Special Issue

Sci.Int.(Lahore),26(4),1431-1435,2014

Kamil J. Kadhim.¹ Nizam Bin Abd Rahman.² Mohd Rizal Bin Salleh.³ and Izani Bin Mohd Zukee⁴

^{2.3,4} Faculty of Manufacturing Engineering, Universiti Teknikal Malaysia Melaka, Durian Tunggal, 76100 Melaka, Malaysia.

^akamil_jawad35@yahoo.com, ^bmdnizam@utem.edu.my, ^crizal@utem.edu.my, ^dizani87@gmail.com

ABSTRACT: This study investigated the effects of various pulsed bias thickness layers on the properties of microstructure and surface topography of AlTiN multilayer coatings. These coatings were deposited on tungsten carbide inserts (WC) (12.7 mm \times 12.7 mm \times 3.18 mm) using a system PVD of cathodic arc vapour by pulsed bias (arc ion plating). The macroparticles in these coatings decreased with the increase in thickness layers. Moreover, the surface roughness values were 0.06157, 0.02855, 0.04868, 0.02439, and 0.1647 µm given the thickness layers of 2.717, 3.089, 3.912, 5.815, and 8.760 µm, respectively. The AlTiN multilayer coatings were crystallized and oriented in the (1 1 1), (2 0 0), (2 2 2), and (3 1 1) crystallographic planes. Results indicate that the microstructure was strengthened at the orientation of (200) direction with the corresponding 2θ of ~43.1465°. Hence, the topographic properties of all of the specimens coated with AlTiN multilayer coatings were enhanced.

KEYWORDS: Arc ion plating, Surface roughness, AlTiN multilayer coatings, thickness layers

1.0 INTRODUCTION

Pulsed bias arc ion plating is an emerging thin film deposition technology developed recently. It is advantageous over traditional arc ion plating technology because it operates under low deposition temperature, can handle residual stress, refines grain, purifies particle and facilitates the effective deposition of multilayer film. In ion plating, the substrate is either concurrently or periodically bombarded to clean the surface of the substrate through sputtering prior to film deposition by atomic- sized vitality particles. During deposition, the properties of the film are also adjusted and controlled by bombardment. Therefore, this procedure must be performed continuously between clean interfaces [1]. TiAlN coatings are relatively hard coatings that are chemically inert, stable thermodynamically, and effectively resist oxidation [2-4].Knotek et al. [5] were the first to deposit Ti coatings on cutting tools through magnetron sputtering. Currently, arc ion plating is widely used to deposit TiAlN coatings [3-7]. Nonetheless, AlTiN multilayer coating [7-12] has also been investigated as a new type of coating. Both of these coating types are deposited by direct current bias. In 1991, Olbrich et al. [13] were the first to deposit TiN coating through arc ion plating with pulsed bias voltage at low temperatures. They reported that this method can reduce the amount of macro particles (MPs) in coating. To date, pulsed bias voltage has been widely used to deposit diamond-like carbon (DLC), which is a coating that is highly resistant to wear. Hence, it is highly suitable for extreme wear conditions and dry machining. This method also facilitates the development of excellent surface morphology and does not damage workpieces coated with different substances [14], including CrN [15], (Cr,Al)N [16], Ni Co CrAlY [17] . However, previous studies focused on pulsed bias voltage, N pressure, and modulation periods and did not emphasize the influence of duty ratio (duty ratio is the fraction of time that indicates the active status of a system in a certain period) on the microstructure and properties of these coating. Although many researchers mixed N_2 and Ar gases as reaction media [6, 17], the current study utilizes pure N as a reactive gas to ensure that fresh Ti and Al atoms bond effectively with N. Furthermore, this study aims to improve system performance by optimizing the pulsed bias voltage. Thus, it is applied at different thickness layers in the deposition of AlTiN multilayer coating on tungsten carbide insert in a pure N atmosphere. Finally, the effects of this thickness layers on the microstructure, orientation, preferred and surface morphologies of AlTiN multilayer coating are investigated.

2.0 EXPERIMENTAL

In this study, experiments were conducted with the ARC system technology by J&L technology Korea, Model Legend H.I.P III. The system comprises of four alloy cathodes with composition ratios Al: Ti 67: 33% and with a pure targets four Ti a

diameter of 150 x 15 mm. The substrate jig holder (turntable) contains six axes with triple rotations refer to (Figure 1). The back of the substrate jig holder houses a thermocouple arranged from high to low level (top to bottom). This thermocouple was located approximately 20 mm- 30mm from the substrate holder.



Figure 1: (a) PVD Arc system technology constructed by J&L technology Korea, Model Legend H.I.P. III. (b) Substrate holder outside the coating chamber

1432

<u>Special Issue</u> ISSN 1013-5316; CODEN: SINTE 8

Sci.Int.(Lah26(4),1431-1435,2014

Table 1: Cleaning process of insert

Tuble It cleaning process of insert							
Tank 1	HT1401 + HT1170 + deionized (DI) water (300 S) (Temp 52 °c) with rotation						
Tank 2	HT1401 + HT1170 + DI water (300 S) (Temp 52 °c) with rotation						
Tank 3	Rotationing DI water sprayer						
Tank 4	HT 1233 + ANTICORR + DI water (150 S) (Temp 52 °c) with rotation						
Tank 5	Ultrasonic rinse in DI water with rotation						
Tank 6	Rinse in DI water with rotation alone						

The AlTiN coating was deposited on the substrate in the coating chamber under gas atmosphere. The ARC of the target material of Ti and AlTi was induced by the bombardment with Ar ions, and the substrate was a WC square cutting insert with a dimension of 12.7 mm x 12.7 mm x 3.18 mm. Prior to coating, the cutting insert was ultrasonically cleaned in a detergent bath given different detergent mixtures and bathing times as listed below in Table 1:

The cutting insert was placed on the rotation turntable. The distance from the substrate to the target was approximately 50 mm, and rotation speed was set to 5 rpm. Throughout the experiment, our coating process consisted of six stages, namely, pumping, heating, ion etching, buffer layer (Ti) deposition, deposition of the main layer of coating (AlTi), and cooling. The Ultimate vacuum pressure required for this procedure was set at 5.0×10^{-5} mbar. Table 2 also presents the setting of these six stages and details the deposition parameters of the multilayer coatings. We analyzed the structural of the coatings through X-ray diffraction (XRD) (Bruker D8 X -ray facility) under Cu Ka radiation. The machine operated at 40kv and 40mA with a glazing incidence angle of 1.5°. XRD is a versatile, non-destructive analytical technique that determines and quantitatively identifies various crystalline compounds in solid materials and powders. These compounds are known as "stages". Diffraction mainly occurs as a result of certain phase relations. Two rays are completely in stage when their path lengths differ by the zero of a whole number of wavelengths (λ =0.1540nm). At a scanning speed of 4°/min, the scanning angel (2°) ranged from 10° to 90°. Surface morphologies were analysed using Scanning Electron Microscope (SEM), and surface roughness was measured according to Ra value through atomic force microscopy (AFM) using Q-Scope 250 (Quaint Instruments Corporation). These measurements were obtained at a square area of (5µm X 5 µm) in contact mode. The thickness of each deposited coating was measured by SEM.

3.0 RESULT AND DISCUSSION**3.1** AFM Analysis

AFM images in 3D were shown in Figure 2, including that of the area coated with multiple layers of AlTiN for comparison [Figure 2(a)]. Furthermore, the roughness values presented in Table 3 are supported by the AFM image in Figure 2, which exhibits the smooth surface area coated with various thickness layers[Figure 2(a)]. The surface of the AlTiN-coated area [Figure 2(d)] is homogenous, although some grooves can be detected. These grooves may have been generated during the cleaning process. Moreover, the rough surface areas were coated with multiple layers of AlTiN at thicknesses of 3.089 and 3.912μ m [Figure 2(b) and 2(c)]. The pits observed on the AlTiN- coated surface in Figure 2(b) were also shown in the 3D AFM image [Figure 2(a)].

3.2 Surface Morphology

Figure 2 displays the morphologies of the surface coated with multiple layers of AlTiN given different pulsed bias thickness layers. However, a major concern related to the deposition of multilayer AlTiN coatings by arc ion plating is the formation of macroparticle (MPs) [19, 20]. These MPs are charged by many electrons and generate strong repulsive force in the specimen with the increase in duty ratio (is defined as the ratio between the pulse duration (T) and the period (P) of a rectangular waveform) because a negative charge dominates the specimen surface as a result of sheath oscillation. Thus, the surface quality of the coatings improved when the amount of MPs reaching the specimen was limited. As shown in Figure 3, the surface roughness decreased from 0.1647µm to 0.06157µm with the increase in thickness layers. The deposition temperature decrease with decrease in duty ratio, as did the diffusion of the incoming ions on the surface. These phenomena deteriorated surface quality (Figure 3) [18].

3.3 Chemical Composition

As shown in Table 3, the atomic ratio of Al/Ti increased from 0.2113 to 1.860 in AlTiN multilayer coatings. With the increase of thickness layers from 2.717 μ m to 8.760 μ m at a bias voltage of -40 V. with the increase in thickness layers from 2.717 μ m to 5.815 μ m,the atomic of N increase from 0.3055 to 0.3183. However, thickness layer increased to 5.815 μ m at a thick layer of 8.760 μ m, also increase atomic N to reach 0.3779. The structure and thickness of AlTiN coatings were studied on a brittle fracture using SEM. Moreover Al content in the multilayer coating decreased. This occurrence can be attributed to the difference in the degree of ionization of Ti and Al [20]; that of Ti vapor was higher (80%) than that of aluminum vapor (50%).

Sci.Int.(Lahore),26(4),1431-1435,2014

<u>Special Issue</u> ISSN 1013-5316; CODEN: SINTE 8

Step	Process	Parameters					
1	Pumping	Rotary, root, and turbo molecular pump					
2	Heating	The induction heater heat the substrate to the set process temperature					
3	Ar bombardment and ion	Ultimate vacuum=5.0x10-5 mbar, pulsed Bias voltage = 900V, Ar gas flow = 100					
	cleaning	sccm, arc source current = $100A$, Time= 30 mints, Temperature = $380 C^{\circ}$					
4	Buffer layer	Bias voltage = 30V, N gas flow = 300 sccm, arc source current=100A, Time = 10					
		mins, Temperature = 380 C°					
5	Main layer AlTiN multilayer	Bias voltage $= 40V$, N gas flow $= 600$ sccm, arc source current 80A, Time $= 45$					
	coating deposition	min, Temperature = 380 C°					
6	Cooling down and venting	Natural cooling, for 30min at 100 °C					



Figure 2: 3D AFM image of the tungsten carbide; (a) 2.717μm; (b) 3.089μm; (c) 3.912μm; (d) 5.815μm; and (e) 8.760μm thick surfaces coated with multiple layers of AlTiN) coating and since is the area take 5 x 5 [μm²] take area.



Figure 3: Surface morphologies of AlTiN multilayer coatings at different thickness layers: (a) 2.717μm; (b) 3.089μm; (c) 3.912μm; (d) 5.815μm; and (e) 8.760μm

International Symposium on Research in Innovation and Sustainability 2014 (ISORIS '14) 15-16 October 2014, Malacca, Malaysia Special Issue

1434

ISSN 1013-5316; CODEN: SINTE 8 Sci.Int.(Lah26(4),1431-1435,2014 **Table 3:** Properties of as-deposited, AlTiN multilayer coatings

Step layer	Film	Elemental composition [wt at%]							Preferred orientation
		Al	Ti	N	Ar	Wt.%	Al/Ti	Ra	
A=2.717µm	AlTiN	35.93	40.96	23.10	0.02	0.877	0.211	0.06157	111,200,220,311,222
B=3.089µm,	AlTiN	33.09	36.29	30.55	0.06	0.9118	1.618	0.02855	111,200,220,311,222
C=3.912 µm	ATliN	33.80	31.63	34.87	-0.31	1.0680	1.898	0.04868	111,200,220,311,222
D-5.815µm	AlTiN	32.84	35.62	31.83	-0.29	0.9219	1.637	0.02439	111,200,220,311,222
E=8.760 µm	AlTiN	31.84	30.39	37.79	-0.03	1.0477	1.860	0.1647	111,200,220



Figure 4: Surface roughness values of AlTiN multilayer coatings at different thickness layers: (a) 2.717µm; (b) 3.089µm; (c) 3.912µm; (d) 5.815µm; and (e) 8.760µm

Thus, Ti ions were attracted to the negatively biased specimens, thereby altering the final atomic ratio of Al/Ti in the AlTiN multilayer coatings [3]. With the increase in thickness layers, ion bombardment also reduced the Al content in multilayer coatings, and their Al contents differed slightly. This difference may be ascribed to the variation in the duty ratios of pulsed bias voltages during deposition, which was too small.



Figure 5: Atomic ratios of Ti/Al and N in AlTiN multilayer coatings at various thicknesses

3.4 XRD Analysis

Figure 6 presents the XRD patterns of the AlTiN multilayer coatings deposited at various thickness layers. All the investigated coatings were highly textured and were preferentially oriented in the (200) direction with a corresponding 20 of ~43.125°. However, the intensities of the (111), (220), (222) and (311) orientations were very low. This finding was consistent with that of Knutson et al. [19]. However, the results of Knutson were obtained after a fixed treatment at 380C°. The radius of Al atom is 0.1440 nm, whereas that of Ti atom is 0.1461 nm. Moreover, the Ti atoms in the TiN lattice of the AlTiN coatings were replaced by Al atoms. The difference in atom radii strengthened the solution, which in turn distorted the lattice and altered the

internal stress of the AlTiN multilayer coatings. Given the increase in Al content, enhanced and improved the hardness of the Al multilayer coating. The optimum with of the textured grain inf the growth direction (111) was very narrow, thus indicating a high degree of crystallinity [20]. For the AlTiN multilayer coatings with different pulsed bias thickness layers, the peaks of the (111) plane display different relative intensities, which suggest that the amount of oriented grains varied.



Figure 6: XRD patterns of AlTiN multilayer coatings on tungsten carbide substrates at different thickness layer

4.0 CONCLUSION

In this study, AlTiN multilayer coatings were deposited on tungsten carbide specimens by pulsed bias arc ion plating, and the effect of thickness layers on the microstructure and properties of these coatings were investigated. With the increase of pulsed bias thickness layers, the strong ion bombardment reduced the amount of MPs in the specimens and improved surface morphology. Furthermore Surface roughness values were minimized to $0.0858\mu m$ at a thickness layer of $3.0818\mu m$.According to XRD analysis, all of the surface coated with multiple layers of AlTiN showed a strong preference for the (200)orientation. These optimal processing parameters can be applied industrially in the future.

ACKNOWLEDGEMENTS

This work was financially supported by Foundation of Education Technical at Ministry Education High of Iraq.

REFERENCES

- S. PalDey, S.C. Deevi, Single layer and multilayer wear resistant coatings of (Ti,Al)N: A review. Materials Science and Engineering. Vol. 342, pp. 58-79, 2003.
- [2] E. Lugscheider, C. Barimani, C. Wolff, S. Guerreiro, G. Doepper, Surf. Coat. Technol. Vol. 86-87, pp. 177-183, 1996

International Symposium on Research in Innovation and Sustainability 2014 (ISoRIS '14) 15-16 October 2014, Malacca, Malaysia

Special Issue

Sci.Int.(Lahore),26(4),1431-1435,2014

- ISSN 1013-5316; CODEN: SINTE 8
- [3] P.J. Mcguiness, M. Cekada, V. Nemanic, B. Zajec, A. Recnik, Hydrogen permeation through TiAlN-coated Eurofer '97 steel, Surf. Coat. Technol. Vol. 205, pp. 2709–2713, 2011.
- [4] X. Li, C. Li, Y. Zhang, H. Tang, G. Li, C. Mo, Tribological properties of the TiAlN thin films with different components fabricated by double-targeted cosputtering, Appl. Surf. Sci. vol. 256, pp. 4272–4279, 2010.
- [5] O. Knotek, M. Bohmer, T. Leyendecker, On structure and properties of sputtered Ti and Al based hard compound films, J.Vac. Sci. Technol. Pp. 2695–2700, 1986.
- [6] S. Paldey, S.C. Deevi, Single layer and multilayer wear resistant coatings of (Ti,Al) N: a review, Mater. Sci. Eng. Vol. 342, pp. 58–79, 2003.
- [7] S.S. Zhao, H. Du, J.D. Zheng, Y. Yang, W. Wang, J. Gong, C. Sun, Deposition of thick TiAlN coatings on 2024 Al/SiCp substrate by Arc ion plating, Surf. Coat. Technol. Vol. 202, pp. 5170–5174, 2008.
- [8] E. Bagci, B. Ozcelik, Investigation of the effect of drilling conditions on the twist drill temperature during step-by-step and continuous dry drilling, Mater. Design. Vol. 27, pp. 446–454, 2006.
- [9] C.H. Hsu, J.K. Lu, K.L. Lai, M.L. Chen, Erosion and corrosion behaviors of ADI depo--sited TiN/TiAlN coatings by cathodic arc evaporation, Mater. Trans. Vol. 46, pp.1417–1424, 2005.
- [10] C.X. Tian a, B. Yang a,b, S.J. Yan a, Z.H. Lu a, Z.H. Huang a, D.J. Fu a" Influence of substrate rotation speed on the structure and mechanical properties of AlTiN/CrN coatings" Surface & Coatings Technology. Vol. 228, pp. S228–S232, 2013.
- [11] C.V. Falub, A. Karimi, M. Ante, W. Kalss, Interdependence between stress and texture in arc evaporated Ti–Al–N thin films, Surf. Coat. Technol. Vol. 201 pp. 5891–5898, 2007.
- [12] M. Braic, M. Balaceanu, V. Braic, A. Vladescu, G. Pavelescu, M. Albulescu, Synthesis and characterization of TiN, TiAIN and TiN/TiAIN biocompatible coatings, Surf. Coat. Technol. Vol. 200, pp. 1014–1017, 2005.

- [13] E. Zoestbergen, N.J.M. Carvalho, J.T.M. De Hosson, Stress state of TiN/TiAlN PVDMultilayers, Surf. Eng. Vol. 17, pp. 29–34, 2001.
- [14] W. Olbrich, J. Fessmann, G. Kampschulte, J. Ebberink, Improved control of TiN coating properties using cathodic arc evaporation with a pulsed bias, Surf. Coat. Technol. Vol. 49, pp. 258–262, 1991.
- [15] R.F. Huang, C.Y. Chan, C.H. Lee, J. Gong, K.H. Lai, C.S. Lee, K.Y. Li, L.S.Wen, C. Sun, Wear-resistant multilayered diamond-like carbon coating prepared by pulse biased arc ion plating, Diam. Relat. Mater. Vol. 10, pp. 1850–1854, 2001.
- [16] X.S.Wan, S.S. Zhao, Y.Yang, J.Gong, C. Sun, Effects of nitrogen pressure and pulse bias voltage on the properties of Cr–N coatings deposited by arc ion plating, Surf. Coat. Technol. Vol. 204, pp.1800–1810, 2010.
- [17] S. Li, J. Song, C. Zhou, S. Gong, Y. Han, Microstructure evolution of NiCoCrAIY Overlay coating for Ni3Al based alloy IC6 turbine vane during long term engine test, Intermetallic vol. 13, pp. 309– 314, 2005.
- [18] Y.Y. Wang, M.S. Wong, W.J. Chia, J. Rechner, W.D. Sproul, Synthesis and char -acterization of highly textured polycrystalline AlN/TiN superlattice coatings, J.Vac. Sci. Technol. Vol. 16, pp. 3341–3347, 1998.
- [19] A. Knutsson, M.P. Johansson, L. Karlsson, M. Oden, Thermally enhanced mechanical properties of arc evaporated Ti0.34Al0.66N/TiN multilayer coatings, J. Appl. Phys. Vol. 108, pp. 44312–44317, 2010.
- [20] C.Mendibide, P. Steyer, J. Fontaine, P.Goudeau, Improvement of the tribological behaviour of PVD nanostratified TiN/CrN coatings – an explanation, Surf. Coat. Technol. Vol. 201, pp. 4119–4124, 2006.

1

Sci.Int.(Lahore),26(4),1431-1435,2014