than 10° (Xiuyong et al, 2014). The superhydrophobic provides the surface with various used in many applications such as self-cleaning, anti-biofouling, anti-icing, drag-reducing, and corrosion resistance (Xiuyong et al, 2014).



Figure 1.1: Droplet of water on Lotus leaf (Bharat, B., and Yong, C. J., 2011)

Whereas, hydrophobic coating is the earlier stage of water repellent coating before achieve a superhydrophobic coating. According to Bharat and Yong (2011) states that hydrophobic is usually used to define the contact of a solid surface with any liquid. The surface will have low energy and build of non-polar molecules. The properties of the hydrophobic coating can be improved to become a superhydrophobic are by increasing the surface roughness and lower the surface energy. According to Kubiak and Mathia (2014), the contact angle of hydrophobic coating is greater than 90° and lowers than 150° . This coating has very good wettability properties.

Various strategies have been develops for coating technologies to aluminum material to prevents it's from harmful effects of water and moisture in the environment. Hydrophobic coating exhibit high water repellency compared to other coatings. The water molecule will roll with a slight applied force from the coating surface. Existing coating that applied on the aluminum material is polymer based paint like spray paint, where in certain duration of times, the painting unable to protect the material any longer when it's exposed to water and harsh environment. Aluminum is widely used in many industrial fields as a basic material because of its good properties in excellent thermal and electrical conductivity but poor in corrosion resistance even it was coated using polymer based paint to improve their mechanical properties. Therefore, aluminum with hydrophobic coating can improved corrosion performance for aluminum material.

The properties of hydrophobic and superhydrophobic coating such as contact angle, surface morphology and the surface roughness can be analyzed on the substrates temperature of the thin film hydrophobic coating. High temperature resistance is one of the main criteria for hydrophobic coating to protect the coating from undergoes oxidation and

resistance to the corrosive. Therefore, effect of temperature on the coating is important to be analyzing because temperature can give impact on the microstructure of the coating, crystalline quality of the coating and the grain size of the coating.

In this work, the samples will be tested over the different temperature and different time of the sample exposure to the heat treatment in order to investigate the effect of temperature of hydrophobic coating on the metal substrate. The contact angle of water will be measure using digital microscope. The surface roughness and the surface morphology of the surface sample are measured and analyzed using Alicona Infinite Focus G^4 . All the result obtained will be evaluates to perform the correlation of this microstructure properties with hydrophobicity of the material.

1.2 Problem Statement

Superhydrophobic and hydrophobic coating has the application of water proofing, self-cleaning, anti-corrosion, and anti-icing. These hydrophobicity concepts have been applied in textiles, paints, electric devices, automotive industry and car glass. Vehicle frame and body in automotive industry usually made from metals like aluminum. The vehicle frame was coated with polymer based paint like spray paint to avoid corrosion and oxides when it contact with water and direct sun. However after certain times, the painting cannot protects the vehicles frame and body anymore when it's exposed to water and harsh environment. Therefore, the best solution to solve this problem by doing a heat treatment on polymer surface to improve its mechanical properties and its hydrophobicity to obtain self-cleaning and anti-corrosion behaviors coating to be applied on the vehicles frame.

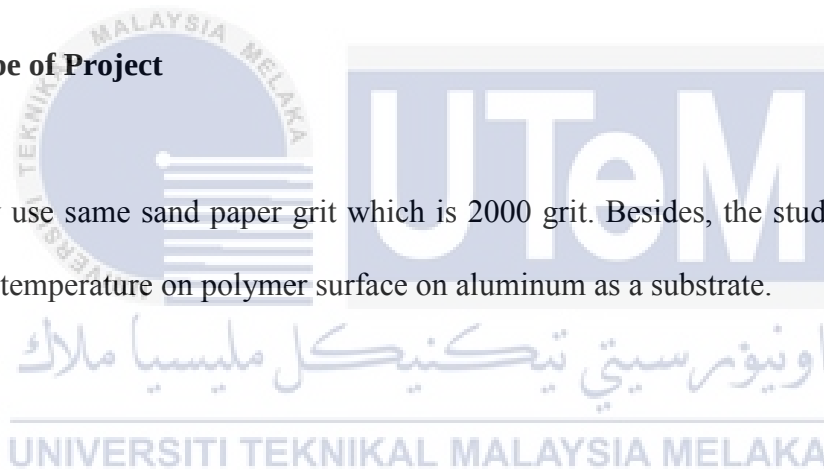
1.3 Objective

The objectives of this project are as follows:

1. To study the effect of temperature for polymer surface on hydrophobic properties at different temperature and time.
2. To analyze the effect of temperature on surface morphology and surface roughness.
3. To perform correlation of the microstructure properties with hydrophobicity of polymer surface.

1.4 Scope of Project

Only use same sand paper grit which is 2000 grit. Besides, the study only focuses on effect of temperature on polymer surface on aluminum as a substrate.



1.5 General Methodology

The work procedures that need to be done to achieve the objective in this project are listed below.

1. Problem statement

The problem will be analyze and investigate to find the best solution in this project.

2. Literature review

Journals, articles, or any materials and equipment regarding the project will be reviewed.

3. Identification

Substrate with polymer coatings will be identified to be tested in the different temperature and time.

4. Sample preparation

Each sample for each experiment will be prepared in specific measurement. Total sample for each experiment are 20 samples for heat treatment with varies temperature and 18 total samples for heat treatment with varies time. Total overall samples are 56 samples.

5. Testing the sample

Sample will be tested with different temperature and different duration time for heat treatment for each experiment.

6. Analysis and result

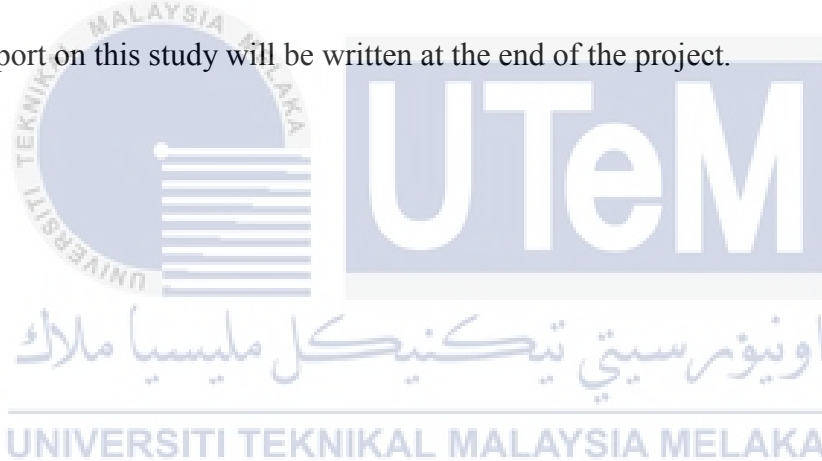
Analysis will be presented on how the temperatures affect the polymer surface on hydrophobicity properties on aluminum substrate according to their contact angle, surface roughness, surface morphology and the correlation of these microstructural properties with hydrophobicity of the polymer surface.

7. Suggestion and recommendation

Suggestion will be proposed based on the analysis.

8. Report writing

A report on this study will be written at the end of the project.



The methodology of this study is summarized in the flow chart as shown in Figure 1.2 below.

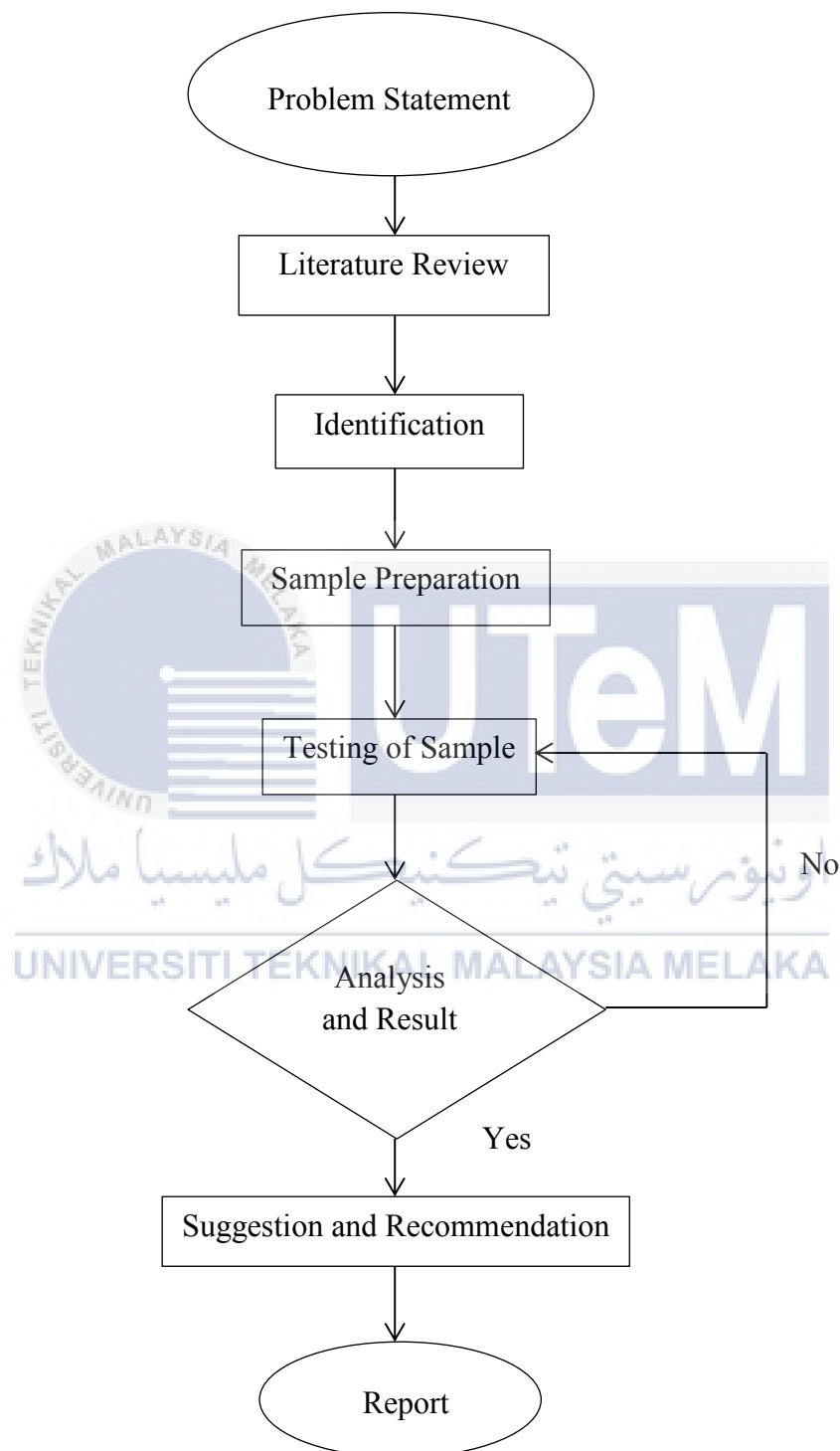


Figure 1.2: Flow Chart of the methodology

CHAPTER 2

LITERATURE REVIEW

2.1 Overview

This chapter presents the review cases study of effect of temperature of hydrophobic coating on aluminum substrate. It includes the hydrophobicity properties which is superhydrophobic and hydrophobic. This review also provides the properties that are used to analyze effect of temperature, which are contact angle, surface roughness, surface morphology and correlation of these microstructural properties with hydrophobicity of the polymer surface. The review is collected from the recent and past journals and references books that have been studied to understand the related topic area of this project. In addition, the review also regarding the function of structural characterization utilizing for this project which is digital microscope and Alicona Infinite Focus measurement.

2.2 Hydrophobicity Properties

Hydrophobicity is the physical property of a surface that avoids water. Generally, carbon and hydrogen atoms are the only molecules in the hydrophobic surface. This surface cannot interact favorably with water and repel water that make a contact with the surface (Ravi, 2016).

2.2.1 Definition of Hydrophobicity

Superhydrophobic coating is a coating that exhibit extremely high water repellency. The degree to which a solid repels a liquid depends on two factors; surface energy and surface morphology. Once the surface energy is lowered, hydrophobicity is enhanced. In superhydrophobic coating, the surface morphology plays an important role in wettability. The main parameter to characterize wetting is the static contact angle, which is the angle that a liquid makes with a solid. There are several factors that can influence the contact angle such as surface energy, surface roughness, and its cleanliness (Bharat and Yong, 2010). Roughening a coating not only enhances its hydrophobicity due to the increase in the solid-liquid interface but also when air is trapped on a rough surface between the surface and the liquid droplet (Adel et al, (2014).

The surface with contact angle between 150° to 180° is called superhydrophobic. Figure 2.1 shows the different contact angle for superhydrophilic, hydrophilic, hydrophobic and superhydrophobic. It exhibit very low water contact angle, smaller than 10° . This causes the water droplet rolling and bouncing from the surfaces. It also will remove contaminants on the surface due to self-cleaning properties of superhydrophobic coating. Water drop are able to capture the dust and dirt particle, while moving on the surface, and can easily remove particle contaminants from the solid surface (Bernagozzi et al, 2013).

Many method have been develops to formulate a superhydrophobic coating such as electrospinning technique, layer-by-layer, electrochemical deposition, spray coating, and sol-gel method. These methods is in conduct by follows two strategies; constructing

hierarchical nano or microscale binary structure or modifying a rough surface with low surface free energy materials (Yongcai et al, 2014). Superhydrophobic are used in many application such as self-cleaning windows, textiles, medical devices and electric devices. The term hydrophobic is from word “hydro-” means water and “-phobia” means fearing or hating in Greek. It refers to water fearing condition. It used to define the contact of solid surface with any liquid (Bharat and Yong, 2011). The value of contact angle for hydrophobic coating is between 90° to 150° as shown in Figure 2.1. Typical applications of hydrophobic coating are condenser, filtration part and food processing parts.

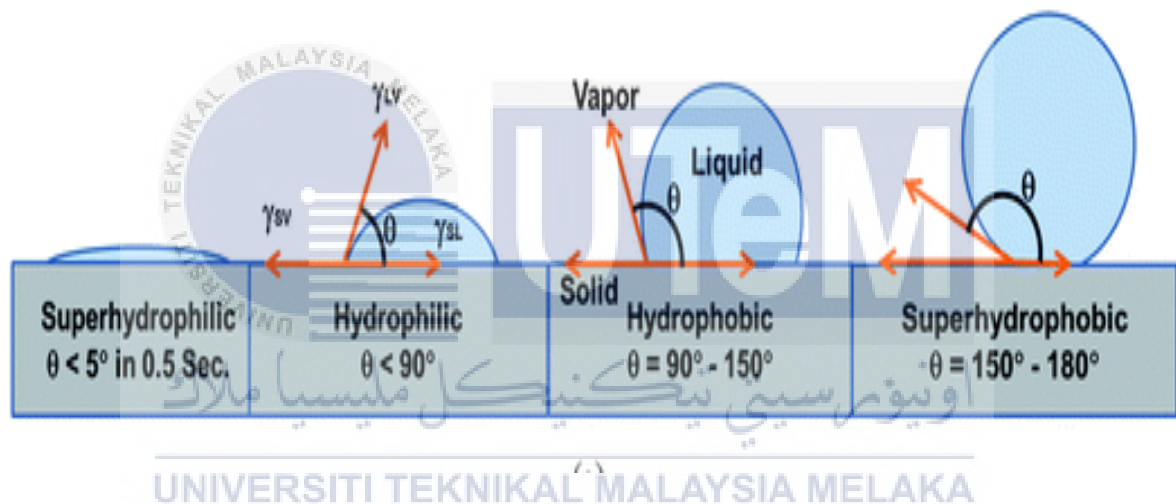


Figure 2.1: Contact angle to determine hydrophobicity (Nurxat et al, 2012)

2.2.2 Hydrophobicity phenomena in nature

The most popular plant that show great water repellency is *Nelumbo nucifera* (Lotus plant), shown in Figure 2.2 below. Other plant is such as *Pistia stratiotes* (Water cabbage), and *Colocasia esculenta* (Wild taro) in Figure 2.2. According to Adel et al (2014), Lotus leaves have shown better stability and perfection as water repellency, eventhough there are many plants exhibits superhydrophobic surface.



Figure 2.2: (a) Lotus leaves (Adel et al, 2014). (b) Water Cabbage (Peru and Sanchez, 2013). (c) Wild Taro (Graves, 2014)

There are some animal evolved with superhydrophobic property, such as water strider. They can walk on water due to surface tension that would suck them in when water were at all attracted to the legs of these animals as shown in Figure 2.3(a) . The leg of water strider are responsible for their water resistance due to the layer of minute hairs that coated on the leg. The resulting large contact angle that will allows surface tension to support the animal walk on water surface (Mark, 2016). Thierry and Frederic (2015) states that many insects are superhydrophobic. Such as, Geikos have feet with high solid-solid adhesion, which make their able to climb on vertical surfaces. It sue to the well-aligned microscopic hair, called setae, on their feet as shown in Figure 2.3(b).

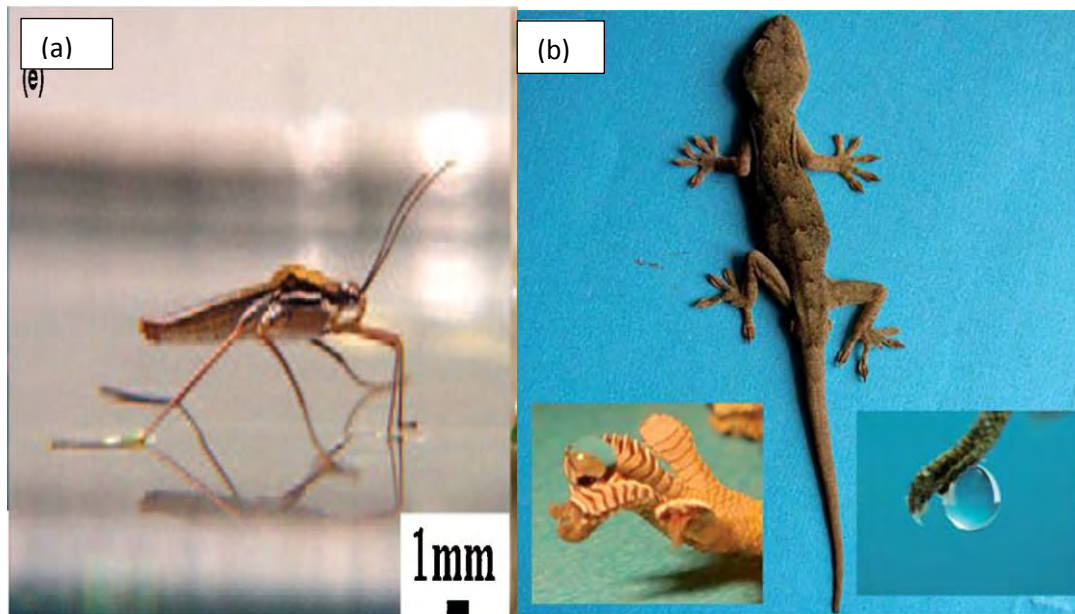


Figure 2.3: (a) Water Strider, (b) Geiko (Thierry and Frederic, 2015)

2.3 Structural Characterization

Aluminum 6061 is chosen as a substrate to test the effect of temperature on hydrophobic coating. This type of aluminum has excellent joining characteristics and good acceptance of applied coatings. It was one of the most versatile from the entire aluminum alloy, which is it have high strength, good workability and high resistance to corrosion and widely available. Aluminum can withstand very high temperature. But at very high temperature, aluminum will experience loss of yield strength. Therefore, this project must be done to analyze the effect of temperature on the hydrophobic coating that applied on aluminum substrate. The analysis is done by investigate the effect of temperature for polymer surface on hydrophobicity properties that applied on aluminum as a substrate by measuring the contact angle, surface roughness, surface morphology and correlation of these microstructural properties with hydrophobicity of the material.

2.3.1 Contact Angle

Contact angle is the most important criteria to analyze the wettability of the surface. Small contact angle, which is smaller than 90° it show high wettability, while large contact angle which is larger than 90° correspond to low wettability. Contact angle can be described as the angle formed by the meeting of liquid with solid surface. The shape of the liquid drop to be measure as contact angle is determined by surface tension of the liquid as shown in Figure 2.4 below. In liquid state, the net force is zero, because each molecule in the bulk is pulled equally in every direction by neighboring liquid molecules. The molecule that exposed at the surface does not have neighboring molecules in all directions to provide a balanced net force. They only pulled inward the neighboring molecules and creating an internal pressure. Therefore, the liquid will contracts its surface area to maintain the lowest surface free energy (Yuehua and Lee, 2013).



Figure 2.4: Surface tension in water droplet (Yuehua and Lee, 2013)

Over 150 years, Thomas Young were proposed treating the contact angle of a liquid with a surface as the mechanical equilibrium of a drop resting on a plane solid surface under the restrains at three condition of surface tension. It forms “Young equation”, where

the angle formed by the solid surface and the tangent of the drop is called as a “contact angle”. Figure 2.5 show the contact action for Young equation.

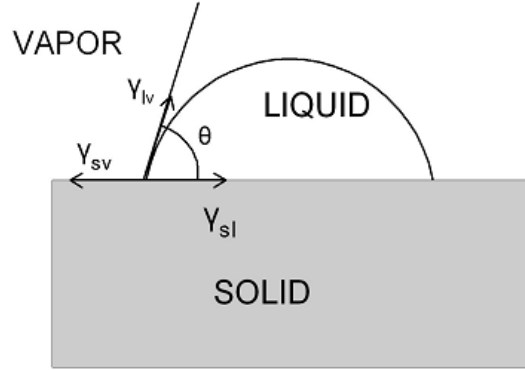


Figure 2.5: Contact angle according to Young Equation (Mittal and Robert, 2014)

Equation 2.1 is relates to the contact angle to the surface free energy of a system containing solid(S), liquid (L), and vapor (V) phases, it referred as Young's equation. Where, γ_{lv} , γ_{sv} and γ_{sl} is represent the liquid-vapor, solid-vapor, and solid-liquid in terfacial tension, and θ_Y is the contact angle (Yuehua and Lee, 2013).

$$\gamma_{lv} \cos \theta_Y = \gamma_{sv} - \gamma_{sl} \quad (2.1)$$

According to Bharat and Yong (2011), Wenzel was formulating a simple model predicting that the contact angle of a liquid with a rough surface is different with smooth surface. Cassie and Baxter present that water vapor which is normally called as “air” in gaseous phase in the literature, may be trapped in the cavities of a rough surface. Therefore, it form solid-liquid-air interface, it opposed to the homogeneous solid-liquid interface. These two theories, explain two condition wetting regimes or states: the homogeneous (Wenzel) and the composite (Cassie- Baxter) regimes. Figure 2.6 show the different of image of water droplet in Wenzel state and Cassie-Baxter state.

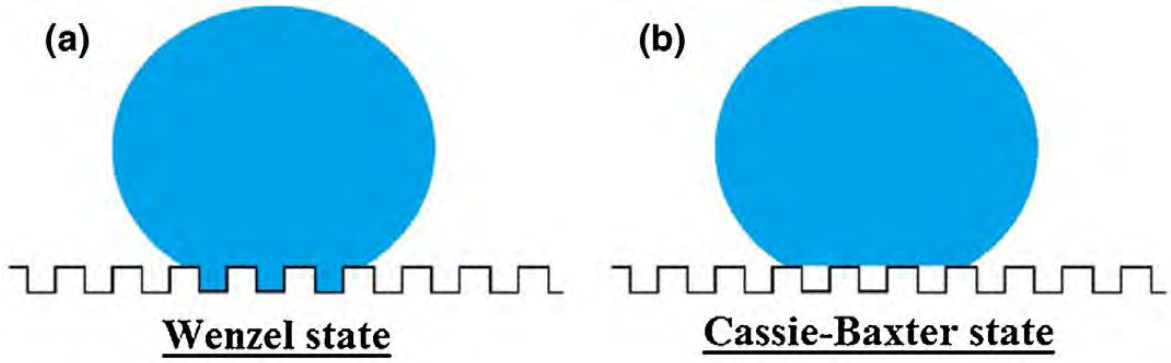


Figure 2.6: Water droplet in (a) Wenzel states and (b) in the Cassie-Baxter states (Thierry and Frederic, 2015)

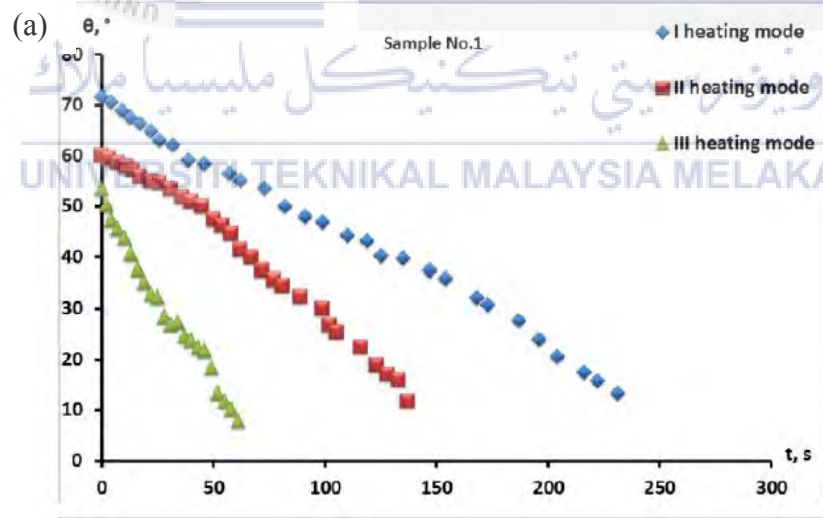
This Wenzel and Cassie-Baxter equation are explained about contact angle of liquid with rough surface. The Wenzel equation, Eq. (2.2) states that the water droplet is in full contact with the surface and θ^Y is amplified by a roughness parameter. Hydrophobic can be obtained when θ^Y is larger than 90° , with high Hysteresis, H and sliding angle, α due to the increase in the solid-liquid interface. It shows that the value of contact angle of a rough surface is dependent on the value of the contact angle of smooth surface.

$$\cos\theta = r \cos \theta^Y \quad (2.2)$$

The Cassie-Baxter equation, Eq. (2.3), indicate the water droplet is suspended on a composite interface made of solid and air trapped between the droplet and the surface. Hydrophobic can be assumed when H is low and α is low due to the increase in the solid-vapor interface. It describe the superhydrophobic surface with contact angle is not dependent to the roughness. Where ϕ is the fraction in contact liquid/solid. Wetting in the Cassie- Baxter state is more suitable to be considered for achieving superhydrophobic rather than Wenzel state (Quanshui and Cunjing, 2014).

$$\cos \theta = \phi_s (\cos \theta^Y + 1) - 1 \quad (2.3)$$

Dmitriy et al (2015) demonstrated the optical method for observing the water drop evaporating on the solid surface. The experiment is to determine the evaporation stages, analysis the effects of surface roughness and temperature reaction on characteristics of drop spreading; the contact angle and their diameter. The substrate used is stainless steel. They used three different surface roughnesses which is, smooth, medium and rough with three different temperature modes; mode I (54°C), mode II (62°C), and mode III (70°C). The result indicate that the contact angle were decreases when the temperature at mode III, where at the highest temperature. .Figure 2.7 show the time dependence of contact angle during drop evaporation for three heating mode in different surface roughness (a) sample I with smooth surface roughness, sample 2 with medium surface roughness, and sample 3 with rough surface roughness.



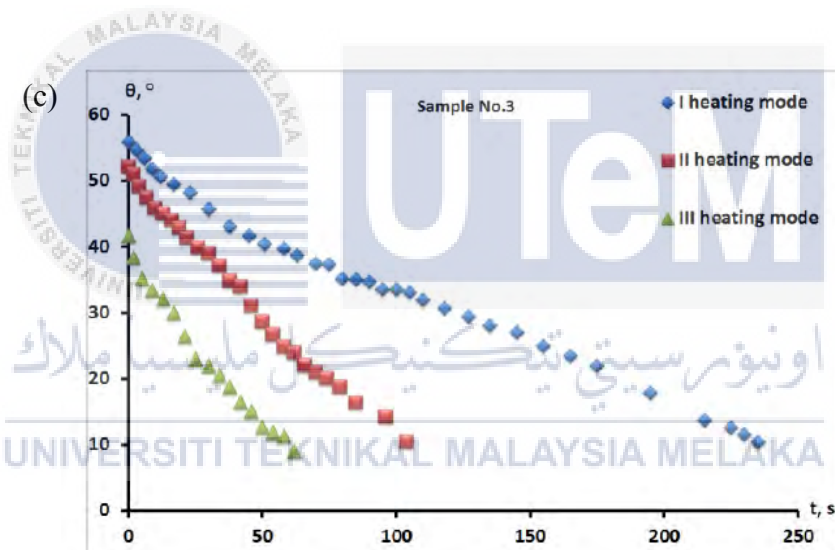
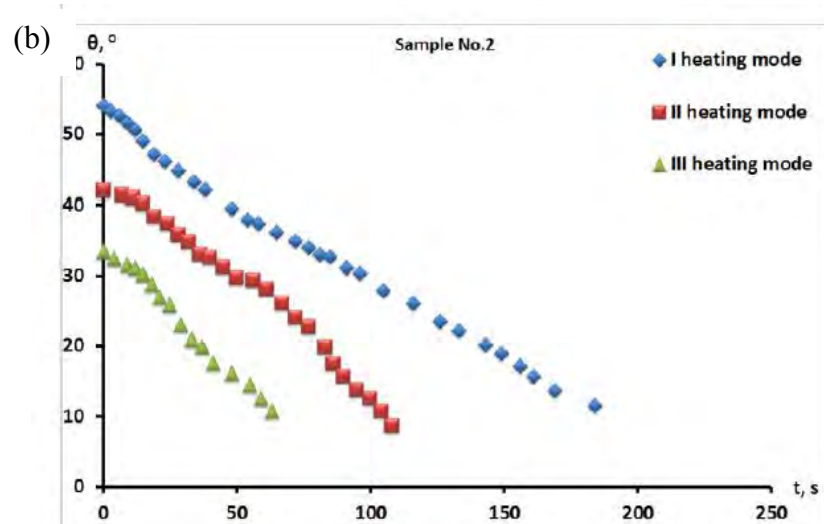


Figure 2.7: Time dependence of contact angle during drop evaporation for three heating mode in different surface roughness (a) sample I with smooth surface roughness, (b) sample 2 with medium surface roughness, and (c) sample 3 with rough surface roughness. (Dimitriy, 2015)

The outline of the graph is due to the contact line begun to reduce, where the radius is decreasing, the drop is “flattening” on the surface. From the experiment, they founds that the surface tension force, which is directed towards the drop center, is reduced in the highest temperature mode compared in mode I, and mode II due to drop temperature is increase. The drop area tends to decrease to the minimum. Thus, this research shows that the higher temperature the more intensively and faster the value of contact angle to decrease and the contact angle is higher on a rougher surface compare to the smooth surface substrates.

Based on analysis that has been done by Wael et al (2015), simple spraying method can be used on fabrication of superhydrophobic metal substrates. The process will lower the surface free energy and increasing surface roughness. Spraying technique is used in this experiment, because its simplicity, low cost and low temperature method. The substrates used in the experiment are copper, aluminum and iron. The analysis including the investigation on the wettability of the surface from hydrophilicity to superhydrophobicity by measured the contact angle of the water droplet on the surface.

Contact angle can be measured using basic digital camera such as Sony, inverted microscope, and telescope-goniometer. Most commonly technique used to measure the contact angle is a direct measurement of the tangent angle at the three-phase contact angle on a sessile drop profile (Yuehua and Lee, 2013).

Sessile drop method is most common used technique to measure contact angle. According to this method, a drop of liquid is place over the surface of the material; it formed three interface; liquid-vapor (drop-atmosphere), solid-liquid (solid material-drop)

and solid-vapor (solid material-atmosphere) interface. The intersection of these three interfaces was form a line called three phase line or contact line (Jonathan, 2013). The tangent of the liquid-vapor interface and the solid-liquid interface in the contact line will formed an angle, which is called as contact angle.

Lomga et al (2017) synthesized a superhydrophobic coating on aluminum sheets using two-step process. First step is by creating surface roughness by chemical etching on the substrate and then decrease the surface energy of roughed aluminum surface. The surface is annealing at elevated temperature from 80°C to 270°C for one hour duration time and 300°C for 24 hours.. Then the contact angle of the sample was measured after the samples were cooled. The result show that when temperature for annealed in range of 80°C to 140°C , the static and sliding contact angle of the samples remains constant. Whereas, when the annealing temperature is more than 170°C the water contact angle start decreasing. During 250°C, the water droplet spreads quickly on the surface. In harsh condition, when the annealed temperature is 300°C, the superhydrophobic aluminum surface lost its superhydrophobic nature. It due to the surface was undergoes complete evaporation of coatings and superhydrophobicity of the surface was turns into superhydrophilicity with static contact angle is 8°.

2.3.2 Surface Morphology

Surface morphology is a high spatial resolution imaging in an advanced form that used sophisticated microscopes to produce images of products, samples and objects that cannot to be seen with the naked eyes, it called as subset of Analytical Imaging. The surface imagining will produced information about surface structure and defects with

atomic or Nano-scale structure. To observe the surface morphology, microscopic techniques capable in atomic-level observation are necessary such as in Infinite Focus Microscope, Scanning Electron Microscopy (SEM), High Resolution Optical Microscopy, Transmission Electron Microscopy (TEM), Atomic Force Microscope (AFM) and Ultra High Resolution-Scanning Electron Microscopy (UHR-SEM). This machine will provide high spatial resolution images that show the relative surface morphology of physical features, defects and particles. Whereas, light microscope is the most common type of microscope where uses a combination of light and lenses to magnify an image. But, this type of microscope does not offer the highest magnification and it has limited structure that can viewed using this microscope.

Surface morphology can be used as one of the element to analyze the effect of temperature on coating by providing the analytical imaging of the surface. Bedia et al (2015), presents the morphological and optical properties of Zinc Oxide (ZnO) thin film that prepared by spray pyrolysis on glass substrates at various temperature. ZnO is commonly used as coating on the surface of material to reduce light reflection and increase light transmission. The surface morphology of the films was evaluated using Scanning Electron Microscopy (SEM). The result shows that the surface morphology of the thin film coating is uniform and evolved with the substrates temperature during the growth. The grain size of the surface is increased and become rougher with the increases of temperature. Besides that, the optical band gap of the surface is increases when the substrates temperature increases. Therefore, it shows that ZnO thin film could be used as antireflection layer in solar cells, and the properties can be enhanced by applying high growth of temperature.

According to Minglin and Randall (2006), found that there are two categories of technique to make superhydrophobic coating; prepare a rough surface from a low surface energy material and modifying a rough surface with a material of low surface energy. It correlated to the relationship between the surface morphology and wetting of the surface. Javed et al (2016), explain that the particle size of the thin film coating increase with the increase of the temperature. It shows effect of temperature on growth mechanism in thin film coating. They used nanostructured Zinc Oxide (ZnO) thin film with high transparency on glass substrates and placed at different temperature to analyze the effect of temperature on the thin film coating.

Shivaraj et al (2015) demonstrate the influence of annealing temperature on crystalline size, surface roughness, surface morphology and photoluminescence properties of Zinc Oxide (ZnO) thin films were prepared by dip and spin coating technique. Thin films were annealed at 400°C and 500°C for one hour each and their structural properties were evaluated using XRD, AFM and SEM. ZnO is prepared on the glass substrate. The result shows when the annealing temperature is increased, the grain size of the surface also increased with the surface relatively rough. This is because, when the temperature is high, the coating atoms had enough diffusion activation energy to occupy the site in the crystal lattice and the lower surface energy grains were becomes larger.

The surface roughness will influence the hydrophobicity of the surface. Tokoro (2016), presented about effect of temperature on the surface roughness that will affect the hydrophobic properties of silicone rubber. He used five silicon rubber samples with different temperature which undergoes heat treatment or without heat treatment. The measurement of the contact angle is used to analyze the hydrophobic condition of the

sample. The result explained that when the temperature is increased, the contact angle also will increase. Then, the surface roughness of the samples is measured. The surface roughness of the sample surface will increase with the increase in value of the contact angle.

2.4 Structural Characterization Utilizing

Structural characterization is a technique used in the experiment to evaluate the process of the experiment. Factors that can be measured for the characterization are contact angle, surface roughness and surface morphology to analyze the correlation of these microstructure with hydrophobicity of the material. The method is by using digital microscope and Alicona Infinite Focus measurement. This specification of technique is important to ensure the technique used is suitable with the process of measurement for the data analysis.

2.4.1 Digital Microscope

Digital microscope is a regular microscope with built in digital camera. The images produced from a digital microscope can be projected to a computer monitor and saved on a computer file (Peter, 2005). Generally, digital microscope is connecting to the computer via a USB port. The images seen through the microscope's eyepiece can be shown on the computer's monitor when the digital microscope is connecting to the computer using USB port. All the images can be saved for the future use. This is the main advantages of digital microscope, which is ergonomics of the instrument. This is because by the image that can be displayed on a monitor, user can view them immediately and analyze the sample image

using software while sitting comfortably (James et al, 2015). Besides that, digital microscope is relatively inexpensive compare with other types of microscopes. But, it provides the ability to view small parts on large screen thus reducing eye fatigue.



Figure 2.8: MSP-3080 USB 3.0 Digital Microscope 8MP X 200 Optical Zoom Full HD 1080p from Panrico

The model of the digital microscope is MSP-3080 USB 3.0 Digital Microscope 8MP X 200 Optical Zoom Full HD 1080p from Panrico as shown in Figure 2.8 above. It provides a superior optical performance with high resolution 8.0 mega pixels sensor. This USB 3.0 will provide very clear color reproduction and precise detail for variety of imaging applications such as plants, insects, circuit board and others. It can capture images from wide range of magnifications, from 5x to 200x magnification using the adjustable stand. This microscope integrated LED illumination with eight white LED light into the system to produce consistent brightness and relief contrast at all levels of magnification. The imaging software offers various application such as live image preview, capture, recording, measurement, annotations and post image enhancement, image zoom in/out, character key in, color and line modified all included. The combination camera and this software were creating a complete solution of the microscope images (Panrico, 2017). The

detail image of digital microscope is shown in Figure 2.9 below. The samples were placed in front of the microscope to capture the image of the water droplet on the coating surface of the sample.

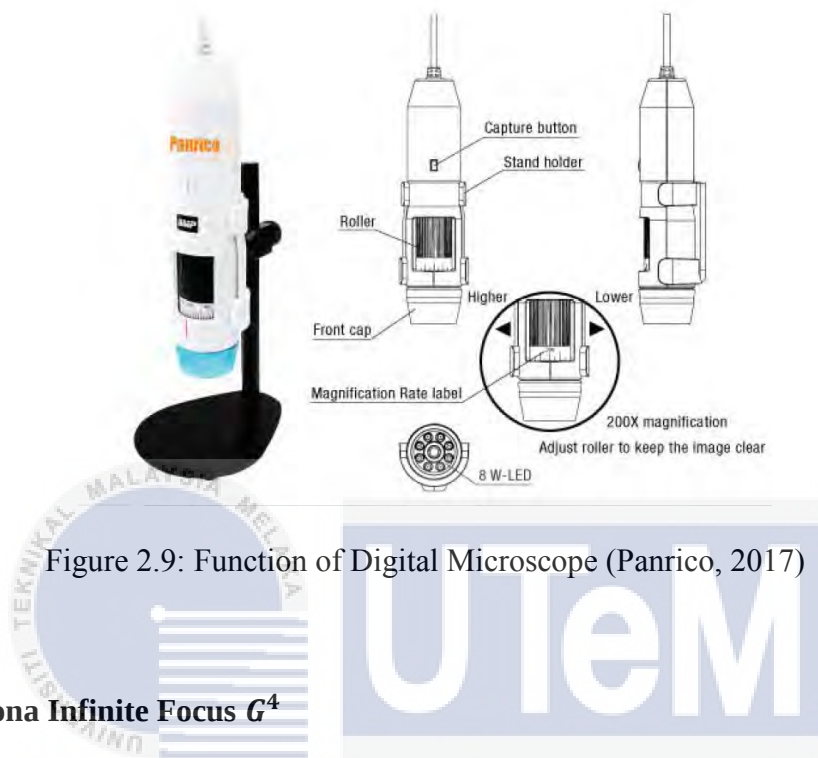


Figure 2.9: Function of Digital Microscope (Panrico, 2017)

2.4.2 Alicona Infinite Focus G^4

Infinite Focus is a high resolution optical 3D surface measurement device which provides the functionalities of an optical profiler and a micro coordinate measurement device in one system (Alicona, n.d.). Full range of surface characterization of measurement of form, roughness, surface texture, volume, edge measurement and 2D image analysis can be obtained from this microscope. Therefore, it will present true colour of sample at high macro, micro and nano level, it can recognize corrosion and blemishes on surface sample. The analysis on surface roughness can be obtained for 2 dimensional and 3 dimensional. Infinite Focus Microscope can be used for all surface and shape, it not only for round shape sample. It can be used for highly structured and very rough surface with steep flanks to shiny, smooth and polished components without changing the handling system (Stefan, 2016). Figure 2.10 below show the image of Alicona Infinite Focus G^4 .

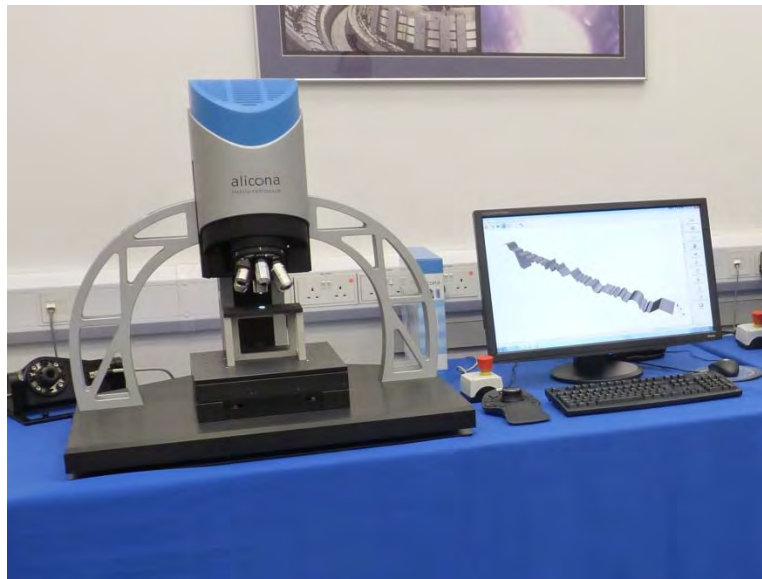


Figure 2.10: Alicona Infine Focus G^4 (David, 2015)



CHAPTER 3

METHODOLOGY

3.1 Overview

This chapter explain the methodology used in this project to achieve the objectives in Chapter 1. The project starts by the material descriptions for the material used in the experiments is listed with the specific explanation were explained in this chapter. It follows by the sample preparation, including the step to determine the dimension and process of the fabrication of the sample with the process of spraying the coating on the samples surface. The process of the experimental set up will also present in this chapter to explain the procedure for these experiment. After that, testing was conduct on the sample to investigate the contact angle, surface morphology and surface roughness.

3.2 Material Description

The material used as a sample in this project is aluminum substrate. The selections of the samples are made based on the original material from the problem statement in Chapter 1, where, aluminum 6061 as a material for car frames material. An aluminum sample is as shown in Figure 3.1.



Figure 3.1: Aluminum substrate

The types of coating used in this project are aerosol, DPI Anchor Spray Paint with light blue color and lacquer, HB Body Auto Clear HS 49 with clear color as shown in Figure 3.2. Lacquer spray is used to create a synthetic coating, to form a high gloss surface of the sample. The lacquer is contains mix of butan-1-ol, acetone, toluene and n-butyl acetate.



Figure 3.2: (a) Aerosol and (b) Lacquer

3.3 Sample Preparation

The aluminum 6061 plate is measured into same dimension which is 10 x 10 x 2mm ($l \times w \times t$) with 20 total sample for heat treatment with varies temperature experiment and 18 total sample for heat treatment varies with times experiments. The dimension for each substrate is as shown in Figure 3.3 below. Shear machine is used for cutting process of aluminum. The substrates, is cleaned using acetone and dried in air.

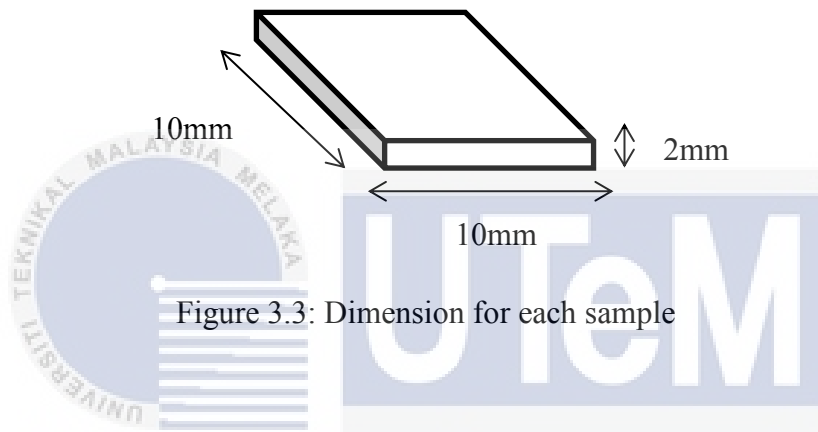


Figure 3.3: Dimension for each sample

For coating process, all the sample were sprayed with one layer of aerosol and let it dry for 10 minutes. Ensure all the spraying technique such as distance of spray, degree of spray and thickness of spray is same for all samples. Spray with one layer of lacquer for extra coating and let it dry for an hour. The samples were placed into a close container with a silica gel to avoid oxidation takes place on the coating of the sample.

All the samples were polish using sand paper with 2000 grit, that attach on grinding and polishing machine with speed 50 rpm to roughen the surface. Clean the surface of the sample and the sample were labelled from 1 to 20 and 1 to 6 for each sample for different experiment. The Figure 3.4 below show the general flow for overall procedure for this process.

Samples were cut in specific dimension and cleaned using acetone.



Samples were sprayed with one layer of aerosol. Let it dry for 10 minutes.



Samples were sprayed with one layer of lacquer. Let it dry in an hour.



Samples were polished using grinding and polishing machine with 2000 grit of sand paper



Samples were ready to start the experiment

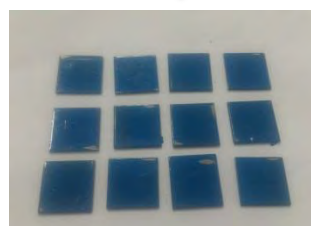


Figure 3.4: Flowchart for general procedure for sample preparation

3.4 Experimental Set Up

The procedures of the experiments are divided into two different experiments. Firstly is heat treatment with varies temperature experiment and secondly is heat treatment with varies time experiment.

3.4.1 Heat Treatment with Varies Temperature

All 20 sample is divided into four different temperature with interval 50°C ; 5 samples for 25°C, 5 sample for 75°C, 5 sample for 125°C and 5 sample for 175°C. Firstly, measure the contact angle of the sample for temperature 25°C. Repeat three times for each sample to get average value of contact angle for temperature 25°C. After that, 5 new samples are placed into the oven at temperature 75°C in 5 minutes. Take out the samples and measure the contact angle for three times and get the average value. The step is repeated by changing the temperature of the oven with 125°C and 175°C. The highest average value of contact angle for each temperature was sent to Alicona Infinite Focus Microscope to measure the surface roughness and surface morphology.

3.4.2 Heat treatment with Varies Time

All 18 samples are divided into three different time exposures to the heat treatment; 6 samples for 10 minutes, 6 samples for 20 minutes and 6 samples for 30 minutes. During the whole experiment periods, the oven is set up at 75°C. Firstly, 6 samples are placed into oven and leaved for the heat treatment with 10 minutes duration of times. After that, the samples were taken out from the oven and the contact angle were measured using the

digital microscope with repeated three times for every sample to get the average value of contact angle. Then, another 6 new sample is placed into the oven and leaves for 20 minutes for the heat treatment. After that, the contact angle is measured with repeated three times. Lastly, the others 6 new samples are leaved in the oven and undergo heat treatment for 30 minutes. The highest average value of contact angle is sent to Alicona Infinite Focus Microscope to analyze the surface roughness and surface morphology. All the procedure of the experiment is repeated using different temperature of heat treatment which is 175°C.

3.5 Testing

Testing on the samples has been done to measure the contact angle, surface morphology and surface roughness of the coating surface.

3.6.1 Contact Angle

Water contact angle is measured using a direct measurement technique. It measured the tangent angle at the three-phase contact point on a water droplet profile. Micrometer pipette is used to form a water droplet with volume $5\mu\text{l}$. MSP-3080 digital microscope was used to capture the image of the water droplet, when it placed horizontally to the sample. The contact angle is measured using MSP-3020 Digital Microscope software as shown in Figure 3.5 below, where the digital microscope is connecting to the computer using USB port. From the software, the measurement is made from the image by aligning the tangent of the water droplet at the point with the surface and the reading will be automatically calculated. The set up used to measure contact angle as shown in Figure 3.5 below.

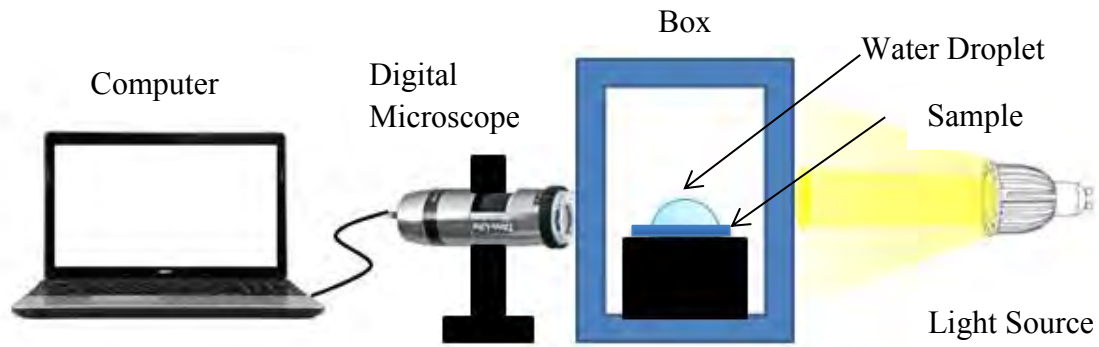


Figure 3.5: Setup to measure contact angle

3.6.2 Surface Morphology

Surface morphologies of the thin film were observed using Alicona Infinite Focus G^4 . It will produced 3D image of the surface sample and then show the change of the surface structure of the sample when the sample was undergoes heat treatment. This microscope also used to measure the surface roughness of the sample. Analysis of the surface roughness and surface morphology of the samples is followed ISO standard 4287, 4288 and 25178 guidelines. Alicona Imaging MeX software is used for analyzing the measurement data. The scale used for analysis the result of surface morphology of all experiments is 200x.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Overview

In this chapter, the experimental results are presented to show the data obtained from the experiment to be analyze in detail. The data present are consists the data for two different experiment. Firstly, experiment of heat treatment with varies temperature. Secondly, experiment heat treatment with varies time using two temperature; 75°C and 175°C. The discussions of the result were also present in this chapter to explain in detail the result obtained from these experiments.

4.2 Heat Treatment With Varies Temperature

The effect of the temperature on the coating can be analyze from the value of the contact angle of the water droplet on the surface sample after undergoes the heat treatment. Table 4.1 shows the results of the contact angle of the coating after undergoes heat treatment with different temperature in 5 minute. From the table, it indicated that the value of the contact angle in increases when the temperature is increases. The value of the standard deviation is calculated to analyze the uncertainty of the result, because the experiment is done on 5 samples for each temperature with repeated at least three times to find the average value.

Table 4.1: Contact angle for heat treatment with different temperature

Temperature (°C)	Contact Angle (°)	Standard Deviation	Uncertainty
25	70.586	3.325	0.744
75	74.664	2.696	0.603
125	87.448	2.219	0.496
175	91.356	1.960	0.438

Figure 4.1 shows the reading of contact angle against temperature. When the temperature is increases, the contact angle is increases. The contact angle at 25°C which is room temperature is 70.586° and at 175°C it increases to 91.356°. It corresponds to the image of the contact angle, where the contact angle of the 5 μ L water droplet with the surface is become larger when the surface is undergoes higher temperature in the heat treatment.

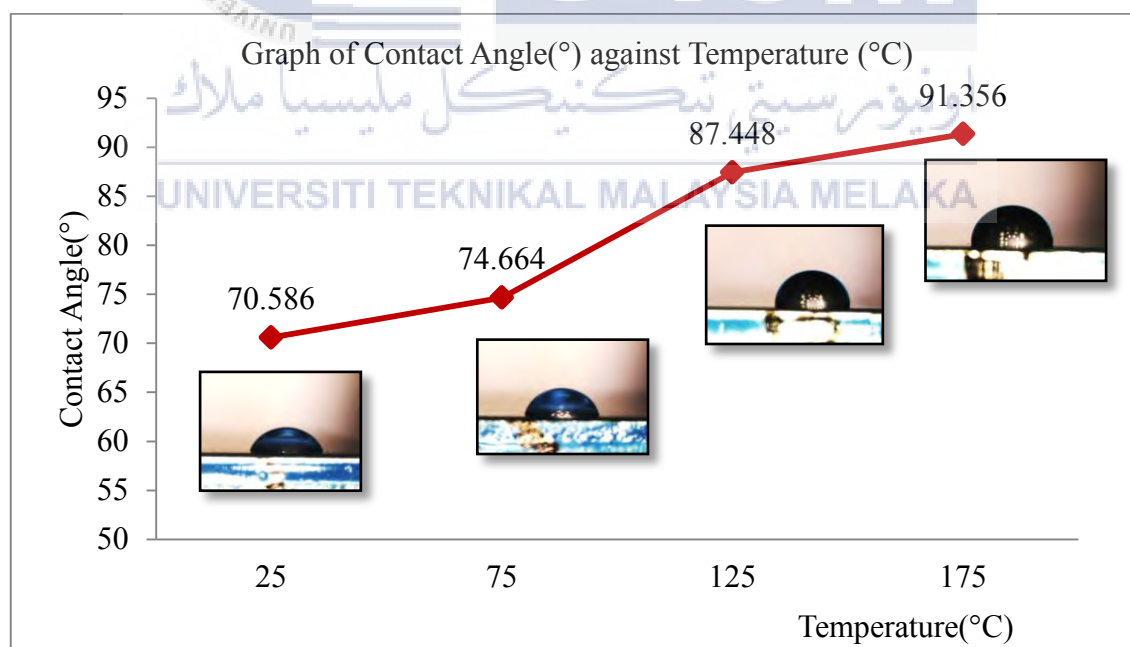


Figure 4.1: Graph of contact angle against temperature with image of the water droplet

The selected samples which have higher average contact angle were observed using Alicona Infinite Focus G^4 to analyze the surface morphology of the surface coating. Figure 4.2(a), (b), (c) and (d) shows the surface morphology image at 25°C, 75°C, 125°C and 175°C respectively with scale 200x. The surface morphology of the sample at temperature 25°C shows a smooth surface of the sample compared with the surface on sample of 175°C. With the temperature increases up to 175°C the surface become rougher and the roughness on the sample almost can be seen clearly from the surface.

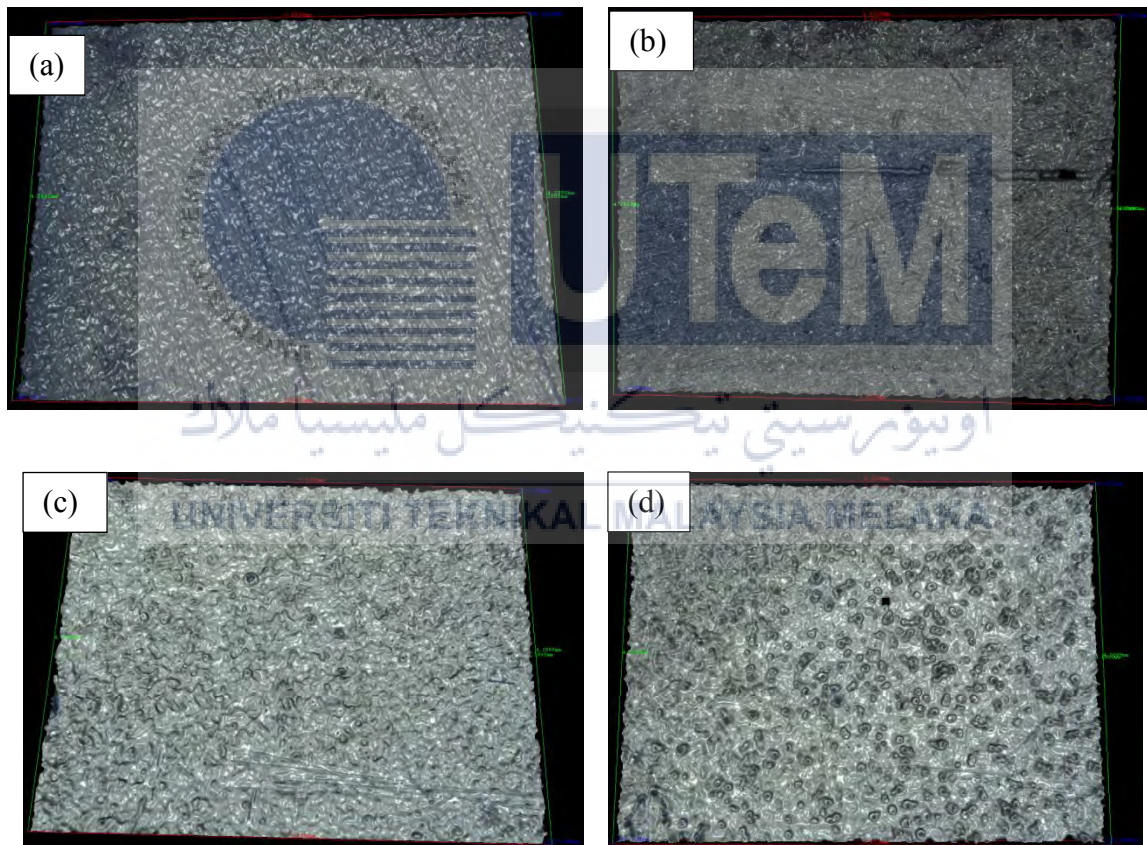


Figure 4.2: (a), (b), (c), and (d) Surface morphology of surface sample with different temperatures 25, 75, 125, and 175°C

Then, the surface roughness was measured using the same microscope; Alicona Infinite Focus G^4 . Figure 4.3(a), (b), (c) and (d) shows the reference location for analyze surface roughness at 25°C, 75°C, 125°C and 175°C.

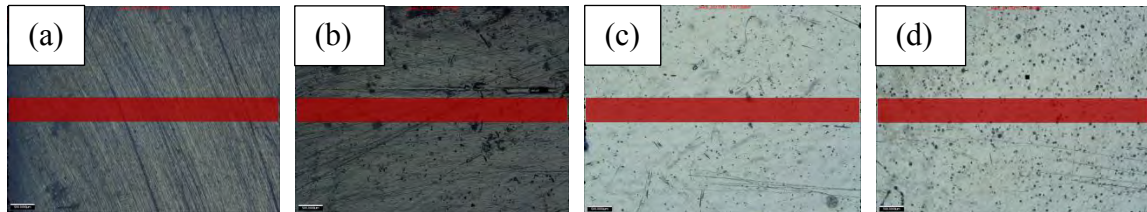


Figure 4.3(a), (b), (c) and (d) The reference location for analyze surface roughness at 25°C, 75°C, 125°C and 175°C

Table 4.2 shows the result of the surface roughness of the sample. While, Figure 4.4 is graph of surface roughness against temperature to explain the relationship between the surface roughness of the sample with the temperature of the heat treatment. The graph indicates that when the temperature increases the surface become rougher.

Table 4.2: surface roughness for heat treatment with different temperature

Temperature (°C)	Surface Roughness, R_A (μm)
25	1.8558
75	3.5194
125	4.5112
175	7.6655

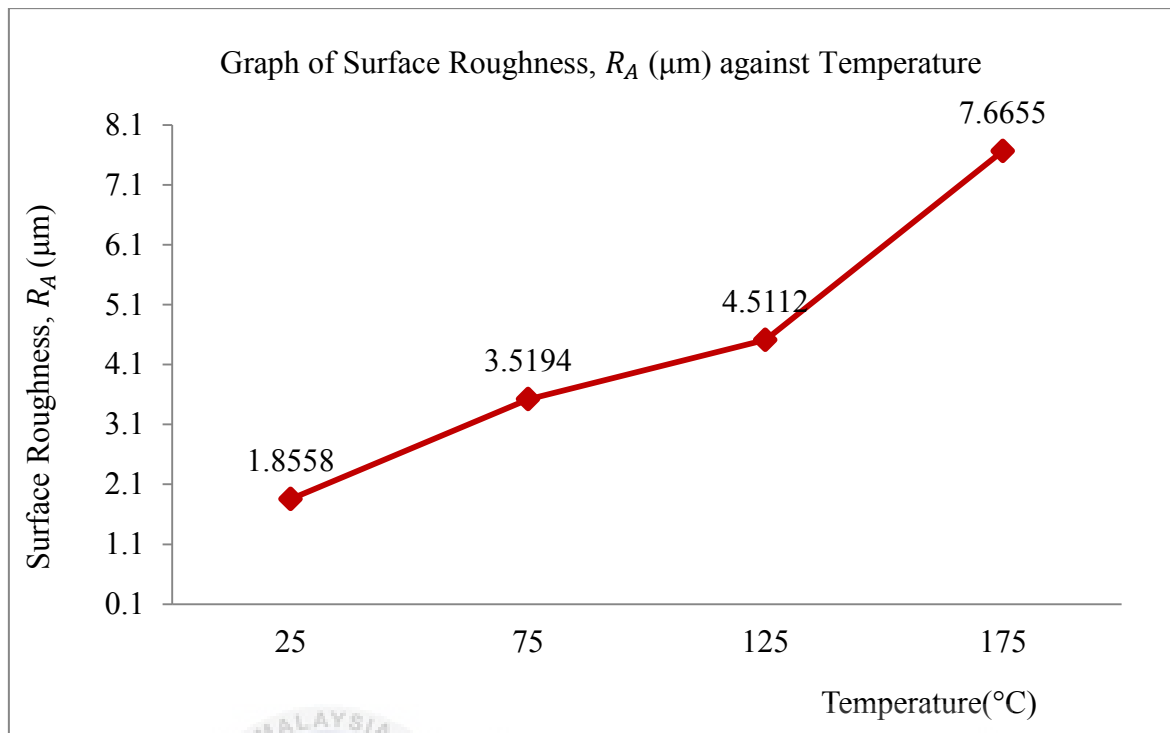


Figure 4.4: Graph of surface roughness against temperature

4.3 Heat Treatment With Varies Time

The result for the experiments heat treatment with varies time is divided into two different experiment, which are the temperature used for the heat treatment; 75°C and 175°C. The experiment is conducted with three different times of exposure of the sample with the heat treatment, which are, 10, 20, and 30 minutes.

4.3.1 Temperature of the Heat Treatment at 75°C

The sample is experience the heat treatment with the same temperature at 75°C but varies in time to analyze the effect of temperature on the coating surface when it undergoes

heat treatment with different times of exposure. Table 4.3 show the result of the contact angle when the samples experience the heat treatment in different duration of time.

Table 4.3: Contact angle for different time of heat treatment

Time (minute)	Contact Angle (°)	Standard Deviation	Uncertainty
10	69.637	1.663	0.679
20	67.285	1.307	0.534
30	66.752	2.255	0.921

While, Figure 4.5 show the graph of the result to show more clear view relationship between values of contact angle against time for heat treatment. The experiment is initiated at heat treatment for 10 minute, where the contact angle is 69.637°. The result indicates that when the longer the time for the heat treatment, the smaller the value of the contact angle. The result for contact angle when the time of heat treatment, 30 minute is 66.752°. The different value of the contact angle for five different duration of time heat treatment is very small, it around 3° only.

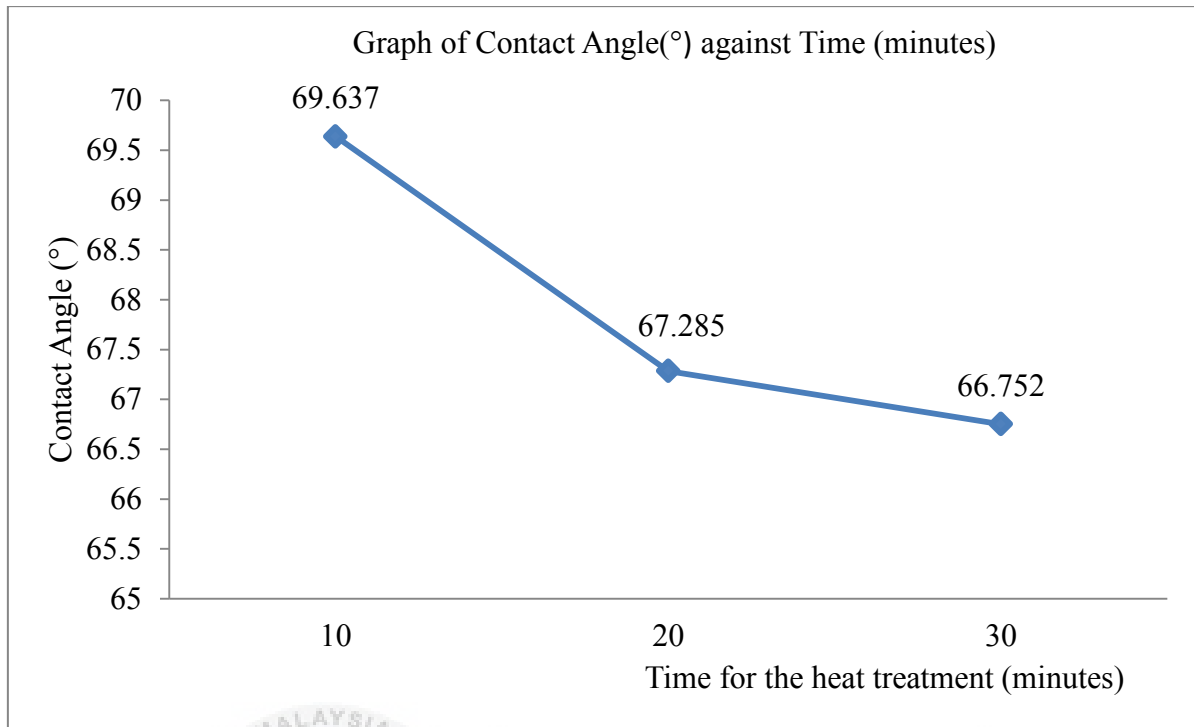


Figure 4.5: Graph of contact angle against duration of time for heat treatment 75°C

Then, the sample is observed using the Alicona Infinite Focus G^4 to analyze the surface morphology. Figure 4.6(a), (b), and (c) show the result of the surface morphology of the coating after undergoes different duration of time of the heat treatment with the image of the water droplet on the surface sample. The images of the surface morphology are almost the same for all samples, it correlate to the value of the contact angle, which is almost the same.

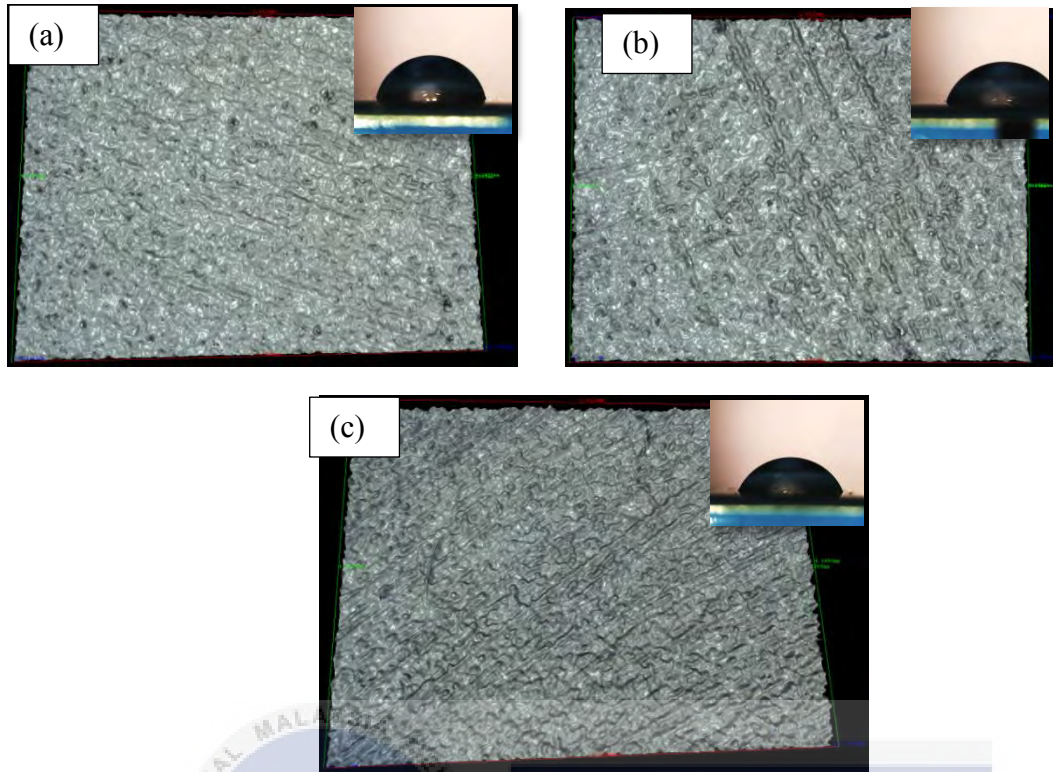


Figure 4.6: (a), (b), and (c) Surface morphology of coated aluminum with difference time exposure to heat treatment 10, 20 and 30 minutes.

Alicona Infite Focus G^4 also used to analyze the effect of temperature on the roughness of the sample coating. Location of the area to be analyze the surface roughness are selected from the same sample that have higher average contact angle, the location is shown in Figure 4.7 below.

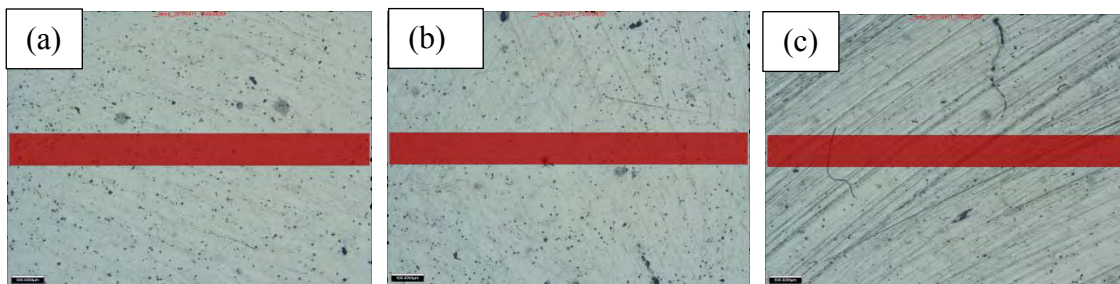


Figure 4.7: (a), (b) and (c). The reference location for analyze surface roughness for heat treatment for 10, 20, and 30 minutes

Table 4.4 show the result of the measured surface roughness of the sample. Then, Figure 4.8 is graph of the result of surface roughness. From the graph shows, it can be notice that when the duration of heat treatment is longer, the surface roughness will become larger. Value of surface roughness can be seen is almost the same for the duration of time of heat treatment 20 and 30 minutes.

Table 4.4: Surface roughness for different time of heat treatment

Time(minute)	Surface Roughness, R_A (μm)
10	3.8895
20	5.2258
30	5.7390

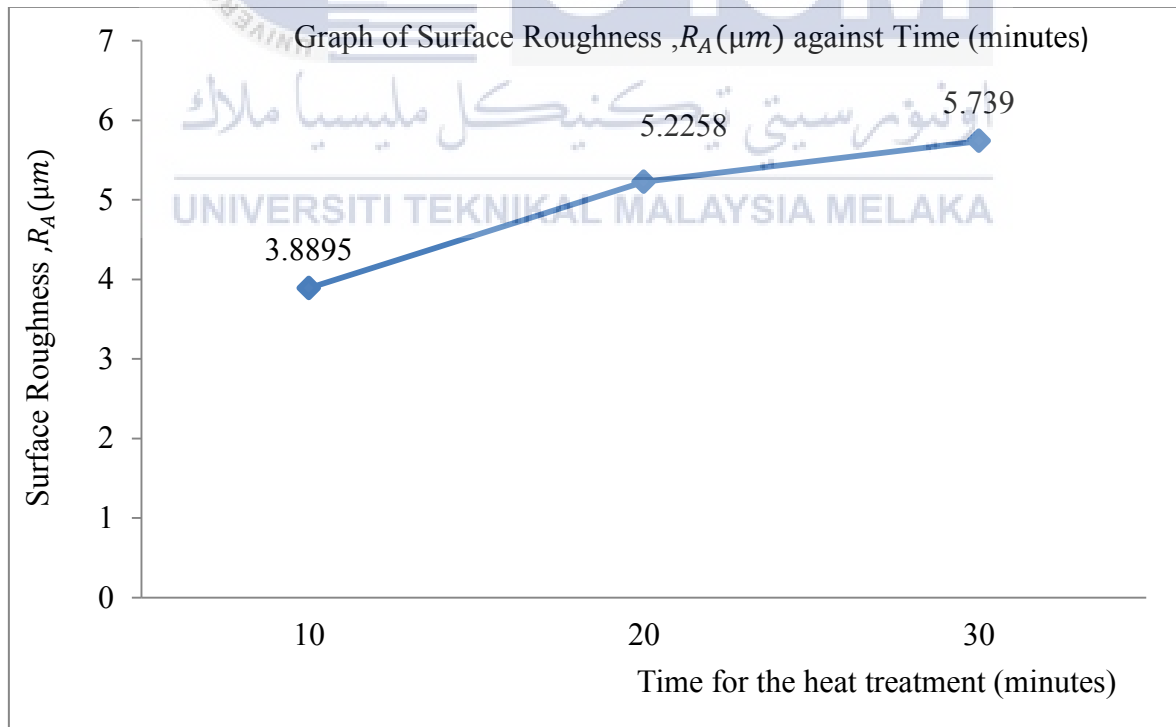


Figure 4.8: Graph of surface roughness against duration of time for heat treatment 75°C

4.3.2 Temperature of the Heat Treatment at 175°C

The sample undergoes heat treatment for same temperature, but with temperature 175°C with varies the duration of time of heat treatment. This experiment is to compare the result of the contact angle for constant temperature 75°C with 175°C whether the trend of the result is similar or getting the opposite result. Table 4.5 presents the result of contact angle of the sample with different time for heat treatment of 175°C.

Table 4.5: Contact angle for different time for heat treatment

Time (minute)	Contact Angle (°)	Standard Deviation	Uncertainty
10	91.185	1.266	0.517
20	87.760	2.656	1.084
30	81.139	0.808	0.330

Figure 4.9 show the graph of contact angle against duration of time for heat treatment 175°C. The graph show consistency of the contact angle decrease with the increase of the duration of time of the heat treatment. Where, when the time for the heat treatment is 10 minutes, the contact angle is 91.185°, it constantly decrease until 30 minute the contact angle is 81.139°

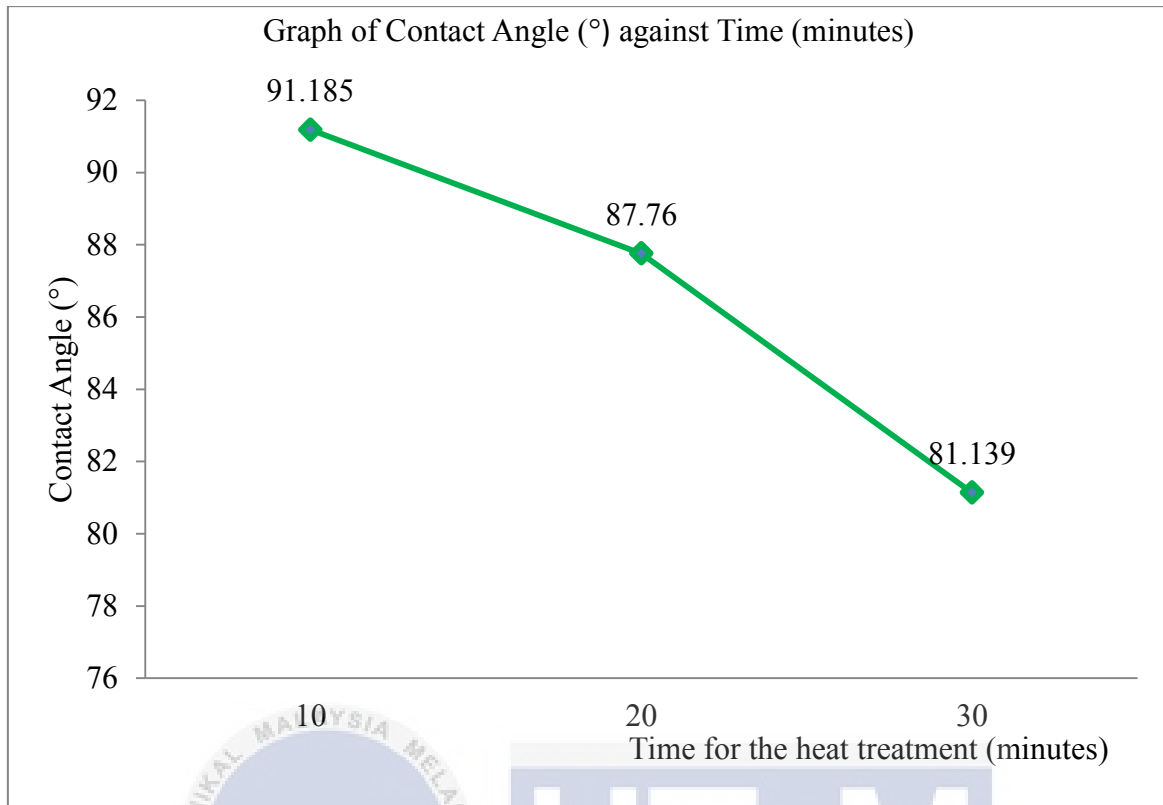


Figure 4.9: Graph of contact angle against duration of time for heat treatment 175°C

The sample is observed under Alicona Infinite Focus G^4 to analyze the surface morphology of the surface sample. All the image show the surface is become rougher due to long exposure of heat treatment at the high temperature as shown in Figure 4.10.

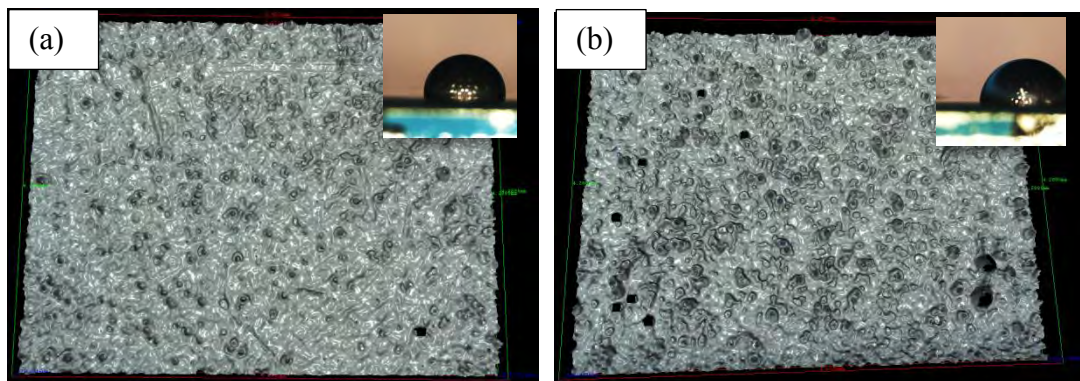




Figure 4.10: (a), (b), and (c) Surface morphology for difference time exposure to heat treatment 10, 20 and 30 minutes.

Then, the surface roughness of the sample is measured with the same microscope. The location of the area to be analyze the surface roughness are selected from the same sample that have higher average contact angle, the location is shown in Figure 4.11 below.



Figure 4.11: (a), (b) and (c) shows the reference location for analyze surface roughness for heat treatment for 10, 20, and 30 minutes

Table 4.6 shows the result of the surface roughness of the sample and Figure 4.12 show the graph of the result. The figure indicates that the surface roughness increased with the gradually increases of the duration of the heat treatment.

Table 4.6: Surface roughness for different time of heat treatment

Time(minute)	Surface Roughness, R_A (μm)
10	6.3568
20	9.5340
30	6.1015

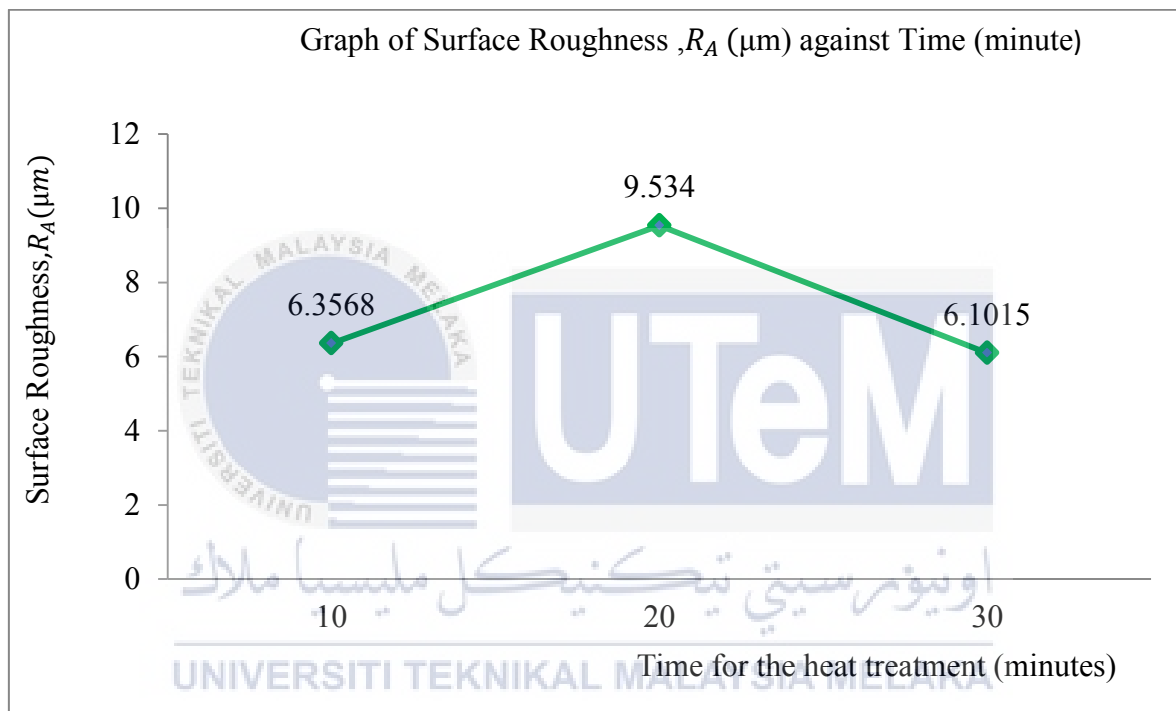


Figure 4.12: Graph of surface roughness against duration of time for heat treatment 175°C

4.4 Discussion

The water contact angle of the samples surface was examined. The results showed that the contact angle increased when the temperature of the heat treatment increased, but the value of contact angle will be decreased when the time of the sample exposed to the heat treatment is increased. Referring to Tokoro (2016), this is because the condition of the

sample is the same except the measurement temperature; as a result the hydrophobic condition has positive temperature dependence. Therefore, when the measurement temperature is increased, the dynamic contact angle increased. It due to the surface free energy has large negative temperature dependence and the effect of the hydrophobic condition of the solid surface is more effective compared to the one of a liquid water droplet.

When the sample is exposed to the heat treatment with a long period, it observed that the coating lose their hydrophobicity. Both experiment that using temperature 75°C and 175°C show that the value of contact angle is decreased with increasing the duration of heat treatment. During experiment with temperature 75°, the contact angle during 10 minutes time exposure is 69.637° and at time 30 minutes the contact angle is 66.752°, while for experiment using temperature 175°C, the contact angle at 10 minutes time exposure to heat treatment is 91.185° and at 30 minutes times is 81.139°. The value of contact angle is depends on the interfaces of the solid surface with the liquid surface.

Alicona Infinite Focus G^4 was used to examine the morphological properties of the sample surface. The surface morphology analysis shows the presence of rough microstructures on the treated surface. The contact angle of non-treatment surface which is at temperature 25°C is about 70.586° and the microstructural peak is smoother compare with the surface that undergoes heat treatment 175°C, which the contact angle is 91.356°, the microstructure peaks much rougher as shown in Figure 4.13 below. It shows a close up image of the surface of the sample. These microstructure peak form air trapped and helps in increasing contact angle of water droplet. Therefore, the contact angle increases with

increasing temperature of heat treatment. The hydrophobic surface is found with contact angle at temperature 175°C, 91.356°. Within the temperature range the microstructure undergoes an evolution of its shape from smooth and uniform pattern of microstructure peak into rough and non-uniform pattern of microstructure.

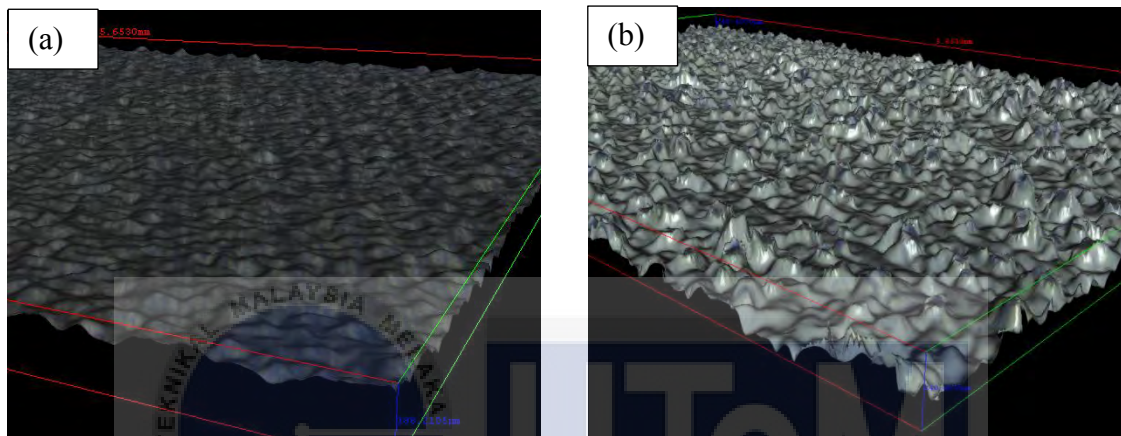


Figure 4.13 Comparison microstructure peak of heat treatment at (a) 25°C and (b) 175°C

When the duration of time of the sample expose to the heat treatment is increased, the contact angles decreased. The trends of the result for both experiment 75°C and 175°C show a similar result. The surface morphology images indicate the surface become smoother when the surface expose in longer time of hat treatment. It observed that the particle size of the thin films decreases when the duration of time of heat treatment is increased. These changes in the particle size at different periods show the effect of time exposure of heat treatment on growth mechanism of the thin film. Figure 4.6 and 4.10 above show the different image of particle size during time exposure 10 minutes with duration time of 30 minutes for temperature 75°C and 175°C. While Figure 4.14 shows the close-up image of the transformation of the surface morphology during duration of

times of heat treatment is 10 minutes and during times is 30 minutes for temperature 175°C.

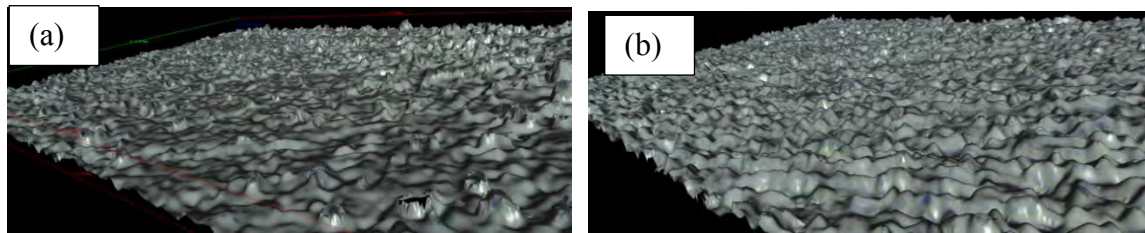


Figure 4.14: Comparison of microstructure peak of heat treatment for (a) 10 minutes and (b) 30 minutes for temperature 175°C

Surface roughness is very important criteria in hydrophobicity evaluation. This is because hydrophobicity phenomenon is depends on surface tension and surface roughness. The hydrophobicity is increased when the surface roughness increased. From the result, the surface roughness of the sample surface increased with the increased of the temperature of the heat treatment and during increasing of the duration time of exposure of the sample to the heat treatment, but slightly decreased during very high temperature 175°C and longer time exposure, 30 minutes. For experiment, heat treatment with varies temperature, the increasing the substrate roughness, give the highest value of contact angle. According to Table 4.2, the average of surface roughness is increased from 1.8558 μm during 25°C and increased to 7.665 μm at temperature 175°C. This clearly shows that the droplet was in Cassie-Baxter state where water vapor which is air is trapped in the cavities of a rough surface (Bharat and Yong, 2010). Where, the water droplet is suspended on the composite interface made of solid and air trapped between the droplet and the surface samples. Silvakumar (n.d.) stated that to obtained high contact angle is depended on the high percentage of liquid-solid interfaces replaced by liquid-gas interface.

The results for both experiment of heat treatment with varies the times show the similar trends of result as shown in Figure 4.15 below. Further increasing of the surface roughness in experiment of heat treatment with varies the times, it resulted the decreasing of the contact angle. It due to the long exposure of the high heat treatment, the water droplet collapses between the asperities and follows the surface topography existing in the Wenzel state. Wenzel state is the condition where formulate that the contact angle of a liquid with a rough surface is different with the smooth surface (Thierry and Frederic, 2015). It explained that the water droplet is in full contact with the surface and amplified by the surface roughness. According to Silvakumar (n,d), Wenzel is the liquid droplet retains contact at all points with the solid surface bellows it.

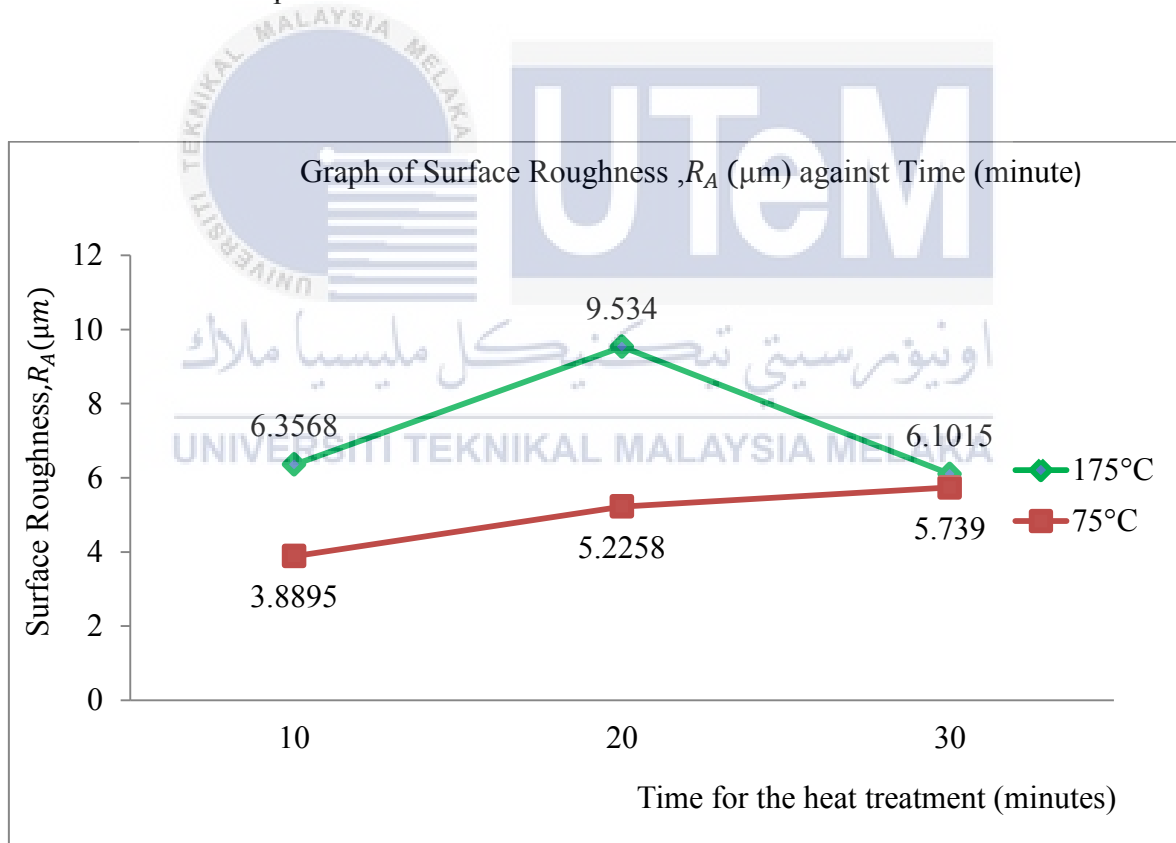


Figure 4.15: Graph of comparison between surface roughness against duration of time for heat treatment 75°C and 175°C

There are clear correlation between the value of contact angle, image of the surface morphology and value of surface roughness with the effect of temperature. First, the increases of the temperature, causes the surface roughness to be enhanced, it can be seen from the image of the surface morphology of the surface. It correlates with the value of the contact angle of the surface, where, the value of contact angle is increased when the temperature of the heat treatment is increased. However, the value of the contact angle start to decreases when the duration of times of heat treatment is increased, and the surface roughness will be increased until at very high temperature and at longer duration of times of heat treatment, the surface roughness starts to decreases. The surface morphology presented that the surface that undergoes longer times of heat treatment return to become smooth from the rough surface during shorter time of exposure to the heat treatment. Therefore, it can be conclude that, to provide the mostly hydrophobic surface, the temperature of the heat treatment must be high with shorter duration time of the heat treatment process to create a high surface roughness with complex surface morphology which is rough surface roughness.

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CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

As a conclusion, the literature study to investigate the effect of temperature of polymer coating on aluminum substrate to observe its hydrophobicity at different temperature and time has been presented in this report. Aluminum substrate with polymer coating were undergoes heat treatment with two different condition; first is heat treatment with varies the temperature and second is heat treatment with varies the times using two different temperature, 75°C and 175°C. This substrate was characterized using contact angle measurement techniques, surface roughness and surface morphology respectively. Surface roughness and surface morphology have been analyze using Alicona Infinite Focus G^4 and the results confirms the presence of the rough microstructure on the treated surface and the contact angle reveals the hydrophobicity of the polymer surface.

Several conclusions can be drawn in regards to the effect of temperature on the surface substrate. It observed that the degree of the hydrophobicity can be increased by increasing the temperature of the heat treatment with shorter time exposure of the heat treatment process. It related to their roughness of the surface morphology. It is experimentally concluded that a correlation exists between increasing the surface roughness will enhanced the hydrophobicity of the surface substrate. The highest degree of hydrophobicity is exhibited by complex surface microstructure. The hydrophobicity state

of the surface was found after the heat treatment at high temperature which is 175°C with shorter time of exposure of the heat treatment which is at 5 to 10 minutes.

The measurement of the contact angle also used to measure the hydrophobicity of the surface. The results is correlate with the value of the surface roughness of the surface and as explained by Wenzel and Cassie-Baxter state, where when the temperature of the heat treatment is increased, the value of the contact angle also increased, which is the contact angle at high temperature 175°C is 91.356° and the surface roughness is 7.6655µm. The hydrophobic surface is remains during temperature of the heat treatment are 175°C for duration of heat treatment is 5 to 10 minutes. Before and after this condition, where the temperature of the heat treatment is lower and the longer duration of time exposure to the heat treatment the contact angle starts decreasing and no longer remains as a hydrophobic surface.

The analysis of the value of contact angle and surface morphology analysis have shown that the surface roughness significantly increase the degree of the surface hydrophobicity. The microstructure change due to the process of heat treatment changes the surface morphology and contributes in the hydrophobicity. The behavior of the microstructure, where become rougher at very high temperature and at very shorter time duration of time of heat treatment will increase the contact angle of the surface and increases the hydrophobicity of the sample.

5.2 Recommendations

There are several aspects can be improved for further investigation. Further tests can be conducted by using different material of the samples to determine the surface reaction of the different material due to the effect of temperature. The problems occurred in this study such as poor spraying technique which is using manually technique. It can be improved by using spray gun in next experimental work to get more consistent coating. Besides that, experiment can be continued using more high temperature than 175° and shorter time of heat treatment, which is shorter than 5 minutes, to analyze the effect of temperature on that condition.



REFERENCES

Adel, M.A., Mohamed, Aboubakr, M.A., and Nathalie, A.Y., 2014, Corrosion Behavior of Superhydrophobic Surfaces: A Review, *Arabian Journal of Chemistry*, 8, pp. 749-765.

Alicona, n.d., *Alicona InfiniteFocus Optical 3D Surface Metrology* [online]. http://www.microcube.ch/attachments/File/IFM_G4_en.pdf [Accessed on 27 April 2017].

Bedia, A., Bedia, F.Z., Ailleria, M., Maloufi, N., and Benyoucef, B., (2015), Morphological and Optical Properties of ZnO Thin Films Prepared by Spray Pyrolysis On Glass Substrates at Various Temperatures for Integration in Solar Cell, *Energy Procedia*, 74, pp. 529-538.

Bernagozzi, I., Antonini, C., Villa, F., and Marengo, M., 2013, Fabricating Superhydrophobic Aluminum: An Optimized One-Step Wet Synthesis Using Fluoroalkyl Silane, *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 441, pp. 919-924

Bharat, B and Yong, C.J., 2011, Natural and Biomimetic Artificial Surfaces for Superhydrophobicity, Self-Cleaning, Low Adhesion, and Drag Reduction, *Progress in Materials Science*, 56, pp. 1-108.

David, B., 2015, *NPL Purchases 3rd Alicona Infinte Focus System*. [online]. Available at: <http://www.gtma.co.uk/npl-purchases-3rd-alicon-a-infinite-focus-system/> [Accessed on 27 April 2017]

Dmitriy, F., Irina , S., Evgeniya, O., and Konstantin, P.,2015, Experimental Research of Wetting and Drop Evaporation Processes on a Heated Substrates as a Method for Research Skills Development for Heat and Power Engineering Students, *Procedia- Social and Behavioral Sciences*, 206, pp. 307-314.

Graves, L., 2014, *Wild Taro* [online]. Available at: <http://www.outdooralabama.com/wild-taro-0> [Accessed on 28 November 2016].

James, D., Georg, S., and Leica, M., 2015, *What You Always Wanted to Know About Digital Microscope, but Never Got Around to Asking* [online]. Available at: <http://www.leica-microsystems.com/science-lab/what-you-always-wanted-to-know-about-digital-microscopy-but-never-got-around-to-asking/> [Accessed on 22 April 2017].

Javed, I., Asim, J., Ziaul, H., Saqib, R., Rashida, J., and Attieh, A.A., 2016, ALD Grown Nanostructured ZnO Thin Film: Effect of Substrate Temperature on Thickness And Energy Band Gap, *Journal of King Saud University-Science*, 28, pp. 347-354.

Jonathan, M.S., 2013, Influence of Experiment Variables on the Measure of Contact Angle in Metals Using The Sessile Drop Method. *Procedia Materials Science*, 8, pp. 742-751.

Kubiak, K.J., and Mathia, T.G., 2014, Anisotropic Wetting of Hydrophobic and Hydrophilic Surfaces – Modelling by Lattice Boltzmann Method, *Procedia Engineering*, 79, pp. 45-48.

Lomga, J., Varshney, P., Nanda, D., Satapathy, M., Mohapatra, S.S., and Kumar, A., 2017, Fabrication of Durable and Rechargeable Superhydrophobic Coatings with Excellent Self-cleaning and Anti-fogging Properties for Aluminum Surfaces, *Journal of Alloys and Compounds*, doi:10.1016/j.jallcom.2017.01.243.

Mark, D., 2016. *Ecological Mechanics: Principles of Life's Physical Interactions*. United States, Princeton University Press.

Minglin, M., and Randall, M.H., 2006, Superhydrophobic Surfaces, *Current Opinion in Colloid & Interface Science*, 11, pp. 193-202.

Mittal, K.L., and Robert, H.L., (2014), *Surface Science Corner*. [online]. Available at: <http://www.plasmablog.com/category/surface-science-corner/> [Accessed on 30 November 2016].

Nurxat, N., Waseem, S.K., Yu, L., Muhammet, C., and Ramazan, A., 2012. Superhydrophobic Electrospun Nanofibers, *Journal of Materials Chemistry A*, 1, pp. 1929-1946.

Panrico, 2017, *Digital Microscope*. [online]. Available at: <http://www.panrico.com.tw/product.php?mode=show&cid=149&pid=1181> [Accessed on 27 April 2017].

Peru, D.,A.,and Sanchez, 2013, *Pistia Stratiotes-Water Lettuce*. [online]. Available at: <http://aquascaping-pe.blogspot.my/2013/02/water-lettuce-pistia-is-genus-of.html> [Accessed on 28 November 2016].

Peter, E., 2005, *The Advantages of Digital Microscope*. [online]. Available at: <http://ezinearticles.com/?The-Advantages-of-Digital-Microscopes&id=67868> [Accessed on 21 April 2017].

Quanshui, Z., and Cunjiang, L., (2014), Size Effects of Surface Roughness to Superhydrophobicity, *23rd International Congress of Theoretical and Applied Mechanics*, 10, pp. 462-475.

Ravi, K.V.V.N., 2016, *What is Hydrophobicity*. [online]. Available at: <https://www.quora.com/What-is-hydrophobicity> [Accessed on 28 November 2016].

Satish, A.M., Pedraza, D.F., Nagaraja, D.H., Pratap, B.W., and Satish, C.G., 2013. Durability and Restoring of Superhydrophobic Properties in Silica-based Coatings, *Journal of Colloid and Interface Science*, 405, pp. 262-268.

Selvakumar, N., Barshilia, H. C., Ramesh, M., and Rajam, K.S., n.d., Effect of Substrate Roughness on Contact Angle of Sputter Deposited Superhydrophobic PTFE Coatings, *Surface Modification Technologies XXIII*.

Shivaraj, B.W., Narasimha, M.H.N., Krishna, M., and Satyanarayana, B.S., 2015, Effect of Annealing Temperature on Structural and Optical Properties of Dip and Spin Coated ZnO Thin Films, *Procedia Material Science*, 10, pp. 292-300

Stefan, S., 2016, *Focus Variation in Optical Metrology-An Interview with Stefan Scherer, CEO of Alicona* [online]. Available at <http://www.azom.com/article.aspx?ArticleID=10330> [Accessed on 27 April 2017].

Thierry, D., and Frederic, G., 2015, Superhydrophobic and Superoleophobic Properties in Nature, *Material Today*, 18, pp. 273-285

Tokoro, T., 2016, Effects of Temperature and Surface Roughness on the Evaluation of Hydrophobic Properties of Silicone Rubber, 978-1-5090-4654-6, Japan: IEEE

Xiuyong, C., Jianhui, Y., Jing, H., Kun, R., Yi, L., Shaoyang, L., and Hua, L., 2014, Large-scale Fabrication of Superhydrophobic Polyurethane/Nano- Al_2O_3 Coatings by Suspension Flame Spraying for Anti-corrosion Applications, *Applied Surface Science*, 311, pp. 864-869.

Wael, I, EL, d., Rafik, A., Wagih, A.S., Abdel, G.M.E.D, and Ahmed, H., 2015, Improved Adhesion of Superhydrophobic Layer On Metal Surface s Via One Step Spraying Method, *Arabian Journal of Chemistry*, pp. 1-10.

Yongcai, T., Jin, Y., Linting, Yin., Beibei, C., Hua, T., Chan, L., and Changsheng, L., 2014, Fabrication of Superhydrophobic Polyurethane/MoS₂ Nanocomposite Coatings With Wear-Resistance, *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 459, pp. 261-266.

Yuehua, Y., and Lee, T.R., (2013), Contact Angle and Wetting Properties, *Surface Science Technique*, XXIII, pp. 663

