UNIVERSITI TEKNIKAL MALAYSIA MELAKA

MICROSTURCTURE OBSERVATION OF PLASMA-SPRAYED TiO_2 COATING ON MILD STEEL SUBSTRATE

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

By

NG SHIH CHIEN

B051610027

960808-01-7410

FACULTY OF MANUFACTURING EMGINEERING

2020

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

Tajuk: **MICROSTRUCTURE OBSERVATION OF PLASMA-SPRAYED TiO_2 COATING ON MILD STEEL SUBSTRATE**

Sesi Pengajian: **2019/2020 Semester 2**

Saya **NG SHIH CHIEN (960808-01-7410)**

mengaku membenarkan Laporan Projek Sarjana Muda (PSM) ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

1. Laporan PSM adalah hak milik Universiti Teknikal Malaysia Melaka dan penulis.
2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.
3. Perpustakaan dibenarkan membuat salinan laporan PSM ini sebagai bahan pertukaran antara institusi pengajian tinggi.
4. *Sila tandakan (√)

☐ SULIT (Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysia sebagaimana yang termaktub dalam AKTA RAHSIA RASMI 1972)

☐ TERHAD (Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/ badan di mana penyelidikan dijalankan)

[√] TIDAK TERHAD

Disahkan oleh:

Alamat Tetap:

126, Jalan Cendana 5,

Taman Rinting, 81750,

Masai, Johor.

Cop Rasmi:

Tarikh: 1/7/2020

Tarikh: ______________________

*Jika Laporan PSM ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali sebab dan tempoh laporan PSM ini perlu dikelaskan sebagai SULIT atau TERHAD.

DECLARATION

I hereby, declared this report entitle "Microstructure Observation of Plasma-sprayed TiO_2 Coating on Mild Steel Substrate" is the results of my own research except as cited in reference.

Signature : ....

Author's Name : NG SHIH CHIEN

Date :1st July 2020

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:

....

(Dr Toibah Binti Abdul Rahim)

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:

....

(Dr Toibah Binti Abdul Rahim)

ABSTRAK

Baru-baru ini, beberapa penyelidikan menunjukkan bahawa ketahanan haus dan kakisan keluli ringan yang rendah mempengaruhi prestasi dan kualiti keluli ringan dalam banyak aplikasi. Untuk mengatasi masalah ini, salutan titanium dioksida (TiO_2) dapat digunakan untuk memberikan lapisan pelindung keausan pada substrat keluli ringan. Dalam kajian ini, salutan penyembur plasma dipilih sebagai kaedah pelapisan untuk menyiapkan lapisan TiO_2 pada substrat keluli ringan kerana dapat menunjukkan ikatan lekatan yang hebat pada substrat. Objektif kajian ini adalah untuk mencirikan serbuk TiO_2 stok suapan yang digunakan untuk menyiapkan lapisan TiO_2 yang disembur plasma pada substrat keluli ringan. Selain itu, struktur mikro lapisan TiO_2 yang disembur plasma pada substrat keluli ringan diperhatikan pada permukaan dan keratan rentasnya untuk mengenal pasti kawasan lebur dan lapisan yang tidak dicairkan dari lapisan yang telah disediakan. Dari pemerhatian SEM, terungkap bahawa serbuk bahan makanan TiO_2 menunjukkan morfologi sudut dan tersumbat. Hasil PSA mengesahkan bahawa serbuk bahan baku TiO_2 bersaiz mikron yang sesuai untuk proses penyemburan plasma. Di samping itu, analisis XRD juga menunjukkan bahawa kedua-dua serbuk bahan TiO_2 dan lapisan TiO_2 yang disembur plasma terdiri daripada fasa rutil dan brookit dan fasa utama adalah rutil kerana rutil adalah fasa yang paling stabil dan biasanya dihasilkan pada suhu proses tinggi. Ketebalan lapisan adalah sekitar 85μm sebagai penyembur plasma yang mampu membentuk ketebalan lapisan tebal berbanding teknik salutan lain. Dari pemerhatian mikrostruktur lapisan TiO_2 yang disemprotkan plasma, didapati bahawa terdapat beberapa liang, zarah lapisan yang tidak dicairkan dan retak terdapat pada permukaan substrat. Oleh itu, disimpulkan bahawa penting untuk mengoptimumkan parameter lapisan penyembur plasma dan keadaan substrat keluli ringan sebelum melakukan proses pelapisan untuk mendapatkan permukaan lapisan yang rata dan rata yang dapat memberikan perlindungan yang diinginkan dan mengekalkan sifat keluli lembut.

ABSTRACT

Recently, several researches have shown that low wear and corrosion resistance of mild steel is affecting the performance and quality of mild steel in many applications. In order to overcome this problem, titanium dioxide (TiO_2) coating can be used to provide wear protection layer of coating to the mild steel substrate. In this study, plasma spray coating is selected as the coating method to prepare TiO_2 coating on the mild steel substrate as it is able to exhibit great adhesion bond to the substrate. The objectives of this study is to characterize the TiO_2 feedstock powders which were used to prepare plasma-sprayed TiO_2 coating on the mild steel substrate. Besides, the microstructure of plasma-sprayed TiO_2 coating on mild steel substrate were observed on its surface and cross section in order to identify melted and unmelted region of as-prepared coating. From the SEM observations, it was revealed that TiO_2 feedstock powders exhibit an angular and blocky morphology. The PSA result confirmed that TiO_2 feedstock powders were in micron-sized which is suitable for plasma spray process. In addition, XRD analysis also showed that both TiO_2 feedstock powders and plasma-sprayed TiO_2 coating consists of rutile and brookite phase and the major phase is rutile since rutile is the most stable phase and usually produced at high process temperature. The thickness of the coating is around 85μm as plasma spray able to form thick coating thickness compared to other coating techniques. From the observation of microstructure of plasma-sprayed TiO_2 coating, it was found that there are several pores, unmelted particles of coating and crack present on the substrate surface. Therefore, it is concluded that it is important to optimize the parameter of plasma spray coating and condition of mild steel substrate before performing coating process in order to get a flatten and even surface of coating which is able to give desired protection and retain the properties of mild steel.

DEDICATION

Only

my beloved father, Ng Kok Ming

my beloved mother, Law Swee Chai

my appreciated younger brother, Ng Sau Ting

for giving me mentally and physically support, cooperation, motivation and also understandings

Thank You So Much.

ACKNOWLEDGMENT

First of all, I would like to express my sincere gratitude to my supervisor, Dr Toibah Binti Abd Rahim for her valuable advices and guidance. Her encouragement and useful suggestions provided me necessary insight into the research problem and paved the way for conducting the report. She helped me a lot in dealing with the obstacles that I faced. The research report was completed smoothly with her clearly direction and proper guidance.

It is a great and unique pleasure to me that I have a chance to thank my beloved family and friends for their encouragement and motivation which helped me a lot in preparation of the report. They gave me strength and right direction towards the completion of report.

In addition, I would also like to express my excessive thanks to all my panels, Associate Prof. Dr. Jariah binti Muhamad Juoi, Dr. Rose Farahiyan binti Munawar and Associate Prof. Ir. Dr. Mohd Asyadi 'Azam bin Mohd Abid for their feedbacks and suggestions to make me avoid from the mistakes in the report. They helped me to adopt new knowledge about the report in order to find the best solution to improve.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST ABBREVIATIONS, SYMBOLS AND NOMENCLATURES

SEM	–	Scanning electron microscope
XRD	–	X-ray diffractometer
PSA	–	Particle size analyzer
TiO_2	–	Titanium dioxide
Al_2O_3	–	Alumina
rpm	–	revolution per minute
cm	–	centimeter
mm	–	millimeter
g	–	gram
sec	–	second
min	–	minute
μ	–	friction coefficient
Fs	–	frictional force
N	–	Newton
Λ	–	wavelength
θ	–	angle
α	–	alpha
γ	–	gamma
°C	–	Degree Celsius
A	–	Ampere
V	–	Voltage

CHAPTER 1

INTRODUCTION

1.1 Research Background

Coating on the surface of materials is mainly used to fulfill the functional purpose for its applications. Coating is essential for optimizing the properties and behavior of the substrate. It helps to give protection on surface to prolong the life period of materials. It also retains the mechanical properties and surface hardness by maximizing the wear performance in order to prevent the damage or crack of materials. It also helps to enhance the surface appearance of the material. In addition, coating helps to limit the speed in corrosion of the metal by reducing the sulfur, chloride, or oxygen content on the surface of metal.

In this study, mild steel is chosen as the substrate material due to it is low cost, high in tensile strength, ductile and malleability. This type of steel is widely used in the industrial applications such as structural steel, machinery part, pipelines, cars and furniture. Mild steel is also known as low carbon steel or plain carbon steel which its percentage of carbon content is between 0.05 to 0.30%. For carbon steel, the carbon exists in the form of iron carbide as it has the ability to increase hardness and strength of the steel. As compared to high carbon steel, the weight of mild steel is lighter and easier to be deformed and machined. Although mild steel is widely used in many industrial applications due to its good mechanical properties, this material experienced poor tribological properties especially severe metallic wear when in contact motion with other materials. Attempts have been made to overcome these problem by applying coating on the surface of the mild steel.

Titanium dioxide (TiO_2) is selected as the feedstock material in the form of powder. TiO_2 consists of one titanium atom and two oxygen atoms which is a white ceramic oxide. TiO_2 is typically inert, non-toxic, non-reactive, white inorganic compound which is odorless and absorbent. Due to its stability, it has enormous applications in photo catalysis, electric appliance, paint, paper, inks and coatings as it can heighten the whiteness and brightness for many materials. TiO_2 exists in 3 crystalline form which are anatase, rutile and brookite. Rutile crystal phases of TiO_2 is more stable compared to brookite and anatase under atmosphere pressure and temperature.

There are several methods can be used to prepare the TiO_2 coating. One of the method is by using plasma spray process as shown in Figure 1.1. Plasma spraying is one of the advanced thermal spray coating technology that can exhibit excellent adhesion to its substrate. Compared with other coating methods such as vapor deposition, electrophoretic deposition and sol-gel, thermal spraying has high flexibility and efficiency in preparation of TiO_2 coating. It also able to provide great thickness of coating on the surface of substrate compared with other coting methods. Many type of materials are available in powder form such as metals, ceramics and alloys, and can be individually selected depending on the substrate. Thus, air plasma spraying is suitable to prepare TiO_2 coatings for mechanical and biomedical applications due to their hardness, wear and corrosion resistance, and biocompatibility.

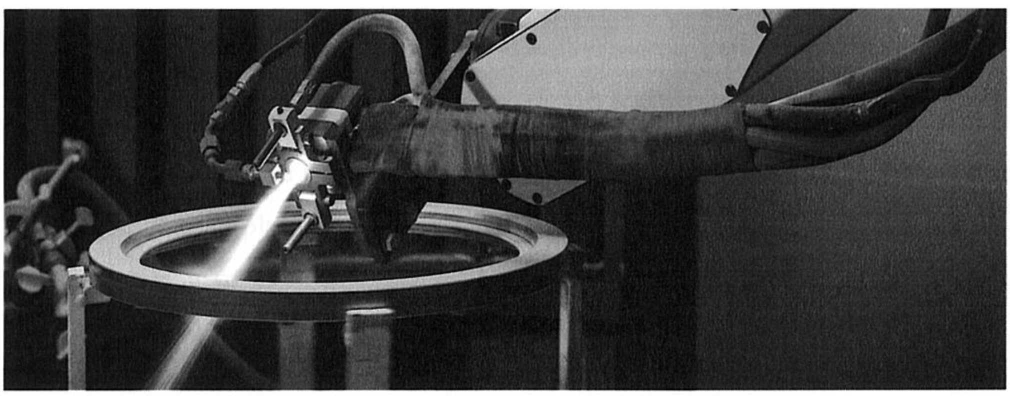

Figure 1.1: Plasma spray coating technique (Oerlikon Metco, 2014).

1.2 Problem Statement

Mild steel is one of the extensively used materials in the engineering applications and constructions industry as it is cheaper and easily to be machined and deformed. But on the other hand, mild steel has limitations which its wear resistance is lower than the other metals and its hardness and tensile strength is lower compared with other types of steel such as high carbon steel and alloyed steel. Mild steel is one type of carbon steel which is also known as low carbon steel since its carbon content is generally lower than high carbon steel. Carbon is the main element that affecting the properties of the carbon steel. The present of carbon is acted as a hardening agent for the steel. It helps to prevent the iron atoms from sliding in the crystal lattice. Because of the lower hardness and tensile strength of mild steel, it will increase the tendency of wear of material. Thus, a harder steel and a stable oxide film layer are able to provide resistance to wear.

Alternative ways such as applying coating, painting, heat treatment or compatible combination of alloy elements would be effective ways to optimize the quality of material and reduce the surface failure problem. Heat treatment is not the most suitable method to solve the problem of wear for mild steel since mild steel has poor high temperature properties while applying painting only helps to improve surface appearance. Besides, the cost of applying coating is generally lower than other solutions. Its high efficiency, flexibility and production rate also the keys to enhance the properties of materials.

Therefore, in order to optimize the performance of mild steel, coating a barrier layer of certain materials on the surface of mild steel is one of the way to overcome problem of mild steel. A protective coating plays the role as a barrier between the surface of material and the aggressive environment or another contacted material. Coating is used as a protective and decorative agent for the surface of material. Titanium dioxide, TiO_2 coating is selected to be coated on the mild steel substrate. TiO_2 feedstock powder will be deposited on the mild steel substrate by air plasma spraying method in order to increase the life time and efficiency of the material.

Coating by using feedstock powder is a durable coating option compared to others method. It is because powders are porous and have smaller surface area which allowing for a better spraying performance in the plasma spray process. By using powder to coat the material, processing times are generally shorter than those used for wet stoving paints since there is no solvent and no flash off period is required. The powder will be melted, flowed and sprayed for creating an even surface of coating on the substrate for modifying the surface and properties of substrate. Besides, powder coated surfaces are more resistant to chipping, scratching, fading, and wearing than other finishes.

The main function of applying the coating are to be decorative and functional of the coated material. Coating is essential in many industrial industries as coating offers surface protection to a number of the products that individuals purchase. Coating helps to increase the wear resistance and prevents corrosion and chemical damage of mild steel. Wear characteristics is highly depending on the mechanical, physical and chemical properties of the material. Wear will affect and weakening the properties of mild steel and its performance. Unlike stainless steel, mild steel is easily to be corroded, rusted and undergo wear which will cause the removal of material on surface of mild steel. Wear is a deformation and continuing loss of material which takes place from one or both two solid surface between contacts. Coating TiO_2 feedstock powder on the mild steel substrate able to create the unique microstructural characteristics in order to exhibit superior physical, chemical, and mechanical properties.

Therefore, in this study, plasma sprayed TiO_2 coatings were prepared on mild steel substrates. The properties of the feedstock powders that were in used in this study were characterized using XRD, SEM and PSA. Then, the microstructure observations were conducted on the surface and cross sectional view of the as-prepared plasma sprayed TiO_2 coating. It is believed that the finding of this study can be used as a viable solution in protection applications and increase the lifespan of mild steel as it is able to produce stable oxide film layer on the substrate which preventing the wear.

1.3 Objectives

The objectives of the project are:

a) To characterize the properties of TiO_2 feedstock powder.

b) To characterize the properties of plasma-sprayed TiO_2 coating on the mild steel substrate.

c) To observe the microstructure of plasma-sprayed TiO_2 coating on mild steel substrate in different views and magnifications on its surface and cross section.

1.4 Scopes

The project will cover the following scopes:

a) Characterization of as-received TiO_2 feedstock powder using several characterization methods such as XRD, SEM and PSA.

b) Characterization of as prepared plasma-sprayed TiO_2 coating using several characterization methods such as XRD and SEM.

c) Comparison on the XDR analysis of TiO_2 feedstock powder and plasma-sprayed TiO_2 coating.

d) Observation on the microstructure view of plasma-sprayed TiO_2 coating on mild steel substrate by SEM in order to identify melted and unmelted region of substrate.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter focusses on the studies and journals that related to the title of this project. Literature review of physical and mechanical properties of mild steel and its limitations by other researchers are discussed in order to identify the problem of mild steel and suitable solutions to solve it. In addition, type of thermal spraying coating, introduction of air plasma spraying coating method and working principles of air plasma spraying are included in this chapter. This chapter also explodes about properties of titanium dioxide (TiO_2) coating and characterization of plasma spray TiO_2 coating by the experiment that conducted by other researchers. Microstructure observation will be focused on by reviewing the surface morphology of the coating.

2.2 Introduction of Carbon Steel

Steel is one of the essential group of material in engineering as it has a wide diversity of uses and applications in industrial. There are several types of steel which are carbon steel, stainless steel, alloyed steel and tool steel. Carbon steel is a type of steel that containing mixture of iron and carbon, with the content of carbon which up to maximum 1.5-2.0%. The carbon will be occurred in the form of iron carbide as it is able to increase the hardness and strength of steel. Besides that, carbon steel contains not more than 0.5% of silicon and 1.5% of manganese which its properties mainly due to its carbon content. There are 3 types of plain carbon steel which are low carbon steel (mild steel), medium carbon steel, high carbon steel. Each carbon steel contains different content of carbon. Different types of carbon steel are used in the various applications (Sanjay *et al.* 2017).

The properties of carbon steel can be enhanced by undergoing suitable heat treatment and process such adding appropriate alloying elements to change physical and mechanical properties of steel. Figure 2.1 shows the effect of carbon content on properties of carbon steel. From the figure, it shows that with an increase in the carbon content in steel, ductility will decrease but the hardness and tensile strength will increase. The maximum increment of tensile strength is achieved at 0.83% of carbon content (Tejero Martin, 2009).

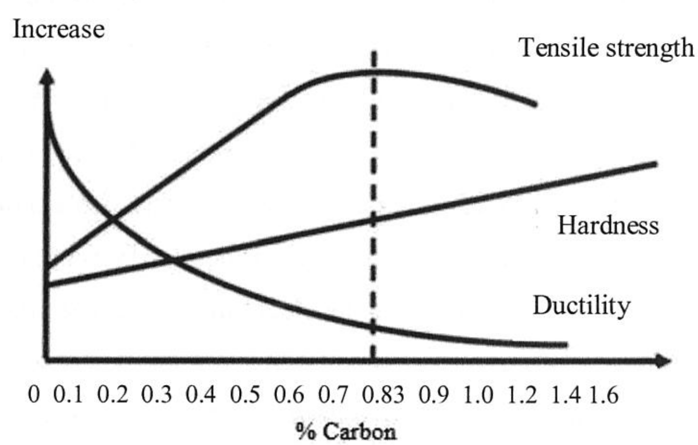

Figure 2.1: Effect of carbon content on properties of carbon steel (Tejero Martin, 2009).

2.2.1 Properties of Mild Steel

Mild steel, also known as low carbon steel or plain carbon steel is a type of steel which its carbon content is about 0.05% - 0.3%. Mild steel contains only iron and carbon as main alloying elements. A maximum percentage contents of 0.25% carbon and 0.4%-0.7% manganese, 0.1%-0.5% silicon and trace of other elements may present in the mild steel. Since the carbon content of mild steel is lower than other carbon steel, its tensile strength and hardness is relatively low. It is cheap, easy to form, malleability and durable steel. Mild steel can be easily machined under many types of processes for example lathe, forging, shaper,