



**INFLUENCE OF ULTRASONIC AMPLITUDE AND SACCHARIN  
CONTENT ON THE PROPERTIES OF ELECTRODEPOSITED  
NICKEL-QUARRY DUST COMPOSITE COATING**

**Submitted in accordance with the requirement of the University Teknikal Malaysia  
Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Hons)**

**by**

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Tajuk: **INFLUENCE OF ULTRASONIC AMPLITUDE AND SACCHARIN CONTENT ON THE PROPERTIES OF ELECTRODEPOSITED NICKEL-QUARRY DUST COMPOSITE COATING**

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## **APPROVAL**

This report is submitted to the Faculty of Manufacturing Engineering of University Teknikal Malaysia Melaka as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Hons).

The members of the supervisory committee are as follow:

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(DR. INTAN SHARHIDA)- Signature & Stamp

## **ABSTRACT**

The research focused on the effects of various ultrasonic amplitude and saccharin content on the surface morphology, hardness, and wear resistance of electrodeposited nickel-quarry dust composite coatings on high speed steels (HSS) substrate. Quarry dust was prepared using a ball milling process and co-deposited on substrate using an electrodeposition process. The nickel modified Watt's bath was prepared using various saccharin content of 5, 10, 15 and 20 g/L. The electrodeposition process was carried out under an ultrasonic agitation at various % amplitude of 20, 40, and 60%. The composite coatings was characterized using Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF). Then, the influence of various ultrasonic amplitude and saccharin content on the composite coatings was investigated using hardness test and wear test. As the saccharin content increase, the hardness of the substrate will increase too. Therefore, from 20% to 40% ultrasonic amplitude shows an increasing trend but at 60% the hardness reduce a little bit. The saccharin content also improve the well resistance of the substrate. The ultrasonic effect to the wear resistance significant to the hardness. As the substrate was applied with 60%, the wear shows a worst track. Ultrasonic amplitude and saccharin content does influence the surface morphology of the substrate as it related to the particles distribution and crystallite size produce.

## ABSTRAK

Kajian menumpukan perhatian kepada kesan pelbagai amplitud ultrasonik dan kandungan sakarin ke atas morfologi permukaan, kekerasan dan daya tahan kehausan saduran komposit nikel- debu kuari melalui proses elektrodeposisi ke atas keluli kelajuan tinggi (HSS). Debu kuari telah disediakan melalui proses penggilingan bola dan di deposit kan ke atas permukaan substrat menggunakan proses elektrodeposisi. Larutan nikel yang telah diubah telah disediakan menggunakan kandungan sakarin yang berbeza iaitu 5, 10, 15, dan 20 g/L. Proses elektrodeposisi telah dijalankan menggunakan amplitud ultrasonik yang berbeza iaitu 20, 40 dan 60%. Hasil saduran komposit telah dicirikan menggunakan pengimbas mikroskop elektron (SEM), difraksi sinar x (XRD) dan pendarflour sinar x (XRF). Kemudian, kesan perbezaan amplitud ultrasonik dan kandungan sakarin ke atas saduran komposit telah dikaji menggunakan ujian kekerasan dan ujian kehausan. Semakin tinggi kandungan sakarin, semakin keras saduran komposit yang terhasil. Bagaimanapun, daripada 20% kepada 40% amplitud ultrasonik menunjukkan kenaikan tetapi 60% amplitud ultrasonik menunjukkan sedikit pengurangan kekerasan ke atas saduran komposit yang terhasil. Kandungan sakarin juga menunjukkan penambahbaikan dalam daya tahan kehausan saduran komposit. Manakala, kesan ultrasonik terhadap daya tahan kehausan sama dengan kekerasan. Apabila substrat dikenakan 60% amplitud ultrasonik, keputusan ujian kehausan adalah paling teruk. Amplitud ultrasonik dan kandungan sakarin mempengaruhi morfologi permukaan substrat disebabkan oleh kaitan taburan partikel dan saiz kristal yang terbentuk.

## **DEDICATION**

For my late father, Shafie Bin Abu Bakar for being the source of my inspiration and motivation to complete my study, my beloved mother, Fatimah Binti Yatim, my beautiful sisters and brothers for being a wonderful support system, giving me money, cooperation , encouragement and understandings all this while.

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## LIST OF ABBREVIATIONS

AFM	-	Atomic Force Microscopy
AIISI	-	American Iron and Steel Institute
ASTM	-	American Society for Testing and Materials
CMMA	-	Composition Modulated Multilayer Alloy
DBT	-	Double Bath Technique
EDM	-	Electrical Discharge Machining
HSS	-	High Speed Steel
HSTFE	-	High Speed Turbulent Flow Electrodeposition
MMCs	-	Metal Matrix Composite
PSA	-	Particle Size Analyzer
PZC	-	Potential Zero Charge
RDE	-	Rotating Disk Electrode
TMAB	-	Trimethylamine Borane
UTeM	-	Universiti Teknikal Malaysia Melaka
SBT	-	Single Bath Technique
SDS	-	Sodium dedocyl Sulfate
SEM	-	Scanning Electron Microscope
XRD	-	X-Ray Diffraction
XRF	-	X-Ray Fluorescence



## LIST OF SYMBOLS

A/dm <sup>2</sup>	-	Ampere per square decimeter
Al <sub>2</sub> O <sub>3</sub>	-	Alumina
cm	-	Centimeter
cm <sup>2</sup>	-	Centimeter squared
Co	-	Cobalt
Cr	-	Chromium
Fe	-	Ferum
g	-	Gram
g/cm <sup>3</sup>	-	Gram per centimeter cubic
g/L	-	Gram per liter
HCl	-	Hydrochloric acid
hr	-	Hour
kHz	-	Kilo hertz
mAcm <sup>-2</sup>	-	Mili ampere per centimeter squared
min	-	Minutes
ml	-	Mililiter
mm	-	Milimeter
Mo	-	Molybdenum
MOS <sub>2</sub>	-	Molybdenum Disulfide
ms <sup>-1</sup>	-	Meter per second
N	-	Newton
NaCl	-	Natrium Chloride
Ni	-	Nickel
rpm	-	Revolution per minutes
Si	-	Silicon
SiC	-	Silicon Carbide
SiO <sub>2</sub>	-	Silica

TiCN	-	Titanium Carbonitride
TiO <sub>2</sub>	-	Titanium Dioxide
V	-	Vanadium
W	-	Tungsten
WC	-	Tungsten Carbide
WO <sub>3</sub>	-	Tungsten Trioxide
ZnCl <sub>2</sub>	-	Zinc Chloride
ZrO <sub>3</sub>	-	Zirconium Trioxide
°C	-	Degree Celcius
%	-	Percentage
% wt	-	Mass fraction
μm	-	Micrometer
μm h <sup>-1</sup>	-	Micrometer per hour

# **CHAPTER 1**

## **INTRODUCTION**

This chapter explained about the synopsis of study, problem statement, objectives, scope of study, significance of study, report organization and an overall summarization for this chapter.

### **1.1 Research Background**

High Speed Steels (HSS) are specially designed for high speed cutting tool applications with an alloying composition mainly consisting of tungsten (W), molybdenum (Mo), vanadium (V), chromium (Cr) and cobalt (Co) (Hadian et al., 2019). Due to the enhanced wear and mechanical properties of the HSS especially at elevated temperatures, the use of them as cutting tool is of interest in recent studies on NbC-Fe based cemented carbides (Hadian et al., 2019). HSS has been widely used in various fields due to their excellent red hardness and good wear resistance (Liu et al., 2015). HSS have traditionally been used as cutting materials and they currently compete in many applications with cemented carbides (Herranz et al., 2014).

A constant extreme rubbing process between the cutting tools and the workpiece will eventually lead to wear and thus lead to failure of the cutting tool (Izzuddin et al., 2015). In order to improve the properties of the cutting tool in term of hardness and wear resistance which could prolonged the life span of the cutting tool, a coating process could be applied. The addition of reinforcements to a metal matrix, such as AISI M2 steel, results in a metal matrix composite material which has intermediate properties between those of steels and cemented carbides or hard metals (Herranz et al., 2014). In order to improve the mechanical properties of HSS as a cutting tool, a composite coating can be introduced to the substrate by electrodeposition process.

Electrodeposition is a technically feasible and greater cost-effective technique acknowledged method for the metal matrix composite (MMCs) preparation. It is because of the numerous advantage which are precisely controlled operation, low cost equipment setups, carried out at a normal pressure, unvarying deposition rate, handling of intricate geometries and simple equipment setup scale-up with ease of maintenance.

The composite coatings properties is depends on the matrix phases and also the amount and distribution of co-deposited particles within the matrix. In this study, quarry dust was used as a reinforcement for the composite coating. Quarry dust is one of the enormous resources of waste materials resulting from crushing process of rocks. It contains high composition of  $\text{SiO}_2$  and considerable amount of  $\text{Al}_2\text{O}_3$  with the density of 1.9 to 2.0  $\text{g/cm}^3$  compared to another reinforcements such as silicon carbide ( $\text{SiC}_p$ ) and  $\text{Al}_2\text{O}_3$  (Oladayo and Samuel, 2017). Quarry dust which contains more  $\text{SiO}_2$  is used as a reinforcement for composite coating as it can improve the surface properties of the substrate.

To enable a uniform dispersion of reinforcement-particle during the electrodeposition, an ultrasonic transducer was introduced to the process. The ultrasonic transducer was used to ensure the agitation of the particles during the electrodeposition process. Many researches have been studied the effect of ultrasound employing to the dispersion of particles in electroplating baths and the sonication impact during the electrodeposition process that may contributed to the characteristics of the resulting composite coatings. It is due to the phenomenon of acoustic cavitation occurs. Besides the ultrasonic, magnetic stirrer is another alternative way to ensure the particle is in suspension during the electrodeposition process.

## **1.2 Problem Statement**

High Speed Steel (HSS) is a subset of tool steels. It is an alloy that gain their properties from either tungsten or molybdenum, often with a combination of the two. HSS usually used as a cutting tool. During the machining process, HSS will worn out due to the friction with the

workpiece. Cutting tool life span usually not long lasting. The life span of the HSS can be prolonged by coating the tool.

To improve the HSS properties, a composite coating reinforced with quarry dust was electrodeposited on the HSS substrate. Quarry dust is one of the enormous resources of waste materials resulting from crushing process of rocks and it contain high composition of  $\text{SiO}_2$  and considerable amount of  $\text{Al}_2\text{O}_3$  with the density of 1.9 to 2.0  $\text{g/cm}^3$  compared to another reinforcements such as SiCp and Alumina (Oladayo and Samuel, 2017).

A considerable amount of work has been published on the effect of ultrasound employing to the properties of composite coatings. The ultrasound was used to agitate the electrolyte in order to achieve a homogenous distribution of the ceramic particle in the coating. However, home of the research study on the effect of ultrasound to the dispersion of industrial waste, such as quarry dust. It is due to the phenomenon of acoustic cavitation occurs. Besides the ultrasonic, magnetic stirrer is another alternative way to ensure the particle is in suspension during the electrodeposition process.

Besides the ultrasonic intensity, this study also focuses more on the effect of saccharin content to the grain size. Saccharin is one of the additive that can be added to the nickel electrolyte bath. It can refine the grain size and in other study also acts as a brighter in the experiment.

### **1.3 Objectives**

The objectives of this study are:

- (a) To study the effect of various ultrasonic amplitude and saccharin content on the surface morphology of electrodeposited nickel-quarry dust composite coating.
- (b) To investigate the influence of various ultrasonic amplitude and saccharin content on hardness of electrodeposited nickel-quarry dust composite coating.

- (c) To examine the effect of various ultrasonic amplitude and saccharin content on wear resistance properties of electrodeposited nickel-quarry dust composite coating.

## **1.4 Scope**

The study only focus on ultrasonic amplitude and saccharin content that is applied during electrodeposited nickel quarry dust composite coating on HSS substrate. The objective for these studies is to investigated the influence of the ultrasonic amplitude and the saccharin content on the electrodeposited nickel quarry dust composite coating toward the surface morphology, hardness and wear resistance. During the electrodeposition of HSS, the ultrasonic probe is used to enable a homogenous dispersion of the quarry dust in suspension during the process. Besides that, these study also focus on the effect of saccharin contain to the properties of HSS.

## **1.5 Significant of Study**

During electrodeposition of HSS, ultrasonic amplitude and saccharin additive is applied to the electrolyte. In this research, ultrasonic is used because of its ability to enable the quarry dust in suspension replacing the magnetic stirrer to improve the uniformity of quarry dust distribution in the composite coating. The surface morphology optimization can be obtained through SEM and XRD. The HSS coefficient of friction can be obtained through the wear test, thus the wear trap can be observed through the software. The optimization of HSS hardness can be determined using the Vickers micro-hardness test.

## **1.6 Organization of Study**

In overall, these researches are majorly consists of three section. The three sections are introduction, literature review, and methodology. For first part is introduction which consists of

the synopsis of this study which refer to research background, problem statement of this study, objective, and lastly scope for this study.

Second section is emphasized on writing on the past related study and research on this topic. This section involves the properties of HSS, quarry dust, the electrodeposition, nickel-composite coating, ultrasonic in electrodeposition, saccharin as additive in electrodeposition, jacketed beaker design and anything that related to this topic.

Last but not least, chapter three which is methodology had covered the method involved in order to explain on the preparation of the experiment, testing method and studies of each sample. This chapter involves the explanation regarding the design of jacketed beaker, the parameter used and sample preparation briefly.

## **1.7 Summary**

It can be concluded that detail and brief information about research background, problem statement, objective, scope of study, significant of study and also structure of report are included in Chapter 1.

## **CHAPTER 2**

### **LITERATURE REVIEW**

This chapter is priority describe the previous theory and research which have been defined and conducted by various researcher years ago. Related information of previous studies are extracted as references and discussion based on their research about electrodeposited nickel composite coating, effect of ultrasonic intensity in term of distribution and wear resistance, saccharin contain on grain size refiner, mechanical and physical properties of the High Speed Steels (HSS) and the glass cell design which related to the electrodeposition using the ultrasonic probe.

#### **2.1 Electrodeposited Nickel Composite Coating**

Electrodeposition is a technique of cost effective and technically feasible acknowledged method for the metal matrix composite (MMCs) preparation. It is because of the numerous advantage which are low cost setup equipment and can be carried out at a normal pressure which the operation can be controlled precisely controlled with unvarying deposition rate, handling of intricate geometries and simple equipment setup scale-up with ease of maintenance. For the component corrosion and wear resistance improvement other than producing a better appearance, nickel electroplating has been a common practice for preparing functional coating (Wang et al., 2018).

Besides the distribution and the amounts of co-deposited particles within the matrix, the composite coatings properties also depends on the matrix. As reported by Merita et al. (2018) composite particles is usually hard inert particle in nanocrystalline structure. These particles can