EFFECTS OF PULSE CURRENT ELECTRODEPOSITION PARAMETERS ON THE PROPERTIES OF NICKEL-QUARRY DUST COMPOSITE COATINGS

This report is submitted in accordance with requirement of the University Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)

by

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I hereby, declared this report entitled "Effects of Pulse Current Electrodeposition Parameters on The Properties of Nickel-Quarry Dust Composite Coatings" is the result of my own research except as cited in references.

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Date : 28 June 2024

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UniversitiTeknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree ofManufacturing Engineering (Engineering Materials) (Hons). The member of the supervisory committee is as follow:

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ABSTRAK

Tujuan kajian ini adalah untuk mengkaji di mana ketumpatan arus dan kitaran tugas yang berbeza mempengaruhi kekerasan, rintangan haus, dan morfologi permukaan salutan komposit yang dihasilkan daripada Habuk Ni-Quari. Salutan komposit yang dihasilkan daripada habuk Ni-Quari telah dicipta dengan menggunakan kaedah elektrodeposisi arus nadi di mana nikel bertindak sebagai anod dan substrat iaitu keluli lembut bertindak sebagai katod. Teknik elektrodeposisi arus nadi dilakukan dengan merendam anod dan katod ke dalam tab penyaduran selama satu jam. Pelbagai ketumpatan arus dan pelbagai kitaran tugas iaitu 1 A/dm^2, 3 A/dm^2, 5 A/dm^2, 7 A/dm^2 dan 9 A/dm^2 dan 25%, 50% dan 75% masing-masing digunakan. Keputusan yang diperoleh menunjukkan bahawa salutan komposit habuk Ni-Quari yang disediakan oleh paparan elektrodeposisi arus denyut meningkatkan kekerasan dan sifat rintangan haus. Mikrograf SEM menunjukkan morfologi permukaan yang diperolehi pada kitaran tugas 25% menunjukkan elektrodeposit yang seragam dan lebih halus dan morfologi permukaan yang diperoleh pada 9 A/dm^2 menunjukkan saiz butiran yang besar dengan terkumpul. Kekerasan mikro salutan komposit diukur dengan ujian mikro-Vickers. Keputusan menunjukkan bahawa pada kitaran tugas 75% dan keadaan 9 A/dm^2 menunjukkan kekerasan yang paling tinggi. Di samping itu, rintangan haus dicirikan oleh pin pada mesin cakera. Keputusan menunjukkan bahawa morfologi permukaan yang seragam dan lebih halus telah menghasilkan COF yang lebih rendah. Oleh itu, salutan komposit dengan nilai rintangan haus maksimum adalah yang dilakukan pada kitaran tugas 25%. Secara keseluruhannya, salutan komposit Ni-Quarry Dust yang disediakan oleh elektrodeposisi arus denyut meningkatkan sifat tribologi dan mekanikal salutan komposit untuk aplikasi automotif seperti galas, aci dan gear.

ABSTRACT

The purpose of this study is to examine where different current densities and duty cycles affect the hardness, wear resistance, and surface morphology of composite coatings produced from Ni-Quarry Dust. Composite coatings produced from Ni-Quarry Dust were created by applying the pulse current electrodeposition method where nickel acts as anode and substrate which is mild steel is acts as cathode. The pulse current electrodeposition technique is done by immersing the anode and cathode into the plating bath for one hour. Various current density and various duty cycles which are 1 A/dm^2, 3 A/dm^2, 5 A/dm^2, 7 A/dm^2 and 9 A/dm^2 and 25%, 50% and 75% are used respectively. Obtained results indicated that Ni-Quarry Dust composite coatings prepared by pulse current electrodeposition display improved hardness and wear resistance properties. SEM micrograph shows that surface morphology obtained at 25% duty cycle shows uniform and finer electrodeposited and surface morphology obtained at 9 A/dm^2 shows large grain sizes with agglomerated. The microhardness of the composite coatings was measured by micro-Vickers test. The results show that at 75% duty cycle and 9 A/dm^2 condition shows the highest hardness. In addition, the wear resistance was characterized by pin on disc machine. The result shows that uniform and finer surface morphology has result in lower COF. The composite coating with the maximum value wear resistance is therefore the one done at 25% duty cycle. Overall, the Ni-Quarry Dust composite coating prepared by pulse current electrodeposition improves the composite coating's tribological and mechanical properties for automotive application such as bearings, shafts and gears.

DEDICATION

This research study is wholeheartedly dedicated to:

My beloved parents who have been my source of strength;

Muhammad Nadzri bin Mat Yusop

Robayah binti Mat Dris

My respectful supervisor,

Dr. Intan Sharhida binti Othman

My siblings and friends,

For their endless support, motivation, and kindness throughout this study.

May Allah S.W.T bless all of you.

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Finally, but just as importantly, I want to express my gratitude to my friends for their support and encouragement throughout the writing of this report. They had offered their insightful advice and opinions all during my research. I cherish our wonderful friendship.

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LIST OF ABBREVIATIONS

ASTM	-	American Society Testing and Materials
COF	-	Coefficient of Friction
FKM	-	Fakulti Kejuruteraan Mekanikal
MMC	-	Metal Matrix Composite
PSA	-	Particle Size Analyzer
SEM	-	Scanning Electron Microscope
UTeM	-	Universiti Teknikal Malaysia Melaka
XRD	-	X-Ray Diffractometer
Ni-QD	-	Nickel-Quarry Dust
EDX	-	Energy Dispersive X-Ray
XRF	-	X-Ray Fluorescence

LIST OF SYMBOLS

A/dm^2	-	Ampere per square decimeter
mm	-	milimeter
µm	-	micrometer
s	-	second
g	-	gram
g/L	-	gram per litre
N	-	Newton
mm/s	-	milimeter per second
°C	-	Degree Celsius
rpm	-	revolution per minutes
%	-	percentage
wt.%	-	mass fraction
Ni	-	Nickel

SiO_2 - Silicon Dioxide

Al_2O_3 - Aluminium Oxide

$NiSO_4$ - Nickel Sulphate

NiCl - Nickel Chloride

NaCl - Sodium Chloride

$Na_3C_6H_5O_7$ - Sodium Citrate

TiC - Titanium Carbide

TiO_2 - Titanium Dioxide

CNT - Carbon Nanotube

ZnO - Zinc Oxide

W - Tungsten

GO - Graphene Oxide

TiN - Titanium Nitride

BN - Boron Nitride

B_4C - Boron Carbide

Si_3N_4 - Silicon Nitride

K_2O - Potassium Oxide

Na_2O - Sodium Oxide

Fe_2O_3 - Ferric Oxide

CaO - Calcium Oxide

CHAPTER 1

INTRODUCTION

This chapter provided an explanation of the background of study, problem statement,objective, and scope of study.

1.1 Background of Study

Mild steel finds extensive use in automobile industries such as metal components. In industrial production, wear failures on the surfaces of metal components such as gears, shaft and bearings. These components are frequent, especially in high-speed, high-load operations. Shorter service lives, higher manufacturing costs, and large financial losses are the outcomes of these working circumstances. Wear failures on workpiece surfaces happen often.

Machine tools and automobiles are only two of the many industries that make heavy use of strong chrome coatings due to their high mechanical and corrosion resistance. Unfortunately, these coatings do not meet the current criteria for environmentally friendly industrial protection, and they emit dangerous levels of hexavalent chromium into the environment during production (Ren et al., 2023). Because of this condition, researchers have suggested alternate coatings and deposition processes.

Recently, the researchers have been looking into a few different techniques to enhance the surface qualities of metal components. These approaches may vary based on the parameters of the procedure. Surface modifications such as electroplating, chemical plating, or laser melting are frequently used to make coatings that have a high resistance to wear or substrates that have surface structures that have been altered with the process (Xia et al., 2020). The application of this approach to impart desirable qualities to surfaces, independent of the material composition of the surface, is standard practice. Among these qualities are solderability, better aesthetics, and resilience to corrosion and wear.

Nickel-based coatings have been used extensively in industry to improve substrate surface properties and wear resistance since they are among the more effective options. Ni alloys' high tensile strength smooth surface, and excellent corrosion resistance resulted to their broad use in the mechanical, chemical, and petroleum industries (Liu et al., 2021). Nickel is capable of being applied as a coating to a broad variety of materials such as metal, polymers, and ceramics. Nickel plating is applied in wide industries including those dealing with mechanical parts such as bearings, gears, and blades as well as the aviation industry, the petroleum industry, the coal business, and the natural gas sector (Xia et al., 2021).

By providing protection to the substrate from outside influences, composite coatings can greatly enhance the substrate's surface characteristics. Additionally, it modifies the metal's surface characteristics, improving its electrical conductivity or hardening and strengthening its resistance to wear. Composite coatings with nickel-based ceramic particles exhibit great strength, high hardness, and resistance to corrosion (Huang et al., 2021). Unfortunately, the performance of nickel-based coatings in terms of wear and corrosion resistance has not been able to keep up with the needs of modern industrial production.

The fine dust produced when minerals and stones are extracted from quarries is known as quarry dust (Shyam Prakash & Rao, 2016). A few variables, such as the rock's geological origin, the method of extraction, and the storage conditions, affect the precise chemical composition of the dust from the quarry. Researchers are searching for new uses for quarry dust to mitigate its detrimental impact on the environment. To give just one example, it is utilized as a raw material in many different businesses, such as those that produce concrete, ceramics, and other building supplies.

Some varieties of quarry dust may be appropriate for usage in the industrial sector due to their high concentrations of silica and alumina (Othman et al., 2019). Depending on theirsize and composition, quarry dust particles may add to the coating's improved hardness. It is commonly known that nickel resists corrosion. Its surface forms a shielding oxide layer that helps keep rust and corrosion at bay. Because of its properties, it can be used in a variety of alloys. According to Zhang et al., (2022), the mechanical, thermal stability, anticorrosive, and wear-resistant qualities have been enhanced by including quarry dust into the Ni matrix as a second reinforcing phase.

Coatings can be deposited onto substrates submerged in electrolytes by the process of electrolysis known as electrodeposition. This process can be carried out in two ways either in an aqueous solution electroplated at room temperature or in a fused salt electroplated at increased temperature. The electrodeposited composite coatings displayed superior mechanical, electrochemical, and oxidative capabilities in comparison to the coatings formed of pure metal (Arunsunai Kumar et al., 2013). The size of the electrodeposited particle and the percentage of the particle that was electrodeposited largely influenced the growing properties. This method's biggest disadvantage is that it can't produce uniform thickness. The other drawbacks include the need to evaluate hydrogen ions and prepare the substrate's surface to a critical degree. Electroplating is commonly employed for low-range corrosion and wear protection as well as decorative coatings. While it is also possible to install high- temperature resistant biological and ceramic coatings by increasing the potential difference in the cell (Karmakar et al., 2021).

Compared to coatings electrodeposited with direct current, those electrodeposited with pulse current have superior mechanical and tribological properties. Metals and alloys can have their microstructures changed by pulse current, which allows one to control and enhance the material's properties. When it comes to depositing these coatings, pulse current electrodeposition is more cost-effective than brushing electroplating, reverse pulse electrodeposition, and direct current electrodeposition. Other benefits include simple operation, high efficiency, safety, and dependability (Xia et al., 2021). Pulse current enables fine-grained control of duty cycle and current density, two plating properties. In pulse current electrodeposition method, the current is zero at T_{ON} (ON time) and has a specified value at T_{OFF} (OFF time).

On the other hand, the direct current electrodeposition technique maintains a consistent current across time. A higher "on" current and a lower or zero "off" current are typical ranges for these currents. Between a pulse's "on" and "off" phases, there is a variation in the current density, which can even reach zero (Ghazanlou et al., 2016). The specific impacts caused on the deposition process are determined by the properties of the pulse, such as the length and amplitude of the "on" and "off" phases. To alter the material's properties during the "on" duration to a specific application, it is imperative to choose the appropriate current density (Kumar Singh et al., 2019).

If there's a chance that the current densities are higher during the "on" time, the composite coating might stick to the substrate more successfully (Kasturibai & Paruthimal Kalaignan, 2014). Conversely, excessively high current densities may result in uneven coatings or inadequate adherence. Coatings tend to be hardened when high current densities are sustained during the "on" phase. There is a chance that this will lead to greater durability.Optimization is necessary because exceptionally high current densities have the potential tocreate brittleness in coatings.

By adjusting the duty cycle of the pulse current electrodeposition process, one may precisely regulate the properties of the deposit material. The duty cycle influences the average current density, the quantity of charge that is passed, and the amount of heat that is created. The percentage of the total pulse cycle time that is spent to the pulses "on" phase is known as the duty cycle. It is made clear that it indicates the portion of the overall cycle duration that is devoted to applying current. There are possible duty cycle values between 0% and 100%. When the duty cycle is 100%, there is no "off" time in the application of the current; conversely, when the duty cycle is 0%, the current is constantly on. The relationship between duty cycle and average current density is shown. Higher duty cycle counts are linked to coatings that adhere better and are more uniform.

1.2 Problem Statement

The automotive industry uses mild steel extensively for metal components. Nevertheless, mild steel is not strong enough for many purposes, and it has a low level of corrosion resistance. Compared to other materials, mild steel is comparatively malleable. They are brittle and easily worn down, especially in applications where they are in contact with abrasive materials or are subjected to repeated mechanical contact. Thus, mild steel can have its mechanical and tribological qualities improved through a process of electrodeposition.

Compared to coatings electrodeposited with direct current, those electrodeposited with pulse current have superior mechanical and tribological properties. The reason for this is that the structure and characteristics of the electrodeposit cannot be more precisely controlled using direct current electrodeposition (Arunsunai Kumar et al., 2013). Because the applied current is pulsating, a change in current density is caused by pulsed current electrodeposition. Among other parameters of the electrodeposition process, this pulse can modify the form, content, and adherence of the material that is deposited. When comparing to direct current electrodeposition, the pulse current electrodeposition approach may generate composites with improved integrated performance, a better microstructure, and finer metal granularity (Ma et al., 2020).

In direct electrodeposition, a low direct current results in coatings with larger grains, whereas a high direct current causes all the bath electrolytes to be absorbed and a high metal deposition causes the coating surfaces to become rough. However, because of the improved physical and chemical properties of the coatings, pulsed electrodeposition offers more benefits than direct current deposition because the current may be adjusted between two pulses. Additionally, the pulse current electrodeposition technique offers enhanced results in terms of fine grain deposition, low porosity of the coating, improved morphologies, hardness, and wear resistance than direct current electroplating does (Kumar Singh et al., 2019).

Direct current electrodeposition does not have parameters that can be altered to get the desired material characteristics. It is possible by the pulse current electrodeposition to modify the electrodeposition process by altering the duty cycle. Duty cycle changes can affect the cooling and crystallization rates, which can affect the microstructure of the coating.The porosity of the coating, which is influenced by changes in the duty cycle, determines how resistant it is to corrosion. Surface morphology is affected by duty cycle changes. The way the coating distributes the quarry dust particles may change if the duty cycle is changed.

1.3 Objectives

- To investigate the influence of various current density on the surface morphology, hardness and wear resistance of Ni-Recycled quarry dust composite coatings prepared by pulse current electrodeposition.
- To study the effect of various duty cycles on surface morphology, hardness and wearresistance of Ni-Recycled quarry dust composite coatings prepared by pulse current electrodeposition.

1.4 Scope

Composite coating consists of nickel and quarry dust produced by using pulse current electrodeposition technique. An electrolyte was prepared by mixing NiSO4, NiCl, NaCl and $Na_3C_6H_5O_7$ for electrodeposition. Mild steel is exposed to electrolyte and serves as cathode while nickel is expose to electrolyte and serve as anode. The electrodeposition variables are current density and duty cycle which are 1 A/dm^2, 3 A/dm^2, 5 A/dm^2, 7 A/dm^2 and 9 A/dm^2 and 25%, 50% and 75% respectively. The disperses of quarry dust to the substrate with the help of magnetic stir bar. The surface morphology of the Ni-quarry dust composite coatings will be analyzed by using scanning electron microscope (SEM) and x-ray diffractometer (XRD). The wear resistance and hardness of the Ni-quarry dust composite coatings will be analyzed by Pin on Disc Machine and Micro-Vickers Test respectively.

CHAPTER 2

LITERATURE REVIEW

This chapter specifically explains the studies and theories that have been established and carried out by researchers over the years. A discussion based on their findings about composite coatings, pulse current electrodeposition, effect of current density and duty cycle on the surface morphology, hardness and wear resistance of the mild steel and related information from earlier studies was obtained and used as references.

2.1 Steel Substrate

Mild steel is a commonly used material in various industrial applications due to its favorable mechanical properties and cost-effectiveness. Because of its low carbon content, mild steel is extremely machinable and has better tensile and impact strengths. It also has outstanding ductility and weldability (Rai & Gupta, 2021). However, mild steel can easily corrode which can shorten its lifespan and affect its structural integrity. To address this issue, researchers have investigated applying protective coatings on mild steel substrates using electrodeposition methods. Electrodeposition technique is a promising technique for improving mild steel substrates' resistance to corrosion.

Rahman Rashid et al., (2017) examined the geometrical properties and metallurgical properties of 316L stainless steel clad that was placed on mild steel substrate. The study revealed that the clad had advantageous characteristics, such as resistance to corrosion, indicating that it might be used in applications that need stronger corrosion protection. In another study, (Ghanbari & Attar, 2015) investigated the effectiveness of epoxy nanocomposite coatings on mild steel substrates that contain nano-silica that has been treated with epoxy silane to prevent corrosion. The study showed that the addition of treated nano- silica increased the epoxy coatings' resistance to corrosion, making them acceptable for preventing corrosion on mild steel.

Mild steel is a substrate used in the electrodeposition process, which offers a wide range of potential applications. The corrosion protection properties of electrodeposited coatings may be useful in sectors such as the automotive, construction, and marine industries where mild steel is commonly employed.

2.2 Coatings

2.2.1 Nickel Coating

Coating is a layer formed on the surface of an object which is called as substrate. The primary function of coatings is to provide protection to the surface from the environment. Coating also enhances the appearance of the surface. They are applied to a wide variety of surfaces, including concrete, metal, plastic, glass, and rubber. Coatings is most employed method for protective applications due to demand for easy application and inexpensive cost (Karthik et al., 2020).

Nickel is a ductile metal with a silvery white color. At room temperature, it exhibits hard paramagnetic properties and great mechanical strength. It takes a while for it to oxidize in air. However, polished surfaces will eventually corrode if exposed to the atmosphere. Nickel is present in a wide variety of everyday products (Razika et al., 2018).

Compared to other alloys, nickel (Ni) and its alloy offer a few benefits, including resistance to wear, corrosion, and hardness. Because of this, scientists and industry are drawn to employ it in the composite coating matrix to improve wear and corrosion resistance, improve magnetic properties, and repair parts (Karmakar et al., 2021). Because nickel gets along well chemically with other metals and alloys, it is the most appropriate metal in all metal matrixes. Additionally, it gives the coated surface very strong corrosion resistance properties (Gupta et al., 2016).

When used as a coating material, nickel exhibits good resistance to corrosion in corrosive environments. However, nickel is not an ideal material to employ in protecting components that are exposed to wear because of its low strength and hardness. The combination of microstructural changes and alloy components increases the hardness of alloy coatings (Doğan et al., 2020). This can be explained by the fine dispersion of nickels in the grain boundary and matrix during the alloying process, resulting in the hardness of the material.

2.2.2 Composite Coatings

Composite coatings including ceramic particles based on nickel such as SiC, Al_2O_3, WC, SiN have excellent corrosion resistance, high hardness, and high strength. Although a lot of research has been done on composite coatings, nickel-based alloy composite coatings are not widely employed because of high stress (Huang et al., 2021). A common matrix used to create electrochemical composite coatings is nickel. Strong corrosion resistance in mildly acidic and alkaline conditions, such as air, along with notable microhardness and favorable tribological characteristics define a nickel matrix. By adding a dispersed phase, some features of the nickel coating could be enhanced (Szeptycka et al., 2016).

Organic polymers and inorganic chemicals combine to form a composite coating. The polymers encase the inorganic ingredients and function as a matrix. Jabbar et al., (2017) noted that in composite electrodeposition techniques, micro/nano particles or fibers are induced into a metal matrix during the electrodeposition process thus this coating is applied to substrates to improve surface qualities and protect them from corrosion. Arunsunai Kumar et al., (2013) noted that electrodeposited composites shown significant benefits when compared to coatings formed of pure metal in terms of mechanical, electrochemical, and oxidative properties.

By employing electrodeposited, metal coatings could acquire new properties that improve their performance, like hardness, wear resistance, lubricity, and corrosion resistance (Jegan & Venkatesan, 2013). With these coatings, one can attain enhanced performance, greater durability, or more targeted functionality because they are made to combine the best qualities of multiple materials. Any substrate can be coated with composite materials.

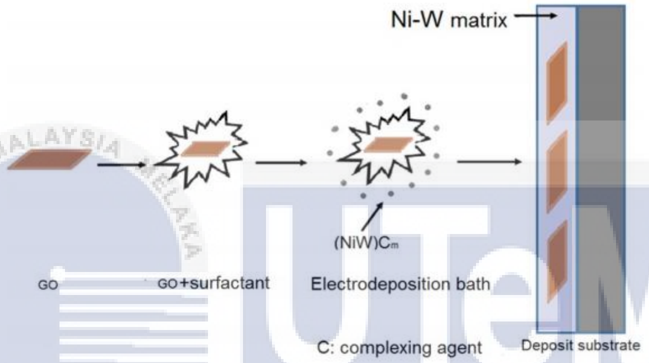


Figure 2.1: Process of composites deposition ((Fan et al., 2016)

Durability, resistance to corrosion, and protection from wear and tear are crucial mechanical conditions in the manufacturing and automotive industries. When using liquid lubricant isn't efficient, this mechanical part slides against each other. Mechanical parts that touch against one another will eventually wear down and need replacement, which might cause the system to fail. According to Kumar Singh et al., (2019) composite coating plays an important role in the protection of sliding mechanical part against each other because composite coatings give protection without adjusting the composition of the substance.

Composite coatings have been a new sort of coating that has been acknowledged by the researchers. Composites coatings have a distinct type based on matrix materials such as metal matrix composite, carbon matrix composite, polymer matrix composite and ceramic matrix composite. Karmakar et al., (2021) noted that metal matrix composite is the most employed in industries due to its versatileness.

2.2.3 Metal Matrix Composite Coating

Metal Matrix Composites (MMC) have exceptional qualities such as hardness, self- lubricating nature, and resistance to wear, oxidation, high temperatures, and corrosion (Kamnerdkhag et al., 2017). MMC coating is a coating in which a reinforcing material is deposited into a metal matrix to improve the surface properties of a substrate. Rai & Gupta, (2021) noted that the reinforcing material typically ceramic, metal alloy powders, metal powder, metal oxide powders, carbides of metals, nitride of metals and organic materials are deposited into the matrix.

According to Zhang et al., (2022) MMC has been studied by scientists because it meets the requirements of mechanical properties to overcome corrosion and wear resistance issues in metals, composite materials, organic materials, and inorganic materials. In addition, according to Karmakar et al., (2020) the surface of the matrix can then be coated to prevent chemical reactions with the matrix. MMC coatings are used to improve corrosion resistance, hardness and wear resistance.

In the industry, aluminum matrix composite is one of the most popular MMC coatings. Its low density, high specific stiffness, and high specific strength are its advantages. A lot of research has focused on the low-cost reinforced particle composite and low density in aerospace, electronics, and automotive industries in recent years. Investigation done by Wang & Zhang, (2023) highlights that the deposited coatings have a compact, crack-free, silver-gray appearance with a reduced surface roughness, and a morphology like colonies. It was confirmed that graphene oxide was present and evenly distributed both inside the nickel covering and on the surface. Since ceramic particle reinforced composite is costly, multiple efforts have been made to produce a low-cost aluminium matrix composite. Reinforcement particles are co-deposited into the matrix to create an inexpensive aluminum matrix composite. Numerous studies have investigated the reinforcing particles that are co-deposited into the metal matrix, such as graphite, TiC, TiO_2, SiC, SiO_2, CNT, Al_2O_3, ZnO, and Gr. In this research, quarry dust is employed as reinforcement particles that are co-deposited into the matrix. An MMC matrix can contain a variety of components, including Fe, Mg, Ni, Al, Co, and Ti. Nickel-based coating is one of those materials for MMC matrix that will be covered in this project.

2.3 Quarry Dust

Researchers have found that hard ceramic particles, such as SiC and Al_2O_3 can be effectively added to coatings to reduce product wear and improve microhardness. The high concentration of SiO_2 and Al_2O_3 in recycled quarry dust makes it an excellent alternative to more costly conventional ceramic particles. Quarry dust waste contributes to 20–25% of total production and is yearly disposed of in landfills (Sridharan et al., 2006). This debris was collected as a byproduct when rocks were crushed in rubble crusher equipment to generate aggregates.

Particles of dust that penetrate the air around quarry operations have a negative impact on the environment. Reusing such waste and converting it into fundamental goods that can be used is one method of reducing environmental pollution (Rani et al., 2020). There has been increased interest in using quarry dust to substitute natural fine aggregates. Utilizing this waste material can reduce environmental effects, lower waste management costs, and enhance concrete quality. There are benefits to using quarry dust as an alternative building material, including increased concrete workability and compressive strength as well as the beneficial disposal of byproducts (Rani et al., 2020).

Prakash and Rao, (2016) found that by substituting 80% quarry dust for fine aggregate, the compressive strength of concrete was enhanced by 16.43%. the compressive strength improved by over 18% by using quarry dust. According to Shyam Prakash & Rao, (2016) higher compressive strength was produced by replacement of 40% of quarry dust. The results indicated that concrete could achieve high strength by partially replacing it with fine aggregate quarry dust.

Xavier & Suresh, (2016) investigated the wear characteristics of composites made from aluminium metal matrix and industrial byproducts. The study shows that wear resistance increased when reinforcement content increased from 10 %wt to 20 %wt. The results implied that wear resistance of composite coatings enhanced by the quarry dust particles. According to Xiao et al., (2020) adding quarry dust improved the wear resistance of composite coatings. This is the result of their research regarding how quarry dust reinforcements affect composite coatings' performance to with stand wear. In addition to increasing wear resistance, they discovered that quarry dust has advantages in oily water separation, air purification, and anti-icing.

2.4 Electrodeposition of Nickel Composite Coating

2.4.1 Overview of Electrodeposition

There are four stages in the metal matrix composite electrodeposition process. The phases are particle-cathode surface contact, mass transfer of the particles onto the cathode surface, formation of surface charge on the particle surface of the suspension, and nanoparticles trapped in the developing metal layers (Ghazanlou et al., 2016).

Kumar Singh et al., (2019), Zhang et al., (2022), Rai & Gupta, (2021) noted that electrodeposition is a favorable technique to fabricate metal-based composite because this technique is simple and economical, ease of use, low setup costs, little production waste, and extensive industrial use.

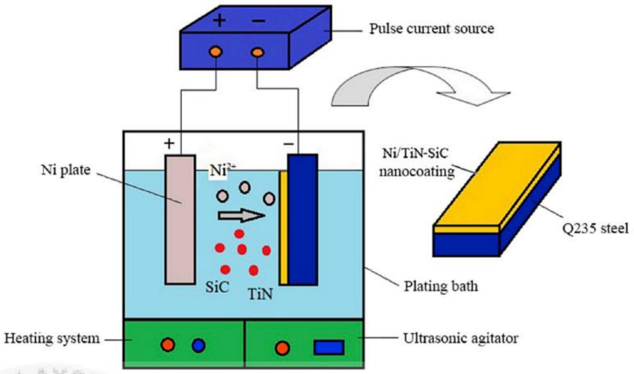


Figure 2.2: Experimental device for preparing Ni/TiN–SiC nanocoatings (Xia et al., 2020)

An electrodeposition technique that discharges the cathode is the most effective way to create nanostructure coatings. Among the variables that could influence nucleation on the cathode are the crystal structure of the substrate, surface free energy, adhesion energy, crystal orientation of the electrode surface, and the substrate's crystallographic lattice (Ghazanlou et al., 2016). Electroplating is commonly employed for low-range corrosion and wear protection as well as decorative coatings. While it is also possible to install high-temperature resistant biological and ceramic coatings by raising the potential difference in the cell (Karmakar et al., 2021).

This technique allows modifying the sizes, forms, and structures of the electrocatalysts on the surface of conducting materials by adjusting the electrochemical deposition conditions. This process is done to change the physical properties of metal such as corrosion protection and increase the wear resistance of a metal. Generally, direct current electrodeposition is used for plating. However, low direct current causes plating to result in coatings with larger grain sizes, while high direct current causes all the bath electrolytes to be absorbed and results in high metal deposition, which produces coating surfaces with roughness (Kumar Singh et al., 2019).

2.4.2 Pulse Current Electrodeposition Method

Pulse current electrodeposition allows for regulated current between two pulses which offers greater advantages than direct current deposition. It is because the coating's enhanced physical and chemical properties. The microhardness of composite coatings was directly impacted by their matrix grain sizes during pulse current electrodeposition (Ma et al., 2020). In addition to that, Kumar Singh et al., (2019) noted that the pulse current electrodeposition method produces better results than the direct current electrodeposition method in terms of fine grain deposition, reduced coating porosity, improved morphologies, hardness, and wear resistance.

The preparation of nickel plating by electrodeposition still has several problems. In truth, this process produces size and surface effects that yield properties that are significantly different from those of the bulk material when the grain size reaches the nanoscale. Furthermore, during electrodeposition, process variables can also change the plating's size and crystal structure, which might impact its mechanical qualities (Wang et al., 2021). Parameters of the electrodeposition that affect coating thickness and surface morphology include bath temperature, electrodeposition time, solution pH, and current density. Since electroplating parameters greatly affect hydrogen evaluation, deposition thickness, and deposition efficiency, they must be carefully managed during the deposition process (Rai & Gupta, 2021).

The goal of pulsed electrodeposition is to create coatings with improved mechanical and anticorrosion qualities. When compared to direct current electro-plating, pulse plating has been shown to increase corrosion resistance and reduce grain size, therefore improving coating quality (Arunsunai Kumar et al., 2013). Furthermore, it was stated that pulse electro- deposition allowed for more control over the composition of the deposit since it was possible to refine the coating's grain down to the nanoscale by increasing the Ni concentration (Kamnerdkhag et al., 2017).

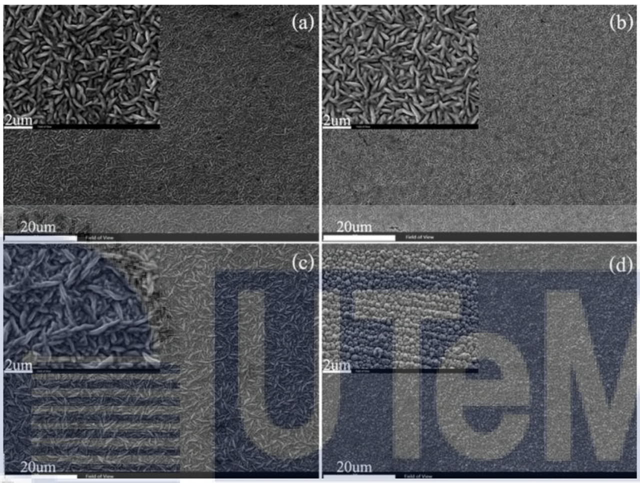


Figure 2.3: SEM images of pulsed Zn-Ni-Al_2O_3 composite electrodeposits at (a) 33% duty cycle; (b) 50% duty cycle; (c) 67% duty cycle; and (d) DC plating (Kamnerdkhag et al., 2017)

Arunsunai Kumar et al., (2013) discovered that pulse current electrodeposition controls more aspects of the electrodeposit's structure and characteristics such as ability to regulate pulse parameters compared to the direct current electrodeposition. Therefore, in contrast to direct current electrodeposition, coatings electrodeposited using pulse current electrodeposition have excellent mechanical and tribological properties. For example, Kamnerdkhag et al., (2017) found that in their study of pulse current electrodeposition of Zn-Ni-Al_2O_3 composite coatings, it was possible to refine the coating's grain down to the nanoscale by increasing the Ni concentration, which allowed for more control over the composition of the deposit. Therefore, pulse current electrodeposition has been shown to increase corrosion resistance and reduce grain size which improves the coating quality.

In another study, Fan et al., (2016) examined the characteristics of Ni-W-GO composite coatings and pulse current electrodeposition. The studies showed that pulse current electrodeposition enhanced the composite coatings' wear resistance and microhardness. Thus, this indicates that pulse current electrodeposition may improve the characteristics of nickel composite coatings.

Pulsed current deposition produced composite coatings with better particle dispersion and a greater loaded percentage of incorporating particles. The wear data indicated that an increase in current density in direct current plated coatings was responsible for a decrease in wear rate. In the case of the pulse current plated composite coating, a negligible effect on the wear rate was seen with increased current density (Bajwa et al., 2016).

Xia et al., (2021) noted that pulse current electrodeposition is more cost- effective compared to brushing electroplating, reverse pulse electrodeposition, and direct current electrodeposition when it comes to deposition. Other than that, pulse current electrodeposition also offers simple operation, dependability, great efficiency, and safety. Studies on pulse current electrodeposition for nickel coating have demonstrated that it is a useful technique for improving the properties of various coatings. Parameters related to pulse current electrodeposition have an impact on the microstructure and mechanical properties of coatings. Thus, this study will examine how pulse current electrodeposition parameters, like duty cycle and current density, affect coating performance.

2.5 Parameters of Pulse Current Electrodeposition

2.5.1 Current Density

One of the parameters in pulse current electrodeposition that influences the microstructure, shape, composition, and properties of coatings is current density. Doğan et al. (2020) highlighted that one of the factors affecting the mechanical, tribological, microstructural, and corrosion properties of the Ni-B/TiN composite coatings during deposition is current density. Therefore, to achieve surface coatings of high quality, current density needs to be regulated.

Among many other desirable properties, pulsed current electrodeposition allows for the creation of deposited materials with improved adhesion, fine-grained structures, and enhanced corrosion resistance (Liu et al., 2021). The current density is one of the variables that affects the nucleation and growth of coating grains. There's a chance that the grains will become finer as the current density rises, which might eventually improve the mechanical qualities. Good corrosion resistance can often be obtained at current densities between moderate and high. Smoother surfaces could be the result of higher current densities.

Kasturibai & Paruthimal Kalaignan, (2014) examined the corrosion characteristics of Ni-Si_3N_4 nanocomposite coatings with pulse electrodeposition. As compared to pure Ni coatings, they found that Ni-Si_3N_4 composite had excellent wear and corrosion resistance. In another study, Yasin et al., (2018) studied how current density affects the mechanical characteristics and morphology of nickel-graphene composite coatings. They found that coatings' corrosion resistance and mechanical quality improved with increased current density. The result of hardness and corrosion resistance of the study by Yasin et al., (2018) are shown in Figure 2.4 and Figure 2.5.

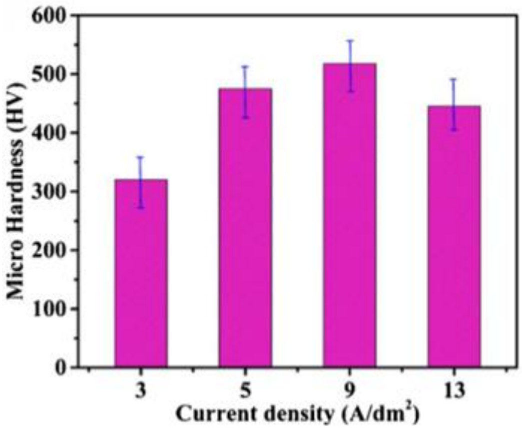


Figure 2.4: Micro hardness of Ni- Graphene composite coating deposited at different current densities (Yasin et al., 2018)

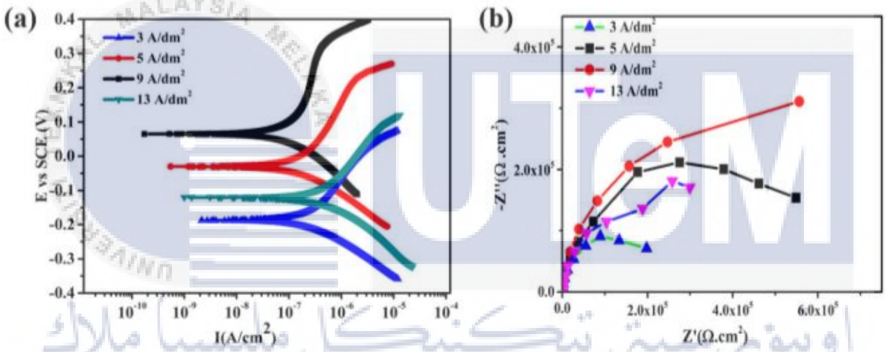


Figure 2.5: Corrosion resistance of Ni- Graphene composite coating deposited at different current densities a) effect of current density on the polarization curves and b) EIS spectra of nickel-graphene composite coatings in 3.5% NaCl (Yasin et al., 2018)

2.5.2 Duty Cycle

The quantity of energy received by the coating varies with the duty cycle. The duty cycle may have an impact on the material's hardness and wear resistance. Duty cycle control could result in uniform coatings by adjusting the duration of the metal deposition process as well as the rest intervals. Carefully regulating the amount of time of the "on" and "off" intervals will result in a consistent and dependable coating deposition throughout the substrate. Higher current densities expedite the deposition process, which results in thick coatings. Thicker coatings are produced at lower current densities.

Duty cycles are another factor that affects the characteristics and qualities of coatings. Duty cycle is the ratio of composite electrodeposition's on to off times. The duty cycle can affect the coatings' microstructure, hardness, wear resistance, and corrosion behavior. Gupta et al., (2016) found that coatings with a 30% duty cycle had better wear resistance and hardness than coatings with other duty cycles during the pulse electrodeposition of Ni-WC composite coatings. This suggested that the selection of duty cycles affected the characteristics of composite coatings. Many studies have examined the effects of duty cycle on the characteristics of nickel composite coatings.

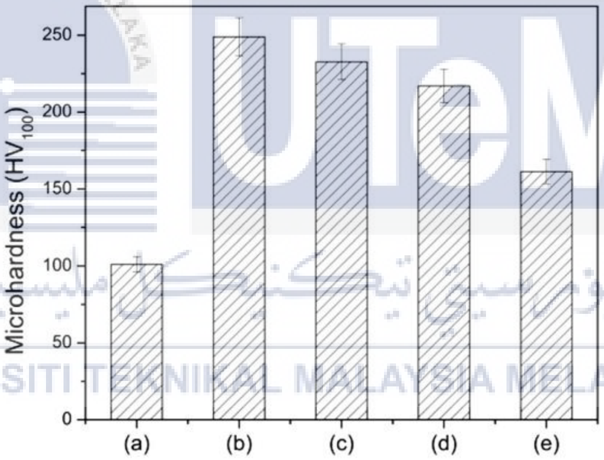


Figure 2.6: Micro-hardness of a mild steel and the electrodeposited Zn-Ni-Al_2O_3 composite coating at (a) a mild steel, (b) pulsed current at 33% duty cycle, (c) pulsed current at 50% duty cycle, (d) pulsed current at 67% duty cycle and (e) DC plating ((Kamnerdkhag et al., 2017)

Sharma et al., (2014) investigated the impact of pulse electrodeposition settings on pure tin coating morphology. Another study by Xue et al., (2017) on how the pulse duty cycle affected the microstructure and mechanical characteristics of coatings made of nickel- graphene composites that were formed by pulse electrodeposition in supercritical carbon dioxide. Both studies showed that higher duty cycle results in finer microstructures and increasing hardness in the composite coating. Figure 2.6 shows the average friction coefficient of the Ni-GO composite coating produced at different duty cycles.

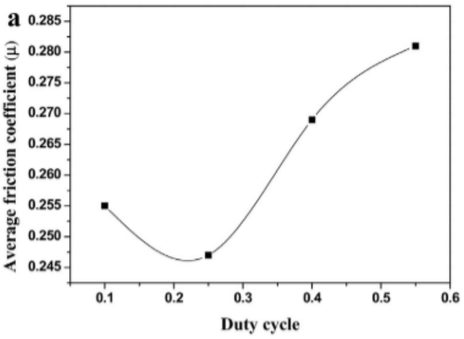


Figure 2.7: The average friction coefficient of the Ni-GO composite coating produced at different duty cycle (Xue et al., 2017)

2.6 Properties of Composite Coating

2.6.1 Hardness

Pulse current electrodeposition of nickel composite coatings results in improved hardness and wear resistance. According to Li et al., (2019) studied the characteristics and microstructure of composite coatings made of Ni-B and SiC. The study shows that the hardness of coatings increased with the increasing of SiC particles. The amount of SiC and B both decreased when the current density increased.

Yasin et al., (2018) investigated the morphology, mechanical characteristics, and corrosion performance of nickel-graphene nanocomposite coatings affected by the deposition current density. The study showed that current density and hardness have a correlation in increasing hardness of nickel-graphene composite coatings.

Sharma et al., (2014) examined how the parameters of the pulse electrodeposition affected the pure tin coatings' morphology. The study discovered that the size of coatings grain decreased as the duty cycle increased. Grain size influenced the mechanical properties of coatings, so duty cycle influences the hardness of the coatings. Another study by Xue et al., (2017) examined how the pulse duty cycle affected the microstructure and mechanical characteristics of coatings made of nickel- graphene composites created by pulse electrodeposition in supercritical carbon dioxide. The study shows that when the duty cycle increased, the hardness and tensile strength of the coatings increased. These results implied that duty cycle influences the hardness of the coatings.

2.6.2 Corrosion Resistance

Nickel composite coatings have drawn a lot of attention in the corrosion protection industry because nickel composite coatings have better qualities than pure nickel coatings. These composite coatings are commonly deposited using pulse current electrodeposition, which provides control over the deposition process and enhances corrosion resistance.

Yasin et al., (2018) presented that the corrosion resistance of nickel-graphene composite coatings was influenced by the current density. Corrosion resistance increased with the increasing of current density. According to Doğan et al., (2020) investigated the corrosion resistance of Ni-B/TiN composite coatings. The studied shows that increased corrosion resistance at a current density of 4.5 A/dm^2. Therefore, both studies implied that corrosion resistance of composite coatings improved by increasing the current density.

In a different study, Xia et al., (2021) examined the impact of duty cycles on the Ni-TiN/SiC nanocoating’s' ability to withstand corrosion. The study discovered that coatings made with different duty cycles and a pulse current density of 4 A/dm^2 had exceptional corrosion resistance. Thus, this indicated that the ability of composite coatings to withstand corrosion resistance improved by increased duty cycles during pulse current electrodeposition.

2.6.3 Wear Resistance

Numerous studies have examined the relationship between wear resistance and current density in composite coatings. Jegan and Venkatesan, (2013) state that Ni/nano- Al_2O_3 composite coatings made by pulse current electrodeposition first had better hardness, wear resistance, and electrochemical corrosion resistance when the current density was increased. However, when the current density increased, there came a value at which the hardness, wear resistance, and corrosion resistance all decreased.

Another study by Doğan et al., (2020) reported that the wear resistance and deposition mechanism of Ni-B/TiN composite coatings are significantly influenced by the current density. The study found that when thickness of coatings increased, the current density increased, and wear resistance decreased.

A great deal of research has been done on the characteristics of composite coatings made by pulse current electrodeposition yet none of these studies has particularly looked at how duty cycle affects wear resistance. Gupta et al., (2016) examined and assessed the characteristics of Ni-WC composite coatings created by pulse electrodeposition. According to Ahmadiyeh et al., (2019), investigated the mechanical characteristics and electrochemistry of coatings made of Ni-B/SiC nanocomposite produced by pulse current electrodeposition. Both studies did not address the impact of duty cycle on wear resistance, even though they offered insightful information about the performance of these coatings. Therefore, further study of how duty cycle influences the wear resistance of composite coatings will be investigated in this project.

CHAPTER 3

METHODOLOGY

This chapter presents the proposed approach for the research, which includes the main procedures that will be used to complete the study. Before providing the materials for the design, processing, testing, and material selection, the specification and details of the previous study will be thoroughly examined. Offering suitable methodologies, recommended equipment, and approaches to complete this investigation is the primary principle of methodology.

3.1 Overview

The purpose of the methodology preparation is to ensure that the electrodeposition of the nickel-quarry dust composite coatings processes proceed according to timelines and objectives. The procedures are put up in accordance with the research scopes to achieve the established goal. Besides, all procedures involving standard instruments, techniques, and specimen testing are measured against the American Society for Testing and Materials (ASTM). Furthermore, the parameters for the operations are likewise predicated on earlier studies. The whole procedure to achieve the expected result starting from the sample preparation, electrodeposition process, characterization of coating, testing, and data analysis are illustrated in the flow chart in Figure 3.1.

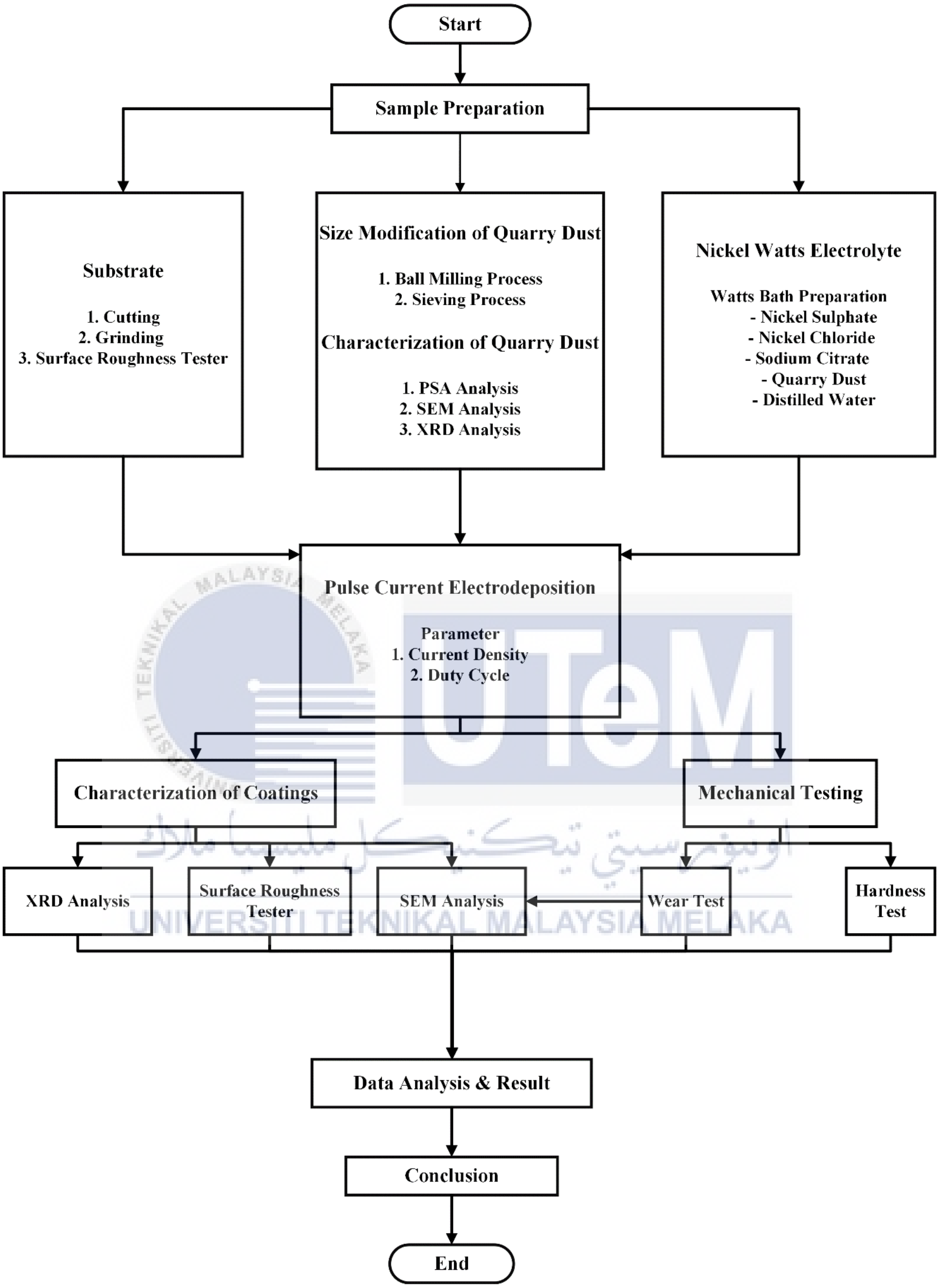


Figure 3.1: Flow chart of experiment

3.2 Preparation of Substrate

3.2.1 Machining Process of Substrate

Mild steel with a thickness of 3 mm were cut into a dimension of 40 mm x 30 mm by using laser cutting machine. Mild steels were then prepared for 20 samples labeled as A1 until D5. Figure 3.2 shows the sample size of mild steel.

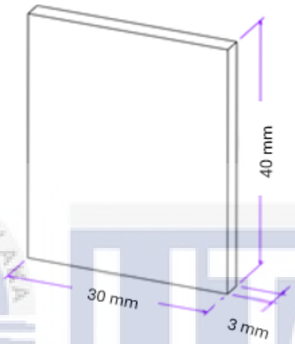


Figure 3.2: Sample size of mild steel

3.2.2 Grinding Process of Substrate

The laser cutting process leaves burrs behind on the surface of the mild steel, thus grinding and polishing were done to have a smooth surface finish. The mild steel was prepared by grinding and polishing the surface with grind using emery grade paper at a speed of 250 rpm. Figure 3.3 shows the grinding machine used in this project. Different grades of emery grade paper started with 240, 400, 600, 800 and 1200 were used. Then, the mild steel was washed by using distilled water and acetone. After the cleaning process, the sample was sent for roughness test.

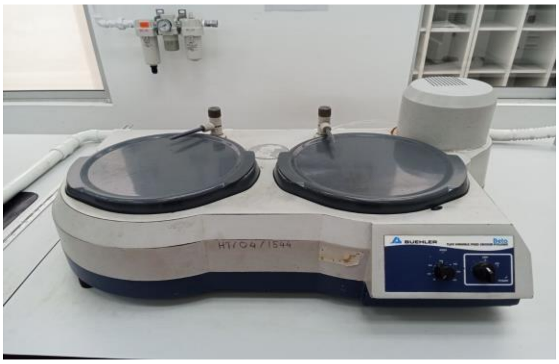


Figure 3.3: Grinding machine

3.3 Preparation of Quarry Dust

3.3.1 Ball Milling Process

The quarry dust was obtained from a quarry in Negeri Sembilan. Quarry dust was crushed to a powder form using a planetary ball mill machine (Retsch PM100). Figure 3.4 shows the planetary ball mill machine. The quarry dust was ball milled for 5 hours with rotational speed of 350 rpm. Alumina jars and alumina balls were used to avoid any contamination during the ball milling process.

Figure 3.4: Planetary ball mill machine

3.3.2 Sieving Process

The grounded quarry dust then was sieved by using siever. Figure 3.5 shows the sieving equipment. The grounded quarry dust was pass through 63 µm sieve. The range of size of quarry dust used in this project is 1-63 µm. The sieving process is done to make sure the size of the quarry dust particles is constant because the size of the quarry dust particles will affect the performance of the material deposit on the substrate.

Figure 3.5: Sieving equipment

3.3.3 Characterization of Quarry Dust

After the quarry dust is sieved to 63 µm, the particles of quarry dust were then sent to particle size analyser (PSA) to check the particle size compare with particle size before sieving process. The surface morphology and structure of the quarry dust were analyzed by using scanning electron microscope (SEM) and x-ray diffractometer (XRD).

3.4 Preparation of Nickel Watt's Bath

To prepare the electrolyte for electrodeposition process, Watts Bath composition was used. The composition of Watts Bath is listed in Table 3.1.

Table 3.1: Bath composition

Watts Bath Composition	
Nickel Sulphate, $NiSO_4$	200 g/L
Nickel Chloride, NiCl	20 g/L
Sodium Citrate, $Na_3C_6H_5O_7$	30 g/L
Quarry Dust	20 g/L
Distilled Water	1L

3.5 Electrodeposition of Nickel-Quarry Dust Composite Coatings

Figure 3.6 shows the electrodeposition setup. The electrodeposition experiment was carried out in a double layer glass cell. The double layer of glass cell was filled with electrolyte in one layer and water in another layer of the glass cell. The thermostatic water bath is to ensure that the plating temperature is 40 °C. Th nickel plate acting as anode was dipped into the center hole of the glass cell while substrate which acts as cathode was attached to the right side of the glass cell. 2 g of quarry dust was added to the electrolyte and the electrolyte was stirred using magnetic stirrer for 10 minutes before the electrodeposition started. The electrolyte solution was stirred at constant speed during the electrodeposition process. The magnetic stirrer will prevent agglomeration of quarry dust particles and help them embedded to substrate. The electroplating time is 1 hour, and the plating parameters are listed in Table 3.2. The experiment is repeated with 20 different samples as shown in Table 3.3.

Table 3.2: Plating parameters

Plating Parameters	Condition
Plating Temperature, °C	40
Pulse Current Density, A/dm^2	1, 3, 5, 7, 9
Duty Cycle, %	25, 50, 75
Electroplating Time, min	60
Stirring Speed, rpm	250

Table 3.3: Parameter matrix table

Current Density Duty Cycle	1 A/dm^2	3 A/dm^2	5 A/dm^2	7 A/dm^2	9 A/dm^2
DC	A1	A2	A3	A4	A5
25%	B1	B2	B3	B4	B5
50%	C1	C2	C3	C4	C5
75%	D1	D2	D3	D4	D5

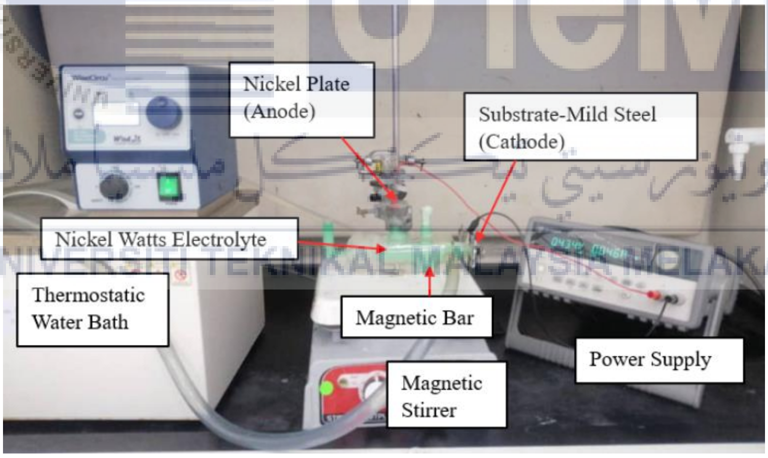


Figure 3.6: Electrodeposition setup

The electrodeposition parameters in this project are current density and duty cycle. Various duty cycles and current densities are set to investigate the effect of duty cycle and current density on the Ni-QD composite coating properties. The value of current density set was 1 A/dm^2, 3 A/dm^2, 5A/dm^2, 7 A/dm^2 and 9 A/dm^2 meanwhile the value of duty cycle set was 25%, 50% and 75%. Duty cycle refers to the ratio of the time that the current is applied (the pulse on time) to the total time of the pulse (the pulse on time plus the pulse off time) during electrodeposition. For 25% duty cycle, the T_{ON} is 250s and the T_{OFF} is 750s for one cycle. For 50% duty cycle, the T_{ON} is 500s and the T_{OFF} is 500s for one cycle. For 75% duty cycle, the T_{ON} is 750s and the T_{OFF} is 250s for one cycle. The cycle is continuous for 1 hour to complete the electrodeposition process. Figure 3.7 shows the rectangular pulse waveform.

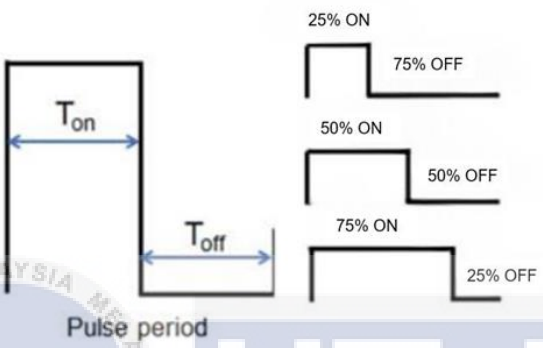


Figure 3.7: Rectangular pulse waveform

3.6 Characterization of Nickel-Quarry Dust Composite Coatings

3.6.1 Surface Morphology of Nickel-Quarry Dust Composite Coatings

Surface morphology and microstructures of the Nickel-Quarry Dust coatings were observed by scanning electron microscope (SEM, ASTM E986) shows in Figure 3.8. SEM shows the average size of the embedded particles inside the coating layers.

Figure 3.8: SEM machine

3.6.2 Structure and Surface Composition of Nickel-Quarry Dust Composite Coatings

The crystalline structures and surface compositions of the nickel and quarry dust were evaluated using X-ray diffractometer (XRD). Figure 3.9 shows XRD machine. The effect of duty cycle on the phase structure of deposited composite coatings were analyzed.

Figure 3.9: XRD machine

3.7 Testing on Nickel- Quarry Dust Composite Coatings

3.7.1 Hardness Testing

The hardness of the coatings was analyzed using a Micro-Vickers tester (ASTM E384) at FTKM. The indenter is placed in 5 different positions on the surface coating using 100g load and a holding time of 15s. The average value of 5 indentations was chosen for the value of hardness in this project.

3.7.2 Wear Testing

The wear behavior of the coating was examined using a Micro Pin on Disk Tribo Tester (CM-9109, ASTM G99). A pin which is 10 mm stainless steel is loaded with 10 N against a flat rotating disc specimen. The specimen is loaded by sliding the pin on the disc at a for 1500 seconds, with a stroke of 2.69 mm and the velocity of friction is set at 5 mm/s. The analysis of the coating's wear behavior involves studying the coefficient of friction (COF) generated during the reciprocal sliding of the ball against the coating layer. The worn morphology was observed using SEM and XRD.

Figure 3.10: Micro pin on disk tribo-tester machine

CHAPTER 4

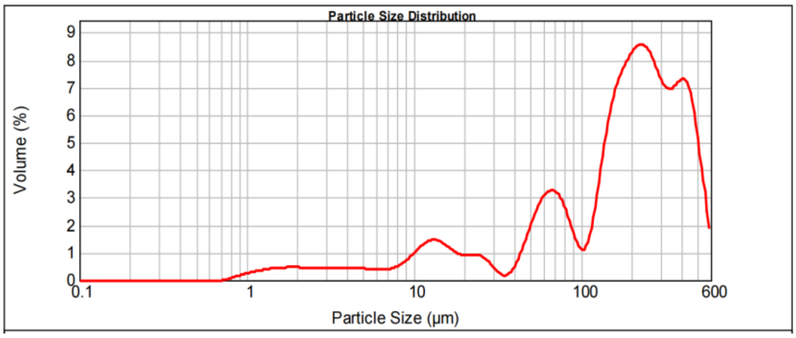
RESULT AND DISCUSSION

This chapter mainly discussed the results obtained after completing the experiment and testing. All the data and findings from Particle size analyzer (PSA), Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), pin on disc machine and micro-Vickers will be analyzed and supported by previous research statement to discuss whether the objectives of the project are achieved.

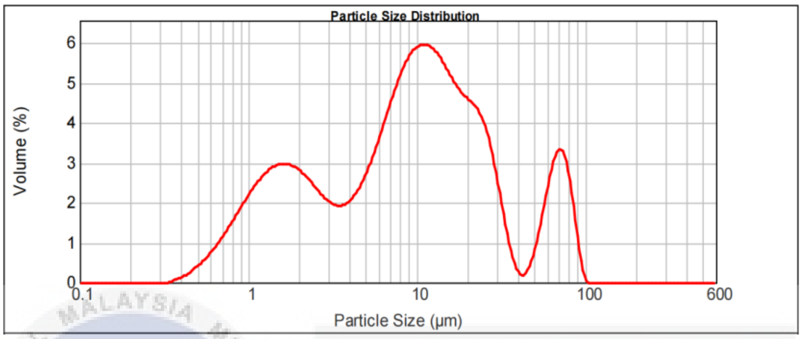
4.1 Characterization of Quarry Dust

4.1.1 Size Modification of Quarry Dust

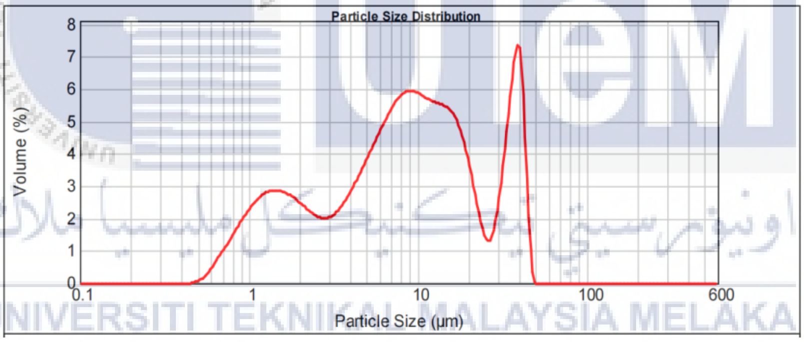
The particles size of quarry dust as received, after 5 hours ball milling and after sieving process are shown in Figure 4.1. The grain size range of quarry dust particles as received is 0.72 µm to 630.96 µm and the grain size range of quarry dust particles after the ball milling process is 0.42 µm to 104.71 µm. Based on the Figure 4.1 (a), the trend shows that the distribution of the particles sizes of quarry dust is on the right which indicates larger particles of quarry dust while based on the Figure 4.1 (b), the trend moves to the left which indicates that the particles sizes of quarry dust has become smaller. This comes from the balls and quarry dust colliding during the ball milling process. However, after the sieving process the range of quarry dust particles is from 0.48 µm to 60.26 µm which is confirmed the range size of quarry dust to be used in this project is from 1-63 µm.



(a)



(b)

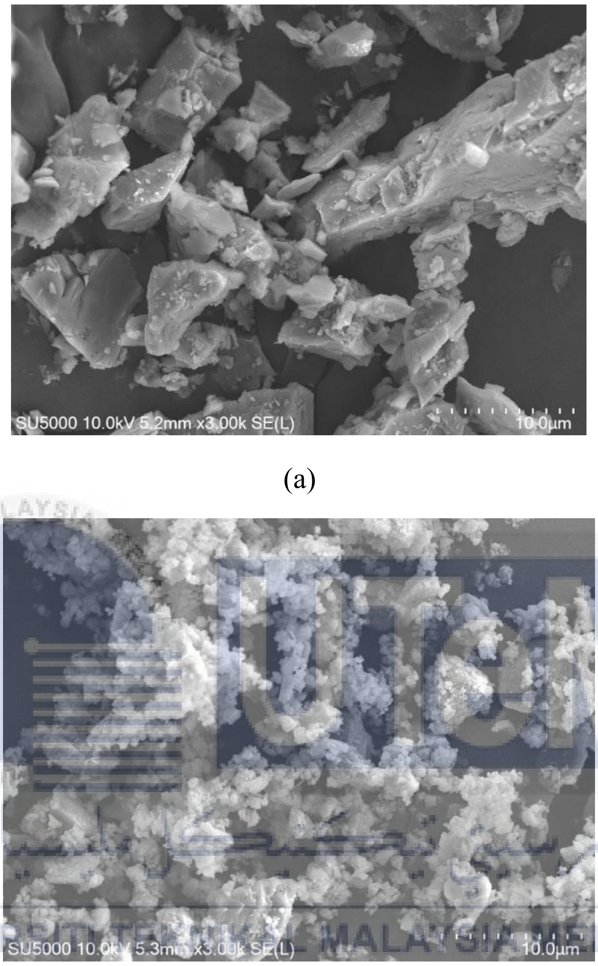


(c)

Figure 4.1: Particle size distribution of quarry dust particles (a) as received (b) after ball milling for 5 hours (c) after sieving

4.1.2 Surface Morphology of Quarry Dust

SEM was used to examine the surface morphology of the quarry dust particles. Figure 4.2 shows the quarry dust particles observed under SEM (a) as received and (b) after ball milling and sieving process. The particles sizes of quarry dust as received are irregular in shape and sizes. The particles sizes are irregular and finer after ball milling and sieving process due to the collision between the alumina balls and quarry dust particles during the ball milling process (Othman et al., 2019).



(a)

(b)

Figure 4.2: Quarry dust particles observed under SEM (a) as received (b) after ball milling and sieving process

4.1.3 Phase Analysis of Quarry Dust

Figure 4.3 shows the intensity of Al_2O_3 and SiO_2 contain in the quarry dust. Silica shows the highest peak of intensity compared to alumina. The XRD result displayed in Figure 4.3 confirms the presence of silica and alumina in quarry dust. Quarry dust particles contain high amounts of silica and alumina (Othman et al., 2019).

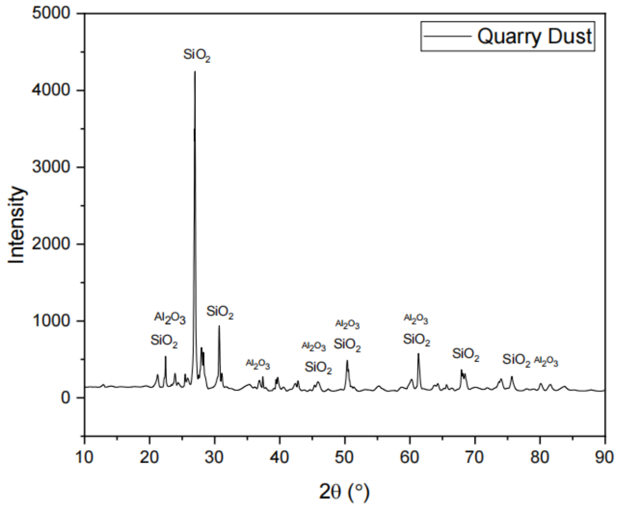


Figure 4.3: XRD analysis of quarry dust

Elemental composition of quarry dust was observed by using X-Ray Fluorescence (XRF). Table 4.1 shows the elemental composition of quarry dust. Silica and alumina are the highest of elemental composition that have been found in quarry dust particles by XRF analysis. Therefore, XRF analysis result is significant with the XRD analysis as shown in Figure 4.2 which shows that the peak of Al_2O_3 and SiO_2 are produced in the phase analysis.

Table 4.1: Elemental composition of quarry dust

Element	Concentration (wt%)
Silica (SiO_2)	72.6
Alumina (Al_2O_3)	15.1
Potassium Oxide (K_2O)	4.9
Sodium Oxide (Na_2O)	3.0
Ferric Oxide (Fe_2O_3)	1.9
Calcium Oxide (CaO)	1.1
Magnesium Oxide (MgO)	0.8
Titanium Dioxide (TiO_2)	0.3
Sulphur Trioxide (SO_3)	0.2
Phosphorus Pentoxide (P_2O_5)	0.1

4.2: Characterization of Mild Steel

4.2.1 Chemical Composition of Mild Steel

The chemical composition of the mild steel is done by using Energy Dispersive X-Ray (EDX). Table 4.2 shows the composition of the mild steel which are iron (76.9%), carbon (20.1%) and oxide (3.0%).

Table 4.2: Composition of mild steel

Element	**Composition (wt. %)**
Iron (Fe)	76.9
Carbon (C)	20.1
Oxide (O)	3.0

4.2.2 Surface Roughness of Mild Steel

Figure 4.4 shows the 3D surface roughness of mild steel. The surface roughness of mild steel shows that it has more red and green color. The red color indicates that the surface roughness is high while the green color indicates that the surface roughness is at intermediate level. The average roughness of the mild steel is 11.12 µm.

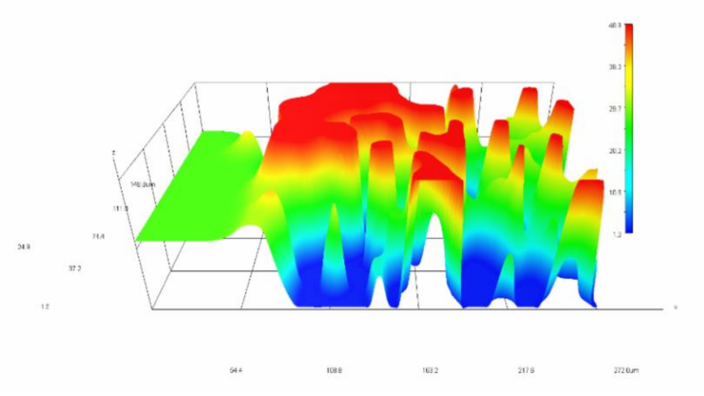
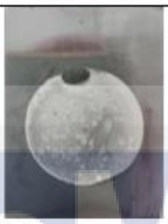


Figure 4.4: 3D surface roughness of mild steel

4.3 Characterization of Ni-QD Composite Coating

Mild steel had been coated with a Ni-QD composite using different duty cycles and current densities. The exposed area of nickel to the coating is 2.25 cm^2. The electrodeposited Ni-QD composite coating on mild steel at different duty cycles and current densities is displayed in Table 4.3.

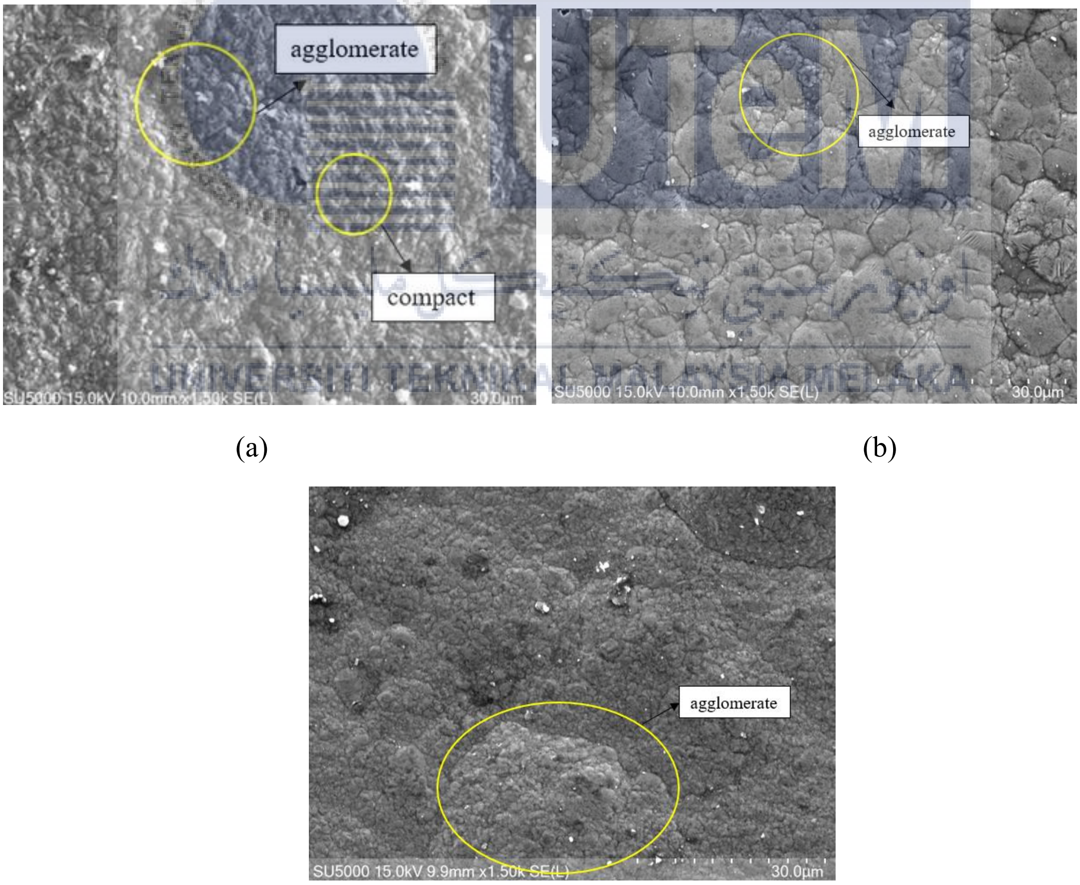
Table 4.3: Electrodeposited of Ni-QD composite coating on mild steel with various current density and duty cycle

Duty Cycle (%)	Current Density (A/dm^2)				
	1	3	5	7	9
DC					
25					
50					
75					

4.4 Effect of Current density and Duty Cyle to Nickel-Quarry Dust Composite Coating

4.4.1 Surface Morphology of Nickel-Quarry Dust Composite Coating

SEM micrograph on Ni-QD composite coating produced using direct current mode with various current density are shown in Figure 4.5. Figure 4.5 (a) shows the grain sizes of coating are compact and agglomerated while Figure 4.5 (b) shows agglomerated particles with coarser grains. Figure 4.5 (c) shows that the grain sizes are larger and agglomerated. When current density is increasing, the deposition rate becomes faster which has led to agglomeration particles. This is aligned to the research by (Raghavendra et al., 2016). These agglomerates are typically attributed to randomly dispersed Al_2O_3 and SiO_2 aggregates on the composite (Rosolymou et al., 2024).

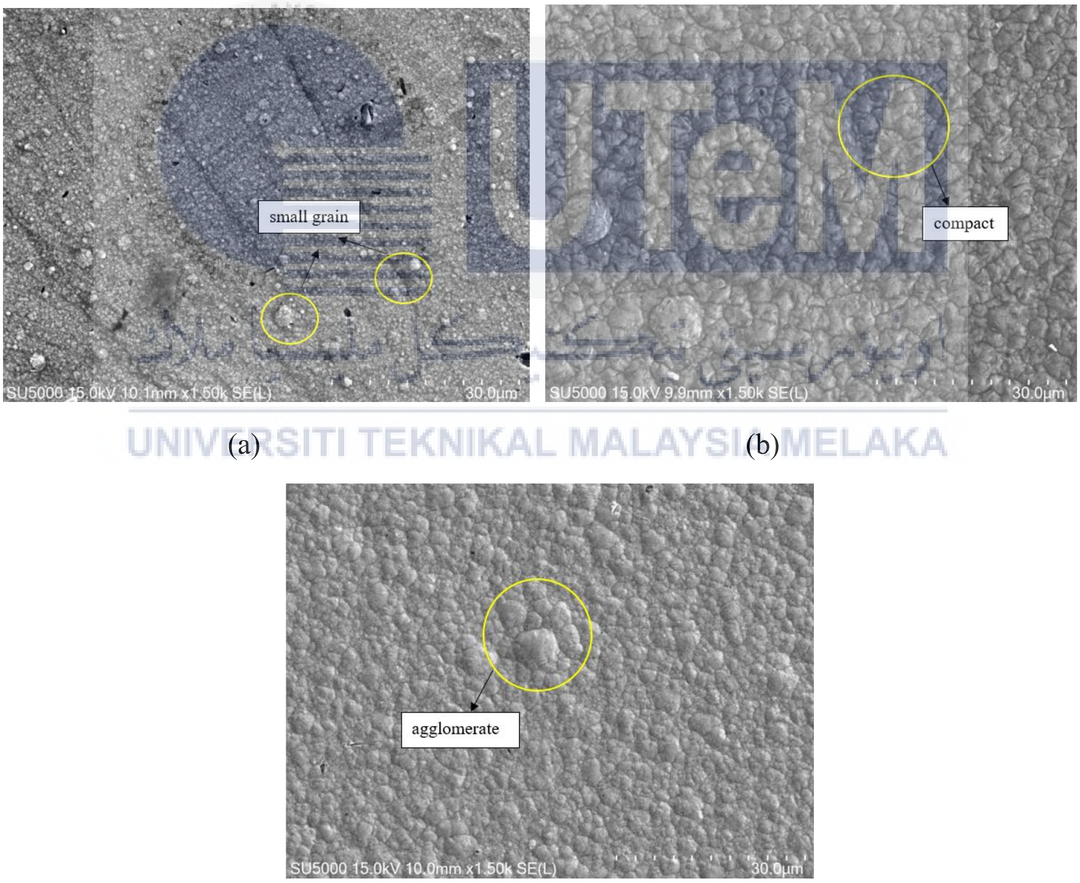


(a) (b)

(c)

Figure 4.5: SEM micrograph on Ni-QD composite coating produced using direct current mode with various current density (a) 1 A/dm^2, (b) 5 A/dm^2 and (c) 9 A/dm^2

SEM micrograph on Ni-QD composite coating produced at 25% duty cycle with various current density are shown in Figure 4.6. Figure 4.6 (a) shows numerous small-sized grains formed on the Ni-QD composite coating. Figure 4.6 (b) shows a uniform, smooth and fine grain structures. Figure 4.6 (c) shows the grain structure is not uniform and severe agglomerated particles are observed compared to Figure 4.6 (b). The electrolyte solution's nanoparticles will be drawn to and embedded in the cathode surface during the T_{ON} of pulse electrodeposition coating. Loosely embedded and agglomerated particles will be returned away from the cathode surface and return to the electrolytic solution during T_{OFF}, when the current supply is shut off. From now on, this procedure will be repeated. A reduction in the duty cycle will result in an increase in the T_{OFF} time, which will lessen particle aggregation and produce finer grains (John et al., 2023).

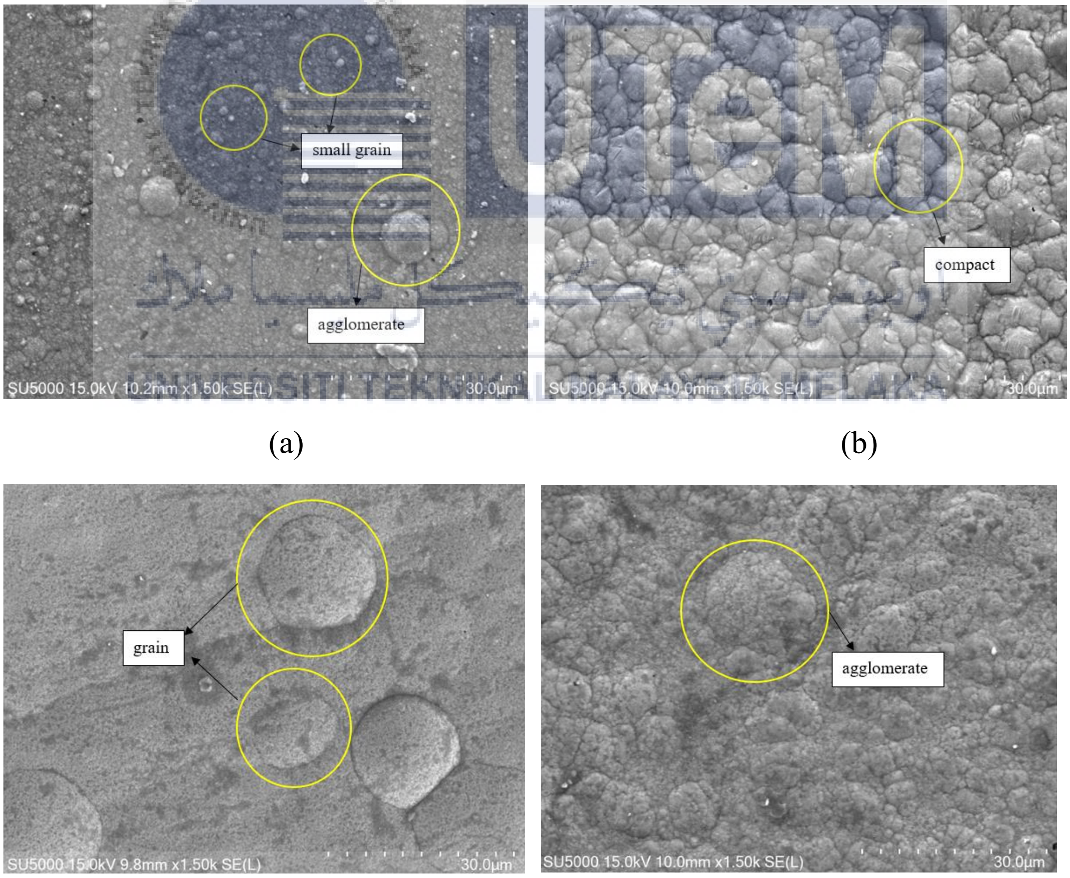


(a) (b)

(c)

Figure 4.6: SEM micrograph on Ni-QD composite coating produced at 25% duty cycle with various current density (a) 1 A/dm^2, (b) 5 A/dm^2 and (c) 9 A/dm^2

SEM micrograph on Ni-QD composite coating produced at 50% duty cycle with various current density are shown in Figure 4.7. Figure 4.7 (a) shows numerous small grain sizes formed on the coating surface. The growth and nucleation of the grains is limited due to the low current density (Sherwin et al., 2021). Figure 4.7 (b) shows a larger, compact and dense grain sizes formed. Figure 4.7 (c) shows grain sizes formed on a layer of a coating. Figure 4.7 (d) shows coarser grain sizes with uneven dispersion due to the agglomeration of particles. At a pulse current density of 6 A/dm^2, the thickness of the hydrogen evolution layer increased due to the significant amounts of hydrogen bubbles that formed at the cathode surface. Consequently, the amount of embedded Al_2O_3 and SiO_2 nanoparticles in Ni-QD nanocoating decreased because of the reduction of the cathode surface's growth of SiO_2 and Al_2O_3 nanoparticles. Additionally, this reduced the nickel grains' inhibitory impact and increased their grain sizes (Xia et al., 2020).

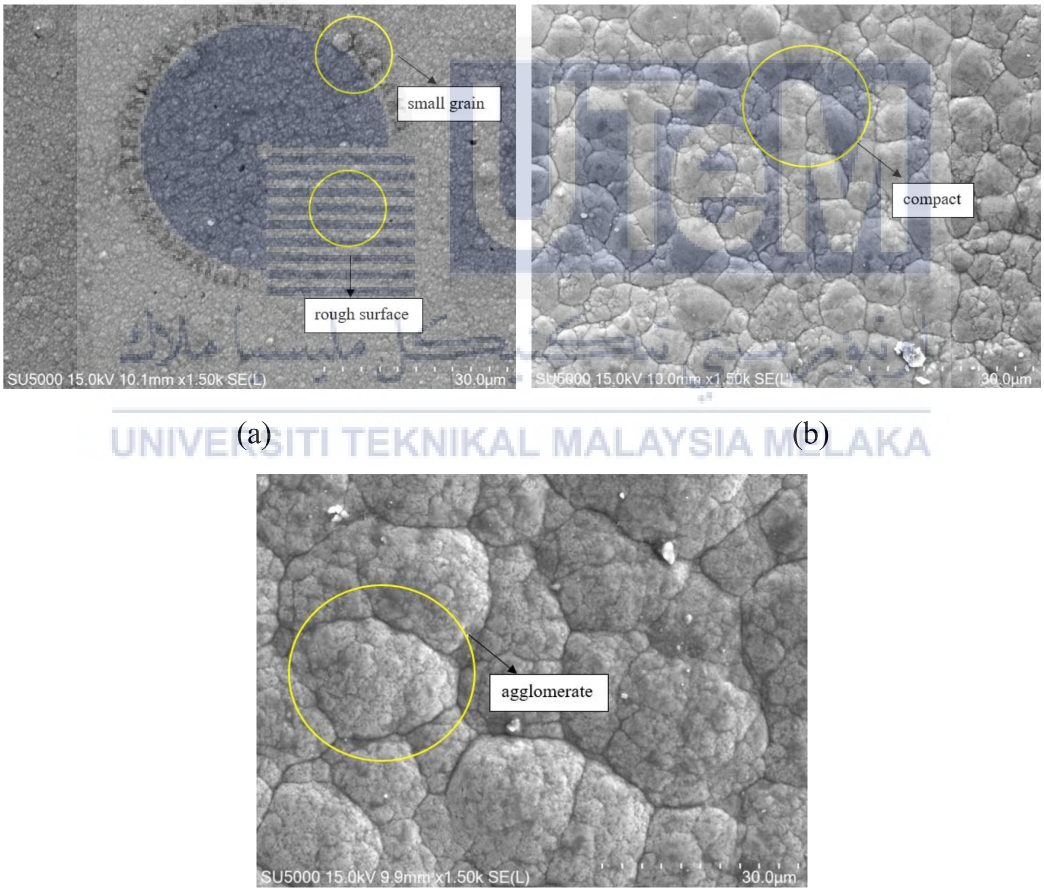


(a) (b)

(c) (d)

Figure 4.7: SEM micrograph on Ni-QD composite coating produced at 50% duty cycle with various current density (a) 1 A/dm^2, (b) 5 A/dm^2, (c) 7 A/dm^2 and (d) 9 A/dm^2

Figure 4.8 shows SEM micrograph on Ni-QD composite coating produced at 75% duty cycle with various current density. Figure 4.8 (a) shows numerous small grains sizes formed on the surface of coating. Low current density causes the coating to contain fewer Al_2O_3 and SiO_2 nanoparticles. Therefore, nickel grain growth was not considerably inhibited by Al_2O_3 or SiO_2 nanoparticles, leading to the creation of multiple large-sized grains and free nickel grain growth (Xia et al., 2020). Figure 4.8 (b) shows a medium coarse grain with evenly distributed particles. Figure 4.8 (c) shows a larger with agglomerated grain sizes. Poor Al_2O_3 and SiO_2 particles could reach double layers more quickly, therefore the number of particles in the alloy matrix decreased as the duty cycle was increased further. However, due to the particles' agglomeration through the brief duration, larger particles did form on the coated surface (Doğan et al., 2023).



(a) (b)

(c)

Figure 4.8: SEM micrograph on Ni-QD composite coating produced at 75% duty cycle with various current density (a) 1 A/dm^2, (b) 5 A/dm^{2}and (c) 9 A/dm^2

4.4.2 Effect of Current Density and Duty Cycle on XRD Pattern of Composite Coating (Phase Analysis)

Figure 4.9 shows the XRD phase analysis of Ni-QD composite coatings produced using 3 A/dm^2 current density at various duty cycles. The highest peak shown is the peak of nickel which is at 52°. This result is aligned with the research by (Xia et al., 2020). The lowest peak shown is the peak of alumina and silica which is at 44°. The presence of alumina and silica peak in the nickel composite coatings suggests that the deposits' nickel matrix has effectively incorporated with the quarry dust particles (Othman et al., 2019).

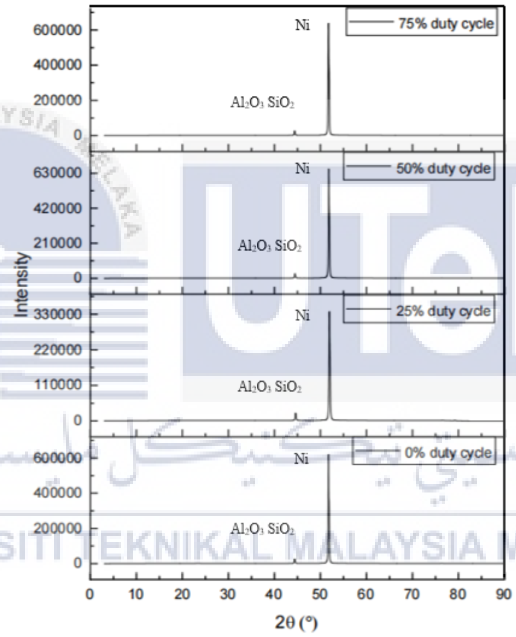


Figure 4.9: XRD phase analysis of Ni-QD composite coatings produced using 3 A/dm^2 current density at various duty cycles

Figure 4.10 shows the XRD phase analysis of Ni-QD composite coatings produced using 50% duty cycle at various current densities. Nickel shows the highest peak of intensity which is at 52° for five different current density. The intensity for alumina and silica shown in the figure fluctuated trend. High current density can cause uneven coating thickness, non-uniform deposition, and variations in the distribution of Ni-QD composite on the substrate. These non-uniformities may result in variations in diffraction intensity at various locations in the coating. The particle is also possible to aggregate which leads to specific regions with higher particle concentrations or uneven distribution.

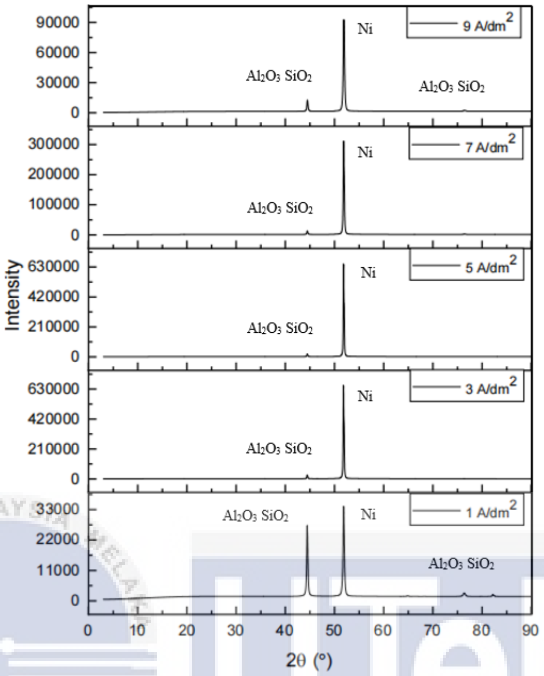


Figure 4.10: XRD phase analysis of Ni-QD composite coatings produced using 50% duty cycle at various current densities

4.4.3 Effect of Current Density and Duty Cycle on Surface Roughness

Figure 4.11 shows the surface roughness of Ni-QD composite coatings at various duty cycles and various current densities. When current density and duty cycle rise, the coating's surface roughness increases (Sherwin et al., 2021). The change in surface roughness was due to grain size variation, resulting from the varying time intervals during pulse coatings (John et al., 2023). At 75% duty cycle with a variable current density shows a significant effect on the surface roughness as the current density increases. High current density shows that the surface morphology is rough. This is due to the varying deposition rates and an increase in electric field intensity that speeds up the mobility of Ni ions (Karadağ et al., 2023). Surface roughness can change in several ways as current density rises at 75% duty cycle.

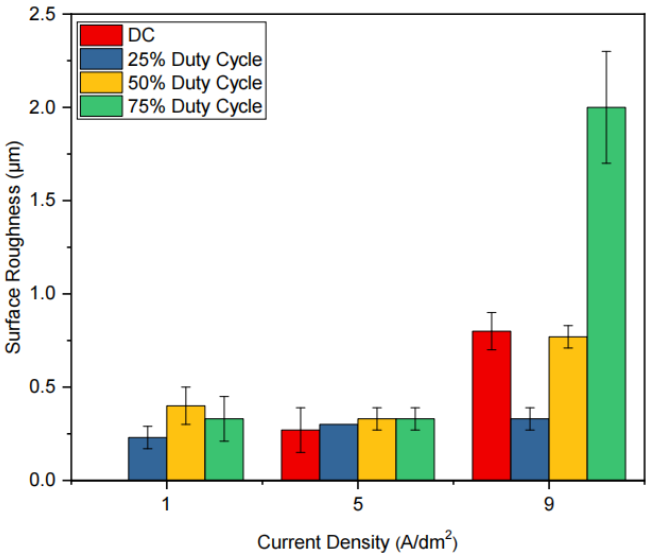


Figure 4.11: Surface roughness of Ni-QD composite coatings at various duty cycle and current densities

4.4.4 Micro-Vickers Hardness Testing

Figure 4.12 shows the hardness values of Ni-QD composite coatings produced at various duty cycles and current densities. Increases in current density and duty cycle result in increasing the hardness of the coating. The force of attraction between the particles and the cathode increases with increasing current density. An increase in amount of particles' co-depositing causes the increase of hardness of the coatings because of the excellent hardness processed by Al_2O_3 and SiO_2 particles. In high duty cycle, T_{OFF} is very short, but T_{ON} is very long, and the conditions are the same as high current density values. So, the number of particles in the coating and microhardness were increased. This result was aligned with research conducted by Ghazanlou et al., (2016). The hardness of the ceramic particles and matrix grains that are embedded in the coatings influences the microhardness of the coating. When current density and duty cycle increases, the contents of ceramic particles yield are higher. Thus, given the same matrix metal grain, coatings with larger ceramic particle amounts should provide coatings with increased microhardness. (Ma et al., 2020). However, the growth rate of crystal nucleation is slowed down by the nanoparticles acting as new growth points, which results in grain refinement. Therefore, the mechanical properties of composite coating are also improved. Due to its finer grain size, the pulse current composite had a more compact structure and decreased coating porosity.

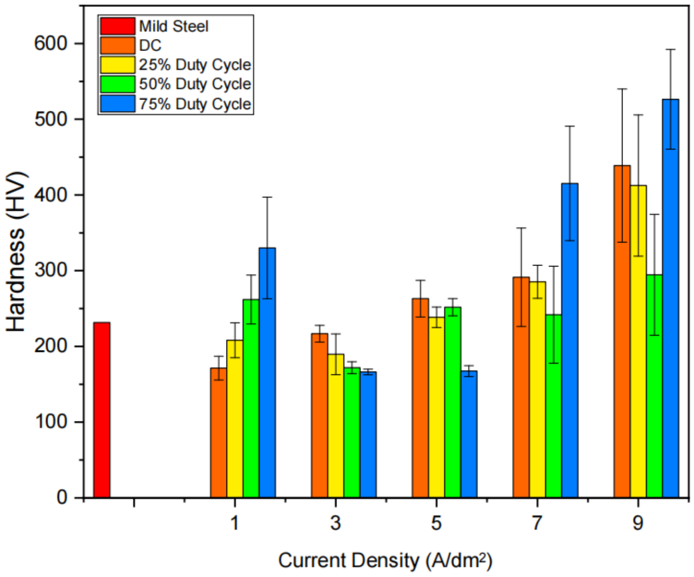
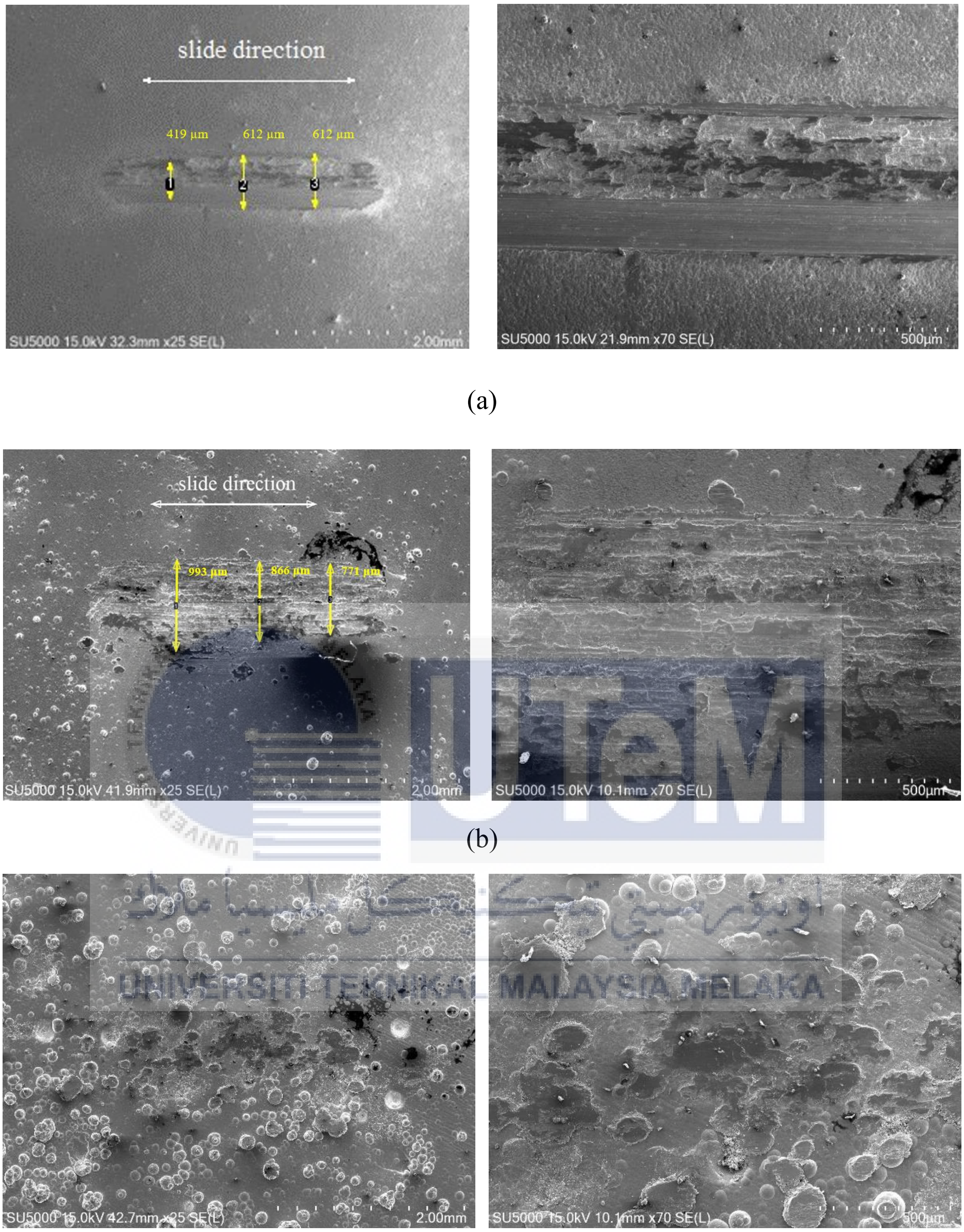


Figure 4.12: Hardness values of Ni-QD composite coatings produced at various duty cycles and current densities

4.5 Tribology of Nickel-Quarry Dust Composite Coating

4.5.1 Surface Morphology on the Wear Track

Figure 4.13 shows the wear track of Ni-QD composite coatings with low and high magnification produced using direct current at various current density. Figure 4.13 (a) shows smooth abrasive grooves with delaminated area on the wear track. Figure 4.13 (b) shows a narrow wear track with more severe abrasive wear. Figure 4.13 (c) shows that the surface morphology of wear track cannot be seen clearly. This is because when current density increases, the surface roughness of the coating increases. So, the sliding track cannot be seen clearly. The presence of Al_2O_3 nano particles has increased wear resistance. A rise in current density allows the particles to agglomerate on the coating, resulting in weaker coating (Ma et al., 2020).



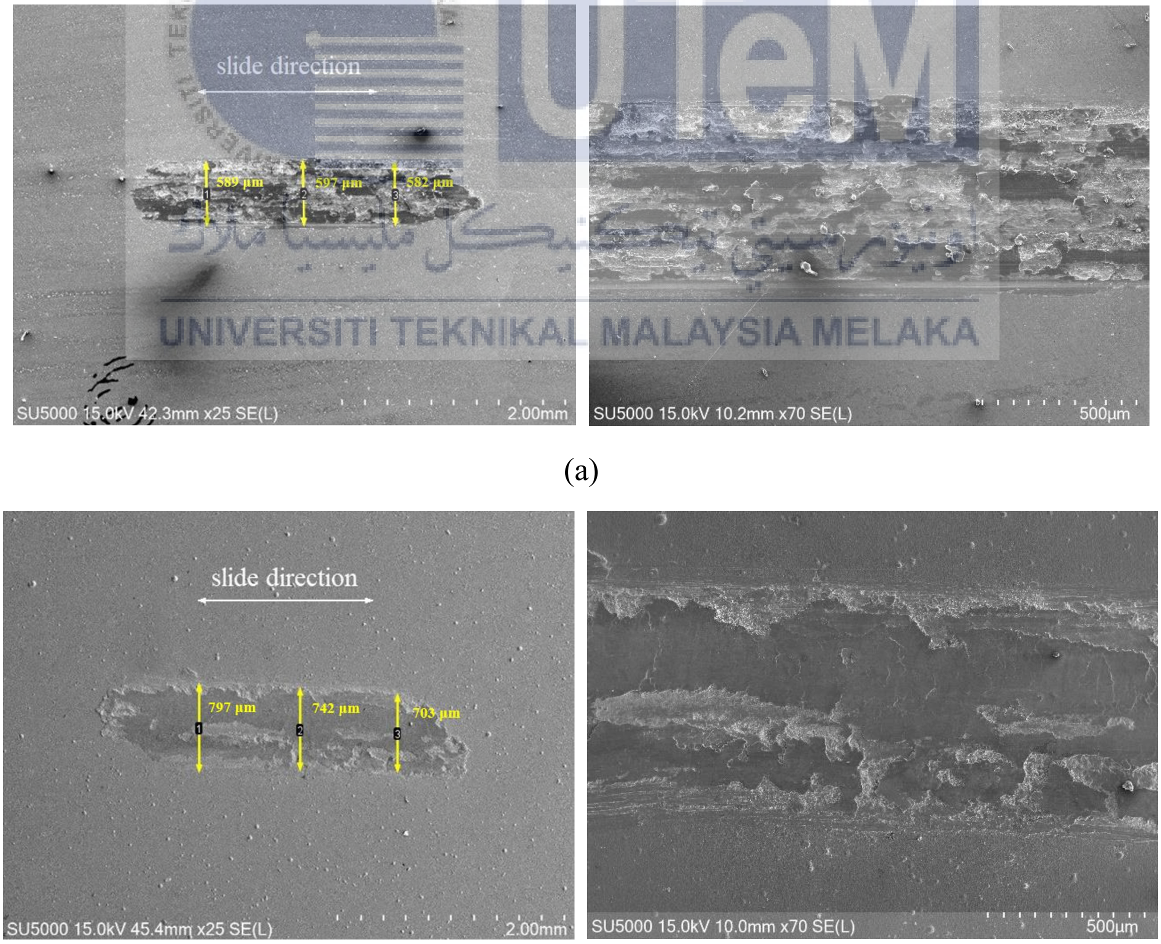
(a)

(b)

(c)

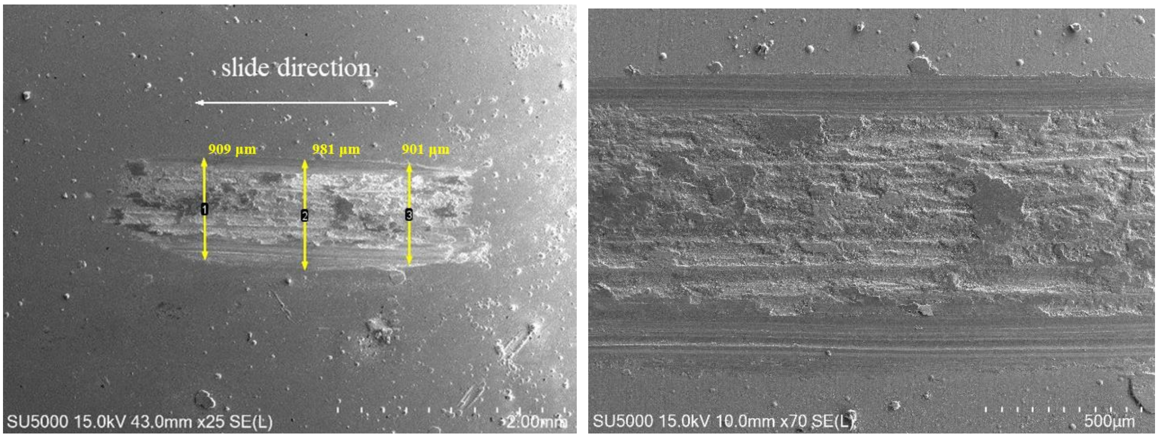
Figure 4.13: Wear track of Ni-QD composite coatings with low and high magnification produced using direct current at various current density (a) 1 A/dm^2, (b) 5 A/dm^2 and (c) 9A/dm^2

Figure 4.14 shows Wear track of Ni-QD composite coatings with low and high magnification produced using 25% duty cycle at various current density. Figure 4.14 (c) shows that the wear track has larger width of wear track while Figure 4.14 (a) shows the smallest width of wear track. Figure 4.14 (b) shows that the surface morphology of wear track is smooth and low abrasive wear. By decreasing the duty cycle, Toff increases and leads to more deposition of nickel and incorporation of alumina particles in coatings. The release of alumina particles from the coatings during the wear test leads to a reduction in wear rate because the sliding wear mode changes to rolling (Xia et al., 2020). As a result, figure (b) has the lowest wear rate. A uniform and fine microstructure was shown by the coating deposited at 5 A/dm^2 and 25% duty cycle, hence preventing any severe damage to the coated surface from hardened steel balls. As a result, exceptional wear resistance was revealed when only a few tiny scratches were seen on the Ni-QD coating surface coated at 5 A/dm2 with 25% duty cycle.



(a)

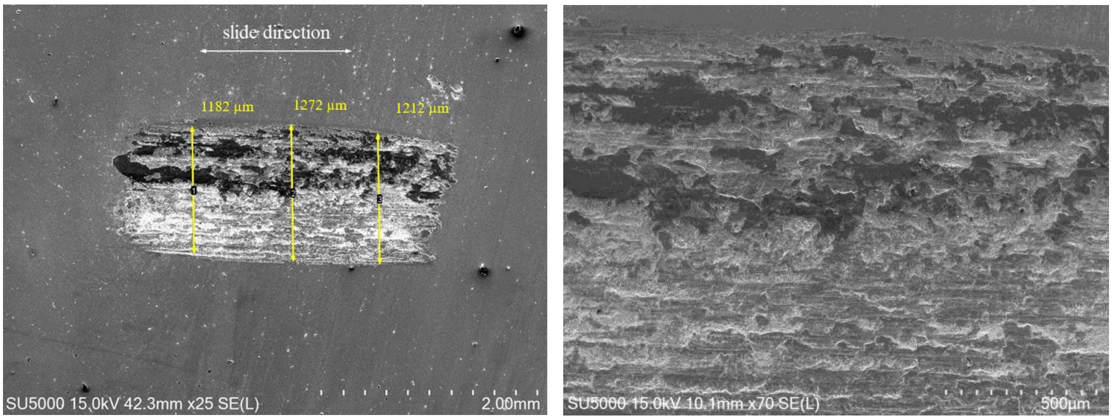
(b)



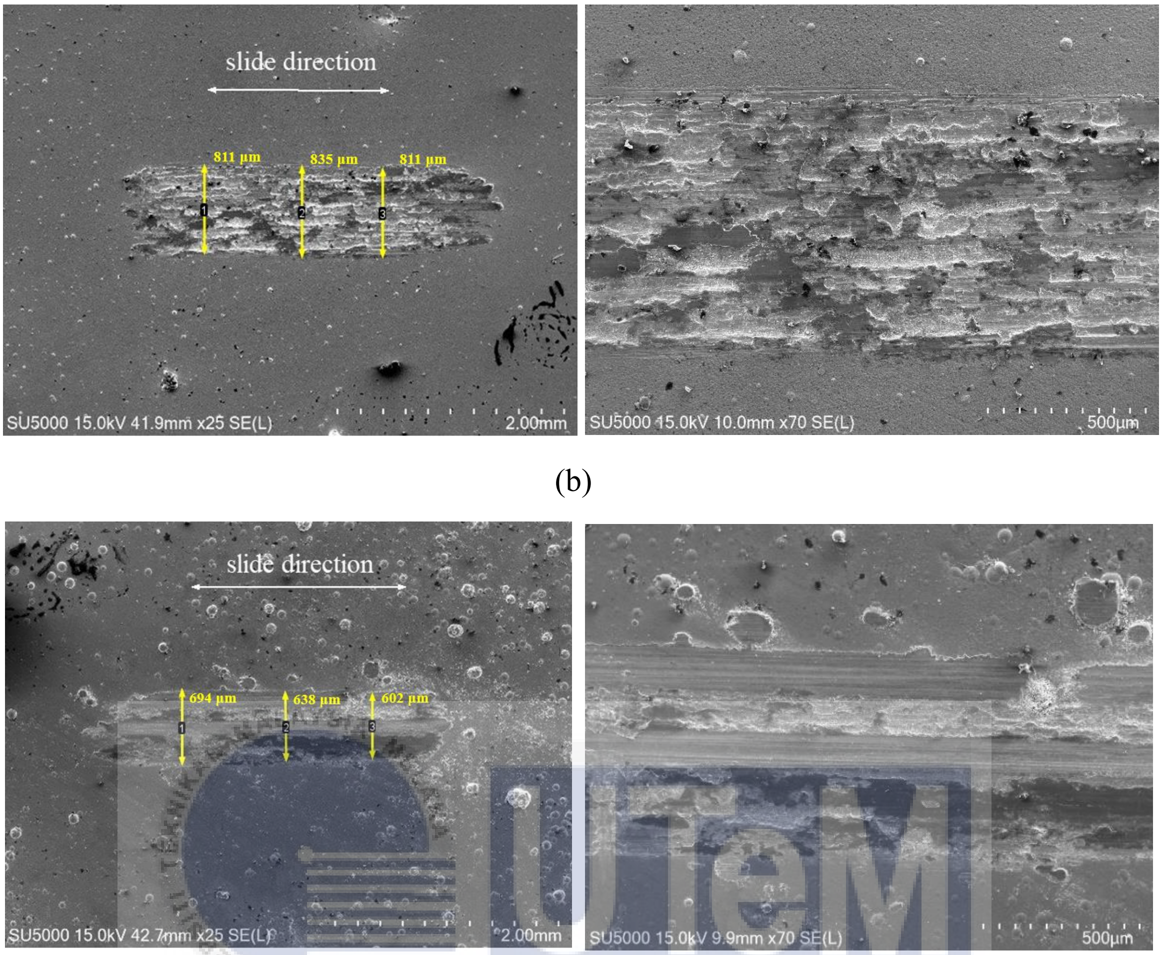
(c)

Figure 4.14: Wear track of Ni-QD composite coatings with low and high magnification produced using 25% duty cycle at various current density (a) 1 A/dm^2, (b) 5 A/dm^2 and (c) 9A/dm^2

Figure 4.15 shows the Wear track of Ni-QD composite coatings with low and high magnification produced using 50% duty cycle at various current density. Figure 4.15 (a) shows that the surface morphology has the largest width of wear track with a deeper abrasive wear. This is because when low current density, the deposition rate of the coating is slow which produce a thin coating (Xia et al., 2020). Figure 4.15 (b) shows that the wear track is deep groove and delamination which are the evidence of poor wear resistance. Figure 4.15 (c) shows moderate width of wear track, but abrasive wear occurs. Depth of grooves are deeper and amount of delaminated area in the composite are smaller which indicates moderate wear resistance.



(a)

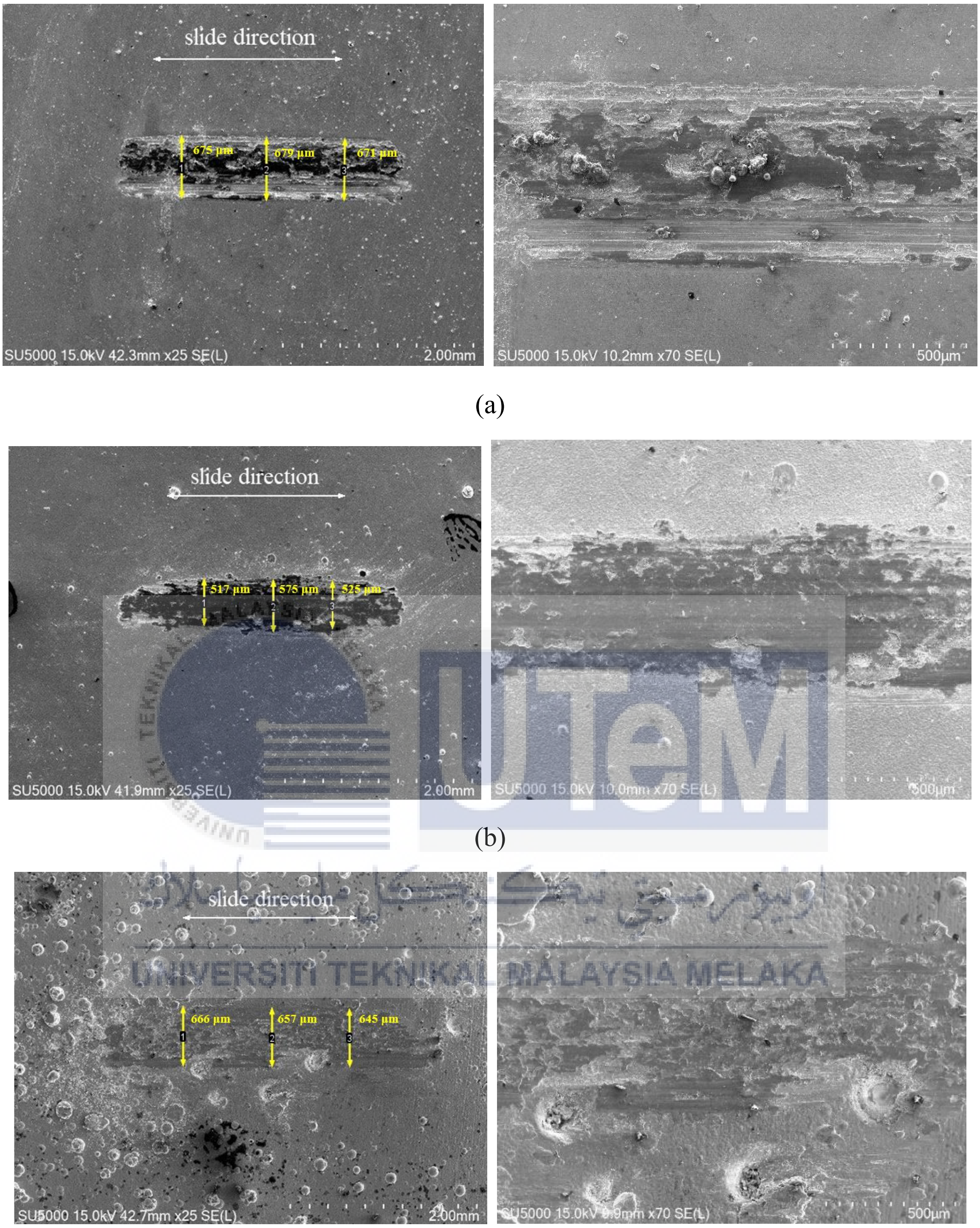


(b)

(c)

Figure 4.15: Wear track of Ni-QD composite coatings with low and high magnification produced using 50% duty cycle at various current density (a) 1 A/dm^2, (b) 5 A/dm^2 and (c) 9A/dm^2

Figure 4.16 shows the Wear track of Ni-QD composite coatings with low and high magnification produced using 75% duty cycle at various current density. Figure 4.16 (a) shows larger width of wear track with greater depth of grooves and more delaminated material. Grooves are deepened on the wear surface of composite coating and appear small wear debris. This indicates that the matrix shows higher wear rate. Figure 4.16 (b) shows deep abrasive grooves and delaminated area are observed. Figure 4.16 (c) shows the surface morphology of wear track cannot be seen clearly. The formation of grooves and small delamination is observed. The debris are the evidence of poor wear resistance. Worn surface morphology under high current density seems severe than low current density (Lin et al., 2015).



(a)

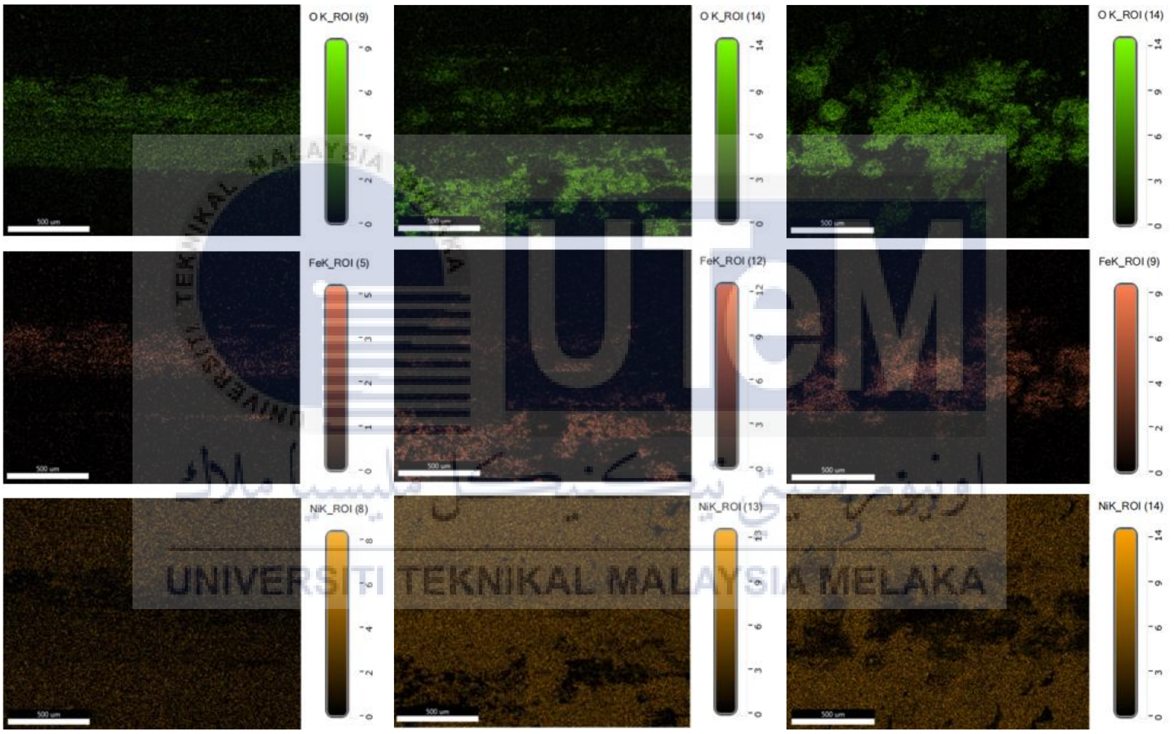
(b)

(c)

Figure 4.16: Wear track of Ni-QD composite coatings with low and high magnification produced using 75% duty cycle at various current density (a) 1 A/dm^2, (b) 5 A/dm^2 and (c) 9A/dm^2

4.5.2 Energy Dispersive X-Ray (EDX) Mapping of the Wear Track

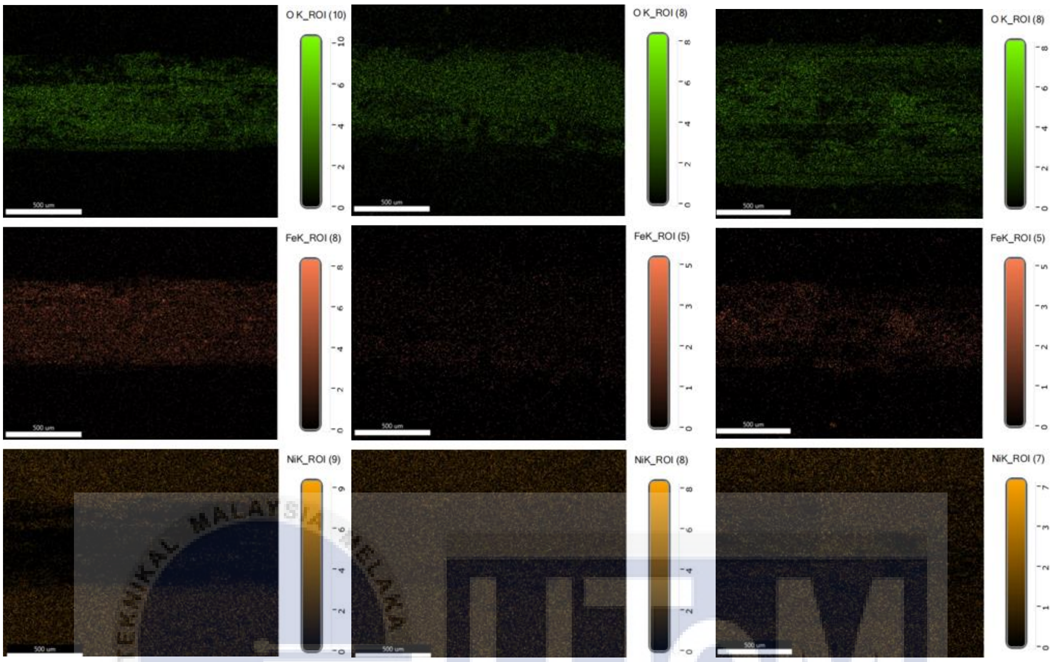
The wear track of Ni-QD composite coatings were analyzed using EDX to show elemental composition on the Ni-QD composite coatings. Figure 4.17 shows that with increasing of current density from 1 A/dm^2 to 9 A/dm^2, the nickel composition on the coating surface is increasing from 82.0% to 84.4%. The iron composition on the coating surface increases from 4.0% to 5.1% when current density is increased. It shows that when current density is increased, more iron is exposed on the surface coatings.



(a) (b) (c)

Figure 4.17: EDX mapping on wear track of Ni-QD composite coatings produced using direct current at various current density (a) 1 A/dm^2, (b) 5 A/dm^2 and (c) 9 A/dm^2

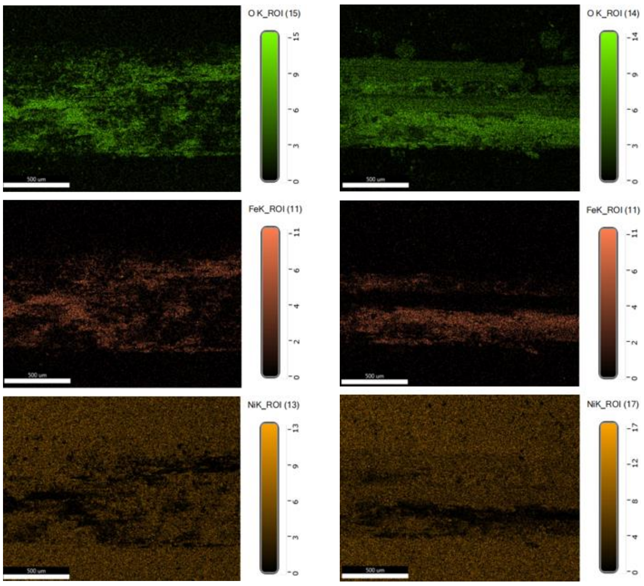
Figure 4.18 (a) shows the highest amount of iron exposed on the surface coating which is 19.0%. This is because due to the low current density and low duty cycle, the coating thickness is low which leads to the coating experienced material removal during the wear test. Figure 4.18 (b) shows the lowest amount of iron exposed which is 2.0% and the highest amount of nickel on the coating surface which are 86.1%. It demonstrates how the wear track of the composite coatings wears out because of the low nickel composition.



(a) (b) (c)

Figure 4.18: EDX mapping on wear track of Ni-QD composite coatings produced using 25% duty cycle at various current density (a) 1 A/dm^2, (b) 5 A/dm^2 and (c) 9 A/dm^2

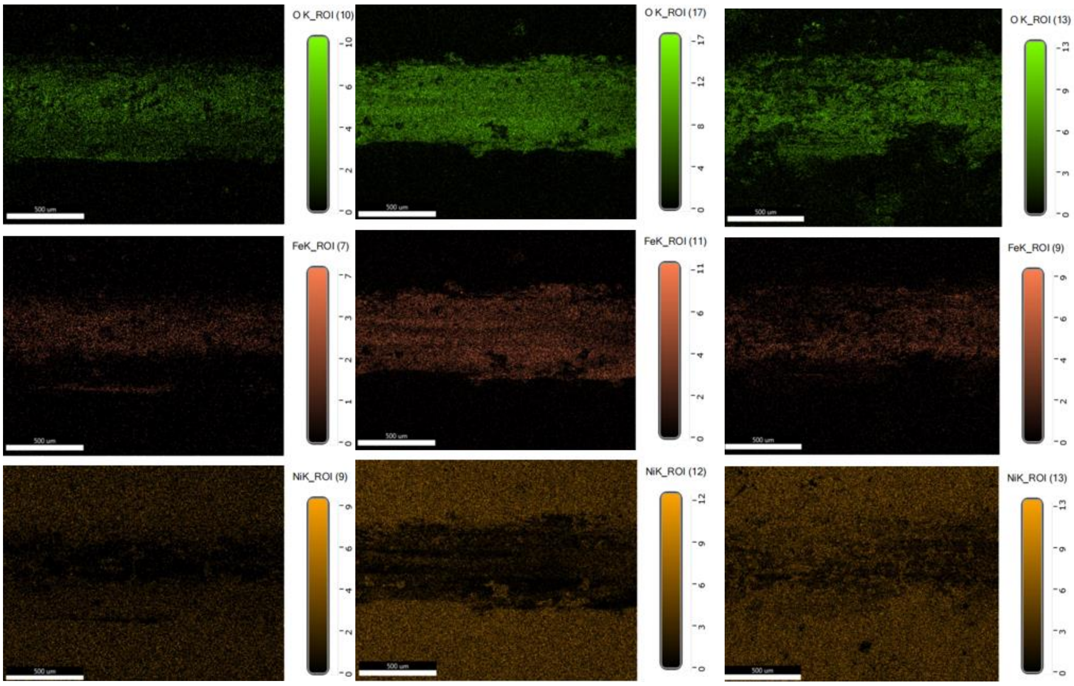
Figure 4.19 (a) shows high composition of iron exposed on the surface coating which is 6.5% compared to figure 4.19 (b) which is 4.3%. Low current density produced thinner coating thus the coating experienced material loss during the wear test. This is correlated to the low composition of nickel on the surface coating shown by figure 4.19 (a).



(a) (b)

Figure 4.19: EDX mapping on wear track of Ni-QD composite coatings produced using 50% duty cycle at various current density (a) 5 A/dm^2 and (b) 9 A/dm^2

Figure 4.20 shows iron composition decreased, and the nickel composition is increased when the current density is increased from 1 A/dm^2 to 9 A/dm^2. However, figure 4.20 (b) shows the highest iron composition and lowest nickel composition. This is probably due to the worn out of the iron from the steel ball during the wear track sliding. So, the surface coating has been contaminated with debris from the worn-out steel ball.



(a) (b) (c)

Figure 4.20: EDX mapping on wear track of Ni-QD composite coatings produced using 75% duty cycle at various current density (a) 1 A/dm^2, (b) 5 A/dm^2 and (c) 9 A/dm^2

4.5.3 Effect of Various Current Density and Duty Cycle to the COF

COF value of Ni-QD composite coatings produced using direct current at various current densities is shown in figure 4.21. Among all five coatings, Ni-QD composite coatings prepared at 7 A/dm^2 shows the smallest average of friction of coefficient which is 0.19. The Ni-QD composite coatings deposited at 1 A/dm^2 shows the highest average of friction of coefficient which is 0.21.

The friction coefficient of Ni-QD composite coatings prepared at 5 A/dm^2 shows the smallest average which is 0.19 shown in Figure 4.22. The main factors that affect the friction coefficient of metal-based composite coatings are surface morphology and the number of ceramic particles in the composite. (Liu et al., 2021). The smooth, fine, and compact surface morphology of the Ni-QD composite coatings, produced at 25% duty cycle and 5 A/dm2 current density, resulted in a low friction coefficient. Wear resistances are related to their surface morphology and microhardness. Higher in microhardness can minimize the wear loss. When compared to uneven and loose coatings, the uniformity and compactness of Ni-QD composite coatings could withstand wear forces. The hardened 10mm steel ball could not tear the metal grains off easily. Therefore, only small scratch observed on the surface of Ni-QD composite coating prepared at 25% duty cycle and 5 A/dm^2 current density (Xia et al., 2021).

Conversely, less embedded nickel, alumina, and silica with loose, uneven microstructures can simply tear nickel grains from the matrix, causing significant wear damage. As a result, after the wear tests, several huge, deeper grooves show up on the worn surfaces with high current density and high duty cycle.

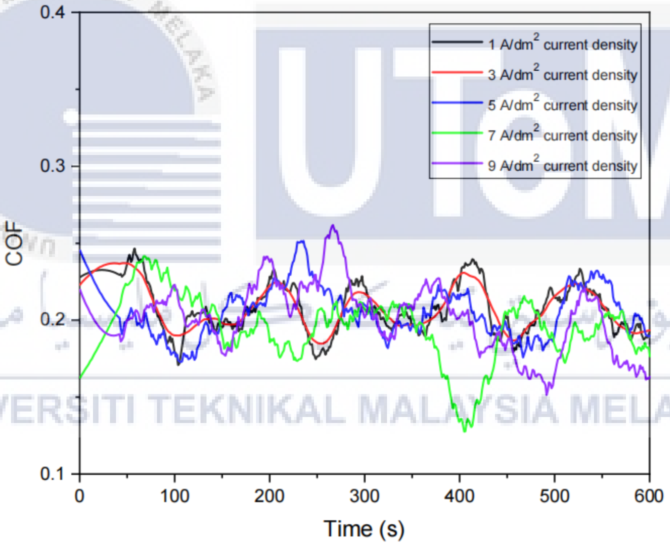


Figure 4.21: COF value of Ni-QD composite coatings produced using direct current at various current density

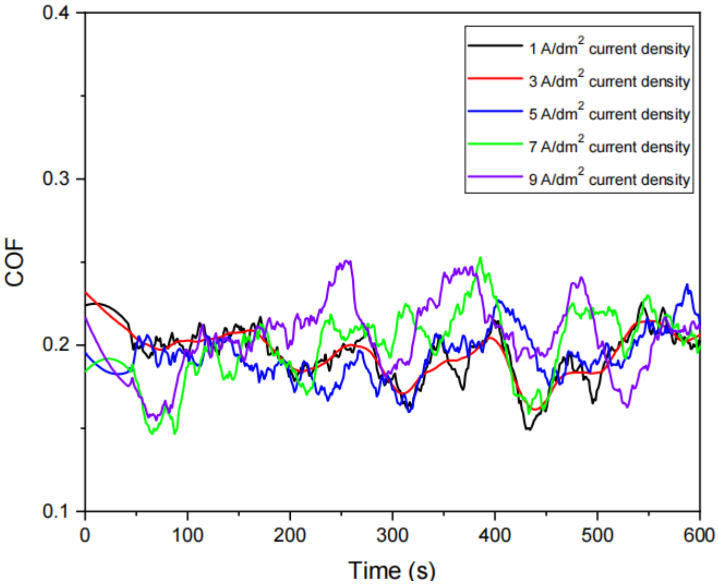


Figure 4.22: COF value of Ni-QD composite coatings produced using 25% duty cycle at various current density

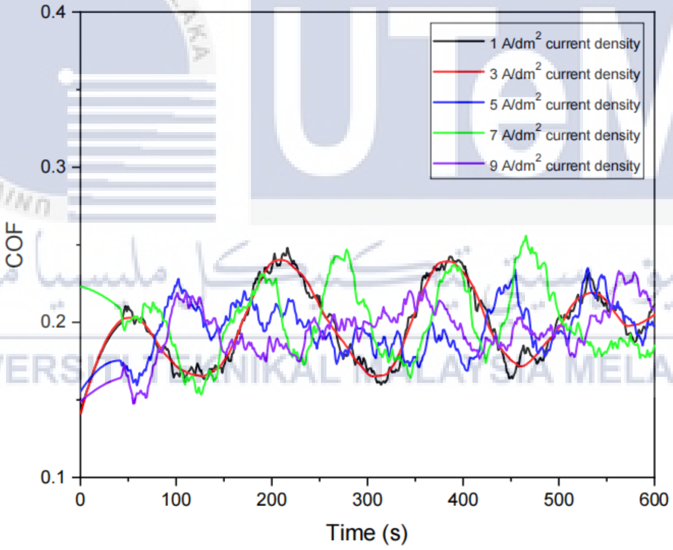


Figure 4.23: COF value of Ni-QD composite coatings produced using 50% duty cycle at various current density

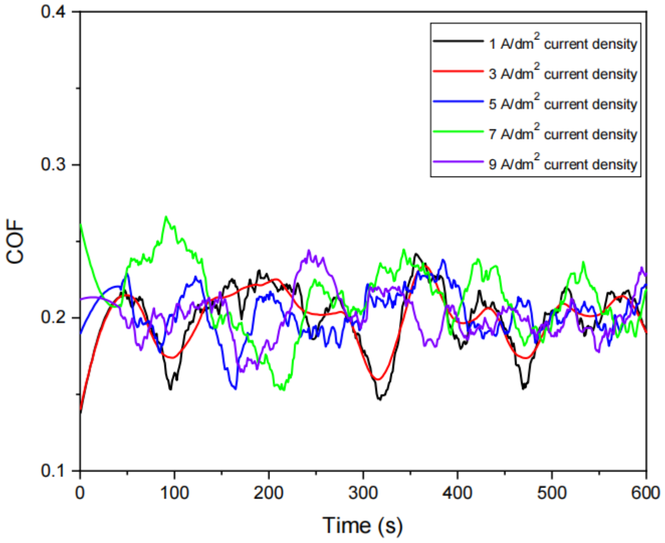


Figure 4.24: COF value of Ni-QD composite coatings produced using 75% duty cycle at various current density

Figure 4.25 shows the COF values of Ni-QD composite coatings produced at various duty cycles and current densities. As the current density increased at 25% duty cycle, the value of COF decreases. Surface morphology of the coating shows fewer abrasive grooves, the area where the coating and counter surface come into contact becomes more uniform, reducing the COF. Smoother surfaces lead to lower COF. The wear properties of Ni-QD composite coating increase when the value of COF decreases (Xia et al., 2021).

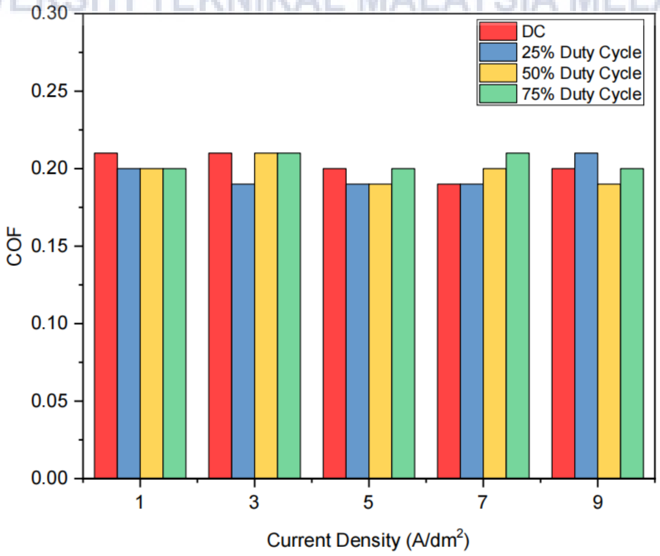


Figure 4.25: COF values of Ni-QD composite coatings produced at various duty cycles and current densities

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The first objective of this project is to investigate the influence of various current density on the surface morphology, hardness and wear resistance of Ni-Recycled quarry dust composite coatings prepared by pulse current electrodeposition. The second objective is to study the effect of various duty cycles on surface morphology, hardness and wear resistance of Ni-Recycled quarry dust composite coatings prepared by pulse current electrodeposition. The data collected has been analyzed and following significant conclusions were reached:

a) Ni-QD composite coating on mild steel has been achieved effectively with different duty cycles and current densities. On the Ni-QD composite coatings, the impact of pulse current parameters which are current density and duty cycles was investigated. High silica and alumina content quarry dust is utilized as reinforcing particles in the nickel matrix to improve the composite coatings' properties.

b) The surface morphology of Ni-QD composite coatings demonstrates that the grains agglomerated, and the surface morphology became coarser as the current density increased. The Ni-QD composite coatings exhibit a smooth and uniform surface morphology at low duty cycles. Because of the growing T_{OFF} in low duty cycle during the experiment, the smooth and uniform surface morphology was observed at 25% duty cycle and 5 A/dm2 current density.

c) As the duty cycle and current density rise, the Ni-QD composite coating's surface roughens. Because of the high current density and duty cycle, which results in rapid deposition, the content of the Ni-QD composite coating increases.

d) The Ni-QD composite coatings hardness increases when the current density and duty cycles are high. The hardness of the ceramic particles and matrix grains that are embedded in the coatings mainly determines the microhardness of the coating. When current density and duty cycles are high, the yield of ceramic particles is increased. Consequently, coatings with higher microhardness should result from higher ceramic particle amounts.

e) The surface shape of the Ni-QD composite coating influences its wear resistance. The surface morphology is more uniform, smooth, and finer at a 25% duty cycle. Less abrasive grooves may be observed in the wear track coating's surface morphology, and as the coating and counter surface's contact area becomes more uniform, the COF decreases. Lower COF is a result of smoother surfaces. When the value of COF decreases, the wear characteristics of the Ni-QD composite coating increase.

5.2 Recommendation

Recommendation for future studies to achieve more reliable results are as below:

a) Proper sample handling of the substrate is essential for future research to prevent corrosion in harsh environments. Thoroughly clean the substrate to remove residues, contaminants, and debris. Use compatible cleaning agents or solvents to prevent damage. Make sure the substrate is not wet to avoid corrosion before keeping it in seal plastics bag.

b) Measure the coating thickness of the samples to examine how the parameter of the study influences the properties of the Ni-QD composite coatings.

c) Conduct corrosion test to evaluate the corrosion resistance properties of the Ni-QD composite coating using potentiodynamic polarization in a 3.5 wt. % NaCl solution at room temperature and a potentiostat/galvanostat (AUTOLAB PGSTAT30, ASTM G44) system.

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APPENDICES

Gantt Chart PSM 1

Activities	W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14	W15
Submission of FYP title	🟧														
Submission Assignment 1			🟧												
Submission of article comparison table				🟧											
Submission Assignment 2				🟧											
Submission Assignment 3					🟧										
Submission of Parameter Matrix Table						🟧									
Submission of list of raw materials, chemicals, equipment, and testing						🟧									
Submission of draft on Chapter 1						🟧									
Submission Assignment 4							🟧								
Submission of draft on Chapter 2								🟧							
Submission of draft on Chapter 3										🟧					
Submission Assignment 5											🟧				
Submission of PSM 1 logbook												🟧			
Mock of poster presentation													🟧		

PSM 1 poster presentation														🟧	
Submission of PSM 1 report compilation														🟧	
Submission of PSM 1 report															🟧

Gantt Chart PSM 2

Activities	W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14	W15
Sample Preparation															
a) Grinding & Polishing	🟨														
b) Roughness Test	🟨														
Quarry Dust Preparation															
a) Ball Milling	🟨	🟨													
b) Sieving Quarry Dust		🟨													
c) PSA Analysis		🟨													
Solution Preparation															
a) Watts Bath Preparation in Bulk			🟨												
Experimentation				🟨	🟨	🟨	🟨	🟨							
Characterization of Coatings															
a) SEM Analysis									🟨	🟨					
b) XRD Analysis									🟨	🟨					
Mechanical Testing															
a) Hardness Test									🟨	🟨	🟨				
b) Wear Test									🟨	🟨	🟨				
c) Corrosion Test									🟨	🟨	🟨				

Report Writing	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛
Result of Experiment			🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨			
Discussion			🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨	🟨			
Submission/Important Deadline	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛	⬛
Logbook												🟨			
FYP Presentation														🟨	
FYP Report															🟨