EFFECT OF NITROGEN PRESSURE ON Ti/TiAl COATING ON 304 STAINLESS STEEL BY PVD-DC MAGNETRON SPUTTERING

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A project report submitted in partial fulfillment of the
requirements for the award of the degree of
Master of Engineering (Material Engineering)

Faculty of Mechanical Engineering

Universiti Teknologi Malaysia

JANUARY 2013

I would like to dedicate my thesis to my beloved parent and my wife and daughters

"You have given me so much, thanks for your faith in me, and for teaching me that I should never surrender"

ACKNOWLEDGEMENT

The author wishes to express his sincere appreciation to all who have helped directly or indirectly in his Masters Project research work. The first is to Allah for his prosperity and guidance. A big thank is to my main thesis supervisor, Prof Dr. Mohd Hasbullah bin Hj Idris, for his passionate assistance, and concern. With his invaluable advices and superb directions, the author has successfully completed his master’s thesis. It is indeed a true honor and privilege for being able to work under the supervision of such a dedicated and enthusiastic lecturer.

Special thankful is due to my co-supervisor Associate Dr.Muhamad Aziz Mat Yazid for her close guidance and assistance throughout the process of carrying out the research work.

Last but not least, the author would like to express his heartfelt gratitude to his family members and friends for their utmost support and motivation throughout this research work. Thanks to all of them.

ABSTRACT

In recent years, for hard coating material studies it was focused on particular coated components for drilling bit and cutting tools such as end mulls, drills and cutting inserts. Titanium nitride (TiN), which is widely used as a hard coating material, was coating on 304 stainless steel substrate materials, because of excellent properties such as adhesion to substrates, high chemical inertness, resistance to elevated temperatures, hard surface (2400 HV) and low By comparison, TiAlN can significantly increase tool lifetime, therefore, it can reduce machine downtime and increases in productivity. In this study, Physical Vapour Deposition method (DC reactive Magnetron Sputtering) was used. This method is widely used for depositing hard coatings on subtract for tool applications. The effect of nitrogen pressure during deposition on microstructure, surface roughness and wear behavior of coating film for both targets was studied. The results FESEM analysis showed columnar structures were formed for both types of coating with thickness of 826.3 nm. From XRD analysis, for TiN the dominant growth plane is (111), whereas for TiAlN is (200). From three-dimensional AFM analysis it was indicated that surface roughness will increase as the N_2 pressure increase. From multi pass scratch test analysis it was showed that the lowest friction and better wear resistance is at N_2 pressure of 10sccm and 8sccm for TiN and TiAlN respectively. Friction coefficient of friction TiN shows limited oxidation resistance and may start to oxidize at temperature above $500°C$, therefore TiAlN become an alternative because it can be withstand at extreme temperature up to 800℃.

ABSTRAK

Sejak akhir-akhir ini, penyelidikan dalam bidang saduran untuk aplikasi mata alat dan mata pemotong adalah sangat tinggi Titanium nitrat (TiN), sangat meluas digunakan sebagai salutan keras yang mana di sadur di atas besi tahan karat 304 kerana mempunyai sifat-sifat yang sangat baik seperti daya lekatan yang kuat ke atas substrat, daya tahan kimia yang baik, daya tahan terhadap perubahan sifat pada suhu tinggi, mempunyai kekerasan yang tinggi (2400 HV) dan mempunyai koefisien geseran yang rendah. TiN memberikan rintangan terhadap pengoksidaan yang terhad dan mula teroksida pada suhu di atas 500℃, oleh itu saduran TiAlN boleh digunakan sebagai pilihan kerana ianya mempunyai daya tahan sehingga suhu 800℃. Secara perbandingan, salutan TiAlN boleh meningkatkan jangka hayat mata pemotong jadi ianya boleh mengurangkan masa penyelenggaraan dan meningkatkan produktiviti berbanding salutan TiN Dalam penyelidikan ini, saduran wap fizikan secara arus terus (PVD - DC) digunakan. Teknik ini sangat meluas digunakan untuk saduran keras di atas substrat untuk aplikasi mata alat. Kesan kandungan gas nitrogen semasa saduran terhadap mikrostruktur, kekasaran permukaan dan sifat kehausan pada kedua-dua jenis saduran yang digunakan akan dikaji. Daripada analisis FESEM mendapati bahawa struktur salutan yang terhasil adalah dalam bentuk kolum untuk kedua-dua jenis salutan yang mempunyai ketebalan 826.3nm. Daripada analisis XRD, kita mendapati, bagi salutan TiN, pertumbuhan salutan adalah dominan pada planar (111), sementara bagi TiAlN adalah (200). Daripada analisis tiga dimensi AFM mendapati permukaan yang terhasil adalah lebih kasar pada tekanan nitrogen yang tinggi. Daripada ujian pelbagai – goresan mendapati, daya geseran terendah dan daya rintangan haus yang baik terhasil pada tekanan nitrogen 10 sccm untuk salutan TiN, manakala untuk salutan TiAlN adalah pada kadar 8 sccm

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LIST OF ABBREVIATIONS

ARC	-	Arc Vapor deposition
AFM	-	Atomic Force Microscope
CVD	-	Chemical Vapor Deposition
DC	-	Direct Current
EDX	-	Energy dispersive X-ray
FCC	-	Face Center Cubic
FESEM	-	Field Emission Scanning Electron Microscope
HV	-	Hardness in Vickers scale
HRC	-	Hardness in Rockwell C scale
IAD	-	Ion Assisted Deposition
MPST	-	Multi Pass Scratch Test
PVD	-	Physical Vapor Deposition
Ra	-	Average Surface Roughness
RF	-	Radio Frequency
SS	-	Stainless steel
OM		Optical Microscopy

CHAPTER 1

INTRODUCTION

1.1 Introduction

In thin film technologies, titanium nitride-based coatings deposited by physical vapour deposition (PVD) methods are widely applied to machining of steel cutting tools to improve tribological performance owing to their superior properties, which include not only high hardness and a low coefficient of friction, but also excellent corrosion, oxidation and wear resistance [1-8].

However, the deformation mechanisms operating under stress, such as cracking, shearing, elastic and plastic deformation, as well as delamination of these hard coatings, including TiN and TiAlN, on ductile steel substrates, are still not well understood. An improved understanding of the deformation mechanisms in these coatings will assist in improved component reliability and enhanced service life.

This project is conducted to study the effect of nitrogen pressures on deposit titanium nitride (TiN) and titanium aluminum nitride (TiAlN) coating via physical vapour deposition direct current magnetron sputtering deposited coating on 304 stainless steel. The characteristics the deposition coating in terms of microstructural and mechanical properties.

This chapter begins with the background of the problem, which covers the issues leading to the problem statement. This is followed by the problem statement, objectives of the study, significance of the study, and scope of the study, an overview of the research methodology.

Chapter two presents the literature view for substrate material and Ti/TiAl targets and mechanical properties of coating films, as well as the types of coating process. Chapter three describes the research methodology for this study, which include a preparation the samples and coating process discusses, the equipment that were used to analysis the coating film. Chapter four discusses result and discussion of. Finally chapter five presents the conclusion from the study.

1.2 Research Background

Very often, coating cutting tools break during operation because of high temperature and wear mechanism. Poor adhesion between the deposited coating and substrate shows sometime poor response in service.

Mechanical components and tools are facing higher performance requirements. The use of surface coating opens up to the possibility for material design in which the specific properties are located where they are most needed. The substrate material can be designed for strength and toughness, while the coating is responsible for the resistance to wear, thermal loads and corrosion. Surface treatment offer remarkable choices for a wide range of tribological applications where the control of friction and wear are of primary concern [1].

The requirement for machine tools namely for cutting purposes and various mechanical parts to have excellent properties have created competition among the industries to produce high quality tools and machine parts. The properties being sought after by end users include toughness, strength, and hardness; wear resistance and high hot hardness. A combination of hard surface and tough matrix is desired in dynamic application. Therefore, many have been trying to find the best combination between the substrate and coting material. In addition, they are also trying to find the various ways and means to improve the material properties (directly correlate with material performance) with relevance to material processing parameters [3, 4].

One of the tool materials that have wide applications in industry is 304 stainless steel since they can adapt well to different kinds of machine tool. Other tool materials have substituted 304 SS in certain application due to its limitations, which include low hot hardness, limited harden ability and wear resistance. Cutting tools and various mechanical parts are often targeted when manufacturers look for improvements in overall productivity of machining systems.

As a consumable item, the selection and use of a correct tool with optimum process setting will bring about considerable savings. The continuous development and improvement of these cutting tool material coupled with that of coating material and coating technology have led to the widespread availability of new tools. These new products can be used at higher speeds and at the same time last longer under increasingly demanding operating condition.

1.3 Problem Statement

Hard coating has been increasingly being used in industry. Numerous studies have been reported on the successful implementation of hard coating, mainly on 304 substrate and on HSS, 316 e.g. tool steel, in order to produce a tool material that has a good combination of tough substrate and hard surface with other material properties, which have a direct impact on the material performance. [3, 4, 5, 6] used TiN and TiAlN as the coating material.

The strength of a metal-ceramic interface is strongly influenced by the chemical composition of the region at or close to the interface. Both structure and bonding across the interface have great effects on the properties of such materials. To design and optimize such metal-ceramic material the first essential question is how to understand atomic structure and bonding mechanisms at the atomic level [7].

According to application of TiN as a coating material, however, has not been potentially used since the problem of adhesion between TiN and substrate, close interrelationship among various properties and some of TiN films are how easy to delaminate after being deposited on the substrate, particularly TiN poor performance cutting above 500℃ In recent years, there have been considerable advances in the development of hard and wear-resistant coatings for applications in the field of cutting tools and other moving parts to improve durability. Addition of Al into TiN or CrN has been shown to improve the oxidation resistance of transition metal nitrides considerably [8]. The properties of (Ti,Al)N film can be controlled by various deposition parameters. The microstructure and mechanical properties of the (Ti,Al)N film are most dependent on bias voltage, nitrogen pressure and the aluminum to titanium ratio. Thus, this project concentrates in the effect of nitrogen partial pressure on the microstructure, mechanical properties and surface roughness of TiN, TiAlN coating on the 304 SS cutting tools. It also emphasizes the optimization of nitrogen pressure in order to obtain batter mechanical properties.

1.4 Objective of study

This project work seeks to:

1. Study the effect of nitrogen pressure in the deposition of Ti, TiAl coating properties
2. Study the microstructure, surface roughness and wear of Ti, TiAl coating on 304 stainless steel

1.5 Scope of study

1. Evaluation of Ti, TiAl coating process based on 304 stainless steel substrate with the changing nitrogen pressure by PVD magnetron sputtering method.
2. To study the microstructure of the coating layers by using SEM- EDX and XRD.
3. To evaluate the mechanical properties of the coating layers based on surface roughness and wear.
4. Compare the microstructures and mechanical properties of TiN and TiAlN coating layers with which deposited on 304 stainless steel.

1.6 Significance of study

There is always ever increasing request from high technological industries for the unique and specialized material and products. The selection of a suitable material for a specific application depends on several processing parameters.

Coatings are widely used to improve the performance of industrial tools. The properties of every material are closely related to its microstructure. The microstructure, however, depends on chemical composition of the material and this in turn is strongly dependent on the material processing and preparation method.

A study of PVD TiN/TiAlN coating tool steel has attracted a lot of research interest because of their applications in both research and industry. TiN/TiAlN coatings have tremendous potential in the field of automobiles and aircraft, especially for cutting tools applications and various mechanical parts, e.g. drilling, milling, dies, etc.

Coating protects the tools initially from direct wear, which lowering the friction between tool and work-piece, reducing the cutting force, and changing the contact geometry and the temperature distribution in the tool and chip. The wear rate is drastically reduced, and higher cutting speeds are made possible [2]. Low process temperature and environment friendly in nature make PVD especially suitable for the coating of finished, heat-treated 304 stainless steel tools.

PVD techniques are widely used as hard coating deposition technique because of increase wear resistance, strong adhesion and high hardness. TiN deposited by PVD technology has been identified as one of the more promising protective coating for cutting tool application. Critical feature of TiN coating include high hardness, better wear, corrosion, heat resistance.

It is expected that the findings from this research would enhance our knowledge thereby providing a better understanding of the effect of nitrogen pressure on deposition film and characteristics, as leading toward the tooling application. It is hoped that the TiN/TiAlN PVD coating tool steels investigate in this study could be successfully applied for cutting tool and various mechanical parts more effectively and productively, thereby providing an alternative to the us of low performance and expensive cutting tools and mechanical part materials.

1.7 Overview of Research Methodology

The achievement of the objectives of the project requires the methodology as outlined in the following:

1. Literature review on related subject
2. Select the appropriate method for material processing, namely the coating process
3. Select the parameter values for coating process
4. Deposition of TiN/TiAlN on 304 tool steel by using magnetron sputtering
5. Metallography study
6. Material characterization by:
   i. X-ray diffraction (XRD) for phase analysis and crystallographic studies.
   ii. Field emission scanning electron microscope (FE-SEM) with energy dispersive X-ray (EDX) for surface image analysis, interface studies, particle morphology, and elemental composition analysis.
   iii. Scratch Tester to determine the strength of adhesion between substrate and coating
   iv. Atomic Force Microscope (AFM) for analysis the surface roughness of the films surface.
7. Data analysis and validation.

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