STUDY OF NICKEL TITANIUM DIOXIDE COATING PREPARED BY ELECTRODEPOSITION

IRNE BINTI ZAINUDDIN
B051410259

UNIVERSITI TEKNIKAL MALAYSIA MELAKA
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This report is submitted in accordance with requirement of the University Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering

by

IRNE BINTI ZAINUDDIN
B051410259
950730 – 12 – 5868

FACULTY OF MANUFACTURING ENGINEERING
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Sesi Pengajian: **2017/2018 Semester 2**

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I hereby, declared this report entitled “Study of Nickel Titanium Dioxide Coating Prepared by Electrodeposition” is the result of my own research except as cited in references.

Signature : ……………………………………
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Date : 3 June 2018

APPROVAL

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

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(Dr Toibah Binti Abdul Rahim)

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(Dr Rose Farahiyan Munawar)

ABSTRACT

Electrochemical deposition process has been extensively studied as it is one of promising processes to form metal matrix ceramic coating without significantly changing crystalline structure of the initial powder material. In sequence to that, most of conventional thermal spray processes change the structure of initial phase powder due to high processing temperature during the coating process. Therefore, this study discuss on how to prepare ceramic coating with minimum time consumption using electrochemical deposition process by utilizing TiO_2 powder as the raw material. The main objective of this study is to deposit TiO_2 coatings on the substrates with enhancing nickel properties using an approach of electrochemical deposition technique. The effects of electrochemical deposition process by varying the composition of TiO_2 powder on aluminium substrates evaluated in this study. TiO_2 as second phase particle was incorporated into the Ni coating to improve the surface mechanical properties. In this study aluminium will act as reference electrode (cathode) and Ni plate acted as the counter electrode (anode), while direct current and electrolyte also have been used to complete the electrodeposition process. Moreover, the deposition mechanism of electrochemical deposition technique also influenced the hardness and surface roughness of substrates. The availability and compositions of coatings were characterized by X-ray diffraction spectrum (XRD), energy dispersive X-ray spectroscopy (EDX), surface roughness machine and surface hardness machine. In the Ni – TiO_2 coating matrix, the results of the tests indicates that with the addition of TiO_2, the microstructure of Ni – TiO_2 coating drastically changes and tends to be in coagulated forms. The presence of TiO_2 in Ni – TiO_2 was confirmed through the test result from EDX and XRD. However, just a small peak of TiO_2 was detected of the Ni –TiO_2 coating based on XRD test. As the incorporation of TiO_2 composition increases the microhardness and surface roughness of the coatings also increase.

ABSTRAK

Proses pengendapan elektrokimia telah dikaji secara meluas kerana ia adalah salah satu proses yang menyakinkan untuk membentuk salutan struktur nano seramik tanpa mengubah struktur asal serbuk. Sehubungan itu, kebanyakan proses semburan haba konvensional mengubah struktur serbuk fasa awal kerana suhu pemprosesan yang tinggi semasa proses salutan. Oleh itu, kajian ini membincangkan cara menyediakan salutan seramik dengan menggunakan masa yang minimum melalui proses pengendapan elektrokimia, menggunakan bahan serbuk TiO_2. Objektif utama kajian ini adalah untuk mendeposit lapisan TiO_2 pada substrat dengan meningkatkan ciri-ciri nikel melalui pendekatan teknik pemendapan elektrokimia. Kerja ini menilai kesan-kesan pembolehubah proses pemendapan elektrokimia menggunakan nisbah serbuk TiO_2 yang berbeza pada substrat aluminium. TiO_2 bertindak sebagai partikel kedua dalam salutan nikel untuk meningkatkan ciri –ciri permukaan mekanikal. Dalam kajian ini aluminium akan bertindak sebagai elektrod rujukan (katod) dan plat nikel bertindak sebagai elektrod balas (anod), manakala arus terus dan elektrolit juga telah digunakan untuk melengkapkan proses elektrodeposisi. Selain itu, mekanisme teknik pemendapan elektrokimia juga akan mempengaruhi kekerasan dan kekasaran permukaan substrat. Komposisi salutan telah dikaji menggunakan spektrum difraksi sinar-X (XRD) dan spekroskopi sinar-X tenaga dispersif (EDX). Dalam Ni – $TiO_{2,}$ keputusan ujian menunjukkan penambahan TiO_2 akan membuatkan saduran Ni – TiO_2 berubah secara ketara dan cenderung untuk bergumpal. Kehadiran TiO_2 dipastikan melalui mesin XRD dan EDX. Tetapi, hanya puncak kecil dapat dilihat melalui mesin XRD. Kekasaran mikro dan kekasaran permukaan lapisan meningkat dengan peningkatan peratusan berat kandungan zarah TiO_2 dalam salutan.

DEDICATION

To all those who have supported, encouraged, challenged, and inspired us. Especially dedicated to my beloved parents, family, honourable lectures and friends for all their guidance, love and attention has made it possible for me to make it up to this point and as well as my dear supervisors who always showed the best possible route by their unmatchable style and by best possible techniques.

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LIST OF ABBREVIATIONS

WHO	-	World Health Organization
TiO_2	-	Titanium dioxide
Ni	-	Nickel
$FeTiO_3$	-	Ilmenite
$CaTiO_3$	-	Perovskite
$CaTiSiO_5$	-	Titanite
UN	-	United Nations
GHS	-	Globally Harmonized System
EPD	-	Electrophoretic Deposition
ELD	-	Electrolytic Deposition
PSA	-	Particle Size Analyzer
SiC	-	Silicon carbide
SEM	-	Scanning Electron Microscope
XRD	-	X-Ray Diffractometer
PSA	-	Particle Size Analyzer
EDX	-	Energy Dispersive X – ray
NaOH	-	Sodium Hydroxide
HNO_3	-	Nitric acid

CHAPTER 1

INTRODUCTION

1.1 Background

In recent years the awareness towards the atmospheric condition is increasing. Moreover nowadays the chemical, system and process is becoming more advance. Two of the world‟s worst toxic pollution problems are indoor air pollution and urban air quality and in year of 2012, WHO reported that air pollution has caused the deaths of around 7 million people worldwide (Robotti *et al.*, 2016). Therefore it is very crucial to solve this problem. One way to protect the world from air pollution is by application of TiO_2 as photocatalyst. Based on Spanou, Pavlatou, and Spyrellis (2009) TiO_2 of metal matrix composite coatings particles exhibit interesting photoelecrochemical and photocatalytical behaviour with enhanced mechanical properties. Owing to this uniqueness, there is many applications using TiO_2 specifically for environmental purposes including self-cleaning surfaces, antifogging materials, water purification, air cleaning, self-sterilization, etc (Salim *et al.*, 2011).

Based on the previous research one of the effective processes to coat the TiO_2 is using the electrochemical deposition process. Recently, many researchers studied on Ni matric composites reinforced with TiO_2 powder particles utilizing electrodeposition technique by either using sulfamate or Watts plating baths. However, electro co-deposition of Ni-TiO_2 is one of the challenged processes as it is difficult to be controlled quantitatively. This is due to the the particles are frequently agglomerated in the metal matrix and also in the electrolyte because of the significant high surface energy (Spanou et al., 2009).

1.2 Problem Statement

In industrial field, especially coating using the nickel has been used widely as it is offers many advantages. Nickel plating has been one of the most investigated processes as it has wide range of application. These applications cover for the use in aerospace industry, automotive parts, decorative doors, musical instruments and etc (Parida *et.al,* 2010). However, nickel surface coating has such low surface mechanical properties such as hardness and wear resistance. Therefore, in order to improve the mechanical properties of nickel, second phase particle such as Al_2O_3, TiO_2, ZrO_2, SiO_2, SiC and TiC etc. are used to incorporate in the coating. For metal-matrix composite coatings, it is well-known that closely dispersed second phase particles have strong dispersion strengthening effect. However, large surface energy of small particles can lead to the agglomeration of fine particles.

In this study, electrodeposition technique was used to develop metal matrix composite coating of Ni – TiO_2. Electrodeposition is chose due to its simplicity and cost effective. Moreover, Watts‟s solution is used to accompany the electrodeposition method. Nevertheless Parida *et.al (*2010) reported that the use of Watts‟s bath may produce stresses to plated material that can cause lower fatigue properties. However, this problem can be overcome by maintaining the ultrasonic agitation during the process which may reduce the stress limit on the plated material.

1.3 Objectives

The current study mainly focus on the success of nickel titanium dioxide (Ni-TiO_2) coating on substrates using electrochemical deposition process in order to improve the surface mechanical properties and also for photocatalytic application. To achieve this objective, there are three sub-objectives that need to be prioritized. This study embarks on the following objectives:

1. To prepare Ni-TiO_2 coating by electrodeposition method by varying the TiO_2 compositions.
2. To characterize the property of the obtained Ni-TiO_2 coating.

3. To correlate the effect of the composition of TiO_2 on the as-prepared Ni-TiO_2 coating on the hardness and surface roughness of the coating.

1.4 Scope

This project covered the following scopes:

a) Study on the effect of different TiO_2 ratio using electrochemical deposition process. Different ratio of TiO_2 can affect the build-up of coating.

b) Use different machines to study on the characterization properties of the Ni-TiO_2 coating based on its morphology, hardness, and surface roughness. From this investigation, the obtained result of pros and cons can be further discussed.

c) The suitable parameters have been chose to match with electrochemical deposition process and Ni-TiO_2 material. The parameter will determine the chemical and mechanical properties of coating that will be formed.

1.5 Project Planning

Project planning is required to identify and to organize the project in order to achieve the objectives in the period of time that has been prescribed. A good planning is a planning that ensures the project run following its actual track. In this project, Gantt chart has been prepared to get the good management time. The Gantt chart illustrates the start time and the end time of the project implementation and summary elements of a project. Appendix A and B shows the Gantt chart for PSM 1 and 2 that need to be followed in order to achieve the aim of the study.

CHAPTER 2

LITERATURE REVIEW

In general literature review is the summarization of the particular sources such as journal, textbooks and internet that have been acquired. This chapter will focus on the theory and research that have been done and approved by the various researcher years ago. Related information of previous studies are extracted as references and discussion based on their research about the Ni/TiO_2 coating itself, the process of coating, mechanical and physical properties.

2.1 Surface Engineering

2.1.1 Coating

Coating is the process of covering the surface of an object, known as substrate. The coating may have two different functions depending on the characteristics of the product; either decorative, functional, or both. The coatings, as for example using paint or chrome will increase corrosion resistance, increase life time, add the value to product and etc. In general coating can be all-over coating which full cover of the substrate or it can just only cover parts of the substrate. The example of these types of coating can be seen at product label on the drinks bottles nowadays. One side of the bottle has an all-over functional coating while the other side has one or more decorative coatings in an appropriate pattern forming the words and images.

The characteristics such as protecting the substrate and being decorative mostly related to paint and lacquers as these types of coating has dual functions. Hence, some artist uses paints only as decoration and the paint on industrial field is presumably only for the function of preventing corrosion. The substrate surface properties can be changed using functional coatings. The examples of substrate surface properties are adhesion, wettability, corrosion resistance or wear resistance.

2.2 Titanium Dioxide

2.2.1 Properties of Titanium Dioxide [TiO_2]

Basically TiO_2 is a material that comes from the group of composites and ceramic. In recent years TiO_2 has been considered in much research due to its advantages in manufacturing industry. Photocatalytic coating and TiO_2 have been a synonym with each other for many years as the TiO_2 can be used widely in photocatalytic reaction. The application of TiO_2 in photocatalytic reaction is widely used for many products and also as well as the research area such as environmental and energy related fields. This proves that the ceramic materials are very advantageous and can be used for many big industries.

According to Gardon & Guilemany (2014) the ilmenite ($FeTiO_3$), rutile, anatase (combine with brookite, is in polymorphic crystal form), perovskite ($CaTiO_3$) and titanite ($CaTiSiO_5$) are the example of the minerals that can be used to extract the TiO_2 materials. In response of this many resources, the annual yield of TiO_2 reach around 6 million tons in 2008 and its industrial success also due to the price of production that lower than others functional metal oxides. TiO_2 has three types of crystal structures which are rutile, anatase and brookite. This three type condition of TiO_2 has the same chemical formula but different crystal structures. Among these three, brookite is rarely used in industrial field as it can change into rutile phase at very low temperatures while the anatase type can easily change to rutile which irreversible when heated above its maximum temperature which is approximately 900 ˚C , shown in figure 2.1 (Bozorgtabar *et al.* 2011).

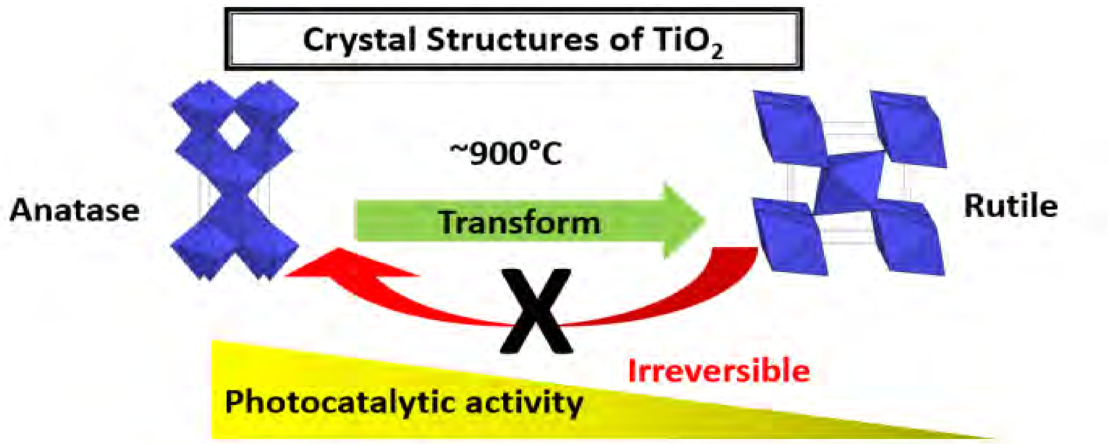

Figure 2.1: Transformation of crystalline structures from anatase to rutile

Based on the United Nations‟ (UN) Globally Harmonized System of Classifications and Labeling of Chemicals (GHS), titanium dioxide (TiO_2) is a white solid inorganic substance and it is thermally stable, poorly soluble, non-flammable and most importantly it is not hazardous. The chemical stability, low cost and lack of toxicity is the terms that best represent TiO_2 material that exhibit the photocatalyst activity (Toibah *et al.* 2016).

2.2.2 TiO_2 as Photocatalytic Application

The world is in need of safe and healthy environment to be a residence for many lives. For that reason, the environmental pollution issue is considered a critical issue and has drawn attention in the world these days. The air pollution problem mostly contributed by forest burning, transportation smoke and industrial activities. The actions have been taken and there are several initiative were proposed and being extensively studied by scientist to overcome it. As the researcher has proved in previous study photocatalytic application of metal oxides is one of the best ways to solve this problem. TiO_2 is the most ideal metal oxide to be used as photocatalyst activity because it has many characteristic that can prevent this problem to occur any further.

Study on TiO_2 has been very broad as it is a cheap, nontoxic, and highly efficient photocatalyst material, thus being applied for many applications such as degradation of organic pollutants, air purification, sterilization and as a demister (Zhong, Chen, & Zhang, 2010). Besides, Gardon *et al.* (2014) highlighted the gradual transition of TiO_2 coating materials from protective surfaces to active functional components has been welcomed and promoted by the whole thermal spray industry, making it widely used in many applications such as bone implants, electric devices, renewable energy, and gas sensors. This is why TiO_2 considered important to coat various materials and used for photocatalytic application.

2.3 Coating Deposition Processes of TiO_2

The coating process has emerged as one of the important process that can provide protection to the product surface and give the product longer life time. There will be three major processes that will be further discussed following the coating industries trend of TiO_2 photocatalytic application nowadays.

In term of coating thermal spray is a group of process used to apply metallic or non-metallic coatings. There are three major categories of the processes including flame spray, electric arc spray, and plasma arc spray. The coating material in form of powder, wire, or rod form will be heated by the energy sources to a molten or semimolten state. Acceleration and thrusting of resultant heated particles toward a prepared surface can occur by mean using either process gases or atomization jets. As the impact occur, a bond forms with the surface, with subsequent particles causing thickness buildup and forming a lamellar structure. Owing to the very high cooling rates characteristic that typically excess of 10^6 K/s for metals, the thin “splats” undergo the changes (Bozorgtabar *et al.*, 2011).

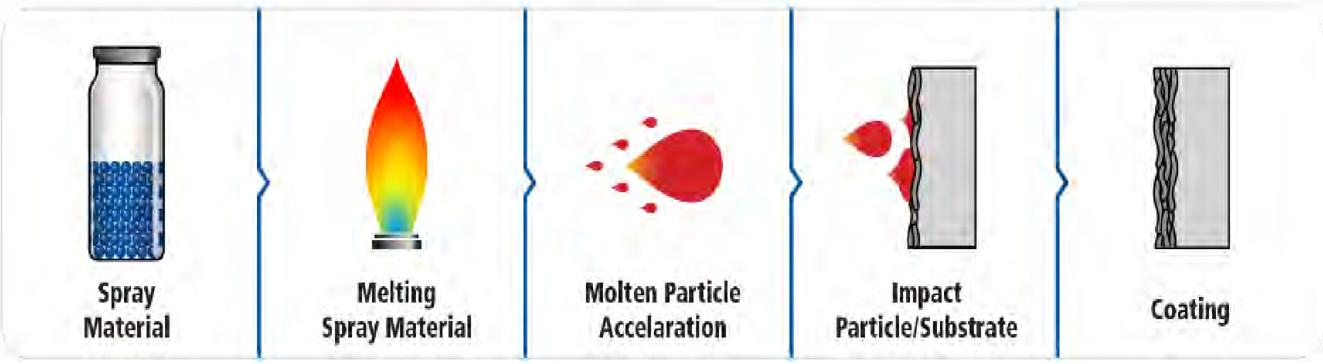

Figure 2.2: The simplified process of thermal spray (Bozorgtabar *et al.*, 2011)

The thermal spray process is one of the promising processes to coat the TiO_2, but it might be a huge challenge to coat this ceramic material. TiO_2 known as a brittle and non-deformable material; make it hard to coat using current thermal process. This is due to the change of anatase phase to rutile under high thermal condition approximately 900˚ C. The change of phase is not favourable for coating because the photocatalytic properties decline by heating.

Based on the Yang *et al,* (2008) another process that has been extensively studied is cold spray process. The cold spray process utilizes the kinetic energy and stands above conventional spray techniques due to its low temperature rates. Pre-heated gas using temperature between (25 – 100 ˚ C) that is lower than melting point of material will helps to accelerate the small particles (5 – 50 lm) and propelled it toward a prepared substrate at supersonic velocities (300 – 1200 m/s). This is the one of the popular process by previous researcher as the structure of powder coating deposition is the same as the starting powder. This can be seen in the figure 2.3 (a) and 2.3 (b) which has the same measurement; 0.2 mm. However, the thickness of deposited coating cannot build up eventhough several pass of deposition was done.

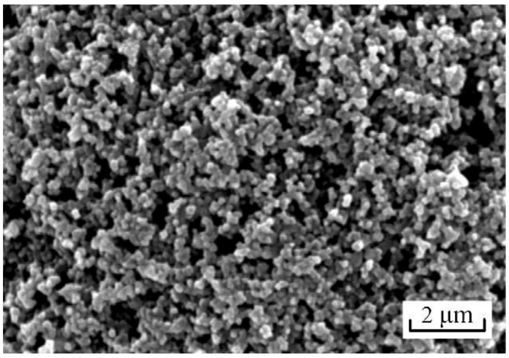

Figure 2.3(a): TiO_2 powder structure (Yang *et al,* 2008)

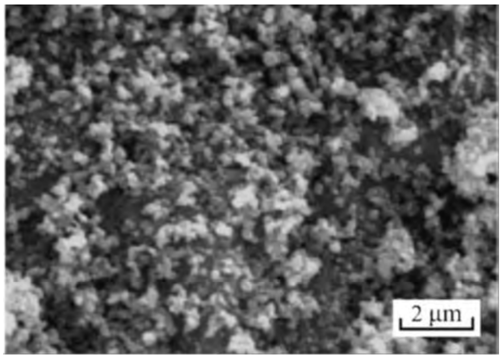

Figure 2.3(b): TiO_2 powder coating deposition (Yang *et al,* 2008)

Apart from that, the electrochemical deposition process is also highly recommended by previous researcher in order to coat the ceramic material onto metallic surface. In sequence to that the electrochemical deposition technique is one of the historical processes as it has been around for a very long time. Rusu *et al*.(2010) stated that electrochemical deposition is a process to coat different metal by depositing a thin layer and enhance or modify the surface properties, by donating electrons to the ions in a solution. Owing to this versatile and unique technique, electrochemical deposition process can be applied to a wide range of potential applications. Moreover, in recent years this technique is spanning throughout the big industries due to its capability in fabricating one-dimensional nanostructures such as nanorods, nanowires and nanotubes.

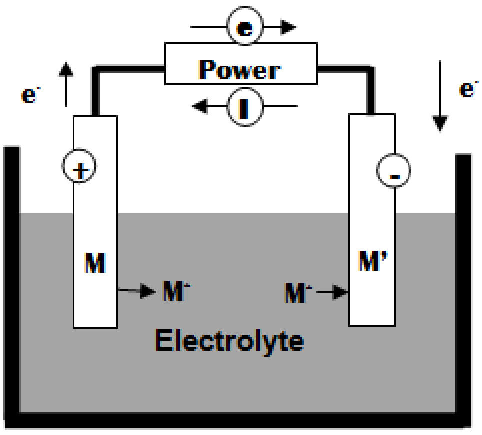

Figure 2.4: Simple electrodeposition system (Rusu *et al*., 2010)