



**Faculty of Manufacturing Engineering**

**EFFECT OF COATING THICKNESS ON THE MICROSTRUCTURE,  
MECHANICAL AND WEAR PROPERTIES OF AITiN COATINGS  
DEPOSITED USING ARC ION PLATING TECHNIQUE**

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**EFFECT OF COATING THICKNESS ON THE MICROSTRUCTURE,  
MECHANICAL AND WEAR PROPERTIES OF AlTiN COATINGS  
DEPOSITED USING ARC ION PLATING TECHNIQUE**

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## DECLARATION

I declare that this thesis entitled “Effect of Coating Thickness on The Microstructure, Mechanical and Wear Properties of AlTiN Coatings Deposited Using Arc Ion Plating Technique” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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## APPROVAL

I hereby declare that I have read this thesis and in my opinion, this thesis is sufficient in terms of scope and quality for the award of Doctor of Philosophy in Manufacturing Engineering.

Signature : .....

Supervisor Name : Associate Professor Dr. Md Nizam Bin Abd Rahman

Date : .....

## **DEDICATION**

To my beloved parents, mother, father, and wife, my sons, and my daughters.

Dedicated, to my beloved, brothers and sisters.

Dedicated, to all my family, and my friends.

Thank you for your support and encouragement.

You all are everything for me.

May Allah bless all of us. Insha'Allah.

## ABSTRACT

AlTiN coatings were deposited on tungsten carbide insert using  $\text{Al}_{0.67}\text{Ti}_{0.33}$  cathodes in cathodic arc plating system. The influence of coating layer thickness on the microstructural, mechanical and wear properties of the coatings was investigated. The AlTiN deposition of hard coatings for tooling applications has many advantages. The main drawback of this technique, however, is the formation of macro particles (MPs) during deposition. The deposited AlTiN coating of various thickness was characterized using X-Ray Diffraction (XRD), scanning electron microscopy (SEM), atomic force microscopy (AFM), Rockwell tester, and ball-on-disc machine, to analyze and quantify the following coating properties: grain size, preferred orientation, atomic elements, thickness, micro hardness, adhesion, surface roughness, and coefficient of friction (COF) of the deposited coatings. Base of this study, the increase in layer thickness was closely related to increase in deposition time. The coating thickness had a significant influence on the width of crater wear. The width of crater wear reduced significantly as the coating thickness increased. However beyond  $5.815\mu\text{m}$ , further increment in coating thickness resulted in increase in crater wear width due to presence of larger macro-particles on the coating surface and reduction of coating hardness. The deposited AlTiN coating also showed a strong preferred orientation of (200) and (111) plane for all coating thickness. The values of various surface roughness measured by *AFM* was at minimum of  $0.07157\mu\text{m}$  for the coating having thickness of  $2.717\mu\text{m}$  whereas; the maximum value of  $R_a$  was  $0.29647\mu\text{m}$  for the coating thickness of  $8.760\mu\text{m}$ . The highest hardness value was  $1939.0\text{HV}$  for coating thickness of  $5.815\mu\text{m}$ . Based on adhesion slope technique, AlTiN coatings of  $3.089\mu\text{m}$  thickness had the best adhesion strength. Correlation study indicated that cutting tools crater wear width has the strongest correlation with the coating atomic percentage ratio of Al/Ti, with coefficient of determination  $R^2$  value of  $0.7251$ . The coating hardness and grain size also indicated some correlation with the crater width wear with  $R^2$  values of  $0.6051$  and  $0.5184$  respectively.

## ABSTRAK

Lapisan AlTiN dideposit pada insert tungsten karbida menggunakan katod Al<sub>0.67</sub>Ti<sub>0.33</sub> dengan sistem penyaduran arka. Pengaruh ketebalan lapisan salutan pada mikrostruktur, mekanikal dan sifat-sifat lapisan telah dikaji. Salutan AlTiN untuk aplikasi alat mempunyai banyak kelebihan. Bagaimanapun, kelemahan utama teknik ini adalah pembentukan zarah makro semasa pemendapan. Salutan AlTiN yang didepositkan dengan pelbagai ketebalan dicirikan menggunakan mikroskop elektron pengimbas, daya mikroskop atom, mesin ujian kekerasan, Rockwell penguji, dan bola ke cakera mesin untuk menganalisis dan mengukur sifat-sifat lapisan berikut: saiz butiran, orientasi pilihan, unsur-unsur atom, ketebalan, kekerasan mikro, rekatan, kekasaran permukaan, dan pekali geseran. Keputusan kajian ini menunjukkan peningkatan dalam ketebalan lapisan berkait rapat dengan masa pemendapan. Apabila masa pemendapan meningkat begitu juga ketebalan lapisan. Ketebalan salutan mempunyai pengaruh yang besar ke atas lebar kehausan kawah. Lebar kehausan kawah berkurangan dengan ketara apabila ketebalan lapisan meningkat. Namun selepas kira-kira enam mikron, kenaikan berikutnya di dalam ketebalan salutan mengakibatkan peningkatan dalam lebar kehausan kawah. Salutan AlTiN yang didepositkan menunjukkan orientasi kristal yang kuat pada (200) dan (111) untuk semua ketebalan salutan. Nilai kekasaran permukaan diukur oleh daya mikroskop atom yang paling minimum ialah 0.07157 $\mu\text{m}$  untuk lapisan yang mempunyai ketebalan 2.717  $\mu\text{m}$ . Nilai maksimum  $R_a$  adalah 0.29647 $\mu\text{m}$  untuk ketebalan lapisan 8.760  $\mu\text{m}$ . Nilai kekerasan tertinggi ialah 1939.0HV untuk lapisan ketebalan 5,815  $\mu\text{m}$ . Berdasarkan teknik lekatan cerun, AlTiN lapisan ketebalan 3.089 $\mu\text{m}$  mempunyai kekuatan lekatan yang terbaik. Kajian korelasi menunjukkan bahawa lebar kehausan kawah alat pemotong mempunyai korelasi yang kuat dengan lapisan nisbah peratusan atom Al / Ti, dengan nilai  $R^2$  0.7251. Kekerasan salutan dan bijirin saiz juga menunjukkan korelasi sederhana dengan memakai lebar kawah dengan nilai  $R^2$  masing-masing ialah 0.6051 dan 0.5184

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## LIST OF ABBREVIATIONS

Å	Angstrom refer to in terms of measurement ( $1 \times 10^{-10}$ meter or 0.1 nm) function
AFM	Atomic Force Microscopy
Al	Aluminum
AlTiN	Aluminium Titanium Nitride
Amorphous	Phases are important constituents of thin films, which are solid layers of a few nm to some tens of $\mu\text{m}$ thickness deposited upon a substrate.
Anticorrosion	Refers to the protection of metal surfaces from corroding in high-risk (corrosive) environments. When metallic materials are put into corrosive environments, they tend to have chemical reactions with the air and/or water. The effects of corrosion become evident on the surfaces of these materials. Therefore, metal equipment lacking any preventive (anti-corrosive) measures, may become rusted both inside and out, depending upon atmospheric conditions and how much of that equipment is exposed to the air. There are a number of methods for preventing corrosion, especially in marine applications.
Ar	Argon
C	Carbon
CNC	Computer Numerical Control
Co	Cobalt
COF	Coefficient of Friction
Cr	Chromium
CVD	Chemical Vapour Deposition
DC	Direct Current
DI water	Purified water is water that is mechanically filtered or processed to be

cleaned for consumption. Distilled water and deionized (DI) water have been the most common forms of purified water.

DLC	Diamond- Like Carbon
EHT	Extra high tension, or high voltage
FCC	Face Centre Cubic
GPa	Giga-Pascal (1GPa= 1,000,000,000Pa)
HT 1170	Deconex is a liquid, mildly alkaline cleaning concentrate used in ultrasonic cleaning systems, with high surfactant content and high degreasing power.
HT 1233	Deconex is a mildly alkaline liquid, cleaning concentrate for the final cleaning step before rinsing and drying, for carbide and HSS parts. It is suitable for both pre-cleaning and final cleaning. However it is very frequently used in the final cleaning step prior to coating.
HT 1401	Deconex is a highly alkaline liquid cleaning concentrate for removing machining oils, corrosion proofing oils. Deconex HT 1401 is primarily used with ultrasonic cleaners during the pre-cleaning step. For use in soaking baths and ultrasonic cleaning systems, especially on corrosion-sensitive metals such as steel, HSS and carbides.
HV	Hardness Vickers Pyramid Number
ICSD	(Inorganic Crystal Structure Database)
MFC	Mass Flow Controller
MPs	Macro-particles
N <sub>2</sub>	Nitrogen
Ni	Nickel
PVD	Physical Vapour Deposition
R <sub>a</sub>	Surface roughness
SCCM	Is referring of standard cubic centimeters per minute indicating cc/min at a standard temperature and pressure. A flow measurement term indicating cc/min at a standard temperature and pressure.
SEM	Scanning Electron Microscopy

Ti	Titanium
TiN	Titanium Nitride
WC	Tungsten Carbide
XRD	X- ray diffraction
Y	Yttrium
Zr	Zirconium

## LIST OF PUBLICATION

### Conference

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## CHAPTER 1

### INTRODUCTION

#### 1.1 Background of study

Single, multi- layers, nitride coatings, are widely used to extend the lifetime of machine parts. The properties of nitride coating improve adhesion and hardness which minimize wear rate and coefficient of friction. This generates significant improvement in performance and prolonged life of the coated tools (Aihua et al., 2012; Biksa et al., 2010).

This research project is part of a program funded by the Iraqi Government to enable their oil companies to find appropriate solutions for the repair of oil drilling equipment. Since nitride coatings have proven to be superior in terms of performance, this research project was geared towards the study of the characteristics and performance of AlTiN coating on tungsten carbide (WC) inserts in cutting duplex stainless steel which was widely used material in oil drilling industry. The AlTiN coating was deposited using arc ion plating technique on to WC insert.

#### 1.2 Introduction

Coating, technique known as physical vapor deposition (PVD), is a process which can be carried out by several techniques such as magnetron sputtering, ion beam assisted deposition, arc evaporation and pulsed laser deposition (PLD), has been successfully used to protect surfaces of mechanical components working under high wear loads during the last five decades (Abu-Shgair et al., 2010)

The coating of thin Ti and Al could be performed as monolayer (Jakubéczyová et al., 2012; Kottfer et al., 2013; Abd Rahman, 2009; Birol et al., 2010 ; Podgursky et al., 2011) multilayer (Ramadoss et al., 2013; Jianxin and Aihua, 2013), and gradient layers

(Chang and Wang, 2007). The thin Ti coating in particular is widely used for its good properties of improving adherence, hardness, reducing wear rate and coefficient of friction (Thornton et al., 1994; Grips et al., 2006; William and Retwisch, 2010; Vamsi- Krishna et al., 2010). A recent work by (Yong-Qiang et al., 2011) suggested that TiN/TiAlN and TiN/AlTiN multilayer coatings deposited on cutting tools could improve mechanical and corrosion resistance. It has been shown that AlTiN coating has better mechanical properties compared to TiN due to the inclusion of aluminum atoms in TiN crystalline structure which results in increasing oxidation resistance by formation of the stable ternary material AlTiN (Verma et al., 2012; Cselle et al., 2009).

Recently, a new coating technology known as pulsed bias arc ion plating (PBAIP) which has advantages over other technologies due to its lower processing temperature requirement, lower residual stress, better grain refinement and particle purification (Soediono 1989; Bunshah, 1994; PalDey et al., 2003). PBAIP also provides the ideal conditions for the deposition of a multilayer film with excellent performance.

The process of ion plating is normally performed by using concurrent or periodic bombardment of the substrate and deposited film prior to deposition which is described as preliminary process called sputtering in order to prepare the substrate (Wei and Gong, 2011; Lackner et al., 2013). It is stated that (Ti, Al) N coating enhances wear and oxidation resistance of tools as, (Arslan et al., 2013; Yıldız et al., 2013).

Titanium Aluminum Nitride (TiAlN), known also as AlTiN, depends on the compositions of Ti and Al and is suitable for coating cutting tools for machining hard to cut materials because the coating can withstand extreme environments such as high temperature and high pressure condition. These properties made TiAlN suitable for use in dry machining and high-speed milling and turning where heat is generated (Chou et al., 2002; Siow et al. 2013; Li et al., 2013; Feng et al., 2013). When properly applied, it