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## DEVELOPMENT AND CHARACTERIZATION OF HARD COATING LAYER ON CP-Ti USING Fe-C-Si POWDER MIXTURE

 $\mathbf{B}\mathbf{Y}$ 

# ADELEKE SAKIRU ADEKUNLE

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> Kulliyyah of Engineering International Islamic University Malaysia

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### ABSTRACT

The growing demand for increasing the wear resistance and hardness of metallic materials has inspired the development of several processes for protective coatings. Tungsten inert gas (TIG) surface modification is one of the most promising processes for this purpose. It enables the formation of a uniform coating by melting the preplaced powder to form the desired composition on the substrate material. In this study, Fe, C and Si (Fe-C-Si) ternary powders were synthesized by preplacement of 1 mg/mm<sup>2</sup> on commercial purity titanium (CP-Ti) substrate using conventional TIG torch arc heat source to form an hard intermetallic compounds. Three different preplaced powder mixtures (PPM) with nominal compositions of 97Fe2C1Si (PPM1), 94Fe4C2Si (PPM2) and 91Fe6C3Si (PPM3) were separately melted on CP-Ti substrate at energy inputs from 945 J/mm to 1350 J/mm in an argon gas environment. The characterization of surface modified CP-Ti was performed using SEM, EDX and analyzer whereas hardness property was measured using MVK-H2 XRD microhardness tester. The tribological behaviour of the surface modified CP-Ti was performed using CSM ball on plate tribometer at room temperature under dry sliding conditions. Results showed that addition of 1 mg/mm<sup>2</sup> of Fe-C-Si powder melting at different energy inputs created a hemispherical melt pool layer with a good metallurgical bonding with the substrate. In general, resolidified melt layers produced different variety of dendrite microstructure whereby the dendrites concentration is more near the surface area compared to the deeper melt depth. The modified surface with PPM2 exhibited maximum hardness of ~ 800 HV 0.5 kgf compared to PPM1 with the hardness value of ~ 530 HV<sub>0.5 kgf</sub> followed by hardness value of 470 HV<sub>0.5 kgf</sub> for PPM3. The hardness developed on the modified surface was found to be strongly dependent on the population of TiC dendrites that segregate at the top of the melt pool layer. The surface modified with different PPM composition showed a significant improvement in the wear resistance. The surface modified with PPM2 is more effective with lowest wear rate of  $1.2 \times 10^{-4}$  mm<sup>3</sup>/Nm compared to CP-Ti which showed highest wear rate of  $5.9 \times 10^{-4}$  mm<sup>3</sup>/Nm. The surface profilometry study showed that CP-Ti processed with PPM2 depicted the lowest wear crater depth of 4.4 µm compared to 65 µm depth for the CP-Ti substrate. Incorporation of Fe-C-Si powder mixture showed a distinctly different friction characteristic under sliding against alumina ball at room temperature, which exhibited a decrease in friction coefficient as compared to untreated CP-Ti substrate. Therefore, TIG melting process can be employed for surface modification of CP-Ti along with other materials in order to improve the hardness and wear resistance properties.

### خلاصة البحث

وقد ألهم الطلب المتزايد على زيادة مقاومة التآكل و صلابة من المواد المعدنية تطوير العديد من عمليات الطلاء واقية التنغستن خاملة تعديل السطح الغاز هي واحدة من أكثر العمليات واعدة لهذا الغرض فإنه يمكن تشكيل طلاء موحد من قبل ذوبان مسحوق لتشكيل تركيبة المطلوب على المواد الركيزة في هذه الدراسة ، والحديد ، والكربون و سي -C )الحديد سي (مساحيق الثلاثي تم توليفها من قبل mg/mm2 على نقـاء التيتـانيوم التجارية (CP-TI) الركيزة باستخدام التقليدية TIG الشعلة قوس مصدر الحرارة لتشكيل مركبات السبائك . ثلاثة مختلفة خلائط مسحوق (PPM) مع التراكيب الاسمية ل\_ل (PPM1) 94Fe4C2Si ، 97Fe2C1Si (PPM1) (PPM2) و PIFe6C3Si (PPM3) و CP-ي في منفصل على -PTي في مدخلات الطاقة من / Q45 Jمم إلى / I350 ملم في الأرجون البيئة الغاز .تم إجراء توصيف سطح تعديل -CPتي باستخدام SEM، SEM و XRD محلل في حين تم قياس صلابة باستخدام خاصية MVK -H2 اختبار الصلادة الدقيقة. تم إجراء السلوك من سطح تعديل -CPتي باستخدام CSMدبوس على القرص في درجة حرارة الغرفة في ظروف الجافة .وأظهرت النتائج أن إضافة 1 mg/mm2 من الحديد -Cسي مسحوق ذوبان في مـــدخلات الطاقـــة المختلفة خلق طبقة تجمع تذوب نصف كروية مع الرابطة المعدنية جيدة مع الركيزة بشكل عام ، تنتج طبقات تذوب متنوعة مختلفة من التغصنات الجهرية حيث تركيز التشعبات هو أكثر بالقرب من منطقة سطح بالمقارنة مع عمق ذوبان أعمق السطح مع تعديل PPM2 أظهرت صلابة الحد الأقصى من 0.5 HV 800 ~كجم ق مقارنة مع PPM1 قيمة صلابة من 530 ~كجم ق HV0.5 تليها قيمة صلابة من HV0.5 كجم ق ل . PPM3 وعثر على صلابة وضعت على سطح تعديلها ل تعتمد بشدة على السكان من التشعبات والتشنج التي تفصل في الجزء العلوي من طبقة تجمع ذوبان أظهرت سطح تعديل مع مختلف PPMتكوين تحسن كبير في مقاومة التآكل السطح مع تعديل PPM2 هو أكثر فعالية مع أدبي معدل ارتداء ( 4-) ^ [ 10 ] × 1.2 ملم 3/Nm ^مقارنة -CPتي والتي أظهرت أعلى معدل ارتداء ( 4-) ^ [[ 10 ]] × 5.9ملم . 3/Nm ^العينة الجهزة مع PPM2 يصور أدبى عمق الحفرة ارتداء 4.4ميكرون مقارنة مع 65ميكرون ل عمق الركيزة -CP تى .أظهرت إدماج الحديد -Cسي مسحوق خليط سمة الاحتكاك تختلف اختلافا واضحا في ظل انزلاق ضد الكرة الألومينا في درجة حرارة الغرفة ، والتي أظهرت انخفاضا في معامل الاحتكاك بالمقارنة مع غير المعالجـــة -CP تي الركيزة .وبالتالي، يمكن استخدام عملية ذوبان TIG لتعديل سطح -CP تي جنبا إلى جنب مع غيرها من المواد من أجل تحسين صلابة و ارتداء خصائص مقاومة .

### **APPROVAL PAGE**

I certify that I have supervised and read this study and that in my opinion, it conforms to acceptable standards of scholarly presentation and fully adequate, in scope and quality, as a dissertation for the degree of Master of Science (Material Engineering).

Md Abdul Maleque Supervisor

Noorasikin Samat

Co-supervisor

I certify that I have read this study and that in my opinion, it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation for the degree of Master of Science (Material Engineering).

Hadi Purwanto Internal Examiner

Mohd Hanafi Bin Ani Internal Examiner

This dissertation was submitted to the Department of Manufacturing and Material Engineering and is accepted as a fulfilment of the requirement for the degree of Master of Science (Material Engineering).

Mohammad Yeakub Ali

Head, Department of Manufacturing and Material Engineering

This dissertation was submitted to the Kulliyyah of Engineering and is accepted as a fulfilment of the requirement for the degree of Master of Science (Material Engineering).

Md Noor bin Salleh Dean, Kulliyyah of Engineering

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Jo my Beloved Parents

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### LIST OF ABBREVIATIONS

AISI	American iron and steel institute
$Al_2O_3$	Aluminium oxide
APS	Atmospheric plasma spraying
$B_4C$	Boron carbide
CP-Ti	Commercial purity titanium
CVD	Chemical vapour deposition
EDX	Energy dispersive X-ray spectroscopy
FeCr	Iron chromide
FeO	Iron oxide
HVOF	High-velocity oxygen fuel
OM	Optical microscope
RSP	Reactive spraying
SEM	Scanning electron microscope
SiC	Silicon carbide
TiC	Titanium carbide
TiN-TiB <sub>2</sub>	Titanium nitride-Titanium diboride
TIG	Tungsten inert gas
TiO <sub>2</sub>	Titanium oxide
Fe <sub>3</sub> C	Iron carbide
PVA	Polyvinyl acetate
PPM	Preplaced powder mixtures
PVD	Physical vapour deposition
RH	Relative humidity
WC-CoCr	Tungsten carbide cobalt chromide
XRD	X-ray diffraction

# CHAPTER ONE INTRODUCTION

#### **1.1 BACKGROUND OF STUDY**

Wear is a process of progressive and undesirable loss or degradation of material from the surface of material components and it is considered as one of the surface failures of material parts. The wear resistance enhancement of component material has encouraged engineers to seek way of improving the surface properties of materials as the resistance to wear is a surface process. Surface modification technique would be an efficient approach to improve the wear property of the components because it can improve the chemical compositions, structures and properties of the metallic and non metallic surfaces without altering the bulk properties of the material. Surface modification helps in cost savings because it involves modifying metal substrate instead of developing bulk alloys. The development of bulk alloy is time consuming and expensive (Dahotre, Sikka and Blue, 2005).

This process is usually employed to make the component better suited in terms of price and service applications. The required surface properties can be achieved by depositing high-wear resistant coatings on the surface of the materials or changing the surface compositions.

Surface modification employs the use of high concentrated heat source which allows the chemical composition and microstructure of a relatively thicker surface layer of a material to be tailored to withstand wear resistance under high loading condition (Hajbagheri, Bozorg and Amadeh, 2008). The use of surface modification is important in increasing the wear resistance of materials because it opens up the possibility where a cheaper and low wear resistance substrate material can be modified to get higher wear resistance without compromising the bulk properties.

Titanium is a transition metal which is abundant on earth. Titanium and its alloys have many attractive properties, including high specific strength and moduli, lower density, and excellent corrosion resistance, primarily due to the formation of a passivate oxide film on the surface of the alloy. These properties have led to the use of titanium alloys in aerospace, marine, biomedical devices, power generation, sports and transportation. However, titanium and its alloys have poor surface properties which restricted the use of these materials in high temperature and high sliding wears applications (Guo, Zhou, Zhao, Wang, Yu, Che and Zhou, 2010). A protective coating or modification of the surface structure is necessary to apply on its surface.

#### **1.2 PROBLEM STATEMENT AND ITS SIGNIFICANCE**

Commercial pure titanium (CP-Ti) and its alloy components are extensively used in aerospace application and their applications are limited because of low wear resistance and high coefficient of friction. CP-Ti and its alloy are being subjected to tribological application which makes it to wear easily with the counterpart material. Another drawback of the CP-Ti is that it is usually being coated with ceramic particles which are very expensive.

In order to improve the wear resistance of CP-Ti with a stable coefficient of friction, material with outstanding high wear resistant and stable coefficient of friction needs to be selected. The surface modification of commercial grade pure titanium (CP-Ti) with Fe, C and Si ternary preplaced powder mixture would offer high wear resistance and a stable coefficient of friction due to the development of new phases on the titanium substrate. The new phases such as TiC and Fe<sub>3</sub>C are responsible for high

hardness and resistance to abrasive wear of this material. Moreover, the presence of silicon would act as oil reservoirs during sliding motion hence, a self lubricating effect to the surface of the parts which may eventually yield a better tribological behaviour of titanium alloy.

Modifying the CP-Ti surface with Fe, C and Si ternary mixture which is cheaper compared to ceramic particles without compromising the intended properties would be of necessity and interesting. Furthermore, a wide variety of methods have been used to produce hard coating layer on Ti and its alloy including PVD, CVD, laser surface cladding, thermal spraying and plasma spraying. All these processes have been successfully used on titanium and its alloy material but they are very expensive and sometimes difficult. Tungsten inert gas (TIG) heat source coating has a potential to be used because as it is cheaper, simpler and usually provide a remarkable enhancement on the wear resistance, corrosion resistance and thermal conductivity without impairing the bulk properties of the material.

### **1.3 RESEARCH OBJECTIVES**

The main goal of this research is to investigate the possibility of forming a hard coating layer on commercial purity (CP-Ti). In order to achieve main objective, the following specific objectives are embarked on:

- To develop hardface coating through incorporation of Fe-C-Si ternary preplaced powder mixture on CP-Ti substrate using TIG torch surface melting technique.
- ii. To study the effect of process variables on surface topography, melt geometry, microstructure and hardness of hard coated layer.

iii. To investigate the wear behaviour of hard coated layer of CP-Ti and compare with uncoated CP-Ti material.

#### **1.4 RESEARCH SCOPE**

The project consists of mainly three different scopes:

- i. In the first part, different compositions of iron, carbon and silicon powder mixture were preplaced on CP-Ti substrate and surface melting was performed using TIG torch with different processing conditions. The electric current and traversing speed were used to control the energy density of the torch.
- In the second part, the metallurgy of the coating layer under different processing parameters thoroughly investigated in terms of microstructure, melt pool configuration and defects under different processing variables.
- iii. In the third part, the hardness and tribological properties of the modified layer are analyzed using microhardness tester and tribometer respectively.

#### **1.5 RESEARCH METHODOLOGY**

A step-by-step frame work has been set up and presented in order to achieve the objectives of this research. The comprehensive flow chart of the experimental work is shown in Fig. 1.1.

- i. Acquiring state of the art knowledge on composite coating on commercial purity titanium in order to develop the hypothesis of the research project.
- Carried out test run for several numbers of preplacement powder with different energy inputs in order to determine the best parameters to be employed.

iii. Sample preparation for characterisation using optical microscopy, SEM, XRD, and EDX. Testing using hardness tester and finally wear and friction study using universal tribometer.



Figure 1.1 Flow chart of the experimental steps

#### **1.6 THESIS ORGANISATION**

The current thesis is divided into five chapters; chapter one presents an introduction to the research work, problem statement and its significance, objective of study with the approach used in the research. Chapter two provides a general overview on surface engineering technology, various techniques for the surface modification of component materials. Also an extensive literature on current state of the art issues concerning wear resistant coating, different techniques that have been used such as plasma spray, thermal spray, laser and tungsten inert gas melting techniques were fully covered.

Chapter three focused on the experimental techniques and preplacement of powder formulation on CP-Ti using tungsten inert gas melting technique. This chapter also discusses the experimental and characterisation analysis on the surface modified commercial purity titanium (CP-Ti). Chapter four presents the results and discussion of the experimental findings. The mechanical properties such as hardness, wear rate and frictional behaviour of surface modified CP-Ti are discussed. In chapter five, a general conclusion on the useful findings and recommendations for further research work are summarised.

# CHAPTER TWO LITERATURE REVIEW

#### **2.1 INTRODUCTION**

Surface engineering technology is an efficient approach to improve the tribological property of metallic and non metallic components by depositing high wear resistant coatings on the surface (Cay, Ozan and Gök, 2011). It is a technique that makes it possible to add a new property, only on the sliding surface, unrelated to the internal properties of the component materials. Because of the wear and corrosion problems, metallic parts are prone to lose their accuracy stability thus making the application of that utilized component to progressively fail (Mridha, Idriss, Maleque and Suryanto, 2012). The use of surface coatings is important in increasing the wear resistance of both metallic and non metallic materials. There are various surface coating techniques, such as nitrocarburizing, plasma nitriding, spark discharging or cladding, hardfacing and chemical and physical vapour deposition processes. Surface modification by means of cladding or alloying is a process by which an alloy powder of a desirable composition and a thin surface layer of the substrate material are simultaneously melted and then rapidly solidified to form a dense coating with strong metallurgical bonding to the substrate. This can produce surface layer that are able to resist extreme wear and corrosion problems, thereby elongating the service life of a part and extend the lifetime of a material part (Kirchgaßner, Badisch and Franek, 2008).

#### 2.2 TITANIUM AND ITS ALLOY

In the past decade, titanium and its alloys have been adopted as standard materials in many industrial applications such as aerospace, chemical plant, petrochemical, biomaterial and automotive parts because of their high strength-to-weight ratio, low density, excellent corrosion resistance and high temperature properties.

There are two most widely used titanium alloys such as Grade 2 commercially pure titanium (CP-Ti) and the age-hardenable Ti6Al4V. The CP-Ti is one of the four grades of pure titanium which differ in impurity level and strength. The component that made from the CP-Ti performs well from the corrosion standpoint but occasionally it has been used in parts or equipments that involve sliding operation (titanium tribosystems). Titanium is an extremely reactive metal and has a poor reputation for tribological application (Budinski, 1991). For this reason they are currently restricted for tribological application. Owing to their poor wear resistance and high coefficient of friction, designers have tried to avoid it in sliding systems. In order to overcome these problems, researchers have used several surface modification methods such as ion- implantation, chemical vapour deposition, physical vapour deposition, laser cladding and thermal oxidation to improve the surface properties of this material.

In this study, an attempt have been made to modify the surface properties of CP-Ti with a preplaced powder mixture of iron, carbon and silicon ternary powder mixture in an argon environment using a novel tungsten inert gas melting technique (TIG).

#### 2.3 TECHNIQUES OF WEAR RESISTANT COATING

Wear resistant coatings consist of carefully applied layers of usually hard materials which are intended to give prolonged protection against wear. There are different techniques by which wear resistant materials or hard coatings can be applied to a metal substrate. PVD, CVD, sputtering and ion plating are used in a similar manner as

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