

**CHARACTERIZATION OF FEEDSTOCK POWDER WITH  
DIFFERENT MORPHOLOGY FOR PLASMA SPRAYED  
TITANIA COATING**

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**UNIVERSITY TEKNIKAL MALAYSIA MELAKA**

**2019**



**CHARACTERIZATION OF FEEDSTOCK POWDER WITH  
DIFFERENT MORPHOLOGY FOR PLASMA SPRAYED TITANIA  
COATING**

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

by

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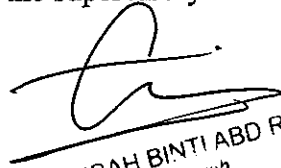
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## APPROVAL

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## ABSTRAK

Kebelakangan ini, beberapa penyelidikan telah menunjukkan bahawa teknik penyemburan haba, terutamanya penyemburan plasma atmosfera (APS), boleh digunakan untuk mendapatkan salutan TiO<sub>2</sub> yang boleh digunakan untuk pelbagai aplikasi dan meningkatkan mutu substrat. Dalam kajian ini, pencirian serbuk bahan mentah dengan morfologi yang berlainan untuk plasma salutan titania yang disembur telah dikaji. Tiga serbuk yang berbeza morfologi iaitu serbuk TiO<sub>2</sub> (6505), serbuk TiO<sub>2</sub> yang bergabung dan dihancurkan (Tayca), dan serbuk TiO<sub>2</sub> yang dihasilkan melalui semburan kering (6233C) digunakan sebagai bahan untuk penyediaan pelet TiO<sub>2</sub> yang padat untuk mensimulasikan salutan titania menggunakan kaedah APS. Sifat-sifat bahan telah disahkan menggunakan penganalisis saiz zarah (PSA), pengimbasan mikroskop elektron (SEM) dan x-ray difraksi (XRD). Kemudian, setiap serbuk ditekan menggunakan 'uniaxial pressing' untuk membentuk badan padat dan disinter pada 1000 ° C untuk mensimulasikan suhu sebenar untuk proses salutan APS. Sifat-sifat pelet TiO<sub>2</sub> yang padat serta telah dibakar dicirikan menggunakan SEM, XRD dan kekerasan diukur menggunakan penguji kekerasan. Kehadiran fasa anatase, brookit dan rutile dalam serbuk TiO<sub>2</sub> telah disahkan oleh XRD. PSA dan SEM mengesahkan bahawa semua serbuk TiO<sub>2</sub> berukuran mikro meter yang sesuai untuk proses semburan plasma. Kajian ini mendapati bahawa tubuh padat sintered yang dihasilkan dari serbuk Tayca yang mempunyai kekerasan tertinggi dengan porositi yang lebih rendah berbanding dengan badan padat yang dibuat dari serbuk 6233C dan 6506 yang disebabkan oleh sifat serbuk awal yang mengandungi nanopartikel. Analisis XRD juga mendedahkan bahawa semua fasa awal TiO<sub>2</sub> telah diubah ke fasa lain selepas proses sintering. Oleh itu, disimpulkan bahawa pembakaran pada suhu tinggi ke atas serbuk TiO<sub>2</sub> yang padat boleh mengubah sifat awal serbuk bahan.

## ABSTRACT

In recent years, several research has shown that thermal spray techniques, especially atmospheric plasma spraying (APS), can be used to get TiO<sub>2</sub> coatings which can apply in various applications and improve properties of the substrates. In this work, the characterization of feedstock powder with different morphology for plasma sprayed titania coating were studied. Three different powder with different morphologies, which were fused and crushed TiO<sub>2</sub> powder (6505), agglomerated TiO<sub>2</sub> powder (Tayca), and agglomerated spray dried TiO<sub>2</sub> powder (6233C) were used as the feedstock materials to prepare sintered dense TiO<sub>2</sub> pellets which mimic or simulate the titania coating using APS method. The properties of the feedstock powders were confirmed using particle size analyzer (PSA), scanning electron microscope (SEM) and x-ray diffraction (XRD). Then, each powder were pressed using uniaxial pressing to form dense bodies and sintered at 1000°C to simulate the actual temperature for the APS coating process. The properties of sintered dense TiO<sub>2</sub> pellets were characterized using SEM, XRD and the hardness were measured using hardness tester. The presence of anatase, brookite and rutile phase in the TiO<sub>2</sub> powders were confirmed by XRD. PSA and SEM confirmed that all the TiO<sub>2</sub> powders were in micro meter sized which are favourable for plasma spray process. This study found that sintered dense body made from Tayca powder having the highest hardness with lower porosity as compared to dense bodies made from 6233C and 6506 powder which due to the property of the initial powder which is agglomerated of nanoparticles. XRD analysis also reveals that all the initial phase of TiO<sub>2</sub> were transformed to other phase after the sintering process. Hence, it is concluded that sintering of dense TiO<sub>2</sub> powders have changed the properties of the initial feedstock powders.

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## LIST OF ABBREVIATIONS

TiO <sub>2</sub>	- Titanium Dioxide
APS	- Atmospheric Plasma Spraying
SPS	- Suspension Plasma Spraying
SEM	- Scanning Electron Microscope
XRD	- X-Ray Diffractometer
PSA	- Particle Size Analyser
H <sub>2</sub>	- Hydrogen
He	- Helium
DE	- Deposition Efficiency





# CHAPTER 1

## INTRODUCTION

### 1.1 Background

Nowadays, the awareness regarding the corrosion problem is rising as the system and process is becoming more exposed to an acidic and oxidation surrounding. Owing to many applications of mild steel in industry, TiO<sub>2</sub> coating are used to coat mild steel to enhance the corrosion resistance properties using plasma spray method. TiO<sub>2</sub> coatings have good electrical and thermal properties and they are resistant to oxidation, corrosion, erosion and wear in high temperature environments (Shanaghi et al., 2009). Plasma spray was seen as the best method to coat TiO<sub>2</sub> onto the substrates due to its fast and economical process for surface modification. Moreover, this coating type is a promising technique which can improve the surface properties of the substrates.

There are few factors that determine the properties of the obtained TiO<sub>2</sub> coating deposited by plasma spray process. The factors are microstructure of the powders, process parameter such as gas, stand-off distance, substrate material and etc which lead to the difference in the properties of the as-deposited coating. Therefore, the interest of this study is to investigate the difference in the performance of coating properties such as hardness, porosity, surface roughness and thickness of the as-

obtained coating due to different type of powder morphology of the feedstock materials. It is believed that powder flowability which influences the continuous feeding when the feedstock materials are used in powder feeder depends on the powder morphology of the starting materials.

## 1.2 Problem Statement

Mild steel is frequently used as a structural material for refining and petroleum production, pipelines, building constructions, and mining (Krishnadevi et al., 2016). However, this type of steel is awfully at risk to corrosion. Corrosion happened when mild steels facing to failure relying on the environmental conditions causing weakening of material surfaces. Advanced ceramics materials,  $\text{TiO}_2$  have been developed over the past half century that exhibit specialized properties such as low modulus, low dense, high corrosion resistance and etc.  $\text{TiO}_2$  usually applied as thermal barrier coatings to protect metal surfaces. Hard ceramic coatings such as  $\text{TiO}_2$  is a good applicants for anti-corrosion applications (Forghani et al., 2014).  $\text{TiO}_2$  coatings are fairly porous and relatively ductile, which can be useful under severe working environments. Advanced coating has been used since 1950s and is the most frequently used technique to deposit thermal barrier coating. According to Jing Xang (2018), APS is one of the advanced hard facing technologies that used to protect various metallic parts, which are exposed to very harsh surroundings. This coating technology also enables preparation of large active coatings that exhibit excellent adhesion to substrates with rather complex shapes and have the widest range of applications that make this process as the most versatile process.

In industrial field, especially coating using the  $\text{TiO}_2$  material has been the most investigated process as it has broad range of application, low cost and chemical stability. Severe research has shown that ceramic materials which are  $\text{TiO}_2$  have unique properties that make them good candidate to use in different fields of applications even under tough environment (Forghani et al., 2013). Hence, titanium

dioxide have been applied as coating on mild steel to improve their hardness, wear, fatigue and corrosion resistance in order to suit many applications. However, the characteristics of the as-sprayed coatings by APS depend not just on the operational conditions but also on the properties of the feedstock powder. Therefore, in this study, three types of TiO<sub>2</sub> powders which possess different characteristic in terms of morphology, particle size and structure were used to investigate its physical and mechanical properties after compacted into dense pellets and sintered at high temperature to mimic the APS process. The properties of the sintered dense body were compared with the initial properties of the TiO<sub>2</sub> powders and also among the different type of TiO<sub>2</sub> powders. The finding of this result might help researcher to decide the type of TiO<sub>2</sub> powder which suitable specific to the desired applications.

### **1.3 Objectives**

The current study mainly focuses on the characterization of feedstock powder with different morphology for plasma sprayed titania coating. To gain this objective, there are three subobjectives that require to be prioritized. This study embarks on the following objectives:

- 1) To characterize various as-received TiO<sub>2</sub> feedstock powders with different morphology specifically produced for plasma spray process.
- 2) To prepare dense cylindrical pellets via uniaxial pressing using TiO<sub>2</sub> powders.
- 3) To characterize the sintered TiO<sub>2</sub> dense pellet by SEM, XRD and hardness tester.

## **1.4 Scope**

This project covered the following scopes:

- a) Characterization of the as-received TiO<sub>2</sub> powders with different morphology using XRD, SEM and PSA.
- b) Sintering of dense cylindrical TiO<sub>2</sub> pellet which were prepared via uniaxial pressing.
- c) Characterization of dense cylindrical TiO<sub>2</sub> pellets using several techniques such as XRD, SEM and Hardness Tester.

## **1.5 Project Planning**

Project planning provides the identification and organization of the project in order to gain the objectives within the period of time that been given. The project will run following its actual track according to the project planning. In this project, Gantt chart has been organized to get the systematic management time. The Gantt chart shows the start time and the end time of the project implementation and summary elements of a project. Appendix A and B shows the detail of the Gantt chart for FYP 1 and 2 so that the aim of the study can be achieved.

## **CHAPTER 2**

### **LITERATURE REVIEW**

This chapter discusses the literature reviews which focus on the past studies by the different researcher that related to the title of this study. Each sources and information of previous studies are extracted as references and discussion based on their research about the TiO<sub>2</sub> coating, TiO<sub>2</sub> feedstock, the coating process and properties of the coating obtained.

#### **2.1 Surface Engineering**

##### **2.1.1 Coating**

Coating is a course of action where the thin layer covering the surface of an object, recognized as substrate. Coating materials are applied to the surfaces either in liquid or powder form. The purpose of the coating is either it can be functional, enhance the aesthetic value, or both which rely on the characteristics of the product. Coatings include paints, drying oils and varnishes, whose primary function is to secure the surface of an object from the environment by increase corrosion resistance, increase life time and etc. (Fauchais et al., 2014). These coatings can also improve the

aesthetic value of an object by complementing its exterior features or even by concealing them from sight. The coating itself may be coating all parts of the product, completely covering the substrate, or it may as it were cover some parts of the substrate (Lech., 1995). In case such as product label on numerous drinks bottles which can be seen these days showed that one side has an all-over functional coating and the other side has one or more coatings in an suitable pattern to create the words and images.

Thus, some artist paints are only for beautification, and the paint used in large industrial sector is only for the function of preventing corrosion. The surface properties of the substrate can be altered using functional coatings such as corrosion resistance, wear resistance or adhesion.

## **2.2 Titanium Dioxide**

### **2.2.1 Properties of titanium dioxide, $\text{TiO}_2$**

$\text{TiO}_2$  is a material that comes from the group of composites and ceramic. In current years  $\text{TiO}_2$  has been given interest due to its advantages in huge industry mainly manufacturing industry. They are fairly porous and generally ductile, which can be useful under extreme working situations (Yilmaz et al., 2007). This verifies that ceramic materials is very beneficial and can be utilized for various industries.

Titanium dioxide is gained from a variety of ores that contain ilmenite, rutile, anatase and leucosene, which are mined from deposits to be found throughout the world (Lyon, 2010). In response of this variety of ores, the yearly yield of  $\text{TiO}_2$  achieves

around 6 million tons in 2008 and its industrial triumph also due to the cost of production that lower than others functional metal oxides (Somi Seong, 2009).  $\text{TiO}_2$  has three crystal polymorphs in nature which are anatase, brookite, and rutile. These three phases of  $\text{TiO}_2$  have the same chemical formula but diverse crystal structures. Rutile is the stable form, whereas anatase and brookite are metastable and are readily transformed to rutile when heated at high temperature (Bozorgtabar et al., 2011). Among these three, brookite is seldomly utilized in industrial field because it can change to rutile phase even at very low temperatures. Meanwhile, the phase transformation from anatase to rutile occurs when the  $\text{TiO}_2$  is heated over its maximum temperature which is at  $\sim 900^\circ\text{C}$  as shown in Figure 2.1. Titanium dioxide ( $\text{TiO}_2$ ) is a white strong inert substance and it is thermally steady, non-flammables, poorly soluble and most imperatively it is not dangerous as stated by the Joined together Nations' (UN) Globally Harmonized System of Classifications and Labeling of Chemicals (GHS). In addition,  $\text{TiO}_2$  has an outstanding resistance in certain marine and chemical environments.

Hence, titanium dioxide's properties include relatively low modulus, low density, wide range of strength and exceptional corrosion resistance over a wide span of environmental conditions.

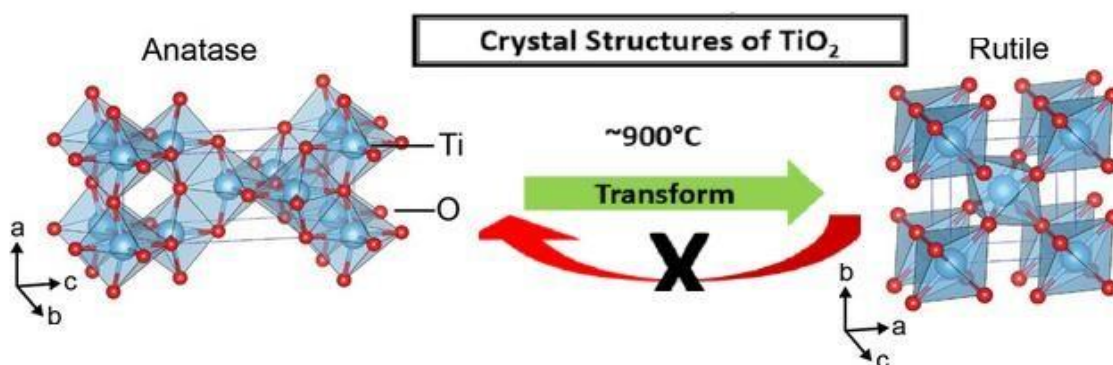


Figure 2.1: Phase transformation from anatase to rutile (Lech, 1995)