THE EFFECT OF SUBSTRATE TEMPERATURE OF TITANIUM ALUMINIUM NITRIDE COATING ON TITANIUM ALLOY (TI-6AL-4V) SUBSTRATE USING PVD METHOD

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TO MY BELOVED PARENTS AND SISTERS FOR THEIR ENDLESS LOVE AND SUPPORT

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ABSTRACT

Titanium and its alloys are widely employed in many industrial fields, due to their high specific strength and good corrosion resistance. On the other hand, these materials show relatively low shear strength and high coefficient of friction. As matter of fact, the poor wear resistance of these materials is still a limit to a wider use. However, these disadvantages mainly concern on bulk materials and can be avoided by using intermetallic coatings. Thin hard coatings have good wear resistance only when they have both good cohesion and adhesion. In this study, the effect of substrate temperature of titanium aluminium nitride (TiAlN) coating on the titanium alloy substrate using PVD method was evaluated. The TiAlN coating was deposited on Ti-6Al-4V substrate by DC magnetron sputtering process at various temperature. The reactive sputter gas with a mixture of Ar (99.999%) and N_2 (99.999%) with flow rate of 20sccm and 10 sccm respectively was used. The substrate temperature was set at 200 °C, 240 °C and 300 °C, and the bias was kept at -60V for all the coatings. Structural characterization of the coating was done using X-ray diffraction (XRD). The XRD measurements showed the presence of Ti₂AlN and also, the results show that its intensity was decreased with substrate temperature, however there was no changes in crystalline structure was occurred. The substrate temperature during the growth, change in crystalline structure will not occur. The surface roughness of the coating was determined using an Atomic Force Microscope (AFM). From three-dimensional AFM analysis it was indicated that the surface roughness will decrease as the substrate temperature increases. And also, Crosssectional FESEM indicated the presence of dense columnar structure. The mechanical properties of the coating layers (cohesion and adhesion) were deduce by both Rockwell-c adhesion test and knife test. These two tests indicated that by increasing substrate temperature, the adhesion and cohesion were also increased.

ABSTRAK

Titanium dan aloi digunakan dengan meluas di industri disebabkan oleh kekuatan spesifiknya yang tinggi dan rintangan kakisan yang baik. Sungguhpun demikian bahan-bahan ini menunjukkan kekuatan ricih yang rendah dan pekali geseran yang tinggi. Jadi disebabkan oleh rintangan haus yang rendah, maka kegunaan bahan ini menjadi amat terhad. Kelemahan ini menjadi amat serius bagi bahan-bahan pukal dan boleh dielakkan dengan menggunakan salutan antara logam. Salutan keras nipis ini mempunyai rintangan haus yang baik sekiranya mempunyai kedua-dua sifat "cohesion" dan "adhesion" yang baik. Dalam kajian ini, kesan suhu substrat aloi titanium dengan salutan titanium aluminum nitrat (TiAlN) menggunakan teknik enapan wap fizikal telah dinilai. Salutan TiAlN ini telah disalut di atas Ti-6Al-4V substrat menggunakan kaedah percikan DC magnetron pada suhu substrat yang berbeza. Percikan gas reaktif dengan campuran Ar (99.999%) dan N₂ (99.999%) dengan kadar aliran 20 sccm dan 10 sccm telah digunakan. Suhu substrat telah ditetapkan pada 200°C, 240°C dan 300°C, dan bias telah ditetapkan pada -60V untuk kesemua proses salutan. Pencirian mikrostruktur telah dilakukan menggunakan pembelauan sinar-x (XRD). Pengukuran XRD menunjukkan kehadiran hablur TiAlN, dan intensitinya berubah dengan suhu substrat, walaubagaimanapun perubahan struktur hablur adalah tidak berlaku. Kekasaran permukaan telah ditentukan menggunakan "Atomic Force Microscope (AFM)". Daripada analisis AFM tiga dimensi ianya menunjukkan bahawa kekasaran permukaan berkurang dengan peningkatan suhu substrat. Juga, daripada FESEM keratan rentas menunjukkan kewujudan struktur kolumnar. Sifat-sifat mekanikal lapisan salutan ("cohesion" dan "adhesion") telah dianalisa menggunakan ujian "Rockwell-C adhesion" dan ujian pisau. Kedua-dua ujian ini menunjukkan dengan peningkatan suhu substrat, "cohesion" dan "adhesion" akan meningkat.

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LIST OF ABBREVIATIONS

| PVD | - | Physical vapour Deposition |
|-------|---|---|
| SEM | - | Scanning Electron Microscope |
| FESEM | - | Field Emission Scanning Electron Microscope |
| AFM | - | Atomic Force Microscope |
| XRD | - | X-ray diffraction |
| TiAlN | - | Titanium Aluminum Nitride |
| TiN | - | Titanium Nitride |
| IBED | - | Ion Beam Enhanced Deposition |
| IBAD | - | Ion Beam Assisted Deposition |
| IVD | - | Ion Vapor Deposition |
| IAD | - | Ion Beam-assisted Deposition |
| HCP | - | Hexagonal Close Packed |
| FCC | - | Face Centered Cubic |
| Zr | - | Zirconium |
| Sn | - | Tin |
| Ti | - | Titanium |
| Al | - | Aluminum |
| Co | - | Cobalt |
| Cr | - | Chromium |
| Mo | - | Molybdenum |
| Mg | - | Magnesium |
| Ca | - | Calcium |
| Ni | - | Nickel |
| Si | - | Silicon |

LIST OF SYMBOLS

α - Alpha
 β - Beta
 °C - Degree celsius
 °F - Degree fahrenheit

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CHAPTER 1

INRODUCTION

1.1 Introduction

Ti–6Al–4V alloy has been widely used for lightweight parts and members in a variety of industrial products since its first production in 1954. At the present time, this typical α + β alloy covers 56% of the titanium market in the USA. The widespread application of Ti–6Al–4V alloy results from its light weight, good corrosion resistance and excellent combination of static strength and ductility. Moreover, this alloy has a potential for further strengthening through tailoring microstructures. In contrast to these advantages, they have relatively poor tribological properties and low abrasive and adhesive wear resistance. A considerable amount of effort has been made to overcome these problems and improve the service life of components by applying several surface treatments such as plasma and laser nitriding, plasma spray, and etc. Although all surface engineering processes with different levels of success have been applied on this alloy, one the promising method for improving these downsides is utilizing coating.

Nowadays coatings, or thin films, are used today in a wide range of applications, from decorative coatings on household items, to highly complex layers in the microelectronics industries. Thin films are mostly created by the condensation of a vapour on the surface to be coated, and through deposition as a thin layer, materials with widely different properties than those achievable in bulk phases are possible. This is the reason for the popularity of thin film processes, as the combination of substrate and film enables properties that would be impossible to achieve with one component alone. Common properties to which thin films provide an improvement or specialization are electrical, magnetic, optical, and most importantly for the work herein, hardness, wear resistance, and thermal stability.

Titanium aluminium nitride is a material which has many remarkable properties, which lend themselves to a wide range of applications. Correspondingly, Titanium Aluminium Nitride coatings have successfully been used as a coating layer for many applications such as biomaterials (Subramanian, Muraleedharan, Ananthakumar, & Jayachandran, 2011), high temperature cutting tools (D. Y. Wang, Chang, Wong, Li, & Ho, 1999), and decorations (Pierson, 1996).

Despite the great appeal of titanium nitride coatings, there are several problems with existing coating methods which limit the use of such coatings. For example, conventional Chemical Vapour Deposition (CVD) methods for depositing nitride thin films require high temperatures (Ohring, 2002) (TiN coating temperature is around 1000°C), which can be quite undesirable for Ti-6Al-4V which is very sensitive to temperature. Also, the high temperatures required for deposition, coupled with two different thermal coefficients of expansion existing for the coating and substrate, result in residual stresses between the coating and substrate that may reduce the fatigue characteristics of the material (Hertzberg, 1996). Furthermore, one of the considerations for using Chemical Vapour Deposition (CVD) is safety, because of utilizing and producing toxic gases such as TiCl₄, HCl and Cl. Thus, Physical vapour Deposition is a dominant method for producing Nitride coatings. Thus, in this study, the process of coating considered Physical Vapour Deposition and the substrate temperatures for this research were as low as possible (100°C, 240°C and 300°C).

The PVD processes may be categorized as either evaporation or sputtering. In the evaporation process, vapours are produced from a source material (Titanium Aluminium when it is vaporised) which is heated by direct radiation, eddy current heating, electron-beam bombardment, lasers, or an electrical discharge. In the sputtering process, positive gas ions (usually argon ions) produced in a glow discharge or neutral atoms bombard the target material (a titanium aluminium cathode when depositing TIAlN), dislodging groups of atoms that then pass into the vapour phase and then deposit onto the substrate surface (Window, 1995).

1.2 Problem of Statement

Owing to their superior mechanical properties, excellent corrosion and wear resistance and good biocompatibility, TiAlN coatings are often used as cutting tools and orthopaedic implant materials in order to extend their life span. The quality of the coating surface is absolutely is dependent on the parameters in the sputtering. Based on importance of TiAlN application, there are some significant reasons to carry out this project. Since substrate temperature plays very important role in the quality of the coating surface, measuring the best temperature is the importance of this study.

Although there have been the numerous studies which discussed the effect of this parameter, the lack of study in this area can be felt. As a result the state of problem of this study is: Titanium alloys are widely used in engineering applications such as aerospace, biomedical but its properties or lifetime can be greatly improved by adding hard layer on its surface. Therefore, by applying a TiAlN layer can become one of the solutions.

1.3 Objective of the Study

This project focuses on wear resistance and quality of TiAlN coated on Titanium alloys especially Ti-6Al-4V, and compares the effect of substrate temperature on the coating surface. So, objective of the study can be classified by:

• To evaluate microstructural analysis and cohesion property on the effect of substrate temperature of TiAlN coating on the Titanium alloys substrate using PVD method

1.4 Research Questions

The following paragraph is the research questions of this study.

- (i) What is the effect of substrate temperature composition on hardness?
- (ii) What is the effect of substrate temperature on the morphology of the surface?
- (iii)How substrate temperature effect on surface roughness?

1.5 Scope of the Study

The scope of study for this research is mentioned as below:

- (i) To perform substrate pretreatment.
- (ii) To produce TiAlN coatings on Ti-alloys using PVD method.
- (iii)To perform microstructural evaluation on surface and cross-sectional of coated Ti-alloys using FESEM / AFM.
- (iv)To perform coating phase identification using XRD.
- (v) To perform mechanical adhesion test of the coated specimen using Rockwell C adhesion test and knife test.

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