UNIVERSITI TEKNIKAL MALAYSIA MELAKA

# Faculty of Manufacturing Engineering

# EFFECT OF ELECTRODEPOSITION CURRENT DENSITY AND QUARRY DUST CONTENT ON THE CHARACTERISTICS AND TRIBOLOGICAL PROPERTIES OF Ni-RECYCLED QUARRY DUST COMPOSITE COATINGS



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**Master of Science in Manufacturing Engineering (Industrial Engineering)**

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# EFFECT OF ELECTRODEPOSITION CURRENT DENSITY AND QUARRY DUST CONTENT ON THE CHARACTERISTICS AND TRIBOLOGICAL PROPERTIES OF Ni-RECYCLED QUARRY DUST COMPOSITE COATINGS

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**A thesis submitted**
**in fulfillment of the requirements for the degree of Master of Science**
**in Manufacturing Engineering**



**Faculty of Manufacturing Engineering**

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**2022**

## DECLARATION

I declare that this thesis entitled "Effect of Electrodeposition Current Density and Quarry Dust Content on the Characteristics and Tribological Properties of Ni- Recycled Quarry Dust Composite Coatings" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.



Signature : ..........................................

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Date : 26/9/2022

## APPROVAL

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

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**DEDICATION**

To my beloved son, Muhammad Aqil Yusuf bin Abdullah Asyraf.

## ABSTRACT

The study experimentally investigated the effect of various current density and quarry dust content on the surface properties and tribological properties of electrodeposited nickel quarry dust composite coatings on High Speed Steel (HSS) substrate. HSS is widely used as a high speed cutting tool due to their excellent red hardness and good wear resistance. Quarry dust is used in this study as a reinforcement because of its high silica and alumina content, which helps to improve the coating's properties. In order to get finer size of quarry dust particles, quarry dust has undergo ball milling process before electrodeposition process. Various current density with range from 2 A/dm$^2$ to 8 A/dm$^2$ and various quarry dust content with range between 15 g/L to 60 g/L were used in this study as the different range of current density and quarry dust content have different outcome. The composite coatings was characterized using Scanning Electron Microscope (SEM) and X- Ray Diffraction (XRD). The influence of current density and quarry dust content was investigated using hardness test and wear test. As the current density and quarry dust content increases, the hardness of the substrate will increases too. The highest hardness value is obtained when current density is at 6 A/dm$^2$ and quarry dust content is 45 g//L. It is same as for the result of wear test as the wear track length is smaller and the wear occur on the surface. With an increase in current density and quarry dust content, the COF value decreased. Therefore, the optimum experiment's parameters are current density at 6 A/dm$^2$ and a quarry dust content at 45g/L.

**ABSTRAK**

Kajian secara eksperimen untuk mengkaji kesan pelbagai ketumpatan arus elektrik dan kandungan debu kuari pada sifat permukaan dan sifat tribologi saduran komposit nikel-debu kuari melalui proses elektrodiposisi ke atas keluli berkelajuan tinggi (HSS). HSS digunakan secara meluas sebagai alat pemotong berkelajuan tinggi kerana kekerasan merah yang sangat baik dan rintangan haus yang baik. Debu kuari digunakan dalam kajian ini kerana kandungan silika dan alumina yang tinggi, yang membantu meningkatkan sifat saduran. Untuk mendapatkan saiz debu kuari yang lebih halus, debu kuari telah melalui proses pengilingan bola sebelum proses elektrodeposisi. Ketumpatan arus elektrik dari 2 A/dm2 hingga 8 A/dm2 dan kandungan debu kuari diantara 15 g/L hingga 60 g/L digunakan dalam kajian ini kerana nilai yang berbeza mempunyai hasil yang berbeza. Hasil saduran komposit telah dicirikan menggunakan Pengimbas Mikroskop Elektron (SEM) dan difraksi sinar X (XRD). Perbezaan ketumpatan arus dan kandungan debu kuari telah dikaji menggunakan ujian kekerasan dan ujian kehausan. Apabila ketumpatan elektrik dan kandungan debu kuari meningkat, kekerasan substrat akan meningkat. Nilai kekerasan tertinggi diperoleh apabila ketumpatan arus elektrik adalah 6 A/dm2 dan kandungan debu kuari 45 g//L. Ia sama seperti keputusan ujian kehausan kerana panjang trek haus lebih kecil dan haus berlaku pada permukaan. Dengan peningkatan ketumpatan arus elektrik dan kandungan debu kuari, nilai COF menurun. Oleh itu, keadaan ideal eksperimen adalah pada ketumpatan arus 6 A/dm2 dan kandungan debu kuari 45g/L

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## LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| AMCHAL | - | Advanced Materials and Characterization Laboratoty |
| CMC | - | Ceramic Matrix Composites |
| CNT | - | Ni- carbon nanotubes |
| COF | - | Coefficient of Friction |
| DC | - | Direct Current |
| EDM | - | Electrical Discharge Machining |
| EPD | - | Electrophoretic deposition |
| FKM | - | Fakulti Kejuruteraan Mekanikal |
| HSS | - | High Speed Steel |
| HVOF | - | High Velocity Oxygrn Fuel Spray |
| MMCs | - | Metal Matrix Composite |
| PC | - | Pulse Current |
| PRC | - | Pulse Reverse Current |
| PSA | - | Particle Size Analyzers |
| QD | - | Quarry Dust |
| SEM | - | Scanning Electron Microscope |
| UTeM | - | Universiti Teknikal Malaysia Melaka |
| XRD | - | X-Ray Diffraction |

## LIST OF SYMBOLS

| | | |
|---|---|---|
| $A/dm^2$ | - | Ampere per square decimeter |
| Al | - | Aluminum |
| $Al_2O_3$ | - | Alumina |
| $Al_2O_3$-$SiO_2$ | - | Mullite |
| cm | - | Centimeter |
| $cm^2$ | - | Centimeter squared |
| C | - | Carbom |
| Co | - | Cobalt |
| Cr | - | Chromium |
| Fe | - | Ferum |
| g | - | Gram |
| g/L | - | Gram per liter |
| HCL | - | Hydrochloric acid |
| Hr | - | Hour |
| Min | - | minutes |
| N | - | newton |
| NaCl | - | Natrium Chloride |
| Ni | - | Nickel |
| rpm | - | revolution per minutes |
| Si | - | Silicon |
| SiC | - | Silicon Carbide |
| $SiO_2$ | - | Silicon Oxide |
| $\mu m$ | - | Micrometer |

# CHAPTER 1

# INTRODUCTION

This chapter explains the overview of background of study, problem statement, objectives, scopes of study and significance of study.

## 1.1 Background of Study

High-speed steels (HSS) are widely used in making high-speed cutting tools, which always require high hardness, good wear resistance, and good thermal fatigue resistance at elevated temperatures (Liu et al., 2021; Michalcová et al., 2021). This kind of wear-resistant and heat-resistant tool steel with secondary hardening characteristics contains a large amount of tungsten, molybdenum, vanadium, chromium, and other alloy elements (Michalcová et al., 2021). At present, HSS cutting tools continue to dominate the tool market (Shaojun et al., 2018). Despite the rapid development of tipped carbide cutting tools and the grinding of cemented carbide cutting tools, other types of cutting tools continue to primarily use HSS material (Shaojun et al., 2018).



Cutting tools are subjected to an extremely severe rubbing process (al Kindi et al., 2018). They are in metal-to-metal contact between the chip and the workpiece which when exposed to high stress and temperatures, causes wear and eventually results in cutting tool failure (Patil & Shinde, 2013). A coating process could be used to improve the properties of the cutting tool in terms of hardness and wear resistance, potentially extending the life of the cutting tool. To improve the mechanical properties of HSS as a cutting tool, an electrodeposition process can be introduced to the substrate by electrodeposition process.

Electrodeposition is one of the most technologically feasible and economically superior technique for producing metal matrix composite coatings (MMCs) (Borkar, 2010). MMCs often exhibit superior surface characteristics including corrosion resistance in a wide range of temperatures, improved physical, mechanical, and tribological properties. Nickel-based composite coatings reinforced with embedded particles (e.g., oxides, carbides, nitrides, and solid-state lubricants) are among the most studied MMCs because of their industrial applications as protective and corrosion-resistant coatings with desirable wear and friction properties (Sajjadnejad et al., 2021).

The properties of composite coatings are determined by the matrix phases as well as the amount and distribution of co-deposited particles within the matrix. Quarry dust was used as a reinforcement for the composite coating in this study. Quarry dust is one of the by-products from the crushing process during quarrying activities, which have gained attention to be used for various application (Kapgate & Satone, 2018; Othman et al., 2019; Zharif et al., 2021). Recently, the utilization of quarry dust which is high in silica and alumina contents have been extended to be used as a reinforcement for MMCs due to high cost of conventional ceramic particles. The quarry dust can be employed as inexpensive strengthening particles which can increase wear resistance and enhanced micro-hardness and have low density (Farhan et al., 2019).

Increasing current density will increase the incorporation of particles content in the composite (Farhan et al., 2019). The deposition of metal matrix with increasing current density is fast enough to entrap and occlude some of the particle and incorporate them into deposits (Farhan et al., 2019). Therefore, this study will investigate the influence of various current densities and various quarry dust content on the characteristics and tribological properties of composite coatings.

## 1.2 Problem Statement

In industrial production, there are many factors that can lead to the failure of machinery and equipment, such as wear and corrosion, which will cause waste of resources, environmental pollution and economic losses. HSS is commonly used as a cutting tool. HSS will wear out during the machining process due to friction with the workpiece. Surface treatment is one of the solutions to overcome this problem. Electrodeposition is widely used as a simple, effective and economical surface treatment technology. Electrodeposition parameters greatly influence the enhancement or decline of the mechanical properties and wear resistance of the fabricated coatings. On the other hand, the right parameter will lead to produce coatings containing well dispersed inert particles in metal matrix. Besides, it is also well established that the combination of multiple types of reinforcing secondary phases makes it possible to tune the properties of the fabricated coatings for the desired application, with a high level of flexibility. However, a limited study has been carried out on nickel reinforced with natural resource by product and effect of electrodeposition parameter to the composite coatings. Quarry dust is one of the by- product from the crushing process during quarrying activities, which contain high percentage of ceramic particles, $SiO_2$ and $Al_2O_3$. The environmental concern is currently rising as one of the main issues that lead to dust pollution and environmental deterioration and by converting the quarry dust into utilizable raw materials for usable application will help to improve the environmental. Therefore, this study will be investigating the effect of various current density and various quarry dust content towards Ni-QD composites coatings.

## 1.3 Objectives

The objectives that have been identified as follow:

1. To study the effect of various current density and various quarry dust content on the surface properties of electrodeposited Ni-QD composite coating.
2. To investigate the effect of various current density and various quarry dust content on the tribological properties of the electrodeposited Ni-QD composite coating.

## 1.4 Scope of Study

The scope of this study is to investigate the influence of different current densities and quarry dust content on the characteristics and tribological properties of composite coatings on Nickel recycle quarry dust substrates. Nickel and HSS were chosen as anode and cathode in this study, respectively. The electrolyte was created by combining a fixed amount of nickel and a variety of quarry dust content. The coatings were electrodeposited on the substrate at various current densities ranging from 2-8 A $dm^2$ and quarry dust compositions ranging from 15-60 g/L. The quarry dust particles and coatings were characterized using scanning electron microscopy (SEM), X- Ray Diffraction (XRD) and a Particle Size Analyzer (PSA). The effect of current density and quarry dust content on the coatings were investigated using hardness test and wear test.

## 1.5 Significance of Study

The findings of this study will help to provide the information on mechanical characteristic and tribological properties of HSS after electrodeposited on nickel quarry dust composite coatings. During the process, various current densities and various quarry dust content were applied into the HSS. The hardness of the HSS can be determined using Vickers micro-hardness test while, the coefficient of friction can be obtained through the wear test.

# CHAPTER 2

# LITERATURE REVIEW

This chapter provides an overview of previous study regarding the electrodeposition process, composite coatings, high speed steel, current densities nickel watt's bath and quarry dust.

## 2.1 Electrodeposition Process

### 2.1.1 Introduction

Electrodeposition is a type of electrochemical process used to modify the surface structure (Mbugua et al., 2020) It is a process of coating a thin layer of one metal on top of a different metal to modify its surface properties, by donating electrons to the ions in a solution (Ubaidah Saidin et al., 2010). This process has several advantages over the other fabricating techniques which includes low cost, simplicity of operations, adaptability, flexibility, high production rate, and industrial applicability (Paul, 2020; Rashidi & Amadeh, 2008). According to Mbugua et al., (2020), surface finish and tribological properties of the coatings can be further improved by the addition of suitable agents and control of deposition operating conditions. Many researchers have studied the electrodeposition of composite coatings in order to develop and fabricate advanced surface coatings that can withstand physical, chemical, and mechanical deterioration (Aliofkhazraei et al., 2021) and have discovered that adding certain chemical agents to the electrolytic solution reduces particle agglomeration and increases particle incorporation into the matrix (Paul, 2020). Lelevic and Walsh (2019) found that electrodeposition of homogeneously dispersed second phase particles within the Ni-P matrix can enhance deposit properties and with the aid of thermal treatment, the hardness of coatings can be improved. Besides, Guo et al. (2008) in the study on influences of surfactants on electrodeposition of Ni- carbon nanotubes (CNTs) found that coatings with surfactants become more homogenous and increased hardness of the composite coatings and improved adherence of the coating onto the matrix.

### 2.1.2 Electrodeposition Parameters

The electrodeposition of metals consists of the reduction of metal ions from different electrolyte solutions on top of a surface to be coated. Figure 2.1 describes the electrodeposition process of the Ni/ diamond coatings under mechanical stirring. In this approach, diamond particle surface was positively charged by absorbing $Ni^{2+}$ ions and with aid of electric field and magnetic stirring, the $Ni^{2+}$ ions and the particles have been transported to and absorbed on the cathodic surface (Li et al. 2021) Then, $Ni^{2+}$ ions acquired electrons and were reduced to Ni atoms. Subsequently, the diamond particles were strongly wrapped by the formed Ni grains. Li et al. (2021) reported that this process is consistent with the Guflielmi model.

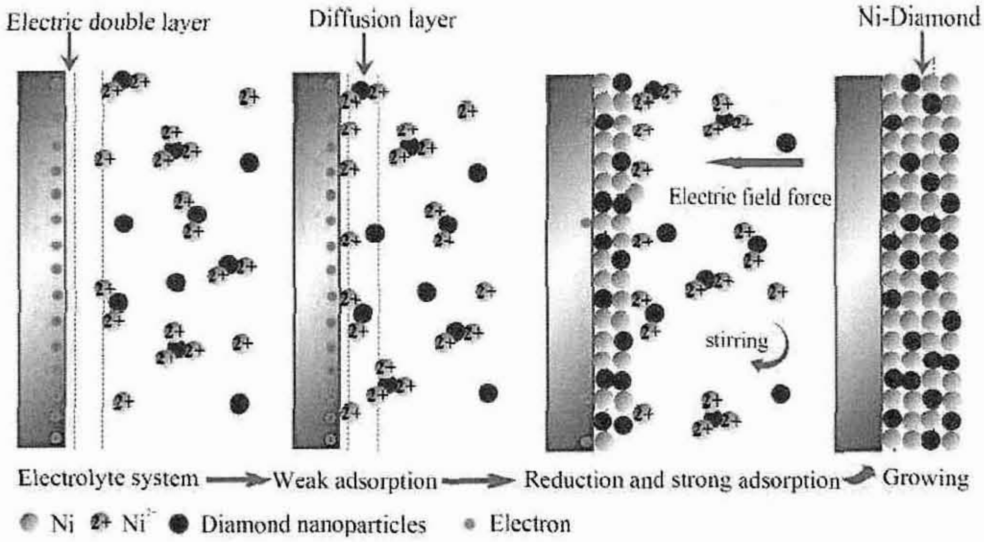



Figure 2.1 : Schematic diagram of electrodeposition process for the Ni/diamond coatings (Li et al., 2021)

**a) Direct Current (DC) electrodeposition**

According to Lelevic and Walsh (2019), constant direct current is the most commonly applied regime in which metallic coatings are deposited.In direct current (DC) electrodeposition, an electric current is continuously transferred through the system without any interruptions (Mbugua et al., 2020). Due to high deposition rates, the DC mode is used to make thick coatings with short deposition times. However, due to irregular composition, grain boundary mismatch, and other structural defects DC mode coatings are also prone to cracking (Paul, 2020). Mandati et al.(2018) highlighted that, apart from its disadvantages, DC electrodeposition remains a top method for the production of single element deposits and binary alloys.

**b) Pulse Current (PC) and Pulse Reverse Current (PRC) electrodeposition**

Pulsed electrodeposition, the deposition is carried out using current pulses of large current densities, the duration of which is of the order of a one to a few hundred milliseconds interspersed with pauses of the same or a higher duration(Wasekar et al., 2016). Nowadays more attention is given to PC and PRC methods because they use simple techniques and give more applications than the traditional DC method (Reddy et al., 2016). Compared to the conventional direct current (DC) electrodeposition, there is much more flexibility in the pulse electrodeposition technique in terms of varying basic electrodeposition parameters such as peak current density (ip), pulse current on-time (TON), and pulse current off-time (TOFF) resulting in unique combination of composition and microstructure in deposited coatings (Borkar, 2010). However, this technique is inefficient and time consuming as the current will "on" and "off" as it helps in ion redistribution in the solution while in the "off" time (Paul, 2020). Pulse reverse current electrodeposition (PRC) is another kind of pulse waveform where anodic pulses follow cathodic pulses(Joseph et al., 2021). The negative current density is proven to be effective in redistributing ions in the double layer and the bulk solution. Besides, this coating has been reported to have fewer pores, cracks, and lower internal stresses. (Paul, 2020). Pulse generators, on the other hand, are expensive than DC unit and this technique needs proper planning in advance with series of procedures in order to attain better results (Borkar, 2010). Figure 2.2 shows the summarization of waveform for different method of electrodeposition process.

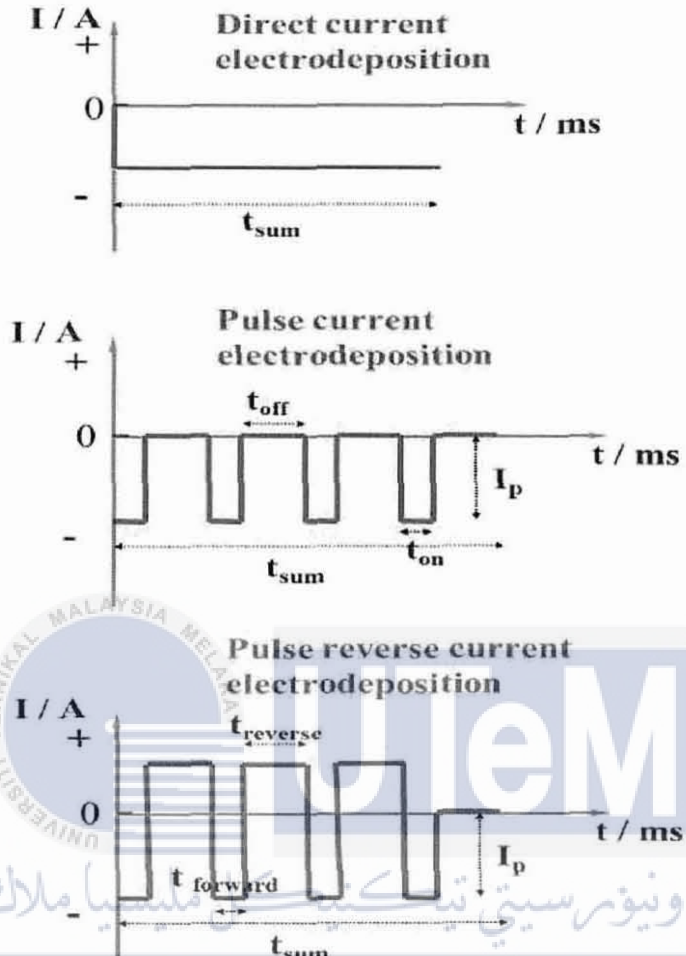





Figure 2.2: Three method of electrodeposition (DC, PC and PRC)

### 2.1.3 Effect of current densities in electrodeposition process

Particles reinforcement in the composite coatings varies with current density. Borkar (2007) in his study, highlighted that at first, incorporation increases sharply at the beginning with increase in current density till it reaches maximum value followed by sharp decrease Therefore, hardness of composite coatings increases due to the combined effect of both grain refining as well as of dispersive strengthening (Borkar 2007) This result is similar to those reported by Rashidi and Amadeh (2008) who writes the coating grains size decreased sharply by increasing the current density Experiment conducted by Wang et al. (2019) also support that the crytalline grain of electrodeposited layer gets fine and compact without pores and cracks as the current density increses. With the increase of current density, the cathode overpotential also increases leading to the improvement of the electrostatic attraction of the cathode to ions (Wang et al. 2019). Besides, Borkar (2007), reported that when electrodeposition at lower current densities, nickel ions dissolved from anode are transported at low rate and hence there is insufficient time for these ions to absorb on particles resuting weak Coulomb force between anions adsorbed on particles leading to lower concentration of electrodeposited particles in the composite coatings.Goral et al. (2015) and Wang et al. (2019) found that hardness of the coating was improved as the current density increased. Table 2.1 shows the summary of effect of current density in electrodeposition process.



Table 2.1: Effect of current density in electrodeposition process

| Author | Findings |
|---|---|
| (Rashidi & Amadeh, 2008) | An increase in current density resulted in a decrease in the average grain size of nickel coatings. |
| (Góral et al., 2015) | 1. The particles distribution was found to be much more uniform and more particles were incorporated in the coatings obtained at the higher values of current density.<br>2. The microhardness values of deposits were found to increase with the increasing of current |

| Author | Findings |
| --- | --- |
| | density.<br>3. The estimated residual stresses in the coatings were of the tensile character and their values have decreased at the higher current density.<br>4. The increase of current density has improved the polarization resistance value and decreased the corrosion current density. |
| (Othman et al., 2018) | As the current density increased the nickel grain size in the Ni-FA composite coating were decreased. The decreasing in nickel grain size resulted in increasing of hardness value. |



### 2.2 Electrodeposition of Composite Coatings

Co-deposition is process of incorporating fine particles of metallic, non-metallic compounds, or polymers from an electrolytic or an electroless bath in the electroplated layer to improve material properties such as hardness, wear resistance, corrosion resistance, lubrication, yield, tensile, and fracture strength (Borkar, 2010). The tailored corrosion protection, hydrophobicity, tailored electronic conduction, corrosion protection, wear resistance and tribological performance offered by electrodeposited composite coatings have resulted in diverse industrial applications in market sectors such as aerospace, automotive engineering, textiles and general engineering (Walsh et al., 2020). Many operating parameters influence the quantity of incorporated particles, including current density, bath agitation or movement of work piece and electrolyte composition. (Low et al., 2006). High incorporation rates of the dispersed particles have been achieved using a high nanoparticle concentration in the electrolyte solution, smaller sized nanoparticles; a low concentration of electroactive species, ultrasonication during deposition and pulsed current techniques (Low et al., 2006). On the basis of the matrix phase, composites can be classified into metal matrix composites (MMCs), ceramic matrix composites (CMCs), and polymer matrix composites (PMCs). The classifications according to types of reinforcement are particulate composites (composed of particles), fibrous composites (composed of fibers), and laminate composites (composed of laminates).

### 2.2.1 Types of composite coatings

**a) Ceramic Matrix Composite Coatings**

Ceramic matrix composites (CMCs) are a subgroup of composite materials as well as a subgroup of ceramics (Basutkar et al., 2015). They consist of ceramic fibres embedded in a ceramic matrix (Basutkar et al., 2015). The matrix and fibres can consist of any ceramic material, whereby carbon and carbon fibres can also be considered a ceramic material. Carbon (C), special silicon carbide (SiC), alumina ($Al_2O_3$) and mullite ($Al_2O_3$–$SiO_2$) fibres are most commonly used for CMCs (Kumar & Vasudev, 2019). The reinforcements used in ceramic matrix composites (CMC) serve to enhance the fracture toughness of the combined material system while still taking advantage of the inherent high strength and Young's modulus of the ceramic matrix (Kumar & Vasudev, 2019).

**b) Polymer Matrix Composite Coatings**

PMCs are composed of different types of organic polymers containing of continuous or short fibers with the different reinforcing agents (Sajan & Philip Selvaraj, 2021). This improves the properties such as stiffness, high strength and fracture toughness of composite materials (Sajan & Philip Selvaraj, 2021). In addition, these materials offer useful design flexibility and comparatively better fatigue and corrosion resistance than many other materials (Kangishwar et al., 2022). Thus recognized PMC as advanced composite materials due to their superior mechanical properties and comparative ease of fabrication. As a result, manufacturers have turned towards these advanced composites for a wide range of applications in variety of industries (Kangishwar et al., 2022; Sajan & Philip Selvaraj, 2021). A polymer matrix reinforced with natural fibers contains a good resistance and inter facial bonding between them helps to maintain their mechanical and chemical identities. In general, the fibers are carriers of charge, while the matrix keeps them in position at the desired orientation, it acts as a means of protects them from environmental damage and transferring the charge between the fibre (Sajan & Philip Selvaraj, 2021). The PMCs have a number of benefits, including low density, lower cost; less abrasive. According to the additives used, the changes could be made to the composite material's properties.

**c)Metal Matrix Composite Coatings**

A metal matrix composite system is generally designated simply by the metal alloy designation of the matrix and the material type, volume fraction, and form of the ceramic reinforcement (Fan & Njuguna, 2016). Electrodeposited metal matrix composite coatings (MMCs) often exhibit superior surface characteristics including corrosion resistance in a wide range of temperatures, improved physical, mechanical, and tribological properties (Sajjadnejad et al., 2021). According to Fan and Njuguna (2016), MMCs differ from other composite materials in several ways. Some of these general distinctions are as follows:

- The matrix phase of an MMC is either a pure or alloy metal as opposed to a polymer or ceramic.
- MMCs evidence higher ductility and toughness than ceramics or CMCs, although they have lower ductility and toughness than their respective unreinforced metal matrix alloys.
- The role of the reinforcement in MMCs is to increase strength and modulus as is the case with PMCs. Reinforcement in CMCs is generally to provide improved damage tolerance.
- MMCs have a temperature capability generally higher than polymers and PMCs but less than ceramics and CMCs.
- Low to moderately reinforced MMCs are formable by processes normally associated with unreinforced metals.

Nowadays, metal matrix composites are being comprehensively used in aero and automotive application because of their impressive properties improved strength, impact resistance, and tailored features (B M et al., 2020). The matrix of MMCs can be wide range of materials including Al, Fe, Mg, Ti, Ni, Be and Co as well and among those range of metal matrix the Ni based MMC coating is very interesting one as the surface modifed by Ni based MMC coating have very high hardness which results in better wear resistance and also shows a good corrosion resistance because of Ni is there (Karmakar et al., 2021).



### 2.2.2 Nickel composite coatings

Nickel electrodeposition is one of the most popular plating techniques and is widely used in industries (Guo et al., 2008). As example, Ni based coating is employed to enhance the surface quality of different engineering parts such as cutting tools, turbines blades, roller and plungers, rolls of rolling mills, extruders, piston head and rods and wearing plates (Karmakar et al., 2021). Nickel-based composite coatings reinforced with embedded particles (e.g., oxides, carbides, nitrides, and solid-state lubricants) are among the most studied MMCs because of their industrial applications as protective and corrosion-resistant coatings with desirable wear and friction properties (Sajjadnejad et al., 2021). Many researchers have investigated the effects of co-deposition of $SiO_2$, $Al_2O_3$, $TiO_2$, SiC and CNT into metal matrix and results showed that the composite coatings possess improved hardness, better wear and corrosion resistance than pure nickel coatings (Jabbar et al., 2017).

In general, many surface modification techniques can be engaged to produce Ni based composite coatings such as Gas tungsten arc cladding, Plasma spraying, Laser cladding, Thermal spraying, High velocity oxygen fuel spraying (HVOF), Electrodeposition, Electroplating, Electroless Plating, Electrophoretic deposition (EPD), Detonation spraying, Cold spraying, Hydrothermal deposition, and Pack cementation (Karmakar et al., 2021).



Recently, the utilization of quarry dust which is high in silica and alumina contents have been extended to be used as a reinforcement for MMCs due to high cost of conventional ceramic particles (Ramesh, 2014). The quarry dust can be employed as inexpensive strengthening particles which can increase wear resistance and enhanced micro-hardness and have low density.

Borkar (2010), in his study highlight that, second phase particles act as physical barrier to dislocation movement and grain boundary sliding resulting into significant improvement in mechanical properties of composite coatings thus nickel composite coatings containing dispersion of second phase particles, such as

$Al_2O_3$, $Si_3N_4$, SiC, $Cr_2O_3$, WC, diamond, PTFE, graphite, or even liquid containing microcapsules help to improve mechanical, tribological, and corrosion resistance properties of nano-composites.

## 2.3 High Speed Steel

High-speed steels (HSS) is a subset of tool steels and widely used in making high-speed cutting tools, which always require high hardness, good wear resistance, and good thermal fatigue resistance at elevated temperatures (Liu et al., 2021). S0ffritti et al. (2020) in the report highlighted these steels must withstand abrasive or adhesive wear and have sufficient hardness, toughness and ductility to prevent chipping, galling and cracking. Typically, HSS consist of carbon steel alloyed with tungsten or molybdenum, together with percentages of chromium, vanadium and cobalt. The M2 tool steel is a widely used material to manufactured cutting tools that have molybdenum additions as one of their primary alloying elements (D. Kumar et al., 2017). The primary utilization of high-speed steels keeps on being in the fabricate of different cutting apparatuses likes drills, taps, processing cutters, instrument bits, adapt cutters, saw edges, planer and jointer sharp edges, switch bits, and etc., in spite of the fact that use for punches and kicks the bucket is expanding (D. Kumar et al., 2017). The addition of reinforcements to the metal matrix helps to improve the mechanical properties of HSS. One method to improve the structure of HSS is through electrodeposition composite coating.

## 2.4 Nickel Watt's Bath

In the electroplating method, a Watts bath is generally used for various applications and products (Kamimoto et al., 2020). A Watts bath is an aqueous electrolyte solution for plating nickel from a nickel anode. Composite plating is a useful technique that can increase the hardness, abrasion resistance, and lubricity through composite formation of a metal coating and fine particles of materials such as silicon carbide, alumina, boron nitride, and polytetrafluoroethylene (Kamimoto et al., 2020). Nickel sulphate, nickel chloride and boric acid frequently used in Nickel watts bath solutions. However, due to environmental and health problem, boric acid can be replaced with others such as sodium citrate. The nickel sulphate provides the proper concentration of nickel ions. The nickel metal content determines the limiting current density for obtaining good deposits. The nickel chloride increases the anode corrosion and helps to produces harder deposits and increases bath conductivity which allows the electrodeposition process to use a lower voltage. Sodium citrate is used as an additive to help in refining the grain size of nickel deposit.

An experimental conducted by Kamimoto et al. (2020) used a Watts- type bath containing 45g/L nickel chloride hexahydrte, 240g/L nickel sulfate, and 30g/L boric acid as the electrolyte on improving the current efficiency of the electroplating process used to prepare nickel-carbon black composites. The current efficiency was greter than 90% during the electrodeposition process compared to Wood's bath. Magdy et al. (2012) conducted an experiment from a Watts bath in the presence of monosodium glutamate (MSG) as a complexing agent. MSG help in improving the appearance of nickel deposited from the Watts bath.



### 2.5 Quarry Dust

Quarry dust is one of the by- product from the crushing process during quarrying activities, which has gained attention to be used for various applications, such as construction industry and manufacturing of building material industry, due to the high percentage of ceramic particles, $SiO_2$ and $Al_2O_3$ (Farhan et al., 2019). About 20 to 25 per cent of the total production in each crusher unit is left out as the waste material (Aleem et al., 2014). Hamid et al. (2018) has defined quarry dust as residue, tailing or other non-voluble waste material after the extraction and processing of rocks to form fine particles less than 4.75mm.

The quarry dust consists of excess fines and is dumped in open fields that cause environmental problem and is commercially unused material (Hamid et al., 2018). Ferronato and Torretta (2019) reported that due to the scarcity of disposal land near industries and growing environmental concerns, dust disposal has become extremely expensive. As a result, it is preferable to obtain environmentally friendly substitutes for by-products, preferably quarry dust, in order to preserve the environment.

A study on quarry dust have been conducted by Hamid et al. (2018) and Othman et al. (2018), and listed its physical and chemical characteristics. The physical and chemical characteristics of quarry dust are presented in Tables 2.2 and 2.3, respectively.

Table 2.2: Physical properties of Quarry Dust (Hamid et al., 2018)



| Property | Quarry Dust |
|---|---|
| Specific gravity | 2.54-2.60 |
| Bulk density ($Kg/m^3$) | 1720-1810 |
| Absorption (%) | 1.20-1.50 |
| Fine particles less than 0.075mm (%) | 12-15 |

Table 2.3: Composition of quarry dust particles (Othman et al., 2018)

| **Element** | **Concentration (Wt %)** |
|---|---|
| $SiO_2$ | 72.6 |
| $Al_2O_3$ | 15.1 |
| $Fe_2O_3$ | 1.9 |
| CaO | 1.1 |
| MgO | 0.8 |
| $Na_2O$ | 3.0 |
| $K_2O$ | 4.9 |
| $SO_3$ | 0.2 |
| $TiO_2$ | 0.3 |
| $P_2O_5$ | 0.1 |

### 2.5.1 Effect of quarry dust composition in electrodeposition process

They are limited research had done studies on quarry dust to be used as an environmentally friendly coating material for surface modification. Most of the research conducted were for replacement material in construction industry ( Aleem et al. (2014), Srinivasan et al. (2014), Tanajirao et al. (2018), Kadir et al. (2018), Jeni and Bakar (2021) and Chitkeshwar (2022))



Othman et al. (2018) and Othman et al. (2019) reported that, as the quarry dust content increases, the hardness and wear resistance also improved, due to the presence of high silica and alumina content in the quarry dust particles. This result supported by Farhan et al. (2019) when Ni-P QD with heat treatment at 200°C shows that successfully improved the wear resistance compared to other composite coatings. Besides, Vickers micro-hardness of the coated surface was improved significantly with the addition of quarry dust Yap et al. (2020).

# CHAPTER 3

# METHODOLOGY

This chapter discussed the method used in the study including the sample preparation, electrodeposition process, method for determining the surface morphology of Ni- QD composite coatings, as well as test on the wear behaviour and hardness of Ni-QD composite coatings on high speed steel substrates.

## 3.1 Methodology

The experiment will begin with preparing the HSS substrates. The HSS will be machined to small rectangular piece and then will be ground using silicon carbide (SiC) papers. Then, the substrate will be chemically surface pre- treated with acetone and ethanol and rinse with distilled water. The purpose of chemical surface pre-treatment is to remove oxide and oil on the HSS substrate in order to improve bath wettability. Next, HSS substrate will be electrodeposited with Ni- QD composite coatings at various current densities and various quarry dust content. Later, all samples will be characterized and tested to know the properties of Ni-QD composite coating deposited. The details of the experiment procedures are illustrated in flowchart in Figure 3.1.

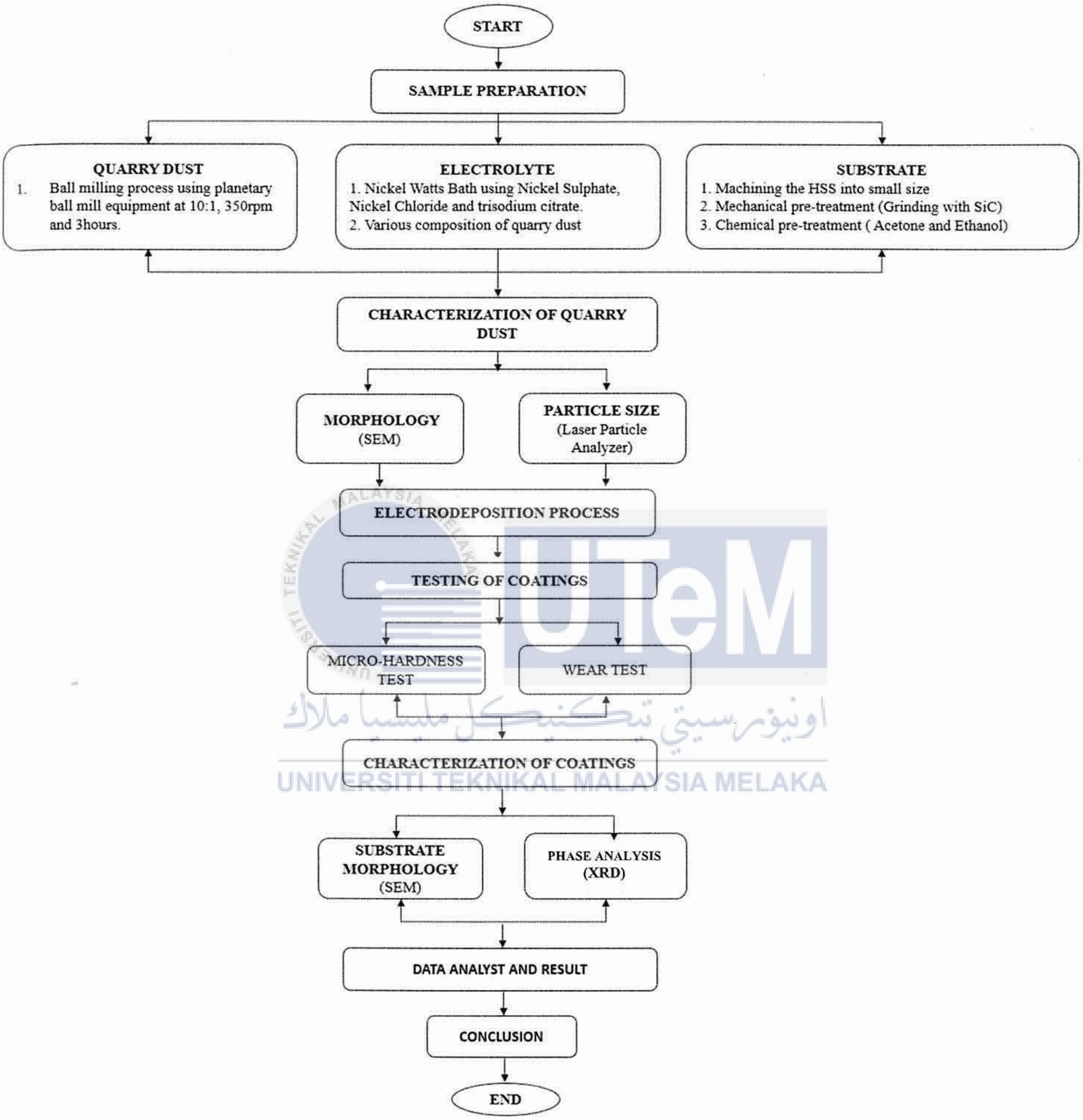



Figure 3.1: Experimental procedure flowchart

## 3.2 Preparation of the Quarry Dust

The quarry dust was obtained from a quarry at Negeri Sembilan. The as- received quarry dust was milled for 3 hours at speed 350 rpm using planetary ball mill equipment as per Figure 3.2. The ball to powder ration is 10:1. The morphology of the quarry dust was determined by using Scanning Electron Microscopy (SEM). The particle size was of the quarry dust was determined by using the particle size analyser (PSA).

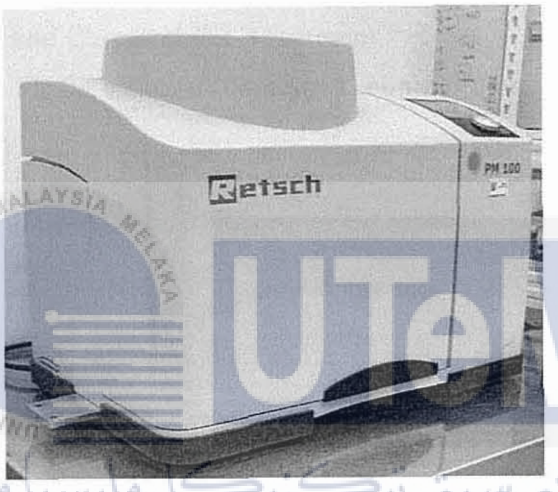



Figure 3.2: Planetary Ball Mill Machine



## 3.3 Preparation of the High-Speed Steel (HSS)

### 3.3.1 Sample Cutting

High speed steel (HSS) is a substrate that been used in this study. The HSS will be machined using Electro discharge machining (EDM) wire cut machine to small rectangular piece with size of 40 mm x 30 mm x 3 mm as per Figure 3.3.

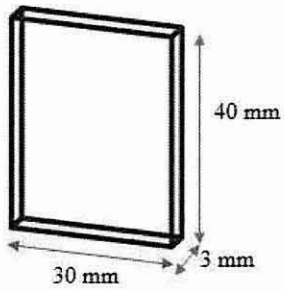



Figure 3.3: Dimension of HSS

### 3.3.2 Mechanical and Chemical Pre-Treatment Process

Surface preparation is the essential first stage treatment of a steel substrate before the application of any coating, and performance of a coating is significantly influenced by its ability to adhere properly to the substrate material. With the aid of a grinding machine as shown in Figure 3.4, the substrates will be ground using abrasive papers of 240, 400, 600, 800, and 1200 grit in order to remove oxides. Figure 3.4 shows the Silicon Carbide paper for the grinding process. The substrates were cleaned in acetone and ethanol and then rinsed with distilled water to eliminate grease.


Figure 3.4: Grinding machine



Figure 3.5: Silicon Carbide paper

**3.4 Nickel Watts Bath**

The electrolyte used for the electrodeposition of Ni-QD comprises of 200 g/l Nickel Sulphate (($NiSO_46H_2O$), 20 g/l nickel chloride ($NiCl_26H_2O$), 30g/l trisodium citrate ($Na_3C_6H_5O_7$) and various composition of quarry dust range from 15g/L, 30g/L, 45g/L and 60g/L.

## 3.5 Electrodeposition Process

A piece of well cleaned HSS substrate will be used as cathode while nickel electrode as anode. The electrodeposition will be carried out with direct current (DC) power. The process parameter of electrodeposition is shown in Table 3.1 while Figure 3.6 shown the diagram of the electrodeposition process. Magnetic stirring at 2 rpm will be used to maintain the homogeneity of the dispersed micro particles. The parameter matrix for the sample as in Table 3.2.

Table 3.1: Electrodeposition parameter condition.

| Operating Condition | |
|---|---|
| Temperature (°C) | 40 |
| Deposition Time (minutes) | 60 |
| Current Density ($A/dm^{-2}$) | 2, 4, 6, 8 |
| Quarry Dust (g/L) | 15, 30, 45, 60 |



Table 3.2 Experiment Parameter Matrix

| Current density ($A/dm^{-2}$) / Quarry dust content (g/L) | 2 | 4 | 6 | 8 |
|---|---|---|---|---|
| 15 | Sample 1 | Sample 2 | Sample 3 | Sample 4 |
| 30 | Sample 5 | Sample 6 | Sample 7 | Sample 8 |
| 45 | Sample 9 | Sample 10 | Sample 11 | Sample 12 |
| 60 | Sample 13 | Sample 14 | Sample 15 | Sample 16 |

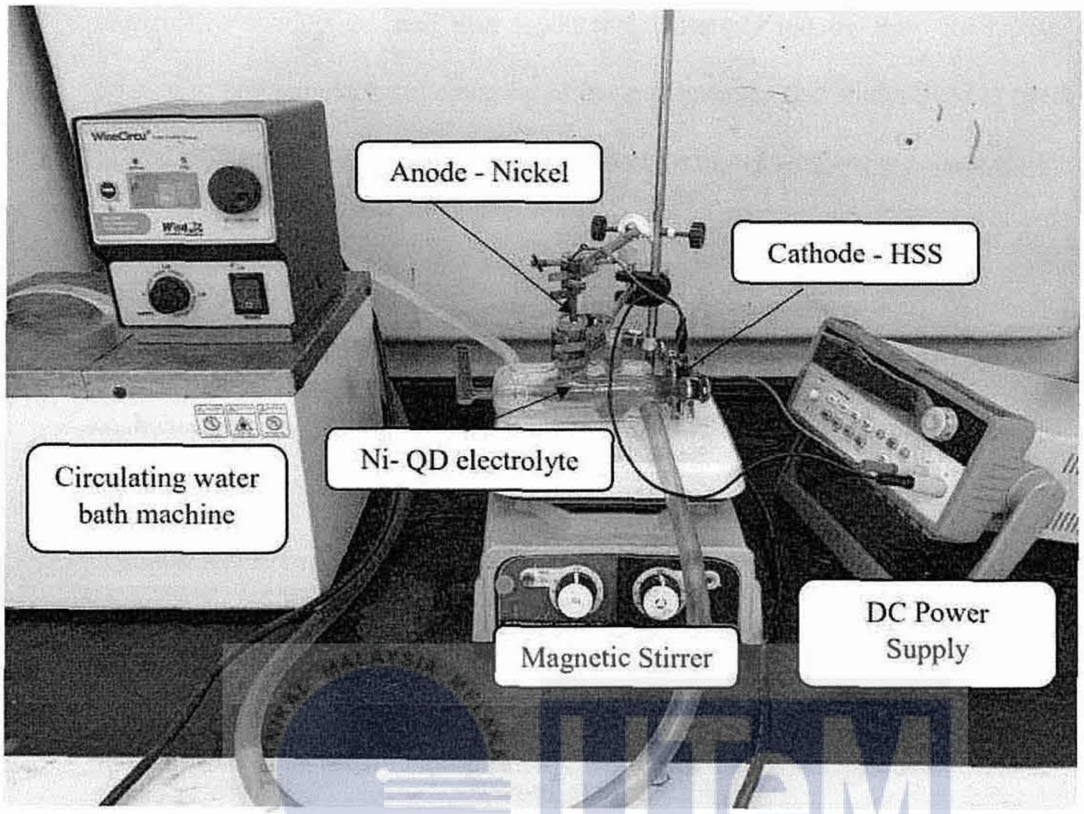



Figure 3.6: Diagram of electrodeposition process



### 3.6 Material Characterization

The morphologies of Ni-QD composite coating on HSS substrate were examined through SEM, XRD and particle size distribution of quarry dust by Particle Size Analyser.

#### 3.6.1 Scanning Electron Microscopy (SEM)

Scanning electron microscope (SEM) is one of the most widely used techniques in characterization of nanomaterials and nanostructures. The signals that derive from electron-sample interactions reveal information about the sample including surface morphology and chemical composition of the sample. Sample must be electrically conductive, and non-conductive materials require thin conductive coating to prevent electrical charging of the specimen. In this study, SEM is used to analyse the effect of Ni-QD towards the surface morphology of the coating.

### 3.6.2 X-ray Diffraction

X-ray diffraction (XRD) is a powerful non-destructive technique for characterizing crystalline materials. It offers details on crystal textures, preferred orientations for crystals, and other structural factors like average grain size, crystallinity, strain, and crystal defects. X-ray diffraction peaks are produced by constructive interference of a monochromatic beam of X-rays scattered at specific angles from each set of lattice planes in a sample. The peak intensities are determined by the distribution of atoms within the lattice. Consequently, the X-ray diffraction pattern is the fingerprint of periodic atomic arrangements in a given material (Bunaciu et al., 2015). In this study, XRD is use to identify grain size and element of crystal structure in the substrate. The crystallite size was calculated using Scherrer's formula.



### 3.6.3 Particle Size Analyzers

Laser diffraction particle size analyzers are used to measure the sizes of particles in both powder and liquid conditions. By measuring the angle of light scattered as the particles move through a laser beam, the size of the particle is determined. Laser diffraction particle size analyzers are frequently used in industrial settings because this method can continuously measure particle sizes across a broad range, from 10 nm to 5 mm. in this study, laser particle analyzer are used to measure the particle size of quarry dust before and after milling process.

### 3.7 Mechanical Testing

After the characterization analysis, the samples will be tested to determine the electrodeposition coating's wear and hardness.

#### 3.7.1 Wear test

A Micro Pin On Disk Tribo Tester machine was used to analysed the wear behaviour of the coating. The machine will reciprocate a 10mm stainless steel ball against the sample while applying a constant load of 10N. The sliding time is 1500 seconds, the set stroke is 2.69mm, and the velocity is 5mm/s. The wear behaviour of the coating will be investigated by analysing the coefficient of friction (COF) generated by the ball's reciprocating sliding against the coating layer. Figure 3.7 shows the Schematic diagram of Wear Tester and Figure 3.8 shows the sliding direction of the machine.



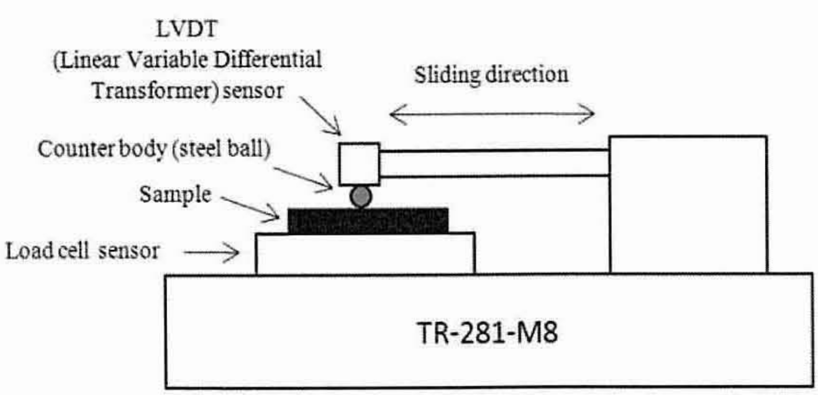



Figure 3.7: Schematic diagram of Wear Tester (Othman et al., 2019).

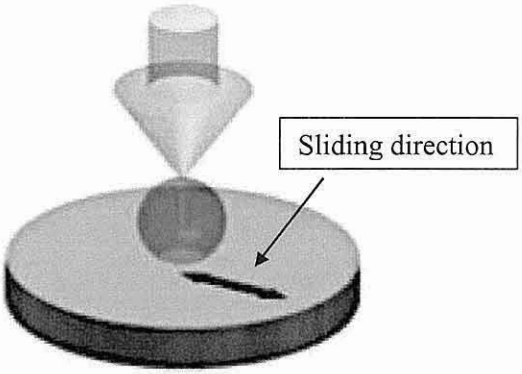



Figure 3.8: Sliding direction (Paul, 2020)

### 3.7.2 Hardness test

The hardness of coatings was measured using a microhardness tester by performing Vickers indentation at a loading force of 50g and a holding time of 15 seconds. A total of five indentations were obtained for each test. The test was conducted at AMCHAL, FKM, UTeM. Figure 3.9 shows the Micro Vickers Hardness Tester. The test used a Vickers indenter pressed into a surface to a specified force. Figure 3.10 shows the schematic of a Vickers indentation probe. After the indentation is finished, the resulting indent is analysed by measuring the dimension of the diamond shape to determine the hardness of the coating material. Figure 3.11 shows the schematic of Vickers indentation.

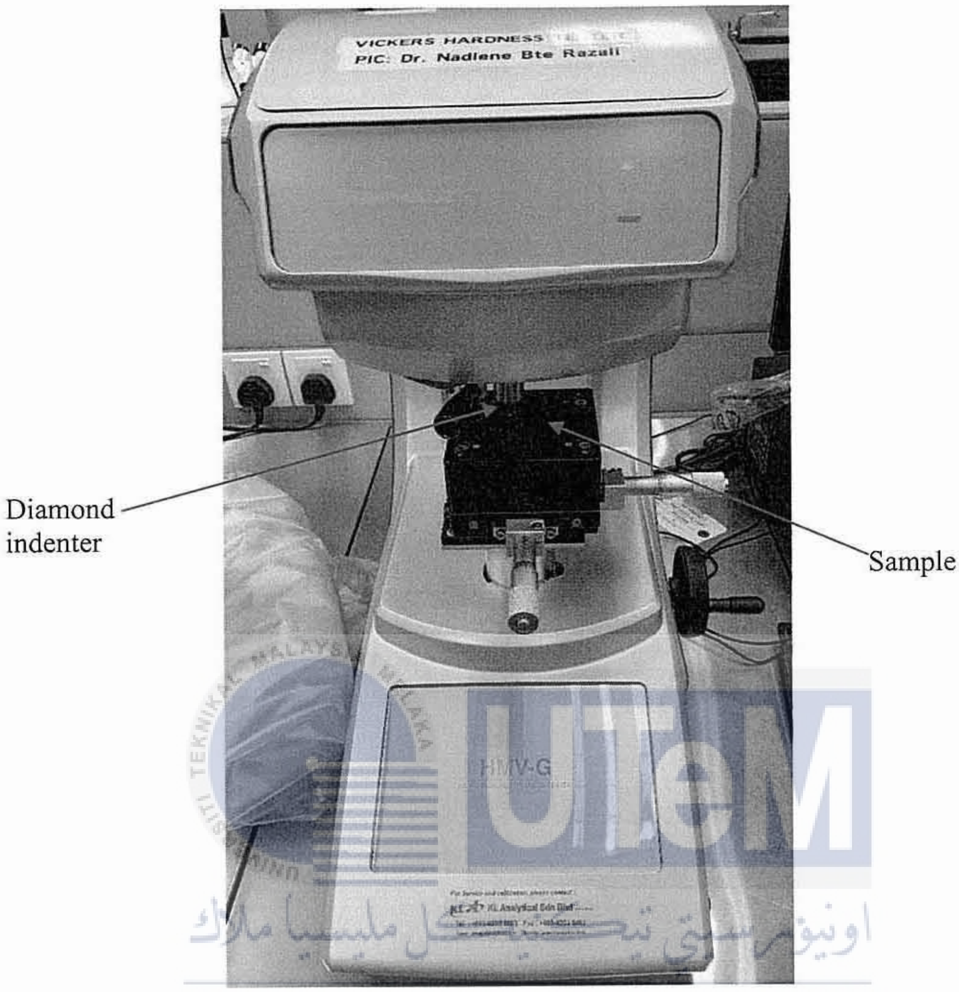





Figure 3.9: Micro Vickers Hardness Tester

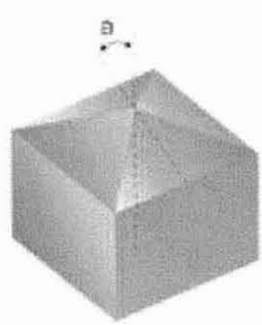



Figure 3.10: Schematic of a Vickers indentation probe.

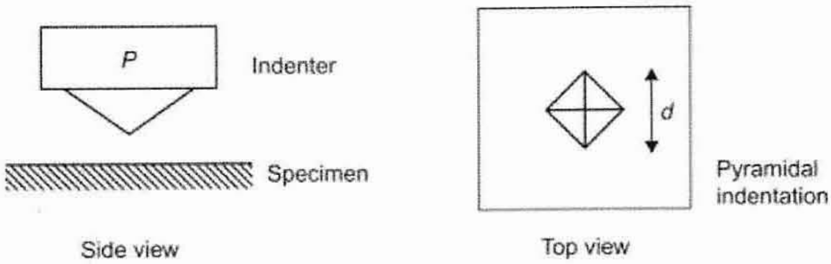



Figure 3.11: Schematic of Vickers indentation.

# CHAPTER 4

# RESULTS AND DISCUSSION

This chapter presents findings of the study followed by a discussion of the findings. The objectives of the study are to study the effect of various current density and various quarry dust content on the surface morphology, hardness and wear resistance of the electrodeposited Ni-QD composite coating. PSA, SEM, XRD was used to characterize the coatings while micro vickers hardness tester and micro pin on disk tribo tester machine respectively for hardness and wear test.

## 4.1 Characterization of Quarry Dust Particles

The particle size of quarry dust was measured in powder form using a particle size analyser (PSA). After a three-hour ball milling process, the particle size was reduced from 362.148μm to 15.157μm. Based on the trend shown in Figure 4.1 (a) and (b), as received quarry dust has size range from 0.810μm to 362.148μm while after milled the quarry dust particles for 3 hours the size become smaller with range from 1.684μm to 15.157μm.



Due to the collision between the balls and the quarry dust particles, the particle size of the quarry dust becomes finer after the ball mill process. It is consistent with the research conducted by Othman et al. (2019) as the results obtained indicate that the quarry dust particle size is influenced by increasing the ball milling duration. The graph of particle size distribution at various quarry dust particles as received and after the ball milling process is shown in Figure 4.1(a) and Figure 4.1 (b) respectively. Figure 4.2 shows the graph comparison of particle size distribution at various quarry dust particles as received and after ball milling process.

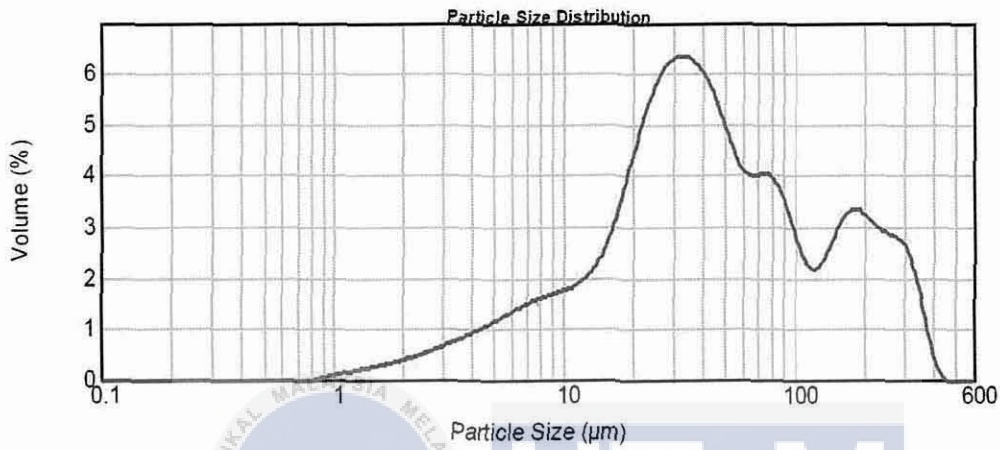



Figure 4.1 (a): Particle size distribution at various quarry dust particles as received.



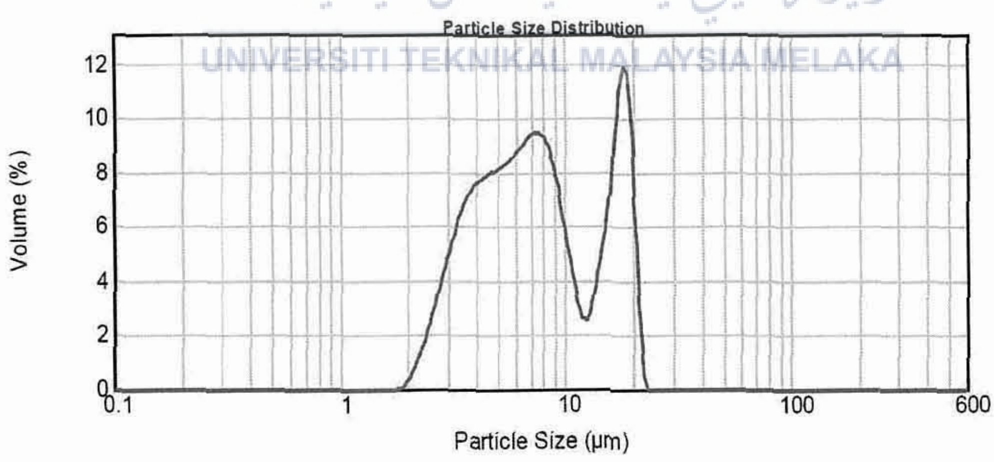



Figure 4.1 (b): Particle size distribution at various quarry dust particles after ball milling process.

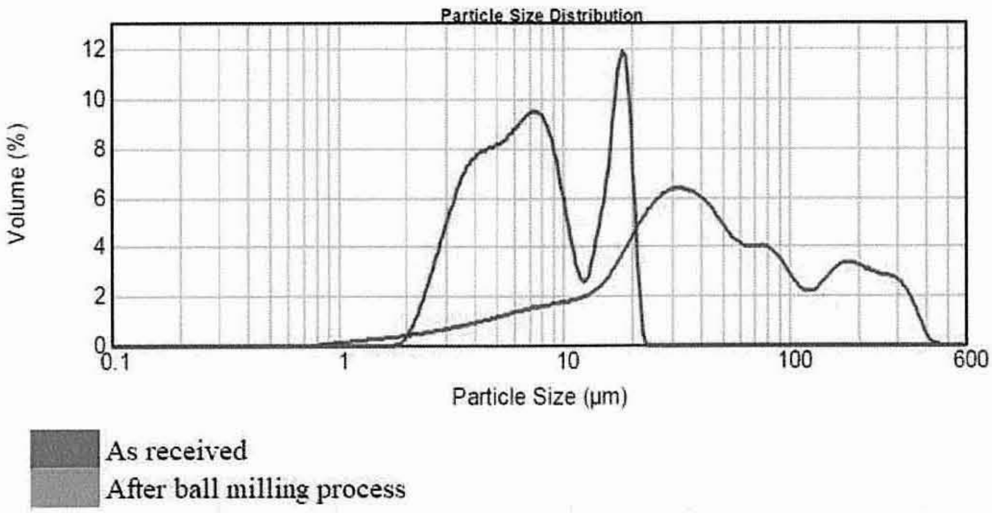



Figure 4.2: Comparison of particle size distribution at various quarry dust particles as received and after ball milling process.

SEM was used to investigate the morphology of quarry dust. Figure 4.3 shows quarry dust particles observed through SEM (a) as received and (b) after 3 hours of ball milling. It can be seen that the quarry dust particles were mostly irregular in shape and size. The size of quarry dust become finer after the ball milling process compared to as received.


Figure 4.3 (a): Quarry dust particles observed through SEM as received.



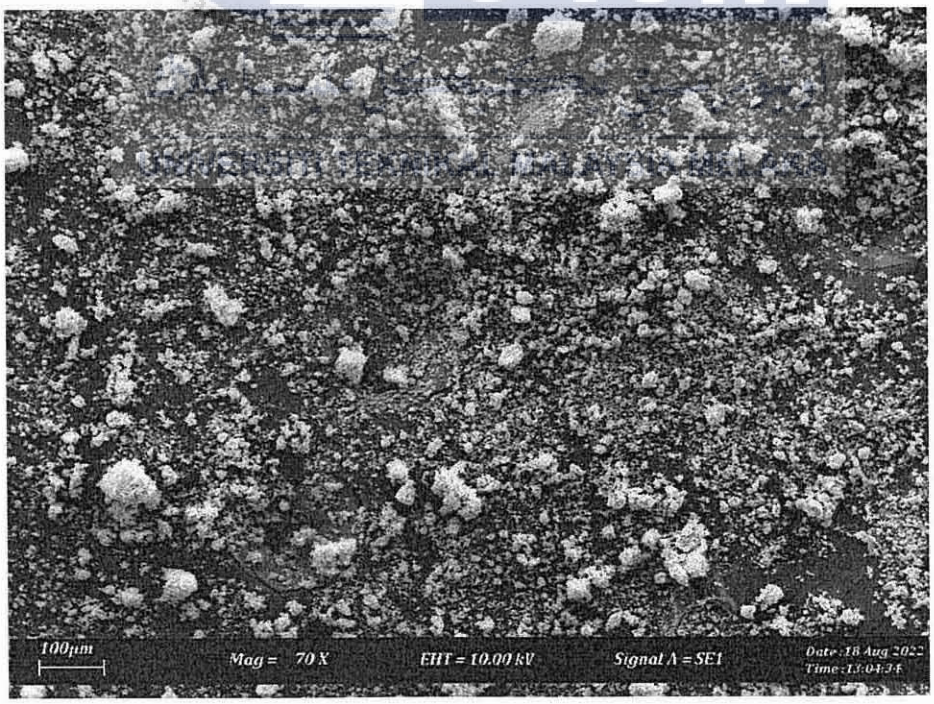



Figure 4.3 (b): Quarry dust particles observed through SEM after ball milling process.

## 4.2 Characterization of Ni-QD Composite Coatings

Ni-QD composite coatings were electrodeposited on HSS substrate at various current densities and various quarry dust content. The exposed area of nickel to the coating is 3.14 $cm^2$. Figure 4.4 show the electrodeposited of Ni-QD composite coatings on HSS with various current densities and various quarry dust content.

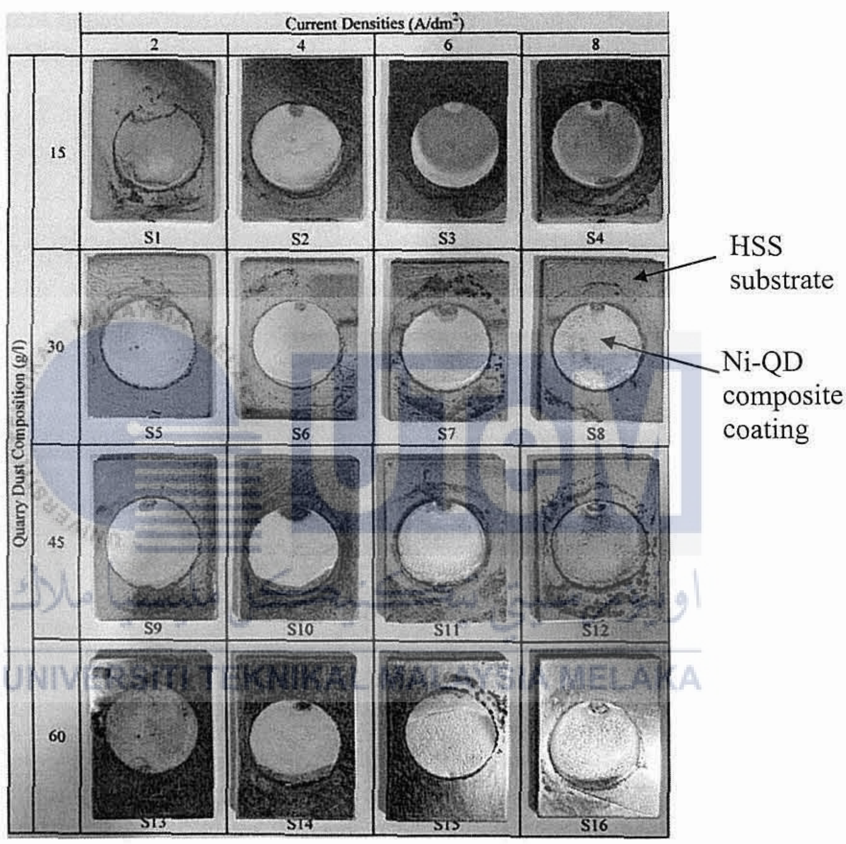



Figure 4.4: Electrodeposited of Ni-QD composite coatings on HSS with various current densities and various quarry dust content.

### 4.2.1 Phase Analysis of Ni- QD Composite Coating

XRD was used to examine the phase analysis of nickel composite coatings. XRD aided in determining the presence of Ni-QD composite coatings on the surface of the HSS substrate. The high intensity of Ni peaks in the XRD pattern makes it difficult to identify small peaks referring to quarry dust particles in the coating. However, the main elements in quarry dust, silica ($SiO_2$) and alumina ($Al_2O_3$) can still be found in each sample. As a result, the presence of quarry dust on the coating was confirmed. Figure 4.5 (a) and (b) shows the intensity of the elements when the current densities and composition of quarry dust increased.

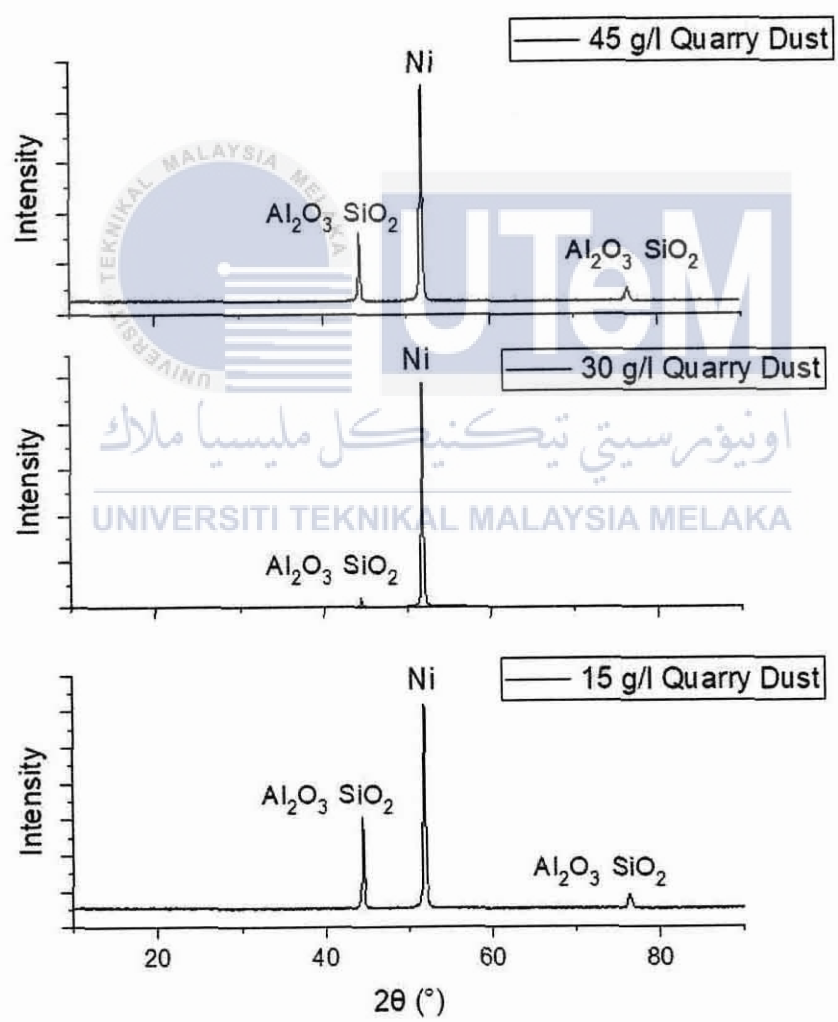



Figure 4.5 (a): XRD phase analysis of various quarry dust content applied with 6 A/dm$^2$ current density.

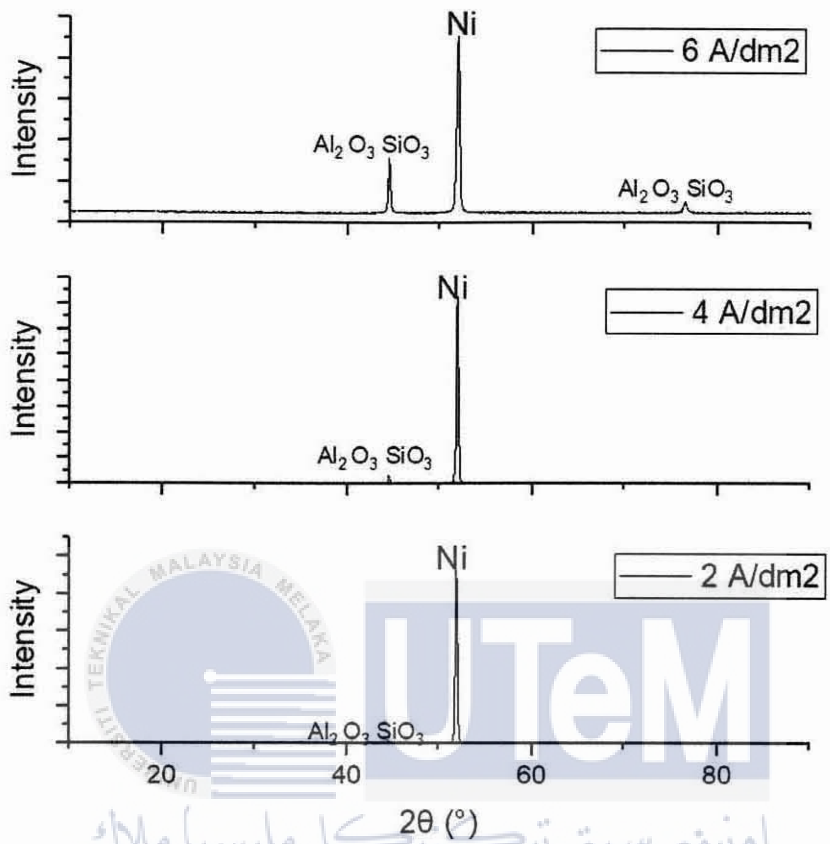





Figure 4.5 (b): XRD phase analysis of various current densities applied with 45g/l quarry dust content.

The crystalized size was studied using Scherrer's equation, $dp=K\lambda/\beta.\cos\theta$ from XRD pattern. From the equation, k is constant (0.94), λ is the wave number of the Cu Ka, 1.5405A, Θ(rad) is the scattering angle and β is FWHM (rad) which is the half way mark of the peak analysed. Table 4.1 (a) shows the crystallite size for the sample applied with various current density range from 2A/dm$^2$ to 6A/dm$^2$ with 45 g/L quarry dust while table 4.1 (b) shows the crystallite size for the sample applied with various quarry dust content at 6 A/dm$^2$ current density.

Table 4.1 (a): Crystallite size for 45 g/L quarry dust sample.

| Current density ($A/dm^2$) | Crystallite size (nm) |
|---|---|
| 2 | 12.8 |
| 4 | 13.31 |
| 6 | 13.07 |

Table 4.1 (b): Crystallite size for 6 $A/dm^2$ current density sample.

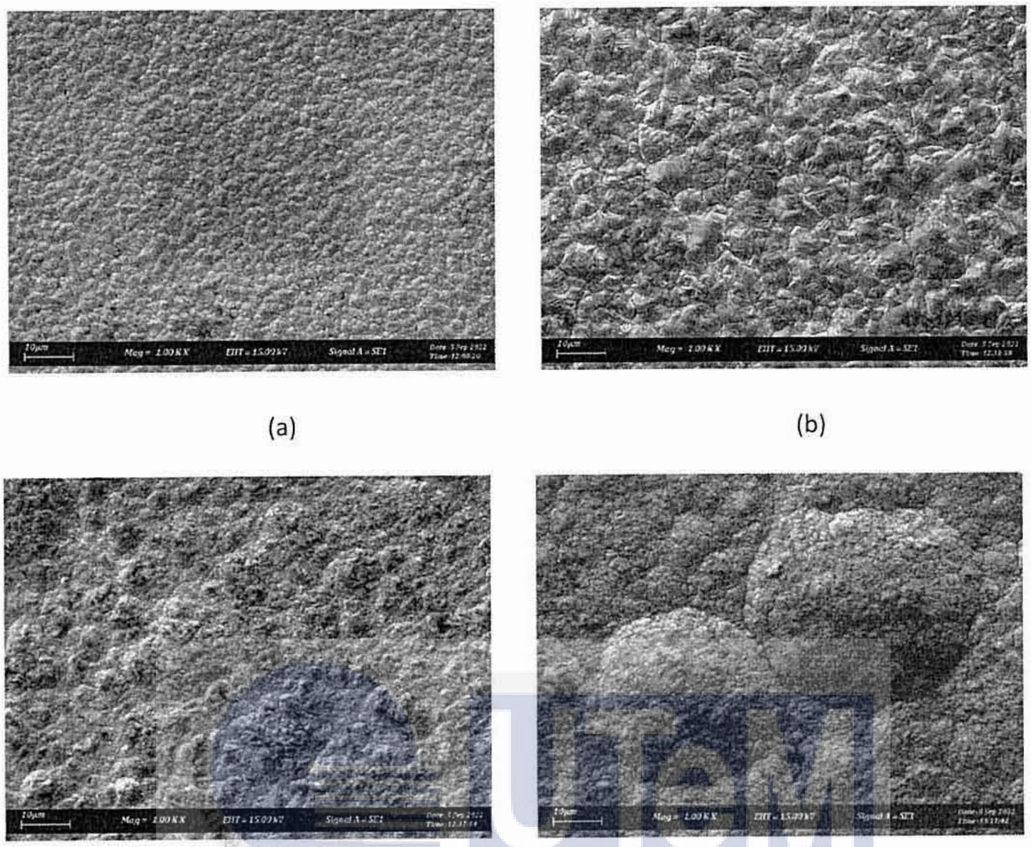
| Quarry Dust content (g/L) | Crystallite size (nm) |
|---|---|
| 15 | 12.17 |
| 30 | 14.93 |
| 45 | 13.07 |

As shown in table 4.1 (a), the crystallite size decreased as the current density increased from 4 $A/dm^2$ to 6 $A/dm^2$. This is due to the grain refining at high current density during the deposition process (Othman et al., 2018). When the quarry dust composition was increased from 30 g/l to 45 g/l with constant current density, the crystallite size decreased as well. The smaller the crystallite size, the more grain boundaries in the coating resulted in improved the hardness properties of the composite coating. (Othman et al., 2019)

### 4.2.2 Surface Morphology of Ni- QD Composite Coating

Surface morphology of pure Ni coating, and composite coatings at various current densities and various quarry dust content was observed and studied by SEM. It was observed that the SEM micrograph images show a colony like morphology that consist lots of nickel grains with have various sizes.

Figure 4.6 shows the effect of current density on the surface morphology at 45 g/L Quarry Dust content. As current density increased, the colonies like morphology become larger and denser. As seen in figure 4.6 at the magnifications of 1k, when 2 $A/dm^2$ current density applied, the SEM morphology show the surface is smooth, uniform, and compact, with finer grains evenly dispersed on it. As current density increased to 4 $A/dm^2$ the structure become larger and when current density 6 $A/dm^2$ was applied the structure become even larger and bumpy surface can been seen clearly. Then, at current density 8 $A/dm^2$ the image shows that the number of colonies like morphology increased and the structure become larger, indicating the relatively rougher surface. Thus, increased in current density result in higher over-potential which in turn increases the density of atomic clusters and so enhances the nucleation rate of coating (Othman et al., 2018)



(a) (b) (c) (d)



Figure 4.6: SEM micrograph on Ni-QD Composite Coating with 45 g/L quarry dust content applied with (a) 2 A/dm$^2$, (b) 4 A/dm$^2$, (c) 6 A/dm$^2$ and (d) 8 A/dm$^2$ current density.

Figure 4.7 shows at the magnifications of 1k, the effect of various quary dust content on the surface morphology at current density 6 A/dm$^2$. Based on the figure, pure nickel coating compared to 15 g/L quarry dust content shows slightly changes in the surface morphology. The changes in the size is slighly smaller. At quarry dust content 30 g/L the structure at the same magnifications look small and blurry. However, when the content of quarry dust increased to 45 g/L the surface become rougher. It can been seen the agglomaration of colonies like morphology due to the hindering of nickel grain growth during the deposition process. As quarry dust content increased to 60 g/L with current density 6 A/dm$^2$ the colonies like morphology become larger due to the co deposition of quarry dust content to the coating and with high current density applied, the deposition of matrix metal is fast enough to entrap and occlude the particles and incorporate into deposit which is aggrementation with the results reportted by Boukhouiete (2021) as higher current densities increase the deposition rate but reduce the controllability of the deposition process. This causes rapid deposition, which poses a risk to the control of crystal growth and the uniformity of particle distribution within the matrix.

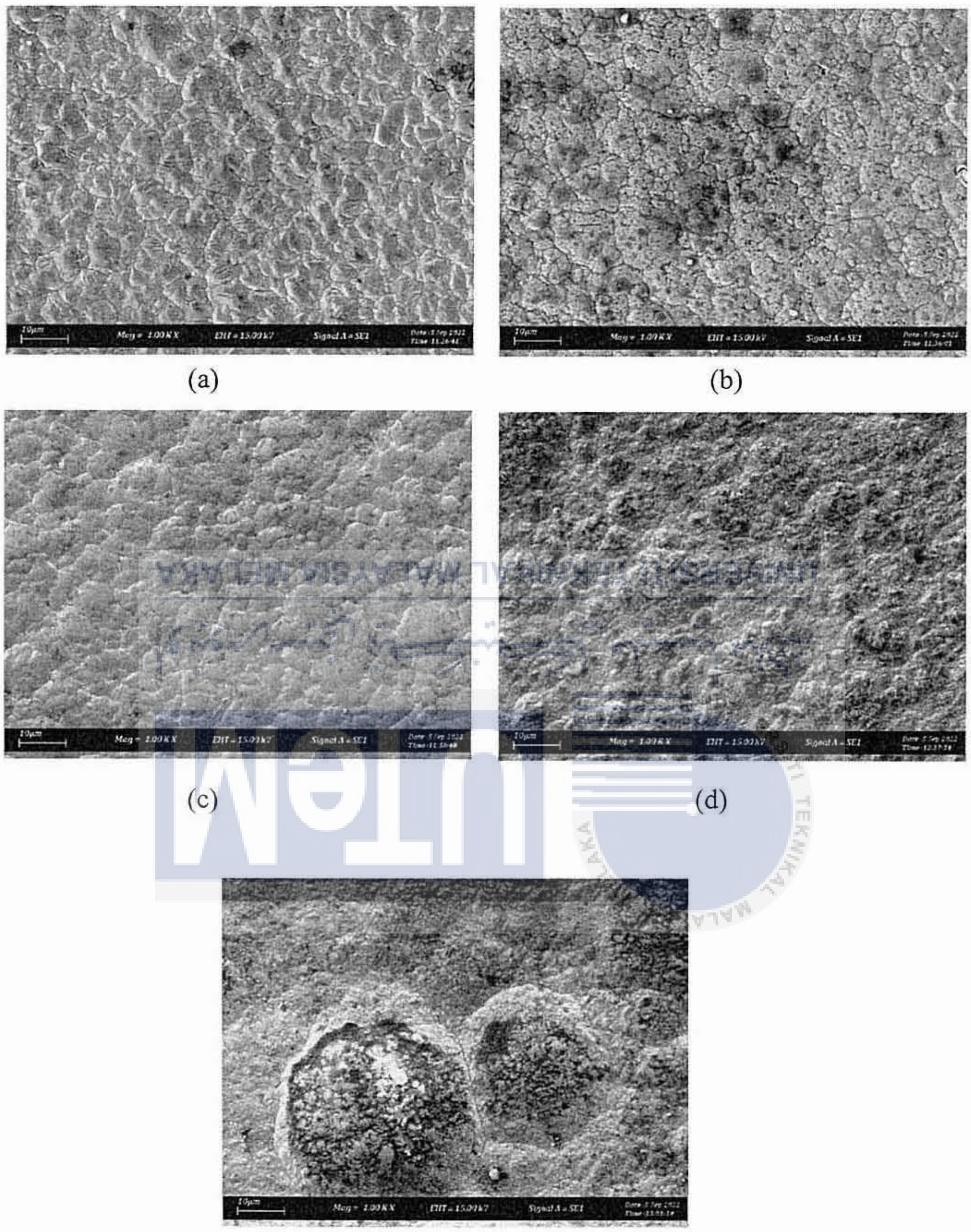



Figure 4.7: SEM micrograph on Ni-QD Composite Coating with current density $6\ A/dm^2$ applied with (a) pure nickel, (b) 15 g/L QD, (c) 30 g/L QD, (d) 45 g/L QD and (e) 60 g/L QD.

## 4.3 Mechanical testing of Ni-QD Composite Coating

### 4.3.1 Micro- Vickers Hardness Testing

Figure 4.8 shows the micro hardness results for different current densities and quarry dust content. When current density 2 $A/dm^2$, 4 $A/dm^2$ and 6 $A/dm^2$ applied to the various quarry dust content range from 15 g/L to 45g/L, the hardness value increased gradually but decreased when reached 60 g/L quarry dust content. The hardness also increased when current density 8 $A/dm^2$ is applied to various quarry dust content range from 15 g/L to 30g/L however when the quarry dust content reached 45g/L the hardness value decreased and increased back when quarry dust content is 60 g/L. The decreased in hardness value when quarry dust content reached 60 g/L is due to the design of jacketed beaker used during the electrodeposition process. The jacketed beaker has a shallow area caused the quarry dust content cannot mix well with the electrolyte even though magnetic stirrer is used during the experimental process. Besides, high content of QD also effect the process as not all the QD successfully mix with the electrolyte resulted a few QD content reached to the substrate. Figure 4.9 shows the design of the jacketed beaker used during the experiment. A flat surface beaker is more preferable for this process as the quarry dust content can mix well with the electrolyte and the aid of magnetic stirrer. The increased in quarry dust content also increased the hardness of the Ni-QD composite coating because of the presence of hard $SiO_2$ and $Al_2O_3$ particles in the deposit, the microhardness of the coating increased with the composition of the quarry dust particles in the deposit. This result consistent with results obtain by Kadir et al. (2018) when quarry dust was used as replacement of sand for the construction industry. Besides, the hardness value also related with the crystallite size as the size decreased the hardness value increased which is in aggrement with result reported by Boukhouiete et al., 2021. Thus, from the graph, the highest hardness can be obtained when the current density is 6 A/dm$^2$ and quarry dust content is 45 g/L with the value of 353HV while bare HSS with the hardness value of 168HV while HSS coating with nickel is 206HV.

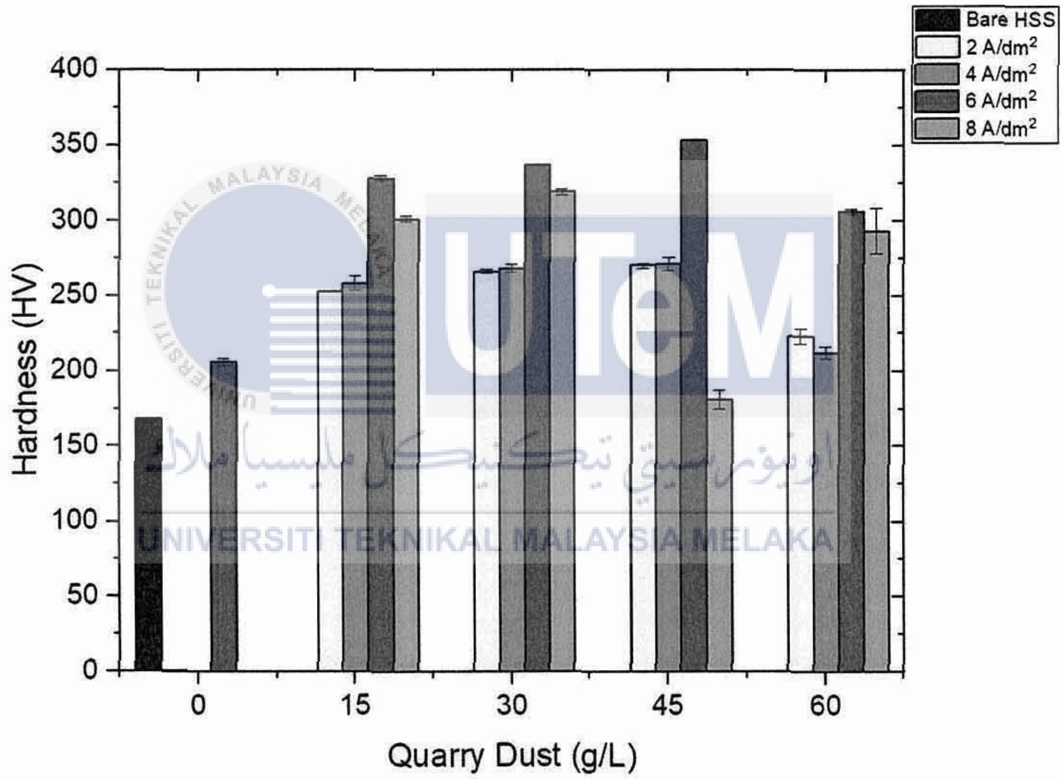



Figure 4.8: Micro hardness of Ni- QD composite coating at various current density and quarry dust content.

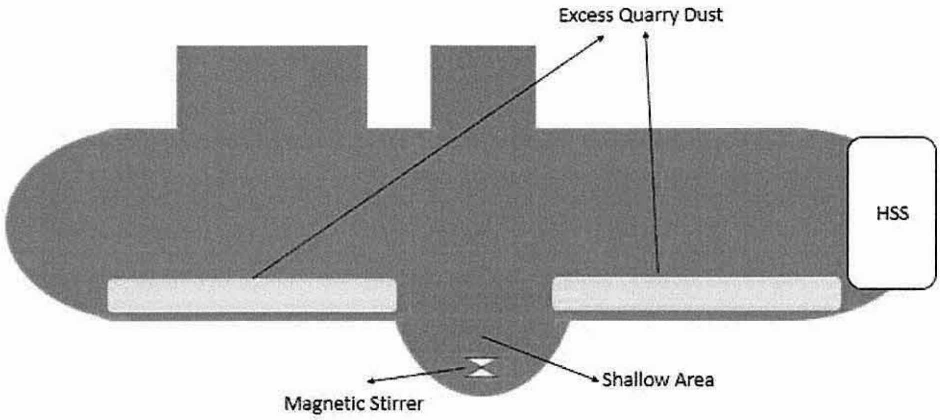



Figure 4.9: Design of jacketed beaker

### 4.3.2 Morphology observation on the Wear Track

The wear resistance properties of the Ni-QD composite coating were examined using wear track. Wear tracks of coated substrate were examined using SEM. Figure 4.10 shows the SEM images of the wear scar formed when various current density applied to the 45g/L quarry dust content. Table 4.2 shows the measurement for the wear scar when various current density applied to the 45g/L quarry dust content.



The wear scar was improved when the current density increased. Figure 4.10 (a) shows the worst result compared to Figure 4.10 (b), (c) and (d). When comparing the wear scare with the surface morphology in Figure 4.6 (a), shows a good distribution and dispersion of particle in the coating good hardness value as per Figure 4.8. However, when compared with the hardness of others quarry dust content with the same current density applied, it shows significant result as hardness increase with the current density and quarry dust content. Figure 4.10 (b) shows the wear track become better compared to the Figure 4.10 (a). The length of the wear track from 2 $A/dm^2$ to 4 $A/dm^2$ are decreased as the current density increased and the hardness value for 4 $A/dm^2$ is slightly higher compared to the 2 $A/dm^2$. Wear track in Figure 4.10 (c) shows better result as the wear only on the surface. The hardness value also increased as per Figure 4.8. The crystallize site also decreased as per Table 4.1 (a) for current density 6 $A/dm^2$. Figure 4.10 (d) shows the wear scare with the lowest width as per Table 4.2. The scar only on the surface, but when compared with surface morphology in Figure 4.6 (d) the structure is larger and hardness value as in Figure 4.8 shows the value is lower compared to others. The result from the wear track in Figure 4.10(d) does not significant to the surface morphology and hardness value this is due to the wear track are done randomly on the substrate, the testing could be done at the area that have sufficient amount of particle reinforcement. Thus, the results show insignificant relations towards others result.

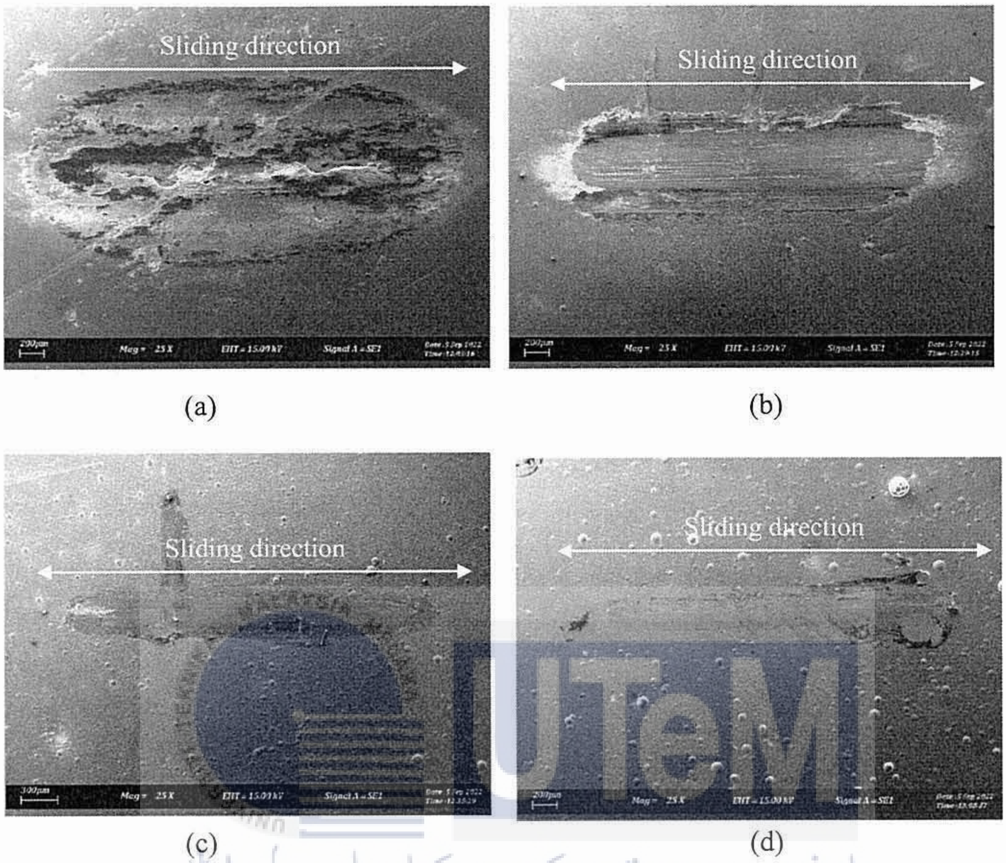



Figure 4.10: SEM micrograph on wear scar with 45 g/L quarry dust content applied with (a) 2 A/dm$^2$, (b) 4 A/dm$^2$, (c) 6 A/dm$^2$ and (d) 8 A/dm$^2$ current density.

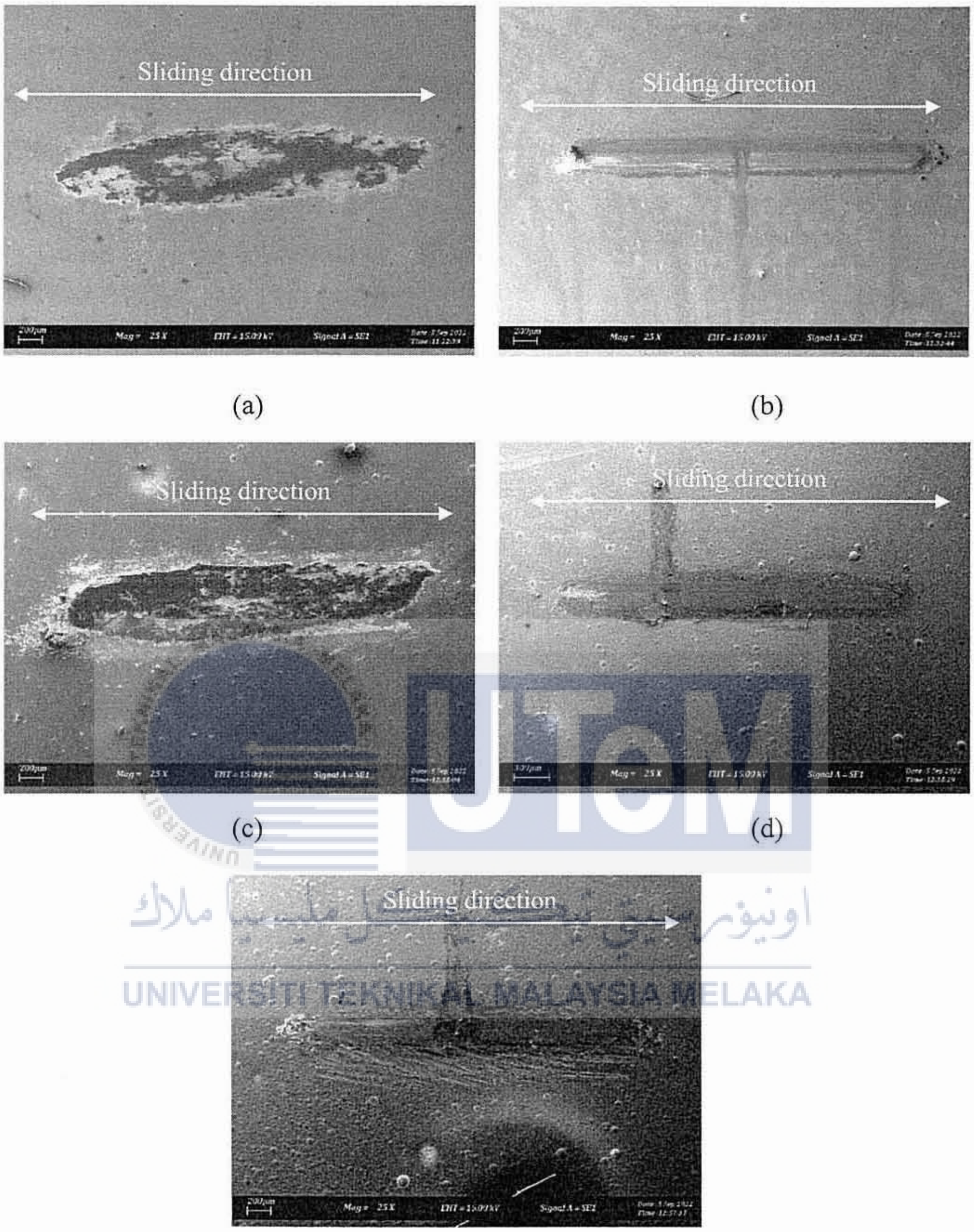


Table 4.2: Measurement of wear scar when 45 g/L quarry dust content applied with various current density.

| No. | Current Density (A/dm$^2$) | Length (μm) | Width (μm) |
|---|---|---|---|
| a | 2 | 3651 | 1545 |
| b | 4 | 3021 | 486 |
| c | 6 | 2905 | 221 |
| d | 8 | 2718 | 204 |

Figure 4.11 shows the SEM images of the wear scar formed when various quarry dust content applied to the 6 A/dm$^2$ current density. Table 4.3 shows the measurement for the wear scar when various quarry dust content applied to the 6 A/dm$^2$ current density.

Figure 4.11 (a) shows the wear track of pure nickel coating. It shows an adhesive wear as the dark structure appear more in the surface morphology. Figure 4.11 (b) shows the wear track structure is smooth and on only on the surface. When compared to the hardness value in Figure 4.8, the hardness value for 15g/L quarry dust content is the lowest compared to others at the same current density. The surface morphology as per Figure 4.7 (b) also show the particle size is smaller. The wear scar result show not significant to the surface morphology and hardness value due to the test conducted randomly on the structure. The wear track in Figure 4.11 (c).shows that the width of 30 g/L quarry dust content is higher compared to 15 g/L. However, when refer to the hardness value on Figure 4.8, the hardness value is higher compared to 15g/L quarry dust therefore the wear track should be better. The test might be conducted at the area that have insufficient amount of particle reinforcement. Figure 4.11 (d) shows the wear track with the lowest length and the value of the highest value of hardness in the same current density applied while Figure 4.11 (e ) shows the wear track only occur on the surface but compared to the hardness value in Table 4.8 the value is the lowest. The surface morphology also shows bumpy surface, large and denser. Thus, the result for Figure 4.11 (e) is not significant.




(e)

Figure 4.11: SEM micrograph on wear scar with 6 A/dm$^2$ current density applied with (a) pure nickel, (b) 15 g/L, (c) 30 g/L (d) 45 g/L and (e) 60 g/L quarry dust content.

Table 4.3: Measurement of wear scar when 6 A/$dm^2$ current density applied to various quarry dust content.

| No. | Quarry Dust (g/L) | Length (μm) | Width (μm) |
|---|---|---|---|
| a | 0 | 3196 | 608 |
| b | 15 | 2970 | 192 |
| c | 30 | 3115 | 600 |
| d | 45 | 2905 | 221 |
| e | 60 | 2635 | 192 |

### 4.3.3 Tribological Result

The effect of quarry dust composition on the coefficient of friction (COF) of composite coatings was evaluate. During the wear test, the COF was determined.

Figure 4.12 shows the COF comparison for 2 A/dm$^2$ current density applied to various quarry dust content. The value of COF for 45g/L quarry dust is highest compared to others. The value is not tally with the hardness result obtain in Figure 4.8. The hardness value shows slightly increase when the quarry dust content increased. Therefore, the result for 45g/L quarry dust with 2 A/dm$^2$ is not significant to the hardness value this is because the test might be conducted at the area that has low amount of particle reinforcement.

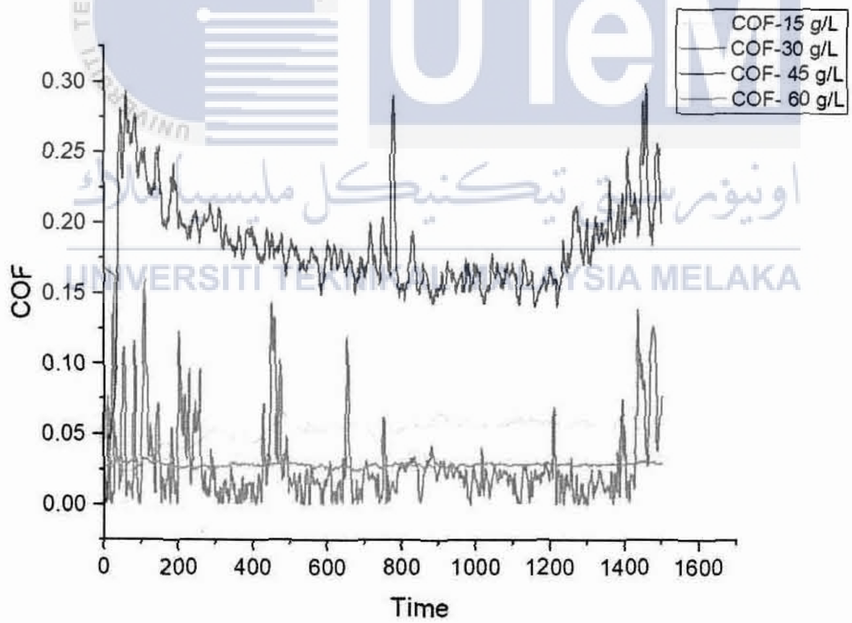



Figure 4.12: Comparison of COF for 2 A/dm$^2$ current density with various quarry dust content.

Figure 4.13 shows the comparison of COF for 4 A/dm$^2$ current density with various quarry dust content. The COF reading are close within the samples. The reading shows decrease in the COF value compared to 2 A/dm$^2$ current density presented in Figure 4.12.

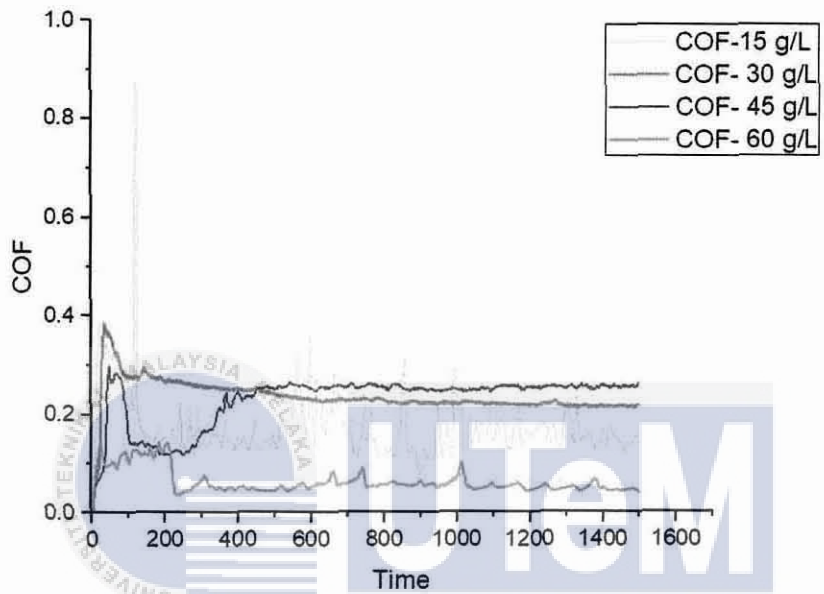



Figure 4.13: Comparison of COF for 4 A/dm$^2$ current density with various quarry dust content

Figure 4.14 shows the comparison of COF for 6 A/dm$^2$ current density with various quarry dust content. The value for 15g/L, 45g/L and 60g/L are close within each other resulted the value on the graph overlap on each samples. However, the value for 30g/L quarry dust content higher compared with others quarry dust content. This result is significant with the wear track as the wear shows both adesive and oxidative wear mechanism operating in the coating area. This result consistent with those of wear resistance of Sn-Plated Ni Coatings (Ahn and Sharma, 2021)

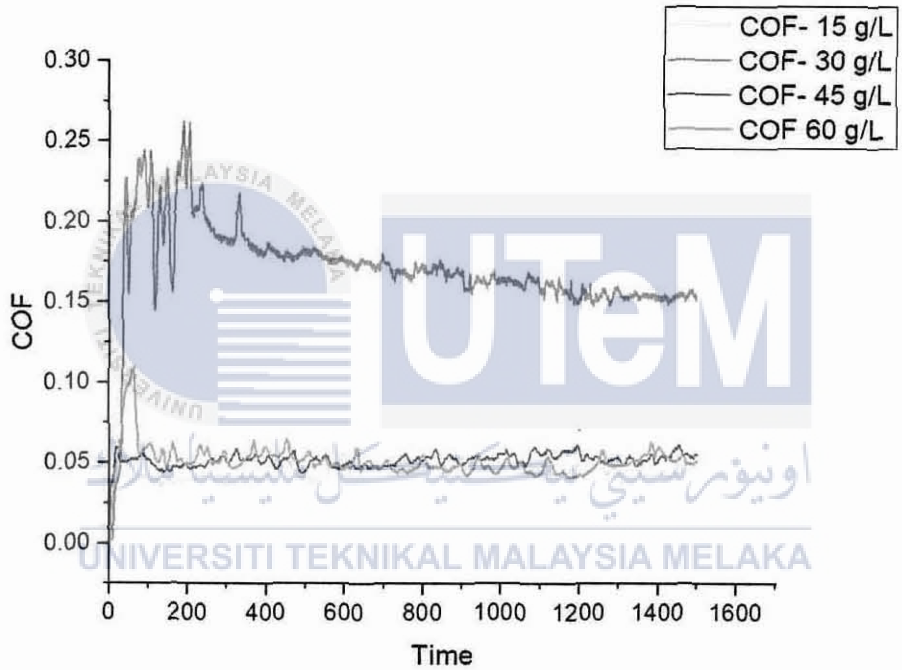





Figure 4.14: Comparison of COF for 6 A/dm$^2$ current density with various quarry dust content

Figure 4.15 shows the comparison of COF for 8 A/dm$^2$ current density with various quarry dust content. The COF value shows decreasing trend except for 30g/L quarry dust content However, when compared to hardness test, the hardness value is the lowest compared to other quarry dust content, thus the result is significant to the hardness test.

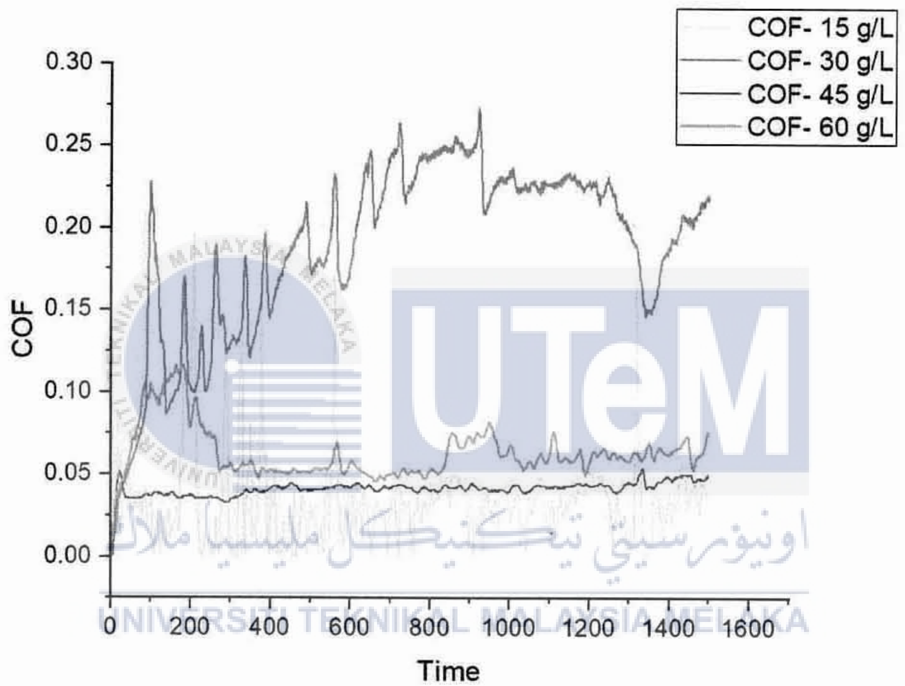



Figure 4.15: Comparison of COF for 8 A/dm$^2$ current density with various quarry dust content

Figure 4.16 shows the summary of COF value. Its shows decreasing in trend for current density 4 A/dm$^2$, 6A/dm$^2$ and 8A/dm$^2$ does it give an effect to the COF value as the current density increased, the COF value decreased. An increase in the current density caused gradual refinement in the grains, however a further rise in current density increased the deposit porosity due to the absorbtion of hydrogen bubles in the costings (Ahn and Sharma, 2021) that caused the COF value slightly increased for quarry dust content 30g/L and 60g/L when reached 8 A/dm$^2$ current density.

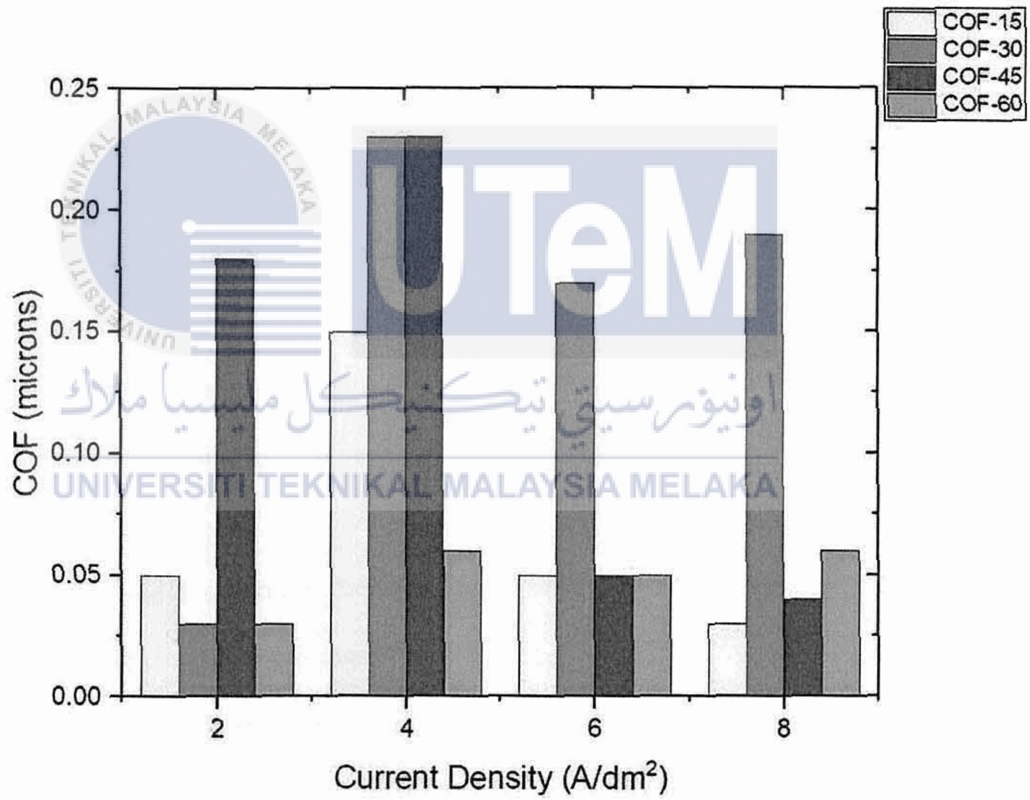



Figure 4.16: COF comparison for current density.

# CHAPTER 5

# CONCLUSION AND RECOMMENDATION

This chapter presents the conclusions and recommendation based on the data analysed in the previous chapter.

## 5.1 Conclusion

Ni-QD composite coating has been successfully electrodeposited and evaluated with varying current density and quarry dust composition. The quarry dust contains a high concentration of $SiO_2$ and $Al_2O_3$, thus this result in improves the properties of the substrate. The following conclusion can be drawn from the experiment:



1. The colonies like morphology become larger and denser as current density increased on the composite coatings.
2. The nickel grain size decreased as the current density increased and composition of quarry dust increased.
3. The microhardness value of the composite coatings increased as the current density increased. Besides, the present of Quarry Dust particles also increased the hardness value due to the presence of $SiO_2$ and $Al_2O_2$ in the Quarry Dust particles.
4. The COF value of composite coatings showed lower compared to the bare HSS. Thus, the wear resistance of the coatings was improved.

As the result, the optimum condition for the current density was determine on 6 $A/dm^2$ and quarry dust content is 45 g/L.

## 5.2 Recommendation

On the basis of the research done for this study, the following suggestions are made:

1. A custom flat beaker should be designed to ensure that all quarry dust particles are consistently stirred during the electrodeposition process, allowing the particles to be deposited on the substrate as much as possible. The suggested design of the beaker as per Figure 5.1.

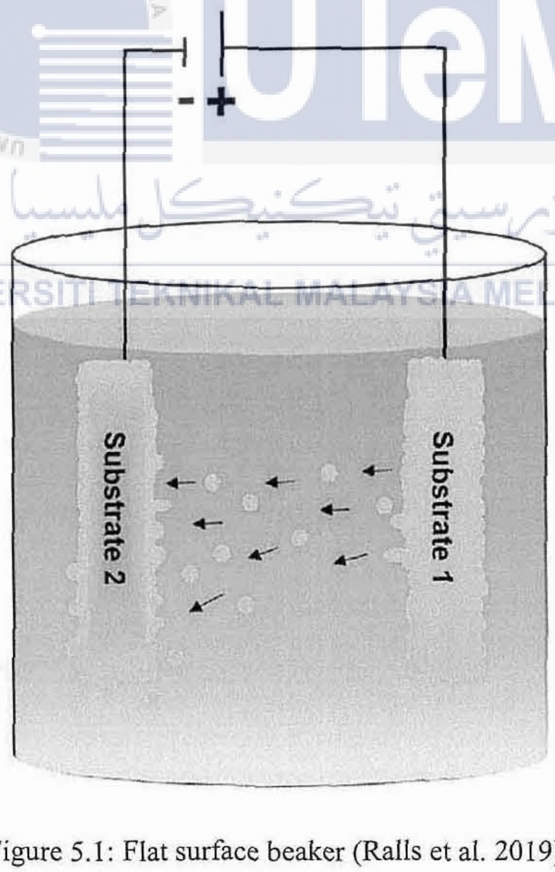





Figure 5.1: Flat surface beaker (Ralls et al. 2019)

2. The pH of the electrolyte should be determined in order to identify the best coating in terms of uniformity, as other electrodeposition parameters such as temperature, stirring rate, and electrolyte bath composition were fixed in this experiment.
3. Using Pulse Current (PC) while conducting the electrodeposition because it has been reported that it improves the surface of the coating by reducing pores, cracks, and internal stress (Paul, 2020).
4. Surface roughness can be conducted to study the influence of parameter on the effect of coating. Besides, wear rate and thickness of the coating should be identified to get better explanation on the result.

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**APENDICES**

Appendix A

GANTT CHART : MASTER PROJECT 1

| No | Activities | Status | Months | | | |
|---|---|---|---|---|---|---|
| | | | March | April | May | June |
| 1 | **PROJECT INTRODUCTION**<br>-Identify problem statement, objectives and scope | PLAN | | | | |
| | | ACTUAL | | | | |
| 2 | **REVIEW LITERATURE WORK**<br>-Findings additional information and knowledge about project | PLAN | | | | |
| | | ACTUAL | | | | |
| 3 | **METHODOLOGY**<br>-Identify the correct method for the experiment | PLAN | | | | |
| | | ACTUAL | | | | |
| 4 | **PRESENTATION OF THESIS PROPOSAL**<br>*-Complete MP1 report* | PLAN | | | | |
| | | ACTUAL | | | | |

Appendix B

GANTT CHART: MASTER PROJECT 2

| No | Activities | Status | Months | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | August | August | August | August | August | September |
| 1 | **QUAARY DUST PREPARATIONS**<br>-ball mill at 1:10; 350rpm, 3hours, 400g | PLAN | | | | | | |
| | | ACTUAL | | | | | | |
| 2 | **SUBSTRATE PREPARATION**<br>-machine the aluminium alloy into small size<br>-surface treatment | PLAN | | | | | | |
| | | ACTUAL | | | | | | |
| 3 | **ELECTROLYTE PREPARATION**<br>-set up the nickel watt bath as per parameter | PLAN | | | | | | |
| | | ACTUAL | | | | | | |
| 4 | **ELECTRODEPOSITION PROCESS**<br>-experiment set up as per parameter | PLAN | | | | | | |
| | | ACTUAL | | | | | | |
| 5 | **ANALYZE AND DISCUSSION**<br>-Analyze data collection<br>-Discussion on the result | PLAN | | | | | | |
| | | ACTUAL | | | | | | |
| 6 | **PREPARATION AND THESIS PRESENTATION**<br>-Conclusion of the experiment<br>-Report writing<br>-Thesis submission | PLAN | | | | | | |
| | | ACTUAL | | | | | | |