UNIVERSITI TEKNIKAL MALAYSIA MELAKA

CHARACTERIZATION OF TiO_2 COATING PREPARED BY THERMAL SPRAY PROCESS

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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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment 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 Toibah Binti Abd Rahim)

ABSTRAK

Fasa anatas TiO_2 mudah mengubah ke fasa rutile melalui proses semburan haba konvensional disebabkan oleh suhu tinggi pemprosesan. Transformasi adalah tidak boleh diterbalikkan dan tidak wajar kerana ia akan mengurangkan aktiviti photocatalytic TiO_2. Oleh itu, teknik salutan yang memerlukan proses yang bersuhu rendah diperlukan untuk memelihara pembentukan fasa anatas dalam lapisan tersedia. Dalam projek ini, salutan anatas TiO_2 telah direka menggunakan proses semburan sejuk. Sebelum di salut, serbuk TiO_2 telah dicirikan menggunakan SEM, XRD dan PSA. Kemudian, serbuk TiO_2 yang bergumpal disuntik ke dalam aliran gas nitrogen dan disalutkan ke atas substrat tembaga. Suhu proses yang terlibat adalah 350° C dan 600° C. Salutan yang telah di sembur telah dicirikan menggunakan SEM, XRD dan mengukur kekasaran permukaan. Ukuran kekerasan juga adalah diukur di antara bahagian lapisan dan substrat. Analisis XRD ke atas serbuk TiO_2 menunjukkan fasa anatas sahaja yang wujud. Pemerhatian di bawah SEM menunjukkan bahawa serbuk tersebut adalah dalam bentuk gumpalan yang terdiri daripada zarah nano sebagai zarah-zarah yang utama. PSA mendedahkan bahawa serbuk TiO_2 dalam taburan rata. Kajian menunjukkan ketebalan lapisan meningkat dengan peningkatan suhu proses semasa pemendapan semburan sejuk. Kekasaran permukaan lapisan adalah menurun kerana suhu meningkat. Selain itu, analisis XRD mengesahkan bahawa fasa anatas telah dikekalkan selepas pemendapan lapisan. Oleh itu, penyemburan sejuk adalah proses yang sesuai untuk penghasilkan salutan TiO_2 fasa anatase.

ABSTRACT

Anatase phase of TiO_2 is easily transform to rutile phase by conventional thermal spray process due to the high processing temperature. The transformation is reversible and not desirable as it will reduce photocatalytic activity of TiO_2. Therefore, coating technique that require low process temperature is needed to preserve the formation of anatase phase in the as-prepared coating. In this project, TiO_2 anatase coating were fabricated using cold spray process. Prior to the coating, the as-received TiO_2 powders were characterized using SEM, XRD and PSA. Then, the agglomerated TiO_2 powder was injected into nitrogen gas stream and deposited onto copper substrate. The process temperature involved were 350°C and 600°C. The prepared coatings were characterized by SEM, XRD and surface roughness. The hardness measurement was also measured on the cross section of the coating. The XRD analysis on the as-received TiO_2 powders showed that only anatase phase was exist. SEM observation showed that the powders were in agglomerated form composed of nanoparticles as the primary particles. PSA revealed that the TiO_2 powders were homogeneously distributed. The study showed that the thickness of coating was increased with increasing of the process temperature during the cold spray deposition. The surface roughness of the coating was decreased as the temperature was increased. Moreover, XRD analysis confirmed that the anatase phase was preserved after the deposition of the coating. Therefore, cold spraying is suitable process to produce TiO_2 anatase phase coating.

DEDICATION

Only

My appreciated father, Mohamad Bin Abu Bakar

My beloved mother, Aminah Abd Ghani

My brothers and sisters

My beloved friend, Maisarah Binti Kursus@Othman

My Supervisor, Dr Toibah Abd Rahim and all the panel

For giving me moral support, encouragement and also understandings

Thank You So Much

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TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

LIST OF FIGURES

LIST OF ABBREVIATIONS

CVD	-	Chemical vapour deposition
PVD	-	Physical vapour deposition
APS	-	Atmospheric plasma spray
TiO_2	-	Titania
Al_2O_3	-	Alumina
HIP	-	Hot isostatic Pressing
HVOF	-	High velocity Oxy-Fuel
SPS	-	Suspension plasma spray
DE	-	Deposition efficiency
$FeTiO_3$	-	Ilmenite
UV-light	-	Ultra violet light
CB	-	conduction band
VB	-	Valence band
E. coli	-	Escherichia coli
SEM	-	Scanning electron microscope
XRD	-	X-ray diffraction
PSA	-	Particle size analyser

LIST OF SYMBOLS

°C	-	Degree Celsius
MPa	-	Mega Pascal
A	-	Ampere
mm	-	Millimetres
V	-	Volt
%	-	Percentage
°F	-	Degree Fahrenheit
kW	-	Kilowatt
g/min	-	gram per minutes
m/s	-	meter/second
$HV_{0.3}$	-	Vicker Hardness
eV	-	electron volt
A/dm^2	-	ampere/square decimetre
µm	-	micro meter
nm	-	nanometer

CHAPTER 1

INTRODUCTION

1.1. Research Background

Research toward photocatalysis is increasing in recent year which involve wide range of application involving titanium dioxise (TiO_2) as the main contribution in photocatalysis reaction. One of the application are anti-tumor activity, self-cleaning, air purification and many more. TiO_2 allow decomposition of toxicological natural mixes in the water and the destructive gases in the environment.

Almost 1.5 million children die every year worldwide according to World Health Organization (WHO) due to low quality of water which specifically bad hygienic level. Water is the key role to the living things. Environmental pollution especially water pollution has destroyed green environment (Gnanasekaran *et al.*, 2015).

In this study, cold spray as one of the thermal spray process was used to coat on copper substrate. Thermal spray is chosen as they offer the possibility of higher scale fabrication and high deposition coating rate compared to chemical vapour deposition (CVD), physical vapour deposition (PVD), and Sol gel method. This technique not only can produce thick coating but also can produce coating with minimum time. Thermal spray processes is one of the best technique to shield the parts and to give hard and thick coating, subsequently life of material expanded.

The different of cold spray process compared to the conventional thermal spray process is the temperature require the process is below the melting temperature of the feedstock materials. In this technique, the feedstock powders are accelerated to very high velocities below the melting temperature of the powders. The powders adhere to the substrate due to plastic deformation during the collision on the substrate. Since the process temperature is usually lower the melting temperature, it is expected that there is no or very minimum melting of particles have occurred during the process. Moreover, preservation of the initial properties especially on the phase contain in the powder is also possible for the fabrication of anatase TiO_2 coating using this method.

1.2. Problem Statement

Copper is the most widely used metal in the world after silicon and aluminium respectively. Copper used mostly in construction industries and machinery manufacturing. After silver, copper has the second highest electrical conductivity of any element and may be alloyed with zinc, producing brass. Copper wires can handle a wider load of electrical power, allowing it to use less insulation and armoring. Copper is ductile material. Over all the advantages, copper have limitation which are corroded and easily rust, costly, and easily scratch. Hence, one of the solution to overcome corrosion and rust is by the coating process.

It is quiet challenging to use nanopowder TiO_2 as feedstock powder for thermal spray process due to nanostructured TiO_2 can clogged the feeding system easily during the deposition. Thus, agglomerated nano size feedstock powder in micro meter ranged is used to prevent the crisis. However, the major defect of nanostructured TiO_2 is the loss of nano-size grain caused by the melting of feedstock during the coating process using the conventional thermal spray process. Therefore, a coating technique such as cold spray is favourable to produce TiO_2 coating for large area with less time consuming which also can avoid phase changes and minimize the grain growth of nanoparticles due to low process temperature.

In this study, TiO_2 coating was deposited using cold spray process. The effect of process temperature was study on the as-deposited coating.

1.3. Objectives

The objectives of the research are:

1. To characterize the properties of the as-received TiO_2 powder.
2. To characterize the properties of as-prepared TiO_2 coating by thermal spray process using different process temperature.
3. To correlate the effect of process temperature on the properties of thermal sprayed TiO_2 coating.

1.4. Research Scopes

This project will cover the following scopes:

1. Characterization of the feedstock powder for thermal spray process using SEM, XRD and PSA.
2. To prepare TiO_2 coating using one of thermal spray method which is cold spray process which require temperature process below the melting temperature of the powder.
3. To characterize the properties of as-prepared TiO_2 coating by thermal spray process using different process temperature based on the thickness, hardness, and surface roughness of coating by using difference types of characterization tools.

CHAPTER 2

LITERATURE REVIEW

2.1. Introduction

This chapter will focus on the research done by the previous researcher related to the thermal spray, TiO_2 feedstock powder, their mechanical properties and physical properties. Thermal spray in this research focused on the cold spray process.

2.2. Thermal Spray

Thermal spray is a group of coating process which classified mainly into three categories such as plasma arc spray, flame spray, and electric arc spray. The coating process of thermal spray can be used to apply metallic and non-metallic coating. Spray processes that uses thermal energy produce by combustion (chemical) or electric discharge (plasma or arc) method to melt the particles. The energy sources utilized to heat the covering material whether in rod, powder, or wire shape (Figure 2.2) to a liquid or semi-molten state. By either gases or atomization jets process, the resultant heated particles are quickened and moved toward the readied surfaced. Upon effect, a bond form with the surface and subsequent particles creating thickness development and forming the lamellar structure (Figure 2.1) (Davis, 2004)