

THE EFFECT OF SUBSTRATE TEMPERATURE OF (Ti,Al)N COATING
ON AISI 304L STAINLESS STEEL AND HIGH SPEED STEEL
USING PHYSICAL VAPOR DEPOSITION (PVD) METHOD

CHOO SHYH JING

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

Faculty of Mechanical Engineering
Universiti Teknologi Malaysia

SEPTEMBER 2013

Specially dedicated to
My beloved family and friends
For their support and inspiration

ACKNOWLEDGEMENTS

Through the overall of the research, firstly I would like to express my sincere appreciation to my thesis supervisor, Dr. Muhamad Azizi Bin Mat Yajid and co-supervisor Mr. Engku Mohammad Nazim Bin Engku Abu Bakar for their encouragement and guidance throughout the entire research. Without their support this thesis would not be able to complete on time.

Besides that, I would also like to thank to all my colleagues and friends who always give me support when I need their help. In addition, I would like to thanks to all the laboratory technicians from the Material Science Laboratory, and Manufacturing Laboratories who giving their technical support while conducting the research.

Last, my special thanks and appreciations go to my beloved family for their encouragement and mentally support through my entire university life. With their continuous support, I was successfully finished my study at Universiti Teknologi Malaysia.

ABSTRACT

The purpose of (Ti,Al)N coating was to provide adherent coating on steel substrates to improve mechanical properties of steel tools in industrial applications. The effect of substrate temperature of physical vapour deposition (PVD) was an important parameter to study which affect the adhesion strength of (Ti,Al)N coating on alloy steel substrates. In this research, two types of substrates were chosen: AISI 304L stainless steel and high speed steel (HSS). The coating was deposited on both substrates at four different substrate temperatures which are 200°C, 240°C, 300°C, and 400°C for 90 minutes deposition time with 200 watt DC power, gas flow rate was maintained at ratio of 10 sccm N₂ : 20 sccm Ar, and 10 mTorr working pressure was used using a pure TiAl target with 50 wt% Ti : 50 wt% Al ratio. The coating morphology was observed as columnar type structure and the surface roughness was decreased with substrate temperatures. From the adhesion test results it shows that the adhesion strength of (Ti,Al)N coating was increased with substrate temperature for both 304L stainless steel and HSS but, the deposition rate of coating was decreased for both substrates.

ABSTRAK

Tujuan penyalutan (Ti,Al)N adalah untuk menghasilkan salutan yang baik di atas substrat keluli aloi untuk memperbaiki sifat-sifat mekanik keluli alat dalam aplikasi-aplikasi industri. Kesan suhu substrat adalah parameter yang sangat penting untuk mengkaji kesan kekuatan lekatan salutan (Ti,Al)N atas keluli aloi dalam pemendapan wap fizikal. Dalam kajian ini, dua jenis substrat telah dipilih: AISI 304L keluli tahan karat dan keluli kelajuan tinggi (HSS). Penyalutan telah dilakukan untuk kedua-dua jenis substrat dengan menggunakan empat suhu substrat yang berlainan iaitu 200°C, 240°C, 300°C, dan 400°C dengan 90 minit masa pemendapan, 200 watt kuasa DC, kadar aliran gas telah ditetapkan pada nisbah 10 sccm N₂ : 20 sccm Ar, and tekanan kerja 10 mTorr telah digunakan dengan menggunakan sasaran TiAl tulen pada kandungan 50 wt% Ti : 50 wt% Al. Morfologi salutan yang terhasil adalah berstruktur kolum dan kekasaran permukaan adalah berkurangan dengan suhu. Daripada ujian-ujian salutan ianya menunjukkan kekuatan lekatan salutan (Ti,Al)N semakin meningkat dengan peningkatan suhu untuk kedua-dua jenis substrat, keluli tahan karat 304L dan HSS, tetapi kadar salutan adalah berkurangan..

TABLE OF CONTENTS

CHAPTER	TITLE	PAGE
	DECLARATION	ii
	DEDICATION	iii
	ACKNOWLEDGEMENTS	iv
	ABSTRACT	v
	ABSTRAK	vi
	TABLE OF CONTENT	vii
	LIST OF TABLES	xi
	LIST OF FIGURES	xii
	LIST OF SYMBOLS AND ABBREVIATIONS	xv
	LIST OF APPENDICES	xvi
1	INTRODUCTION	1
	1.1 Introduction	1
	1.2 Background of Study	2
	1.3 Problem Statement	2
	1.4 Objective of The Study	2
	1.5 Scope of The Study	3
	1.6 Significant of the Study	3
2	LITERATURE REVIEW	4
	2.1 Introduction	4

2.1.1	AISI 304L Stainless Steel	5
2.1.2	High Speed Steel	5
2.1.3	Properties of AISI 304L Stainless Steel and HSS	6
2.1.4	Application of AISI 304 Stainless Steel and HSS	7
2.2	Overview of (Ti,Al)N Coating	8
2.3	Physical Vapor Deposition (PVD)	9
2.3.1	Vacuum Deposition	10
2.3.2	Ion Plating	11
2.3.3	Sputter Deposition	11
	2.3.3.1 Direct Current (DC) Magnetron Sputtering Process	12
	2.3.3.2 Radio Frequency (RF) Magnetron Sputtering Process	13
2.4	Coating Parameters	14
	2.4.1 Effect of Gas Pressure	14
	2.4.2 Effect of Substrate Temperature	15
2.5	Coating Adhesion Strength	16
2.6	Generation Of (Ti,Al)N Coating	19
3	RESEARCH METHODOLOGY	20
3.1	Introduction	20
3.2	Overall Methodology Flow Chart	21
3.3	Substrate	22
3.4	Substrate Preparation	23
3.5	Coating Parameters	23
3.6	Sample Preparation	24
	3.6.1 Power Chain Saw	24
	3.6.2 Buehler Linear Precision Saw Machine	25
	3.6.3 Grinding and Polishing Machine	25
	3.6.4 Ultrasonic Cleaner Machine	26
	3.6.5 Magnetron Sputtering Machine	27

3.7	Analytical Instruments	27
3.7.1	Lego Glow Discharge Spectrometer (GDS)	27
3.7.2	Field Emission Scanning Electron Microscope (FESEM)	28
3.7.3	X-ray Diffractometer (XRD)	29
3.7.4	Atomic Force Microscope (AFM)	30
3.8	Mechanical Testing Instruments	31
3.8.1	Digital Rockwell Hardness Tester Machine	31
3.8.2	Knife Test	32
4	RESULTS AND DISCUSSIONS	33
4.1	GDS Stainless Steel Composition Analysis	33
4.2	FESEM-EDX High Speed Steel Composition Analysis	34
4.3	Coating Layer Analysis	35
4.3.1	AFM Surface Topography Analysis	35
4.3.1.1	Surface Topography Of (Ti,Al)N Coating	35
4.3.2	FESEM (Ti,Al)N Coating Layer Analysis	39
4.3.2.1	Elemental Distribution Of (Ti,Al)N Coating	39
4.4	(Ti,Al)N Coating Characterisation	42
4.5	(Ti,Al)N Coating Adhesion Test	44
4.5.1.	Rockwell C Indentation Test	45
4.5.2.	Knife Test	50
4.6	(Ti,Al)N Coating Thickness	55
5	CONCLUSIONS AND RECOMMENDATIONS	61
5.1	Introduction	61
5.2	Conclusions	62
5.3	Recommendations	63

REFERENCES	64
-------------------	----

APPENDICES A-U	68-79
-----------------------	-------

LIST OF TABLES

TABLE NO.	TITLE	PAGE
2.1	Chemical composition of AISI 304 and 304L stainless steel	7
2.2	Chemical composition of different types of HSS	7
2.3	Physical properties of AISI 304 and 304L stainless steel	7
2.4	Applications of AISI 304 stainless steel and HSS	8
3.1	DC Magnetron Sputtering (Ti,Al)N deposition parameters	24
4.1	Material Composition of AISI 304L Stainless Steel	33
4.2	Chemical composition of high speed steel in terms of atomic and weight percentage	34
4.3	Arithmetic surface roughness (Ra) of (Ti,Al)N coating at different substrate temperatures	38
4.4	(Ti,Al)N coating thickness after DC magnetron sputtering	60

LIST OF FIGURES

FIGURE NO.	TITLE	PAGE
2.1	Ternary phase diagram of Ti-Al-N at 1000 °C	9
2.2	Schematic diagram of DC magnetron sputtering process	12
2.3	Principle of operation of RF sputtering deposition process	13
2.4	Thornton's structure zone model for magnetron sputtering coating, T is substrate temperature, T _m is coating melting point, and function of Ar gas pressure	15
2.5	Schematic of the cross-section of an indentation on a diamond-coated cemented carbide substrate showing the crack pattern at the interface as well as the crack diameter	17
2.6	Adhesion strength quality HF1 to HF6	18
3.1	The Overall Research Methodology Flow Chart	21
3.2	Schematic diagram of substrates 304L stainless steel and HSS	22
3.3	BEHRINGER Power Chainsaw	24
3.4	BUEHLER Linear Precision Saw Machine	25
3.5	BAINPOL MECTCO grinding and polishing machine	26
3.6	Branson 2510 Ultrasonic Cleaner Machine	26
3.7	The mechanism of DC magnetron sputtering process	27

3.8	The LECO Glow Discharge Spectrometer (GDS) Machine	28
3.9	The LECO Glow Discharge Spectrometer (GDS) Machine	28
3.10	X'Pert PRO XRD machine	29
3.11	The Working Principle of XRD	29
3.12	SPA300HV model of AFM machine	30
3.13	The working Principle of the AFM	30
3.14	Digital Rockwell Hardness Tester	31
4.1	304L stainless steel 2 dimensional and 3 dimensional surface topography (Ti,Al)N coating at (a) 200°C, (b) 240°C, (c) 300°C, and (d) 400°C	36
4.2	High speed steel 2 dimensional and 3 dimensional surface topography (Ti,Al)N coating at (a) 200°C, (b) 240°C, (c) 300°C, and (d) 400°C	37
4.3	Graph of average surface roughness (Ra) of coating at different substrate temperatures (a) AISI 304L stainless steel and (b) HSS	38
4.4	FESEM-EDX Mapping chemical element distribution for (Ti,Al)N coating on 304L stainless steel coating with substrate temperature at (a) 200°C, (b) 240°C, (c) 300°C, and (d) 400°C	39
4.5	FESEM-EDX Mapping chemical element distribution for (Ti,Al)N coating on high speed steel coating with substrate temperature at (a) 200°C, (b) 240°C, (c) 300°C, and (d) 400°C	41
4.6	Grazing X-ray diffraction pattern of (Ti,Al)N coating on 304L stainless steel with substrate temperature at 200°C, 240°C, and 300°C	43
4.7	Grazing X-ray diffraction pattern of (Ti,Al)N coating on high speed steel with substrate temperature at 200°C, 240°C, and 300°C	43

4.8	FESEM micrograph VDI 3198 indentation test of (Ti,Al)N coating on 304L stainless steel with substrate temperature at (a) 200°C, (b) 240°C, (c) 300°C, and (d) 400°C	45
4.9	FESEM micrograph VDI 3198 indentation test of (Ti,Al)N coating on high speed steel with substrate temperature at (a) 200°C, (b) 240°C, (c) 300°C, and (d) 400°C	47
4.10	50x magnification micrograph D6677-07 knife test of (Ti,Al)N coating on 304L stainless steel with substrate temperature at (a) 200°C, (b) 240°C, (c) 300°C, and (d) 400°C	50
4.11	50x magnification micrograph D6677-07 knife test of (Ti,Al)N coating on high speed steel with substrate temperature at (a) 200°C, (b) 240°C, (c) 300°C, and (d) 400°C	52
4.12	FESEM micrograph of (Ti,Al)N coating thickness for 304L stainless steel with substrate temperature (a) 200°C, (b) 240°C, (c) 300°C, and (d) 400°C	55
4.13	FESEM micrograph of (Ti,Al)N coating thickness for high speed steel with substrate temperature (a) 200°C, (b) 240°C, (c) 300°C, and (d) 400°C	57
4.14	Average (Ti,Al)N coating thickness for 304L stainless steel and high speed steel at substrate temperature of 200°C, 240°C, 300°C, and 400°C	59

LIST OF SYMBOLS / ABBREVIATION

TiN	Titanium Nitride
CrN	Chromium Nitride
(Ti,Al)N	Titanium Aluminium Nitride
AISI	American Iron and Steel Institute
XRD	X-Ray Diffractometer
EDX	Energy Dispersive X-ray Analysis
AFM	Atomic Force Microscope
FESEM	Field Emission Scanning Electron Microscope
304L	304 Low Carbon
HSS	High Speed Steel
PVD	Physical Vapour Deposition
RF	Radio Frequency
DC	Direct Current
GDS	Glow Discharge Spectrometer
SCCM	Standard Cubic Centimeters Per Minute
RMS	Root Mean Square
Ra	Arithmetic Average

LIST OF APPENDICES

APPENDICES	TITLE	PAGE
A	FESEM EDX Mapping results for AISI 304L stainless steel after (Ti,Al)N coating with 200°C	68
B	FESEM EDX Mapping results for AISI 304L stainless steel after (Ti,Al)N coating with 240°C	69
C	FESEM EDX Mapping results for AISI 304L stainless steel after (Ti,Al)N coating with 300°C	69
D	FESEM EDX Mapping results for AISI 304L stainless steel after (Ti,Al)N coating with 400°C	70
E	FESEM EDX Mapping results for AISI high speed steel after (Ti,Al)N coating with 200°C	70
F	FESEM EDX Mapping results for AISI high speed steel after (Ti,Al)N coating with 240°C	71
G	FESEM EDX Mapping results for AISI high speed steel after (Ti,Al)N coating with 300°C	71
H	FESEM EDX Mapping results for AISI high speed steel after (Ti,Al)N coating with 400°C	72

I	Glazing X-Ray Diffraction pattern for AISI 304L stainless steel after (Ti,Al)N coating with 200°C substrate temperature	72
J	Glazing X-Ray Diffraction pattern for AISI 304L stainless steel after (Ti,Al)N coating with 240°C substrate temperature	73
K	Glazing X-Ray Diffraction pattern for AISI 304L stainless steel after (Ti,Al)N coating with 300°C substrate temperature	73
L	Glazing X-Ray Diffraction pattern for AISI high speed steel after (Ti,Al)N coating with 200°C substrate temperature	74
M	Glazing X-Ray Diffraction pattern for AISI high speed steel after (Ti,Al)N coating with 240°C substrate temperature	74
N	Glazing X-Ray Diffraction pattern for AISI high speed steel after (Ti,Al)N coating with 300°C substrate temperature	75
O	AFM analysis AISI 304l stainless steel after (Ti,Al)N coating with 200°C substrate temperature	75
P	AFM analysis AISI 304l stainless steel after (Ti,Al)N coating with 240°C substrate temperature	76
Q	AFM analysis AISI 304l stainless steel after (Ti,Al)N coating with 300°C substrate temperature	76
R	AFM analysis AISI 304l stainless steel after (Ti,Al)N coating with 400°C substrate temperature	77
S	AFM analysis high speed steel after (Ti,Al)N coating with 200°C substrate temperature	77

T	AFM analysis high speed steel after (Ti,Al)N coating with 240°C substrate temperature	78
U	AFM analysis high speed steel after (Ti,Al)N coating with 300°C substrate temperature	78
V	AFM analysis high speed steel after (Ti,Al)N coating with 400°C substrate temperature	79

CHAPTER 1

INTRODUCTION

1.1 Introduction

Alloy steels are one of the most commonly used and cost effective structural materials in modern industry. However when steel is used for critical wear resistant components and machining tools such as bearings, taps, dies, twist drills, saw blades, and other cutting tools in harsh environments, accelerated damage are usually occur because of its low wear resistance properties.

Nowadays due to the low mechanical properties of alloy steel, a hard coating such as TiN, CrN, and (Ti,Al)N was introduced as a protective coating on mechanical tools. The exceptional mechanical and corrosion resistant properties of these hard coatings such as high hardness and wear resistant, and good oxidation resistance at high temperature make the surface of the hard coating to be potentially ideal as coatings for wear resistant components and machining tools. Since the early failure of tool steels is always on the outermost surface, hence with well-adhered surface hard coating films deposited on the steel can lead to major improvements of life time and performance of such tool steels.

1.2 Background of Study

Physical vapour deposition (PVD) is one of the preferred techniques for the development of hard coating due to many advantages such as better control of process parameters and stoichiometry of the deposition coating. Besides that, it is also more cost effective as compared to plasma enhance chemical vapour deposition and low pressure chemical vapour deposition [1,2]. 304L stainless steel and high speed steel were chosen as the substrate material in this research and (Ti,Al)N hard coating was deposited because it has excellent high oxidation resistance, high hardness and high corrosion resistance. Besides that, this coating is suitable used for cutting tools especially for dry and high speed machining application [3,4].

1.3 Problems Statement

The (Ti,Al)N coating was deposited on the steel substrate using physical vapor deposition (PVD) METHOD. However, the PVD parameters will influence the (Ti,Al)N coating adhesion strength to the steel substrate. Hence, in this project there is a necessary to conduct a research to investigate an optimum PVD parameter in order to produce good (Ti,Al)N coating on the surface of stainless steel and high speed steel without delamination.

1.4 Objective of the Study

The objective of this research is to study the effect of substrate temperature of (Ti,Al)N coating on AISI 304L stainless steel and high speed steel using Physical Vapor Deposition (PVD) method.

1.5 Scope of the Study

1. To produce (Ti,Al)N interlayer on AISI 304L stainless steel and high speed steel using direct current (DC) magnetron sputtering method.
2. To do microstructural evaluation on the coated specimen using XRD, EDX, and AFM.
3. To conduct Rockwell C indentation test and knife test to evaluate the coating adhesion strength.

1.6 Significant of the study

Nowadays, ferrous metals or steels are become one of the most popular used material for engineering applications. Although the properties of steels are suited for most of the engineering applications such as taps, dies, twist drills, and other cutting tools, but in order to fulfil more demanding application of steels, the (Ti,Al)N deposition as an adherent coating on steel surface will gain more advantages. With the combine properties of steel and (Ti,Al)N coating, the new properties of the system will provide much better than the normal steel, besides that the performance and the life span of the tools will also improved.

However, in order to deposit a good adhesion (Ti,Al)N coating on steel, the deposition parameters for (Ti,Al)N coating on steel substrate become important to study. Thus, this research will focus on the study of the effect of substrate temperature of (Ti,Al)N coating on AISI 304L stainless steel and high speed steel using PVD method.

8. Chemical and Physical Properties of AISI 304 Stainless Steel (2011). Atlas Steels Australia. <http://www.azom.com/article.aspx?ArticleID=965>.
9. L.A.Geller (1961). Instrumental'nye stali 2nd Edition. Moscow.
10. Product Data Bulletin of 304/304L stainless steel (2007). AK STEEL Cooperation.
11. High Speed Steel Tool Data (1994). ISC Cutting Tools INC.
12. R.Polini, F.P.MAntini, M.Amar, W.Ahmed, and H.Taylor (2005). Effects of Ti- and Zr-based interlayer coatings on the hot filament chemical vapor deposition of diamond on high speed steel. *Thin Solid Film* 494.116.116-122
13. C.Harish, Barshilia, M.S.Prakash, A.Jain, and K.S.Rajam (2004). Structure, hardness and thermal stability of TiAlN and nanolayered TiAlN/CrN multilayer films. *Vacuum* 77, 169-179
14. F.Quesada, A.Marino, and E.Restrepo (2006) TiAlN coatings deposited by r.f. magnetron sputtering on previous treated ASTM A36 steel. *Surface & Coating Technology* 201, 2925-2929
15. S.Paldey, and S.C.Deevi (2002). Single layer and multilayer wear resistant coating of (Ti,Al)N: a review. *Material Science and Engineering A* 342, 58-79
16. R.Polini, F.P.Mantini, M.Braic, M.Amar, W.Ahmed, H.Taylor, and M.J.Jackson (2005). Effects of Ti- and Zr-based interlayer coatings on the hot filament chemical vapour deposition of diamond on high speed steel. *Thin Solid Film* 494.116.116-122

17. E. Lugscheider, C.Barimani, C.Wolff, S.Guerreiro, and G.Doepper (1996). Comparison of the structure of PVD-thin films deposited with different deposition energies. *Surface and Coatings Technology*. vol. 86-87, pp. 177-183.
18. D.M.Mattox (1997). *PVD Hand Book*. New Mexico
19. L.Q.Jie (2008). *Silicon Self-Assembled Nanodots Fabricated Using A Radio-Frequency Magnetron Sputtering Method*. Universiti Teknologi Malaysia: Master Thesis
20. H.N.Shah, R.JAyaganthan, D.Kaur, and R.Chandra (2010), Influence of sputtering parameters and nitrogen on the microstructure of chromium nitride thin films deposited on steel substrate by direct current reactive magnetron sputtering. *Thin Solid Films*. 518, 5761-5768
21. P.C. Jindal., *Thin Solid Films* 154 1987. 361–375
22. W.Heinke, A.Leyland, A.Matthews, G.Berg, C.Friedrich, and E.Broszelt (1995), Evaluation of PVD nitride coatings, using impart, scratch and Rockwell-C adhesion tests. *Thin Solid Films*. 270, 431-438
23. J.Gerth, and U.Wiklund (2008). The influence of metallic interlayers on the adhesion of PVD TiN coating on high speed steel. *Wear* 264, 885-892
24. O.Golzman, G.HAlperin, I.Etsion, A.Berner, D.Shectman, and G.H.Lee (1999), Study of wear behavior and adhesion of diamond films deposited on steel substrates by use of a Cr-N interlayer. *Diamond and Related Material* 8,859-864

REFERENCES

1. J.musil, P.Baroch, J.Vicek, K.H.Nam, and J.G.Han (2005). Reactive magnetron sputtering of thin films: present status and trends. *Thin Solid Films* 457,208
2. I.Safi (2000). Measurement of energy transfer at an isolated substrate in a pulsed magnetron discharge. *Journal Of Apply Physic* 127,203
3. A.K.Singh, N.Kumari, S.K.Mukherjee, and P.K.Barhai (2013). Atomic force microscopy analysis of effect of RF/DC power ratio on the properties of co-sputtered $Ti_xAl_{1-x}N$ thin films. Jharkhand, Inda.
4. J.C.Oliveira, A.Manaia, and A.Cavaleiro (2008). Hard amorphous Ti-AlN coatings deposited by sputtering. *Thin Solid Films* 516p5032
5. W.D.Calister (2007). *Materials Science and Engineering: An Introduction* 7th Edition. John Wiley & Sons, Inc.
6. H.K.D.H. Bhadeshia (2006). *Steels* 3rd Edition: Microstructure and Properties. Elsevier Ltd.
7. A.Ahmad (2011). *Surface Morphology Studies of Silicon Interlayer On Stainless Steel Substrate Using Physical Vapor deposition (PVD) Method.* Degree Thesis. Universiti Teknologi Malaysia.

25. Standard test method for evaluating adhesion by knife. ASTM International Designation : D6677-07 (2012)
26. S.R.Jiang, D.L.Pend, X.Y Zhao, L.Xie, and Q.Li (1995), Study on the mechanical and chemical properties of (Ti,Al)N films prepared by DC magnetron sputtering. Applied Surface Science. 84, 373-377
27. K.Singh, P.K.Limaye, N.L.Soni, A.K.Grover, R.G.Agrawai, and A.K.Suri (2005), Wear studies of (Ti-Al)N coatings deposited by reactive magnetron sputtering. Wear 258, 1813-1824