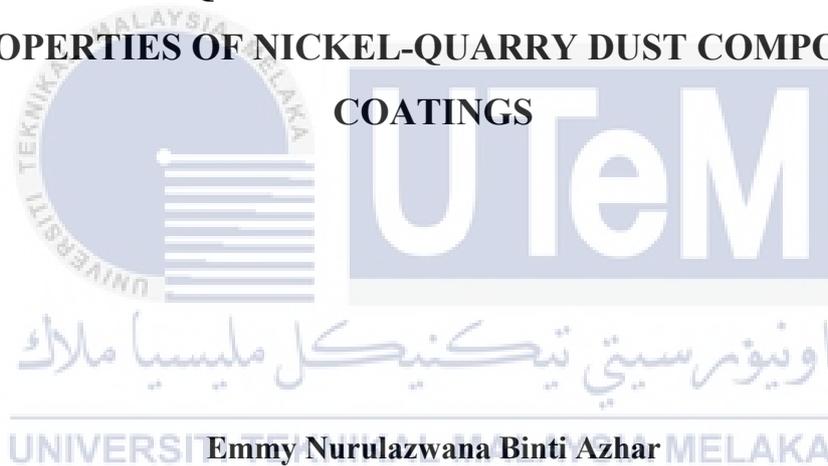




Faculty of Manufacturing Engineering

**EFFECT OF QUARRY DUST COMPOSITION ON THE
PROPERTIES OF NICKEL-QUARRY DUST COMPOSITE
COATINGS**



Master of Manufacturing Engineering (Quality System Engineering)

2018

**EFFECT OF QUARRY DUST COMPOSITION ON THE PROPERTIES OF
NICKEL-QUARRY DUST COMPOSITE COATINGS**

EMMY NURULAZWANA BINTI AZHAR

**A thesis submitted
in fulfillment of requirements for the degree of Master of Manufacturing Engineering
(Quality System Engineering)**



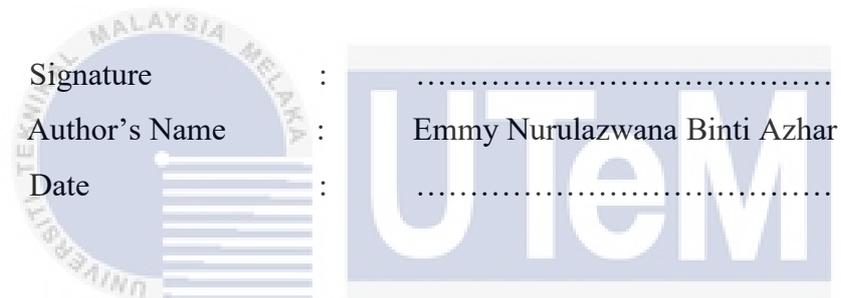
UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2018

DECLARATION

I declare that this thesis entitled “Effect of Quarry Dust Composition On The Properties Of Nickel-Quarry Dust Composite Coatings” is the results of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in term of scope and quality for the award of Master of Manufacturing Engineering (Quality System Engineering).

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ABSTRACT

The rationale of this research is to study the effect of quarry dust composition on the properties of nickel-quarry dust (Ni-QD) composite coating on aluminium alloy 6061 (AA6061) substrate. AA6061 is widely used in aircraft industries due to its low density to weight ratio and high strength. As aluminum operates in aggressive environments in atmospheric air, the aluminium must be coated to prevent corrosion. However, aluminum and its alloys have a high affinity to oxygen which means it always be covered with a thin oxide film growing rapidly. Thus, surface pre-treatment should be done before proceed to electrodeposition. The composition of quarry dust in nickel Watt's bath is varied from 10g/l to 50g/l. The characterization of quarry dust particles and Ni-QD composite coating on AA6061 substrate was characterized using X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF) and Scanning Electron Microscopy (SEM). Ni-QD composite coating also being tested to investigate the wear and hardness properties. From the results, the hardness of Ni-QD composite coating increased by increasing the quarry dust composition. Other than that, Ni-QD composite coatings also showed a good result in wear testing where the average of COF of Ni-QD composite coatings is lower than bare AA6061.

ABSTRAK

Rasional kajian ini adalah untuk mengkaji kesan komposisi debu kuari ke atas sifat salutan komposit debu kuari dan nikel pada aloi aluminium 6061 (AA6061) substrat. AA6061 digunakan secara meluas untuk pembuatan kapal terbang kerana ketumpatan yang rendah kepada nisbah berat badan dan kekuatan yang tinggi. Oleh kerana aluminium beroperasi dalam persekitaran yang agresif dalam udara atmosfera, aluminium mesti bersalut untuk mencegah kakisan. Walau bagaimanapun, aluminium dan aloi mempunyai afiniti yang tinggi kepada oksigen yang bermakna ia sentiasa ditutup dengan filem oksida. Oleh itu, pra-rawatan permukaan perlu dilakukan sebelum meneruskan untuk penempatan. Komposisi debu kuari adalah berbeza-beza dari 10g/l sehingga 50g/l. Pencirian zarah debu kuari dan salutan komposit debu kuari dan nikel pada aloi dilakukan menggunakan XRD, XRF dan SEM. Ujian juga dilakukan bagi mengenalpasti sifat haus dan kekerasan salutan komposit debu kuari. Berdasarkan hasil ujian, kekerasan salutan komposit debu kuari dan nikel meningkat dengan meningkatkan komposisi debu kuari itu. Selain daripada itu, salutan komposit debu kuari dan nikel juga menunjukkan keputusan yang baik dalam ujian haus di mana purata COF lapisan komposit debu kuari dan nikel adalah lebih rendah daripada AA6061 terdedah.

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DEDICATION

To my beloved family and Iswandie, I love you ❤️



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First and foremost, all praise to the Most Gracious and the Most Merciful Allah for His generous blessings and strength given to me complete my Master Project successfully.

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LIST ABBREVIATIONS, SYMBOLS, NOMENCLATURE

%	-	Percent
°C	-	Degree Celsius
μ	-	Micro
A/dm ²	-	Ampere per decimetre squared
Al	-	Aluminium
cm	-	Centimetre
g/l	-	Gram per litre
HV	-	Hardness Vickers
ml	-	Millilitre
mm	-	Millimetre
N	-	Newton
NaCl	-	Sodium Chloride
NaOH	-	Sodium Hydroxide
Ni	-	Nickel
wt.%	-	Weight percent

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Increasing applications in aerospace and allied fields of aluminium alloy 6061 (AA6061) is due to the inherent properties like lightness and good strength-to-weight ratio. It replaces many metals in applications where weight is considered as a major factor since its density is one third of the steel. As one of the important applications, AA6061 is employed in aerospace structural fastened joints where wear and fatigue damages can cause catastrophic failures under fluctuating loads (Gupta et al., 2014).

Aluminium is a reactive metal that owes its corrosion resistance to a thin, protective oxide surface layer, which is generally stable in air and aqueous solutions. Although aluminium oxide is generally protective, pores and other defects caused from alloying elements can lead to localised corrosion (Paloumpa et al., 2004). One possibility to increase the durability and performance of materials for different applications is to protect those using composite coatings. In recent years, there has been a growing interest in the development of composite coatings (Shrestha et al., 2001).

Hafiz et al. (2010) mentioned that due to the low mechanical strength, pure aluminium cannot be used as a structural component. Usually, it is combined with various alloying elements to produce grades which are suitable for a large number of applications. Due to their light weight and high thermal conductivity, aluminium alloy have also been considered for

tribological application for example in engine parts and sports products. However, their use has been limited because of rigidity and lack of wear resistance and adequate strength. Also, due to their low melting points, exposure of aluminium alloy to moderately elevated temperatures can cause extensive surface degradation. There is a need for surface protection systems with adequate combined mechanical strength and heat resistance capability for demanding application.

One way to improve the response of aluminium alloys to friction and surface degradation is to cover the surface of aluminium alloys with nickel coatings. Previously, cadmium and hard chromium have been used extensively as protective coatings but cadmium is highly toxic in its metallic form. Both coating and the deposition process are potentially harmful. In this study, nickel is used to replace hazardous chromium and cadmium. Nickel which possesses high tensile strength and good toughness is a popular choice as a matrix material and also it can disperse both soft and hard reinforcements (Gupta et al., 2014).

Significant interest on electrodeposition of nickel on AA6061 has increased considerably due to the fact that coating can eliminate the disadvantages of uncoated aluminium to some extent. Among various process technologies for composite coatings, electrodeposition has advantages such as cost-effectiveness relative to spray and sputtering processes (Chen et al., 2002). This technique can provide special surface properties, like increase hardness, abrasion and corrosion resistance, low friction coefficient to meet the requirements of advanced materials.

Therefore, in this study, nickel-quarry dust (Ni-QD) composite coating will be deposited on AA6061 through electrodeposition technique using various compositions of quarry dust. The effect of various composition of quarry dust on Ni-QD composite coatings to tribological, hardness and corrosion resistance will be covered.

No research has been done on studying the effect of quarry dust as reinforcement to the properties of the composite coating. Therefore, the literature review is only referring to the composite coating reinforced with conventional ceramic particles or fly ash.

1.2 Problem Statement

Nickel matrix composites have commonly been reinforced with most conventional ceramic particles such as SiC, Al₂O₃, TiC, B₄C, MgO, Si₃N₄ and AlN. However, the cost of manufacturing these composites is high and this limits their utilization in several engineering designs. Hence, continuous attempts being made to produce low cost of metal matrix composites. Recently, aluminium alloy composites reinforced with fly ash, a waste by-product of combustion, has been engineered (Rohatgi *et al.*, 2010), as potential substitutes to conventional composites in a few applications.

Tremendous efforts have been done in the area of coating technology to study the utilization of by-products and waste material which can be used as inert particles that act as reinforcement of metal matrix. Quarry dust as by-products from crushing of coarse aggregates during quarrying activities has received considerable attention to enhance the properties of nickel matrix composite coatings.

Quarry dust is crushed dust obtained from stone boulders in stone crushers during the production of coarse aggregates. The quarry dust consists of excess fines and is dumped in open fields that cause environment pollution. In the recent past good attempts have been made for the successful utilization of various industrial by products such as fly ash, silica fume, rice husk ash, foundry waste to save environment (Yap *et al.*, 2013).

Quarry dust have gained attention to be used in various industries such as construction

industry manufacturing of building material industry due to the high percentage of ceramic particles in quarry dust itself, SiO_2 and Al_2O_3 . Due to the high contents of SiO_2 and Al_2O_3 in quarry dust, fundamental study on the formulating of nickel matrix composite coating utilizing quarry dust as reinforcement will be formed. Limited research has been carried out on nickel reinforced with natural resource by product. However, the information regarding composite coating that use quarry dust as reinforcement element in metal matrix composite coating has not been reported.

Therefore, this research will be focused on the effect of varying quarry dust content on the Ni-QD composite coatings deposited on AA6061 through electrodeposition technique in order to enhance the mechanical properties of pure AA6061.

1.3 Objectives

1. To characterize the effect of various composition of quarry dust particle on the surface morphology of Ni-QD composite coating by using Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF).
2. To investigate the effect of various composition of quarry dust on the wear properties of electrodeposited Ni-QD composite coatings on AA6061 substrate.
3. To examine the effect of various composition of quarry dust on the hardness of electrodeposited Ni-QD composite coatings on AA6061 substrate.

1.4 Scope of Study

The scope of this study is to investigate the effect of varying the composition of quarry dust as reinforcement and annealing treatment process on the hardness, wear behaviour, and

corrosion resistance of Ni-QD composite coating on AA 6061 substrate. In this study, nickel and AA6061 were selected as anode and cathode respectively. The electrolyte was prepared by using fixed amount of nickel salt and various composition of quarry dust. AA6061 substrate was undergone surface pre-treatment using mechanical pre-treatment which is grinding and polishing prior to the electrodeposition process. The samples were produced using five composition of quarry dust (10g, 20g, 30g, 40g, and 50g) in order to find the optimum composition of quarry dust. The characterization of the quarry dust particles and coatings was carried out using SEM, XRD and XRF. The effect of quarry dust composition on the coating properties will be investigate using a hardness test and wear test.



CHAPTER 2

LITERATURE REVIEW

2.1 Composite Coating

2.1.1 Introduction

Composite is a material made from two or more constituent materials with significantly different physical or chemical properties that, when combined, produce a material with characteristics different from the individual components. The individual components remain separate and distinct within the finished structure.

Composites coatings fulfill many technical and decorative functions. Composite coatings working initially on systems designed for helicopters. Since then, many company has cooperated with designers and engineers from aerospace companies to provide coatings for the variety of the composites now used in many aircraft.

Composite coating has been well developed. The advantages of such coating are quite clear. Composite coatings are widely applied to mechanical component due to their high mechanical, chemical and tribological properties, as well as excellent corrosion resistance (Wan et al., 2015). Kan et al., (2015) found that the simultaneous deposition of the non-metal inclusions and metal phases to form composite layers brings a significant improvement to physical and mechanical properties of the coatings. Particles- reinforced composite coatings produced by electrodeposition have been widely used due to their high hardness, good wear and corrosion resistance compared to pure metal or alloy coatings (Ewa et al., 2010).

2.1.2 Types of Composite Coatings

(a) Ceramic Matrix Composite Coatings

Ceramic matrix composite (CMC) coatings are outstanding in their ability to withstand high temperatures, in addition their hardness and wear resistance. As mentioned by Gavalda et al. (2017), CMC have increased in demand in many different industries because of their excellent mechanical characteristics such as the strength-to-weight, stiffness-to-weight, corrosion and fatigue resistance.

Commonly, one of the materials acts as a matrix and the other one as reinforcement. The matrix is in charge of spreading the stress to the fibres and protecting them from external damages while providing the final shape of the component. Besides, reinforcement provides greater mechanical properties (Teti, 2002).

(b) Polymer Matrix Composite Coatings

Polymer matrix composites (PMCs) are comprised of a variety of short or continuous fibres bound together by an organic polymer matrix. Unlike CMC, in which the reinforcement is used primarily to improve the fracture toughness, the reinforcement in a PMC provides high strength and stiffness.

PMCs are often divided into two categories which are reinforced plastics and advanced composites. The distinction is based on the level of mechanical properties (usually strength and stiffness). Reinforced plastics, which are relatively inexpensive, typically consist of polyester resins reinforced with low-stiffness glass fibres. Advanced composites, which have been in use for only about 15 years, primarily in the aerospace industry, have superior strength and stiffness, and relatively expensive (Rodney et al., 1988).

(c) Metal Matrix Composite Coatings

Metal matrix composite (MMC) coatings possessing excellent tribological properties, good corrosion resistance and higher fatigue life are gaining widespread popularity (Ramesh et al., 2003).

Mentioned by Panagopoulos et al. (2011), MMC have been the subject of significant research and development effort over past decades, as they offer many advantages over conventional metallic alloys. Their most important characteristics are high specific strength and stiffness, enhanced mechanical properties, increased elevated temperature strength, improved wear resistance and control over thermo-physical properties (thermal expansion and conductivity).

Therefore, MMC coatings are considered as important engineering materials for many industrial and technological applications. However, in order to reduce the cost and improve the properties of MMC coatings, new materials and production produced have been used. The utilization of natural resources such as fly ash, quarry dust and etc which is by-product material that contain most conventional ceramic particles such as Al_2O_3 and SiO_2 for the production of MMC coatings was introduced by Rohatgi (Panagopoulos et al. 2011).

2.1.3 Nickel Composite Coatings

Nickel, being an engineering material, is the widely used metal matrix in aerospace and automotive industries due to their high specific strength, good toughness and corrosion resistance (Zhong et al., 2012). Besides, nickel which possesses high tensile strength and good toughness is a popular choice as a matrix material and also it can disperse both soft and hard reinforcements (Pradeep et al., 2014).

It is well known that the properties of the composite coatings are heavily dependent

upon the degree of particles incorporation in deposit. Higher incorporation percentages and more uniform distribution of inert particles in the metal matrix lead to the improvement of the mechanical, tribological, anti-corrosion and anti-oxidation properties coatings. (Qiuyuan et al., 2008).

Mechanical and chemical properties of nickel can be improved by adding the second phase particles into nickel based composite system (Hongmin et al., 2015). Furthermore, Chang et al. (2015) stated composite coatings containing fine abrasive particles like SiC, Al₂O₃, Si₃N₄, diamond etc. can be remarkable to increase the hardness and reduce the friction coefficient as well as the wear rate of the mechanical component.

Addition of ceramic particles to synthesize composite coatings act as a barrier to dislocation movement and grain boundary sliding thus enhanced hardness of composite coatings. The hardness of composite coatings mainly influenced by two aspects such as hardness of metal matrix which is determined by microstructure of coatings and volume content of reinforced particles in the composite coatings (Ramesh et al., 2003).

A comparison of Vickers microhardness for a wide range of nickel composite coatings is shown in Figure 2.1. It clearly shows significant improvement in hardness of composite coatings compared to pure nickel coatings. The microhardness of composite systems increased with increase in volume percent of the reinforcements.

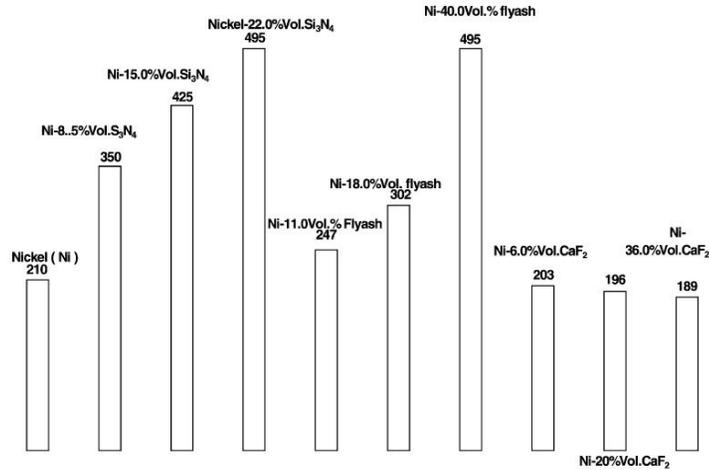


Figure 2.1: Hardness of nickel composite coatings. (Ramesh et al., 2003)

The improvement in microhardness as well as wear resistance of composite coatings mainly depends on reinforced second phase particles in the nickel matrix act as a physical barrier to nickel grain growth and plastic deformation of nickel matrix under loading promotes grain refining and dispersive strengthening effect to improve microhardness as well as wear resistance composite coatings (Vaezi et al., 2008). The increase in microhardness and decrease in the wear rate of composite coatings is due to combination of grain refining and dispersion strengthening effect.

The variation of the wear rates of nickel composite coatings with completely different volume fractions of calcium fluoride, silicon nitride, and fly ash are shown in Figure 2.2 It is observed that all composite coating systems studied exhibits lower wear rates in comparison with nickel coating. Wear rates of all the composite coatings decreases with increase in volume fraction of the reinforcement. Nickel–calcium fluoride composites possess the lowest wear rate.

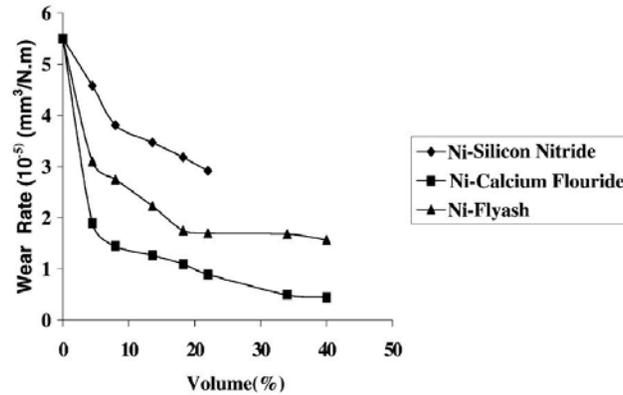


Figure 2.2: Variation of wear rate of coatings with volume fractions under sliding velocity of 1.57 m/s and contact pressure of 1.23MPa. (Ramesh et al., 2003)

Nickel composite coatings have better corrosion resistance than pure nickel coatings because reinforced second phase particles disturb the regular growth of nickel crystal and promote new nucleation site. This makes structure of composite coatings more finely crystalline and hence improved corrosion resistance of composite coatings (Szczygiel et al., 2005). Addition of these particles in composite coatings will decrease corrosion current density and will shift corrosion potential of composite coatings.

2.2 Electrodeposition of Nickel Coatings

2.2.1 Introduction

Electrodeposition is an important way to prepare MMC coatings with the merits of low cost, capability of achieving well controlled coatings at room temperature and coating complex component (Low et al., 2006 and Chen et al., 2015). A homogeneous and well-dispersed ceramic particle reinforced micro-structure with high ceramic particles concentration has been one of the central concerns in the MMC to optimize their mechanical performance

(Miserez et al. 2004).

2.2.2 Fundamental of Electrodeposition

Deposition of a layer of nickel on a substrate involves the dissolution of one electrode (the anode) and the deposition of metallic nickel on the other electrode (the cathode). Direct current is applied between the anode (positive) and the cathode (negative). Conductivity between the electrodes is provided by an aqueous solution of nickel salts.

When nickel salts are dissolved in water, the nickel is present in solution as divalent, positively charged ions (Ni^{2+}). When current flows, divalent nickel ions react with two electrons ($2e^-$) and are converted to metallic nickel (Ni^0) at the cathode. The reverse occurs at the anode where metallic nickel dissolves to form divalent ions. The electrochemical reaction in its simplest form is:



Because the nickel ions discharged at the cathode are replenished by the nickel ions formed at the anode, the nickel electrodeposition process can be operated for long periods of time without interruption.

2.3 Aluminium

2.3.1 Introduction

Aluminium is the most abundant metal in the earth and the third most abundant element, after oxygen and silicon. It makes up about 8% by weight of the earth's solid surface. The chief source of aluminium is bauxite ore. Its atomic number is 13. Aluminium is a soft, durable, lightweight, ductile and malleable metal with appearance ranging from silvery to dull

gray, depending on the surface roughness. Aluminium is nonmagnetic and non-sparking. Aluminium has about one-third the density and stiffness of steel. It is easily machined, cast, drawn and extruded (Daniel et al., 2013). Corrosion resistance can be excellent due to a thin surface layer of aluminium oxide that forms when the metal is exposed to air, effectively preventing further oxidation (Narinder et al., 2010).

2.3.2 Aluminium Alloy

Aluminium and its alloys are desirable materials due to high strength stiffness to weight ratio, good formability, and recycling potential for use in automobile industries as components of internal combustion engine (Daniel et al.,2013). Recently, aluminium alloys are increasingly being used to improve fuel consumption of vehicles by reducing their weight, along with their excellent workability and corrosion resistance (Makoto et al., 2009). In order to improve their surface properties, plating onto aluminium alloys has been commonly adopted.

Since pure aluminium is quite soft compared to other metals, thus, the metal can be alloyed with another metal to overcome the weakness of aluminium. Most of the aluminium that has been alloyed with at least one other element is already reaching the market place (Jeyaraj et al., 2015). This can be proved that the combination of aluminium alloy with other element can give positive feedback to the users. There are few series in aluminium alloy as shown in the Table 2.1 below.

Table 2.1: Series of alloys and its characteristics (aluminium.org, 2015)

Alloy Code	Characteristics
1xxx Series	<ul style="list-style-type: none"> • Contains no alloying elements • The proportion of aluminium is 99.3-99.9%

	<ul style="list-style-type: none"> • Suitable for applications mainly in electrical and heat-power industry • Materials of this series are considered non-hardenable alloys • Tensile strengths of 40-60 Mpa
2xxx Series	<ul style="list-style-type: none"> • The alloying element is copper • High strength alloys • The strength is achieved by the heat treatment process • The tensile strength is about 400 Mpa • Not suitable for surface treatments and poor for welding
3xxx Series	<ul style="list-style-type: none"> • The alloying element is copper • Moderate strength • Good formability • Suitable for anodizing and welding
4xxx Series	<ul style="list-style-type: none"> • The alloying element is silicon
5xxx Series	<ul style="list-style-type: none"> • The alloying element is magnesium • Moderate in strength (200-350 Mpa) • Excellent resistant to corrosion in aggressive atmosphere and seawater
6xxx Series	<ul style="list-style-type: none"> • The alloying elements are magnesium and silicon • Moderate in strength (200-350 Mpa) • Can be easily anodized
7xxx Series	<ul style="list-style-type: none"> • The alloying element is zinc • The highest strength among all series • The tensile strengths ranging between 450-500 MPa may exceed 600 MPa in some cases. stress corrosion, especially when welded
8xxx Series	<ul style="list-style-type: none"> • The alloying element is other than for the other series (including lithium)

2.3.3 Aluminium Alloy 6061

The 6XXX series alloys are a kind of ultra-high strength aluminium alloy, widely used in both aviation for the construction of plane structures such as wings and fuselages and civilian industries because of their excellent specific strength, and welding performance (Modi

et al., 1998). They have been considered as a new era of lightweight and high-strength structure materials (Haga et al., 2006) AA6061 is an aluminium alloy with zinc as its primary alloying elements. The presence of zinc as major alloying element will increase the strength and allow hot or cold precipitation hardening. The composition of elements present in AA6061 is shows in Table 2.2

Table 2.2: Composition of alloying elements in AA6061 (wikipedia, 2018)

Element	Content (wt. %)
Aluminium	95.85-98.56
Silicon	0.4-0.8
Iron	≤ 0.7
Copper	0.15-0.4
Manganese	≤ 0.15
Magnesium	0.8-1.2
Chromium	0.04-0.35
Zinc	≤ 0.25
Titanium	≤ 0.15
Others	0.05-0.15

Aluminium alloy 6061 has been the standard workhorse 6XXX series alloy to be broadly used in the aerospace industry ever since. It was the first successful Al-Zn-Mg-Cu high strength alloy using the useful effects of the alloying addition of chromium to improve good stress-corrosion cracking resistance in sheet products (Franco et al., 2015). However, alloy 6061 remains the baseline with a good balance of properties required for aerospace applications even other 6XXX alloys have since been developed with improved specific properties. In the Table 2.3 below shows the material properties of aluminium alloy 6061 .

Table 2.3: Material properties of AA6061 (MakeItFrom.com, 2008)

Properties	Value
Density	2.70 g/cm ³
Young's modulus	68.9 GPa
Specific heat capacity	897 J/Kg-K
Tensile strength	124-290 MPa
Elongation at break	12-25%
Poisson's ratio	0.33
Melting temperature	585 °C

2.4 Quarry Dust

2.4.1 Introduction

The extraction of aggregates, coal and other mineral resources produces large volume of waste material. These wastes arise due to a number of factors including contamination of the natural resource, the existence of local fault zones and overburden, and the processing of the mineral resource itself (Woolley, 1994; Rockliff, 1996; Wainwright, 2002).

Quarry dust is a by-product in the production of concrete aggregates during crushing process of rocks. Quarry dust used in concrete as fine aggregates for protecting the natural environment. About 20-25% of total production in each crusher unit is left out the quarry dust as waste material. It becomes a useful additive to the natural soil to improve its strength characteristics (Sridharan et al., 2006).

As a result of sustained research and developmental works undertaken with respect to increasing application of the industrial waste, the level of utilization of quarry dust in the industrialized nations like Australia, France, Germany and UK has been reached more than 60% of its total production (Ilango et al. 2008). Quarry dust is use for road construction

and manufacture of building materials such as lightweight aggregates, bricks, tiles and autoclave blocks. The sizes of quarry dust are smaller than the 200 μ m sieve compared with the river sand (Ramesh et al., 2013).

2.4.2 Chemical Composition

Quarry dust consists of chemical components like silicon dioxide and alumina in major quality. Apart from these substance, a reactive magnesium oxide, sodium oxide, calcium oxide, iron oxide, potassium oxide, titanium dioxide, phosphorus pentoxide and manganese oxide are also found in major quantity. Table 2.4 below is the chemical composition of quarry dust particles as observed by using XRF technique.

Table 2.4 Chemical composition of quarry dust particles (Naganathan et al., 2011)

Composition	Concentration (wt%)
SiO ₂	69.94
Al ₂ O ₃	14.60
Fe ₂ O ₃	2.16
TiO ₂	0.26
MgO	0.38
CaO	2.23
Na ₂ O	2.40
K ₂ O	6.91
P ₂ O ₅	0.71
MnO	0.07

2.4.3 Formation and Physical Characteristic

Quarry dust is fine rock particles. When boulders are broken into small pieces quarry

dust is formed. It is gray in colour and it is like fine aggregate (Anitha et al.,2013). The physical properties of quarry dust are listed in Table 2.5.

Table 2.5: The physical properties of quarry dust (Anitha et al.,2013)

Property	Quarry Dust
Specific gravity	2.60
Absorption (%)	1.30
Bulk relative density (Kg/m ³)	1700
Fine particles less than 0.075mm (%)	14

2.4.4 Environmental Issues

Quarrying involves the production of significant amounts of waste. Some types of quarries do not produce large amounts of permanent waste, such as sand and gravel quarries, whereas others will produce significant amounts of waste material such as clay and silt. The good news is that they are generally inert and non-hazardous, unlike the waste from many other processes. However, there is still potential for damage to the environment, particularly with water contamination (Vincent et al., 2012).

For example, suspended particles. Even though they are chemically inert but it may imbalance freshwater ecosystems. Large amounts of solids can also exacerbate flooding, if it is dumped on the flood plains. The accumulation of waste by-products will still need to be stored and managed somewhere that will not affect the environment in an adverse manner. Furthermore, the treatment and disposal of the waste may produce more negative impacts on the environment.

While quarries can cause significant impact to the environment, with the right planning and management, many of the negative effects can be minimised or controlled and in many

cases, there is great opportunity to protect and enhance the environment, such as with the translocation of existing habitats or the creation of new ones.



CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter will discuss on the methodologies that will be used to carry out the analysis. This includes the method used to determine surface morphology of Ni-QD composite coatings, the wear behaviour, and hardness of Ni-QD composite coatings on AA6061 substrate.

The experiment starts with preparing 5 samples of substrates. Then, follow by surface pre-treatment. Surface pre-treatment divided into 3 stages which are alkaline cleaning, acid cleaning and zincating treatment. Next, aluminium substrate will be electrodeposited with Ni-QD composite. Later, all samples will be characterized and analysed to know the properties of Ni-QD composite coating deposited. The details of the experiment procedures is illustrated in flowchart in Figure 3.1

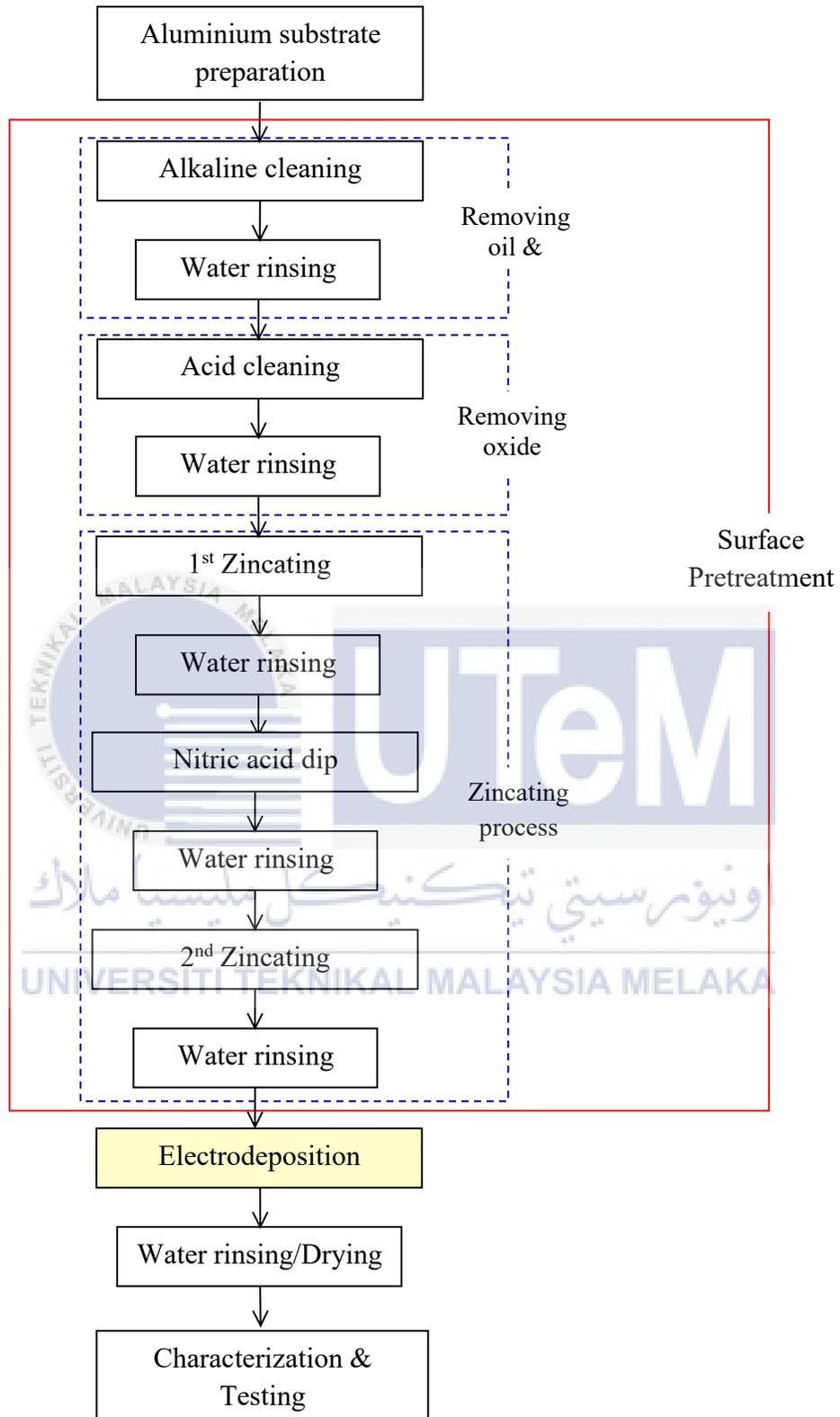


Figure 3.1: The experimental procedure flowchart

3.2 Aluminium Substrate Preparation

3.2.1 Sample Cutting

The substrates used in this study were rectangular AA6061 with dimension 40mm x 30mm x 5mm. 5 pieces of aluminium plate will be prepared.

3.2.2 Mechanical Pre-Treatment

All AA6061 specimens were grind with SiC papers with increasing finishes (180, 240, 320, 400, 600, 800, and 1200) and cleaned by ultrasonic vibration with acetone for 15 minutes, and then rinse by distilled water. The aim of the grinding is to attain better adherence of the coating and substrate due to strong mechanical interlocking (Hiramatsu et al., 1997). The AA6061 substrate then will be polished until obtained shiny surface. Polishing is important stage in preparing a specimen for microstructure analysis.

3.3 Surface Pre-Treatment

As it well known, aluminium and its alloys have high attraction to oxygen, which means that the freshly cleaned aluminium will always be covered with a thin oxide film. Therefore, it is difficult to coat the substrates covered with such an oxide film with good adhesion, so suitable surface preparation is required. Surface pre-treatment have three stages, which are alkaline cleaning, acid cleaning, and zincating.

3.3.1 Alkaline Cleaning

Polished AA6061 substrates are degreased in 50g/l NaOH solution for 10 seconds. Substrates then cleaned using distilled water. The purpose of alkaline cleaning removes oils

and greases from the substrates surface.

3.3.2 Acid Cleaning

Since aluminium exposed to air is expected to be covered by dense oxide coating, it is removed by dipping it in 50 vol% nitric acid solution for 20 seconds. During this step, nitric acid solution removes existing oxide layer and a thin, uniform oxide film on the AA6061 surface.

3.3.3 Zincating Pre-treatment

Pre-coated thin Zn films protect the aluminium against re-oxidation. Prior to the deposition of Ni-QD composite coatings, a double zincating process was conducted on the substrate at 45 seconds for the first zincating and at 15 seconds for the second zincating. Acidic etching for 15 seconds in HNO₃ solution at room temperature was performed between consecutive zincating operations. The chemical composition of the zincate solution is presented in Table 3.1.

Table 3.1 Chemical composition of zincate bath

Composition	Concentration (g/l)
Sodium hydroxide, NaOH	525
Zinc sulphate, ZnSO ₄ .7H ₂ O	100
Sodium potassium tartrate	9.8
Ferric Chloride, FeCl ₃	1

3.4 Electrodeposition Process

Cathode is the AA6061 substrate, whereas the anode is a nickel electrode as shown in

Figure 3.2. The composition parameters are given in Table 3.2. In addition to the surfactants, stirring at constant speed using mechanical stirrer will be used to maintain the homogeneity of the dispersed micro particles. The bath will be stirred for minimum of one hour using stirrer before proceed to electrodeposition process in order to avoid the particles to be agglomerated at the bottom of the bath.

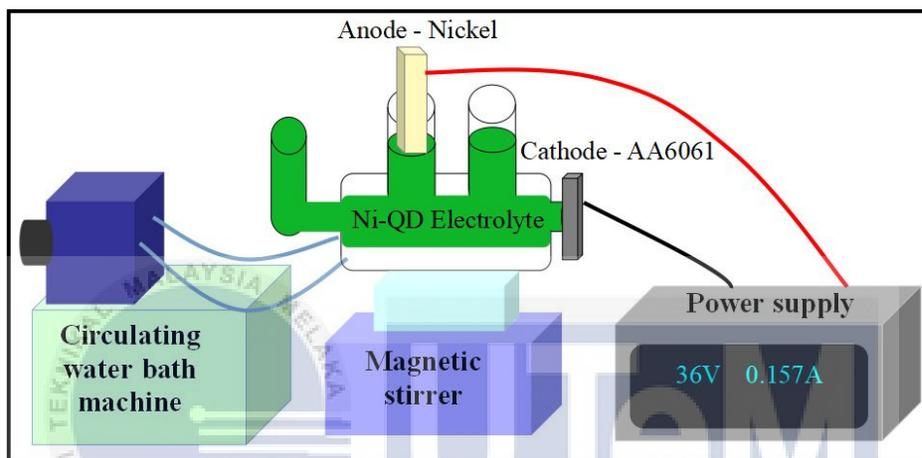


Figure 3.2 Schematic diagrams of electrodeposition process

Table 3.2

Chemical composition of Ni-QD Watt's bath solution and electrodeposition parameter

Composition	Concentration (g/l)
Nickel sulphate hexahydrate	200
Nickel chloride	20
Sodium citrate	30
Operating condition	
Temperature	40°C
Deposition time	1 hour
Current density	3 A/dm ²
Composition of quarry dust (g/l)	10, 20, 30, 40, 50

3.5 Material Characterization

The characterization of Ni-QD composite coating on the aluminium substrate is carried out to characterize the presence of nickel as the deposited coating layer on the AA6061 substrate and to analyse the surface morphology of the coating. The characterization is done by using SEM, EDS, XRD and XRF.

3.5.1 Scanning Electron Microscopy (SEM)

SEM is electron microscopes that use a focused beam of high-energy electrons to form highly magnified image. To produce an image on the screen, the electron beam scans over the area to be magnified and transfers this image to the screen. Sample must be electrically conductive, and non-conductive materials require thin conductive coating to prevent electrical charging of the specimen.

In this research, SEM is used to analyse the effect of Ni-QD towards the surface morphology of the coating. However, most of the nickel baths are acidic, and with nickel being very positive to aluminium on the electromotive scale, immersion deposits of nickel and chemical attack on the aluminium substrate will happen. In order to protect the aluminium surface during processing, the samples will undergo sputter process before being put under SEM.

3.5.2 Energy Dispersive Spectroscopy (EDS)

EDS makes use of the X-ray spectrum emitted by a solid sample bombarded with a focused beam of electrons to obtain a localized chemical analysis. Qualitative analysis involves the identification of the lines in the spectrum and is fairly straightforward owing to

the simplicity of X-ray spectra. Quantitative analysis (determination of the concentrations of the elements present) entails measuring line intensities for each element in the sample and for the same elements in calibration Standards of known composition.

3.5.3 X-Ray Diffraction (XRD)

XRD is an analytical technique looking at X-ray scattering from crystalline materials. Each material produces a unique X-ray "fingerprint" of X-ray intensity versus scattering angle that characterize crystalline atomic structure.

3.5.4 X-Ray Florescence (XRF)

XRF analysis conducted in this experiment to identify compound that existed in quarry dust. The primary radiation excites the atoms within the element causing electrons to be ejected from their shells. The vacancy left by each electron is then filled by another electron from the adjacent outer shell of the same atom. Because the energy levels of the electron orbits in each element are unique, the corresponding range of x-ray emissions produced by that element is also unique. Thus, energy the emitted will tell which element it represents. The analysis is conducted at Kolej Kemahiran Tinggi Mara (KKTM), Masjid Tanah.

3.6 Mechanical Testing

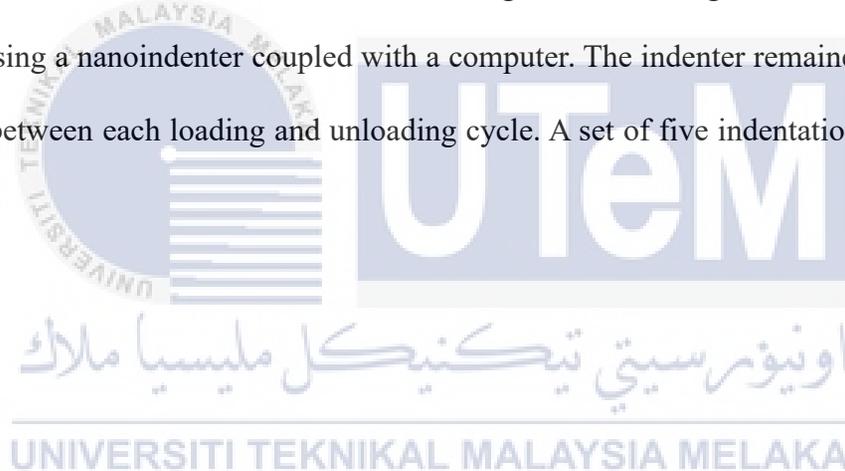
After the characterization analysis, the samples will be tested to determine the properties of wear, corrosion resistance, and hardness of the electrodeposited coating.

3.6.1 Wear Test

The wear behaviour of the coating will be analysed using ball-on-disc wear test. A 10mm stainless steel ball will be moved in reciprocate manner by the machine against the sample under a constant load of 10 N. The sliding time will be set to 600 seconds and wear track distance is 5 mm. The wear behaviour of the coating will be done by analysing tribology parameters resulted from the reciprocatory sliding of the ball against the coating layer.

3.6.2 Hardness Test

Vickers microhardness tests conducted using a load of 100g. Nanoindentation testing performed using a nanoindenter coupled with a computer. The indenter remained stationary for 15 seconds between each loading and unloading cycle. A set of five indentations was acquired for each test.



CHAPTER 4

RESULTS AND DISCUSSION

4.1 Characterization of Quarry Dust Particles

The chemical composition of quarry dust particles was investigated with aid of XRF techniques. Table 4.1 show the composition of quarry dust particles as found by XRF technique. The observation can be possibly attributed to the fact that quarry dust is consisted of many oxides and other compounds.

Table 4.1 Composition of quarry dust particles as found by XRF technique

Chemical Element	Concentration (Wt%)
SiO ₂	72.59
Al ₂ O ₃	15.19
Na ₂ O	5.16
K ₂ O	3.51
Fe ₂ O ₃	1.36
MgO	0.96
CaO	0.76
TiO ₂	0.21
SO ₃	0.15
P ₂ O ₅	0.07

It is found that the particles are having a size in the range of 0.1 to 600 μm . It has been

shows that the quarry dusts used in this study have an average size of 300 μm as shown in the Figure 4.1



Figure 4.1 Particle size distribution of quarry dust

The morphology of quarry dust was studied by SEM. It can be seen clearly from Figure 4.2 that the quarry dust particles were composed mostly in irregular shape with various sizes.

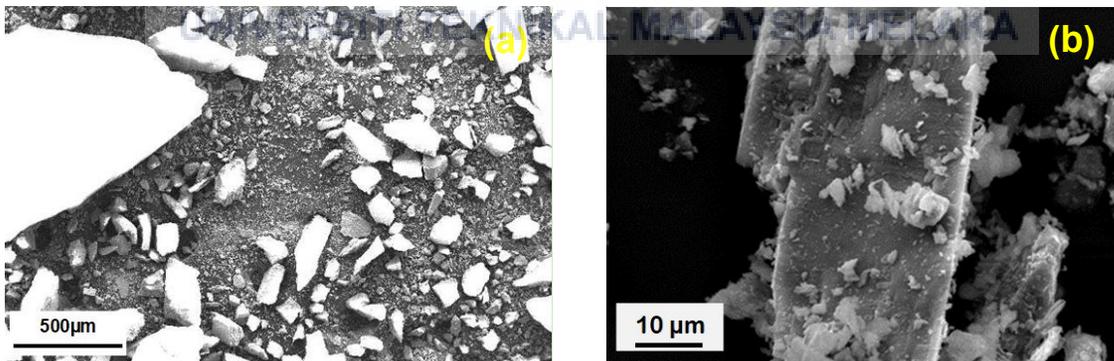


Figure 4.2 SEM image of quarry dust particles (a) before crushing (b) after crushing

4.2 Characterization of Ni-QD Composite Coatings

Figure 4.3 shows Ni-QD composite coatings electrodeposited on AA6061 substrate at various quarry dust compositions. All samples show the bright appearance of coating.

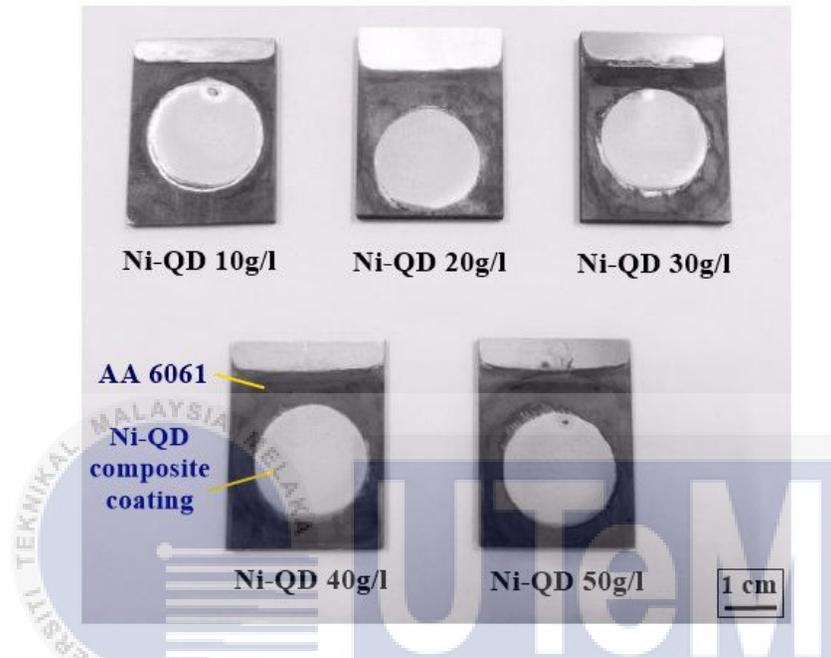


Figure 4.3: Electrodeposited Ni-QD composite coating on AA6061 with various composition of quarry dust

4.2.1 Phase Compositions of Ni-QD Composite Coatings

Surface of the electrodeposited Ni-QD composite coating has been analyzed using XRD. By using this machine, the Ni-QD composite coatings can be proved to be on the surface of the aluminium alloy substrate. Figure 4.4 shows the XRD pattern of the electrodeposited Ni-QD composite coatings on AA6061 substrate. XRD pattern shows the high intensity of Ni peaks hinder the identification of small peaks referring to quarry dust particles into the coating. Small peaks are referring to the low quantity of quarry dust particles into the coating. However, according to the peaks present, the main elements of quarry dust

like silica and alumina does exist on each samples. Thus, the presence of quarry dust was confirmed on the coating

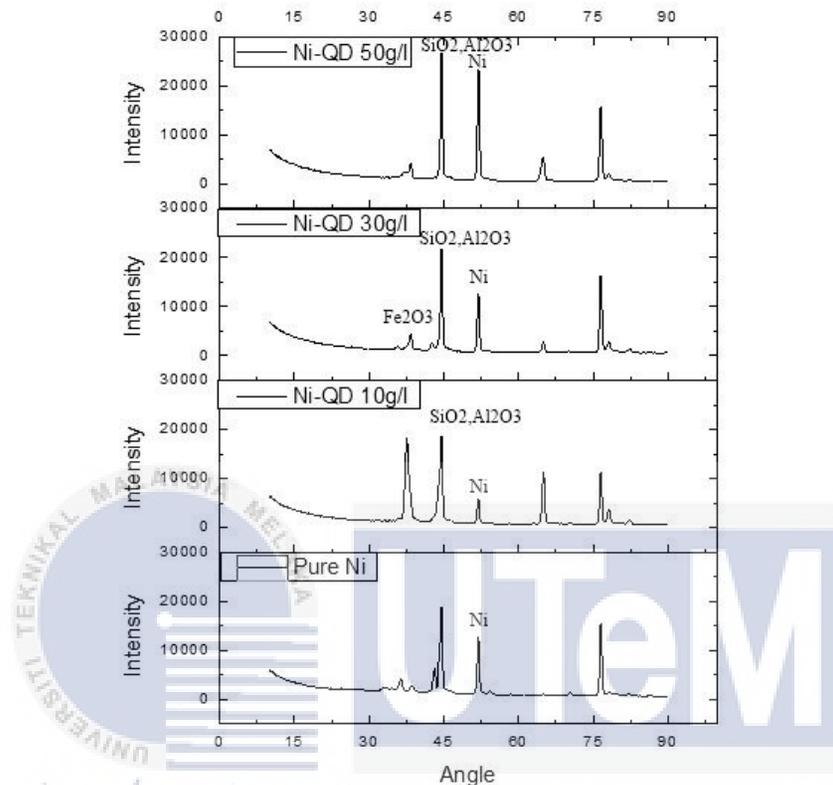


Figure 4.4: XRD patterns of electrodeposited Ni-QD composite coating

Figure 4.5 below shown the grain size of nickel electrodeposited on AA6061 substrate. Nickel grain size is reduce as the composition of quarry dust increase.

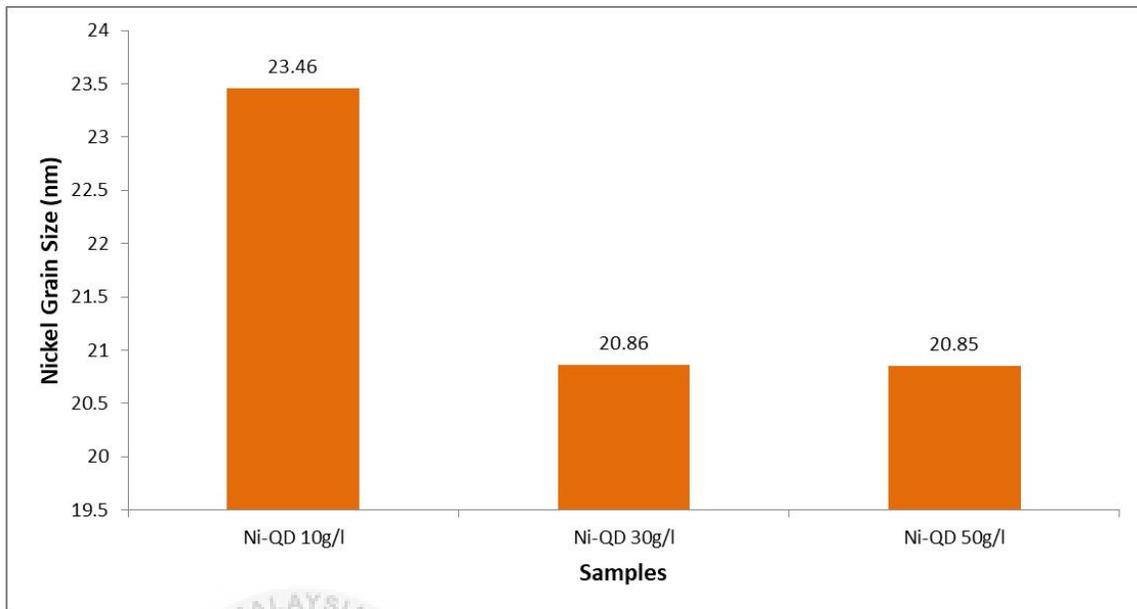


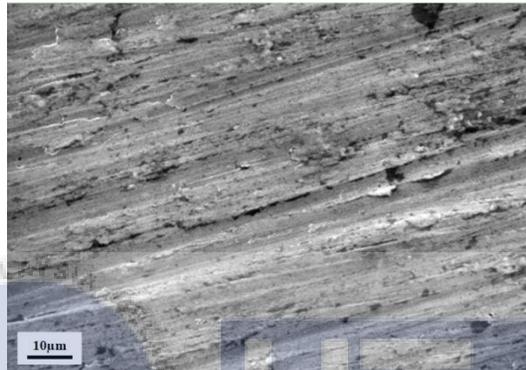
Figure 4.5 Grain size of nickel electrodeposited on AA6061

4.2.2 Surface Morphology of Ni-QD Composite Coatings

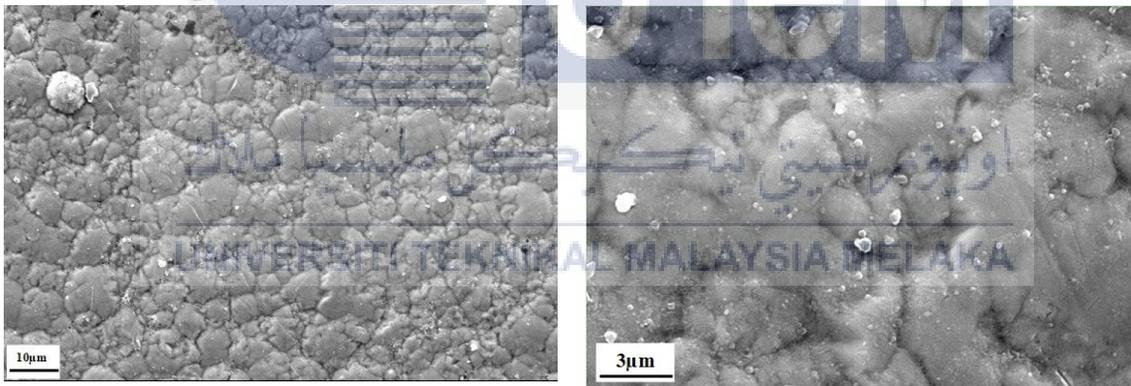
Surface morphology of the bare AA6061, pure Ni coating, and composite coatings at various quarry dust compositions are shown in Figure 4.6. The images show the coating at low and high magnification which is 1000 and 5000. From the figure, it was observed that the crystalline structures of nickel coating became finer in the presence of quarry dust particles. It was observed that the Ni-QD composite coatings already cover the surface of AA6061 and produce bumpy surface.

The co-deposition of quarry dust particles at 10 g/l shows a rough surface morphology compared to pure nickel coating. However, as the quarry dust content increased to 30g/l and 50g/l, the coating surface became rougher, due to the formation of colonies like morphology that consist lots of nickel grains with various sizes. These images also show the agglomeration

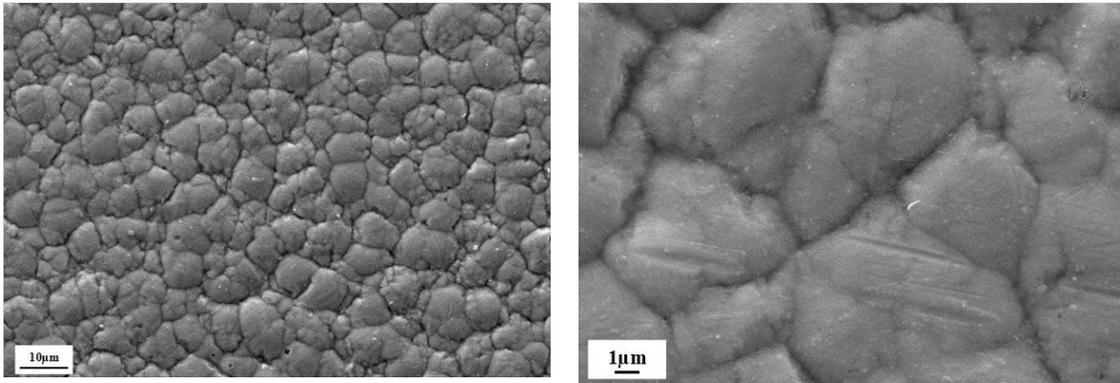
of colonies like morphology. It seems possible that these results are due to the hindering of nickel grain growth during the deposition process. Each crystal could be found to contain several smaller grains. The images indicate that the morphology changed from large to small crystal shape and it shows a growth of nickel crystal. (Rashidi et al., 2008)



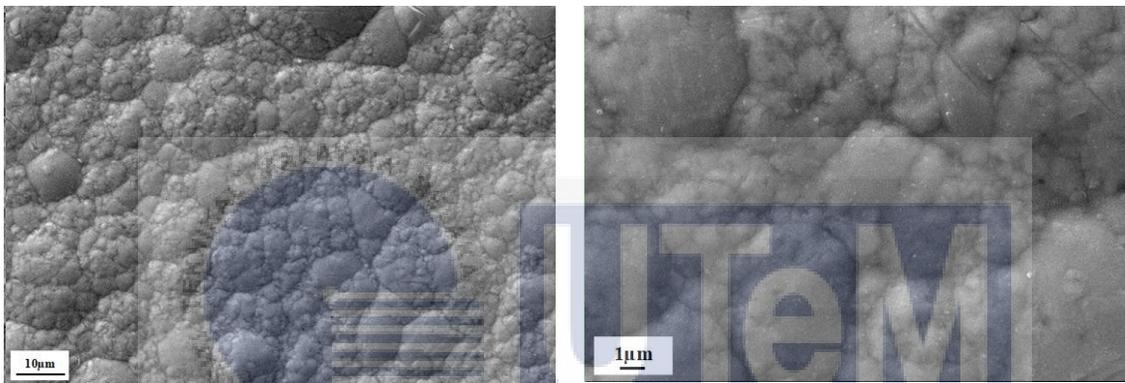
(a)



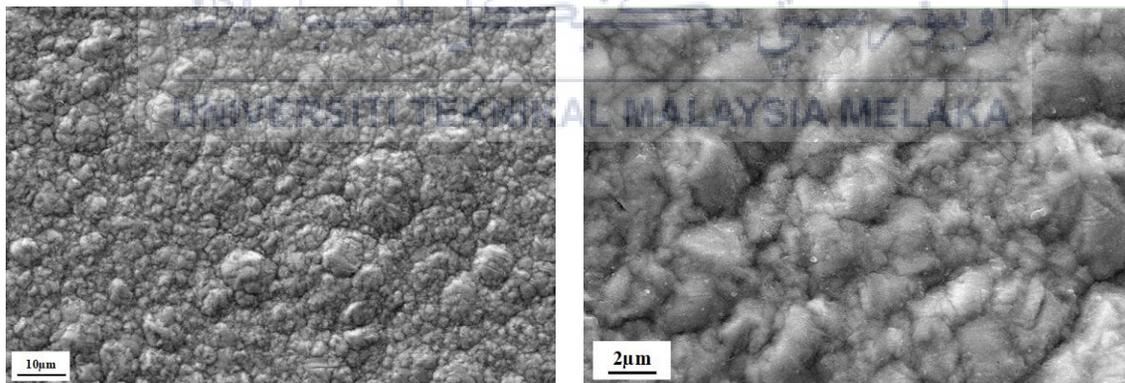
(b)



(c)



(d)



(e)

Figure 4.6: SEM micrographs of (a) Bare AA6061 (b) Pure Ni (c) Ni-QD 10 g/l
(d) Ni-QD 30 g/l (e) Ni-QD 50 g/l

4.3 Mechanical Testing of Ni-QD Composite Coatings

4.3.1 Micro-hardness

Micro-hardness studies showed that Ni-QD composite coating exhibit much higher hardness as compared to bare AA6061. The micro hardness of composite coating increased with increasing in quarry dust composition as shown in Figure 4.7. The improvement in hardness of these composite coatings can be attributed to the higher hardness of nickel and quarry dust. By increasing the quarry dust composition in Watt's bath solution, the microhardness of the composite coating increased due to an increased content of quarry dust incorporated to deposit. The micro-hardness of coating increased with the composition of quarry dust particles in deposit due the presence of hard SiO₂ and Al₂O₃ particles. Viet et al. (2013) found that by increasing the quarry dust concentration in arrangement, the microhardness of the composite coatings increased because of an expanded substance of QD incorporated to deposit

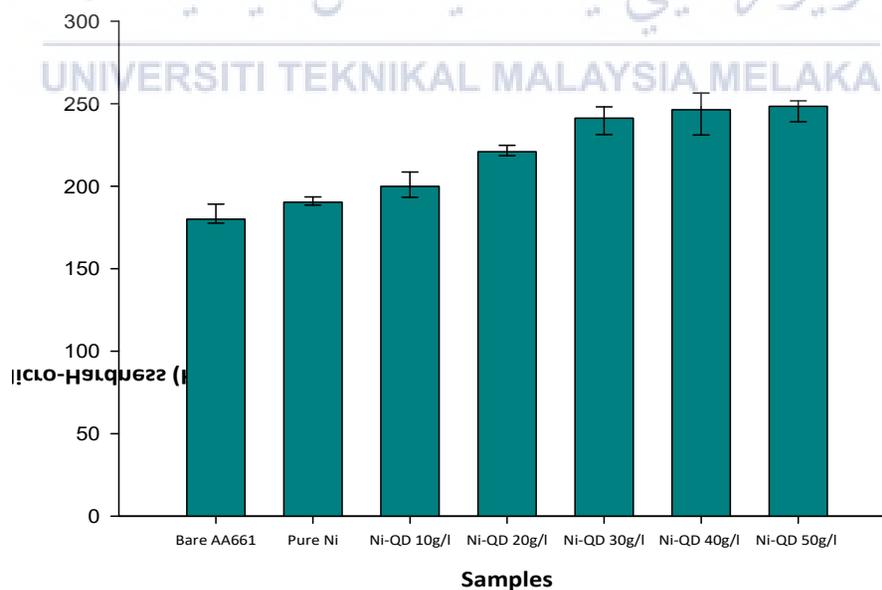


Figure 4.7 Micro-hardness value of Ni-QD composite coatings

4.3.2 Tribological Result

The effect of quarry dust composition on the coefficient of friction (COF) of composite coatings is shown in Figure 4.8. The figure shows the average value of COF with increasing quarry dust composition. It observed that the average of COF of Ni-QD coating is decreased as the composition of quarry dust increase. The average value of COF of bare AA6061 is 0.5694 while the lowest average value of COF of Ni-QD composite coatings is 0.34109 which the composition of quarry dust is 50g/l. Based on the data, by increasing the composition of quarry dust, can increase the wear resistance. Wear resistance of coatings increase due to the decreasing of coefficient of friction obtained.

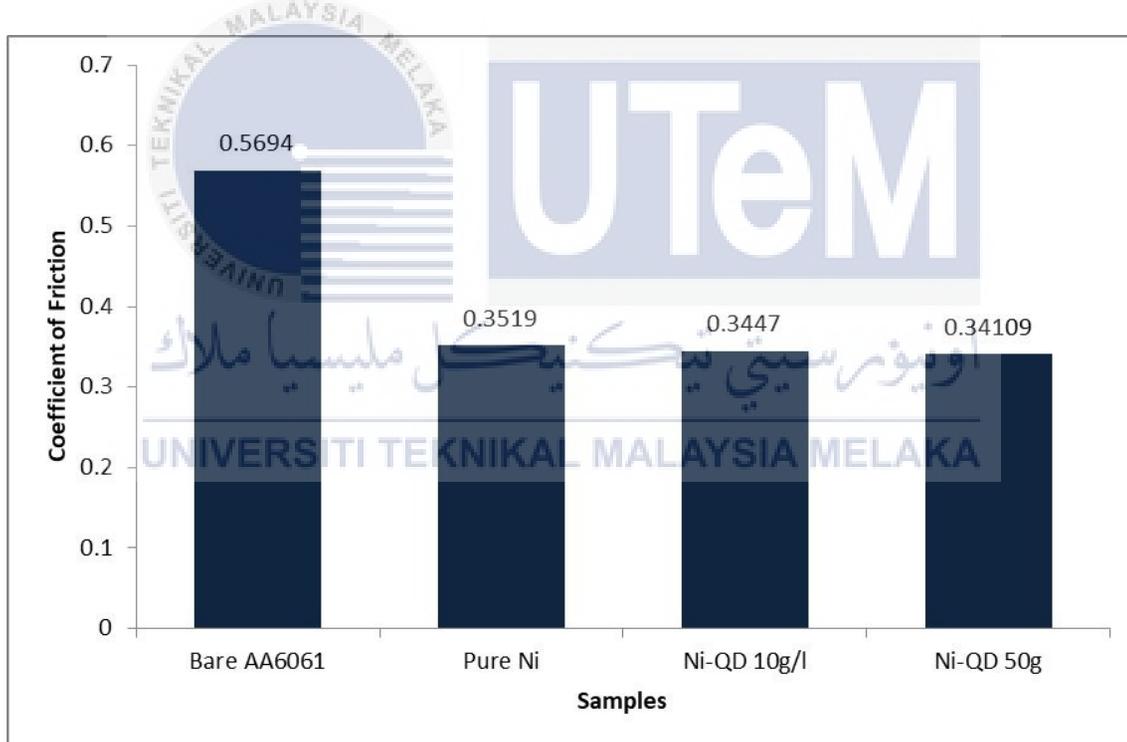


Figure 4.8 Average value of coefficient of friction

Figure 4.9 shows the weight loss of Ni-QD composite coatings samples after completed wear testing. Based on the bar chart below, it clearly shown the total weight loss of

samples decrease as the composition of quarry dust increase.

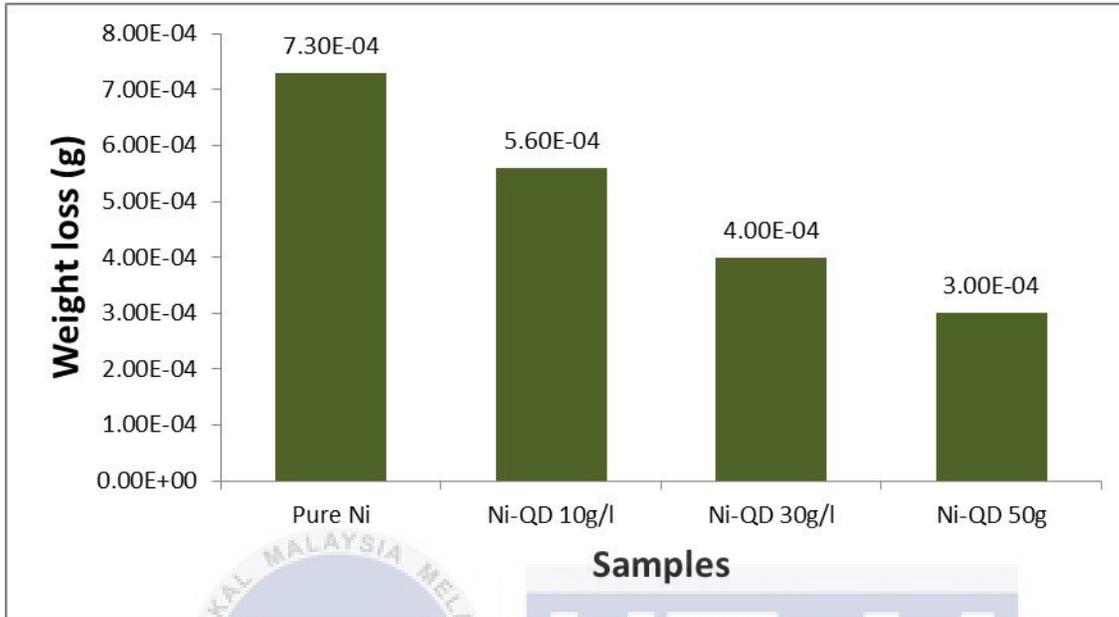
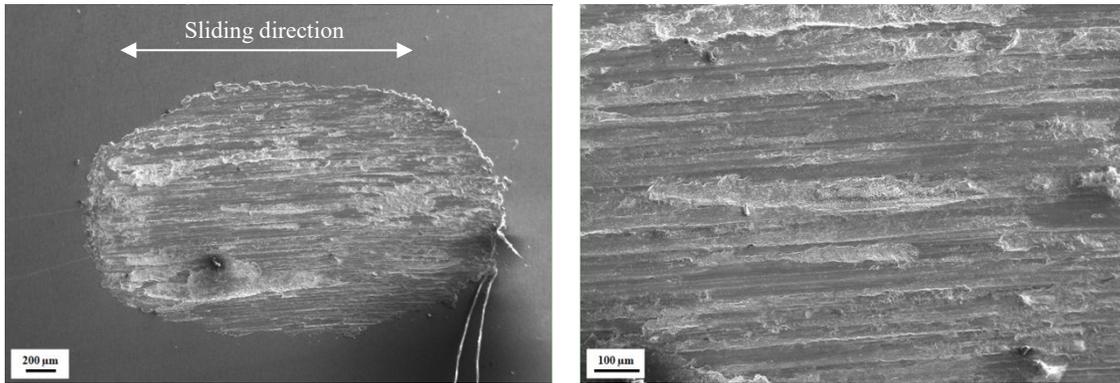


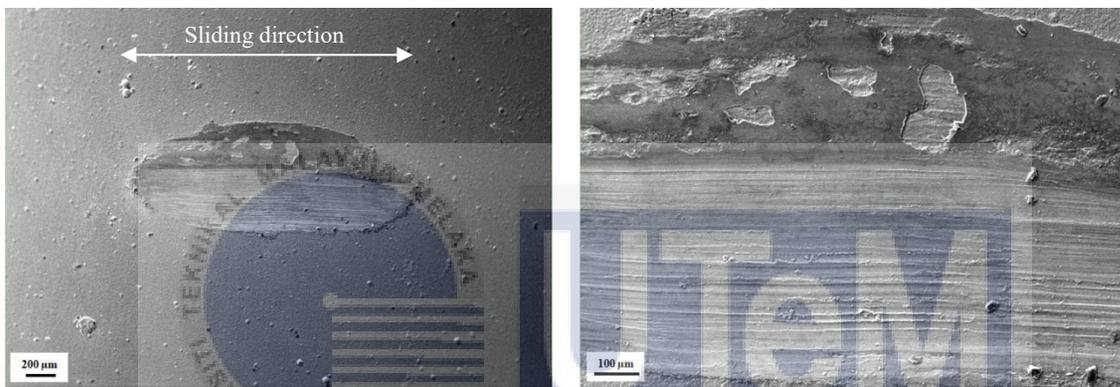
Figure 4.9 Value of weight loss after completed wear testing

The effect of increasing the composition of quarry dust on the wear track of composite coatings is shown in Figure 4.10. The figure demonstrates the sliding direction which is length 2cm. The width of wear track on Ni-QD coatings is diminished as the composition of quarry dust increase. The width on wear track of bare AA6061 is greater compare with others while.

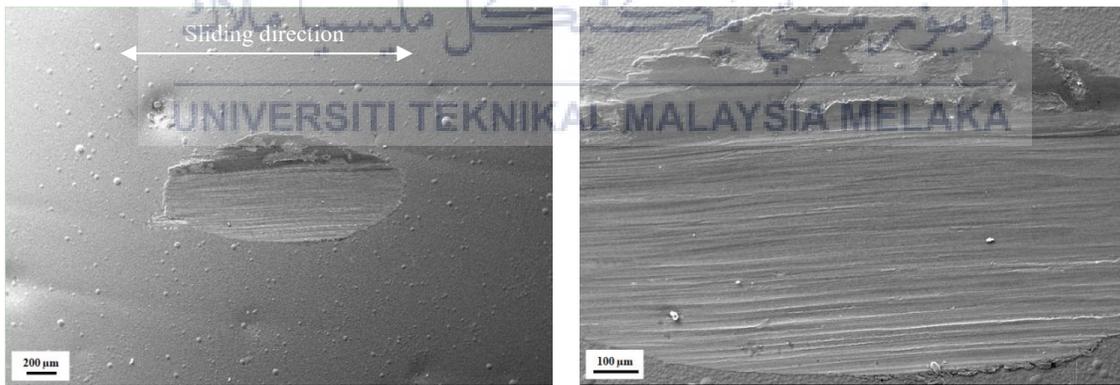
Ni-QD 30 g/l composite coatings exhibited the narrowest wear width and shallowest plough lines compared to other coatings. These findings indicated that nickel co-deposited with 30 g/l of quarry dust has improved the wear resistance of bare AA6061.



(a)



(b)



(c)

Figure 4.10: SEM image of wear scars (a) Bare AA6061 (b) Pure Ni (c) Ni-QD 30g/l

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

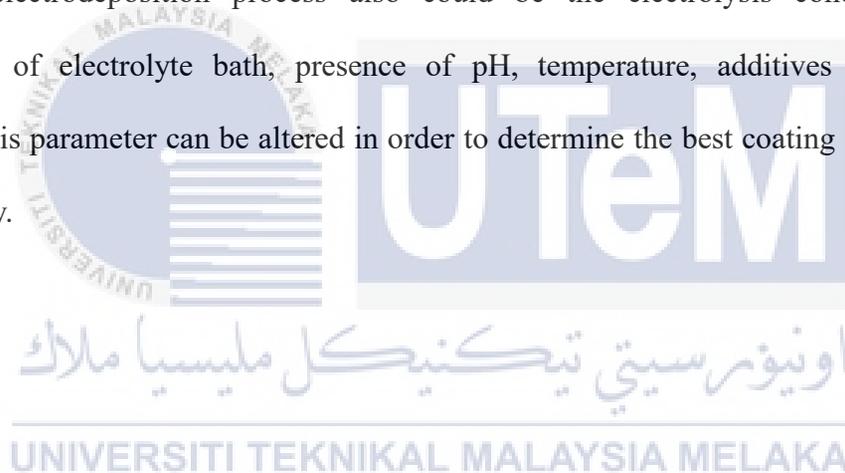
Ni-QD composite coating managed to be prepared by electrodeposition process. The composition of quarry dust has been set as manipulated variables and the effect to the properties of Ni-QD composite coating were analysed. Generally, quarry dust is consisted of many oxides and other ceramic compound. The investigation reveals the following conclusion:

1. The crystalline structures of Ni-QD became rough as the composition of quarry dust increase.
2. The hardness of Ni-QD composite coating increased by increasing the quarry dust composition. The hardness of bare AA6061 is 180.5HV compared to Ni-QD 50g/l, the hardness is 245.3HV.
3. Ni-QD also showed a good result in wear testing where the average of COF of Ni-QD composite coatings is lower than bare AA6061. COF of Ni-QD 50g/l is 0.34109 while COF of bare AA6061 is 0.5694.

In a nutshell, the experimental procedures have been successfully done to prepare Ni-QD composite coating on AA6061 substrate. The effect of quarry dust composition clearly enhanced the properties of electrodeposited Ni-QD composite coating on AA6061 substrate.

5.2 Recommendation

As the recommendation, research can be done by study the effect of heat treatment towards the Ni-QD composite coating and thus determining the optimum temperature that will give the best coating properties. The temperature at which the coating will be uniform and crack-free should be determined. Besides that, a study on the size of quarry dust can be made in order to identify whether it affects the mechanical and chemical properties. The size at which the coating will be homogeneous should be determined. Another parameter that would affect the electrodeposition process also could be the electrolysis condition such as, composition of electrolyte bath, presence of pH, temperature, additives and electrolyte agitation. This parameter can be altered in order to determine the best coating such as in terms of uniformity.



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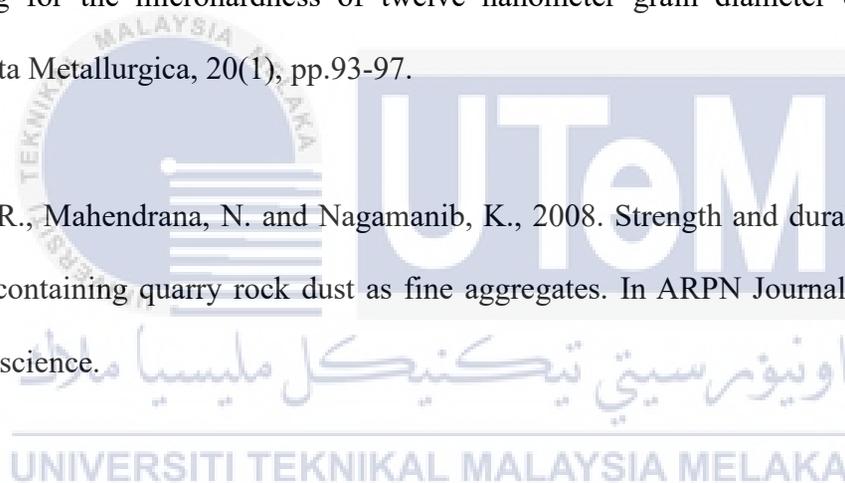
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