Current Applied Physics 10 (2010) 471–474

Contents lists available at ScienceDirect

Current Applied Physics

journal homepage: www.elsevier.com/locate/cap

Effect of cathodic arc PVD parameters on roughness of TiN coating on steel substrate

M. Ali [a,*], E. Hamzah [b], I.A. Qazi [c], M.R.M. Toff [d]

[a] Department of Physics, COMSATS Institute of Information Technology (CIIT), Park Road, Islamabad, Pakistan
[b] Faculty of Mechanical Engineering, Universiti Teknologi Malaysia (UTM), 81310 Skudai, Johor, Malaysia
[c] National University of Sciences and Technology, Tamiz-ud-din Road, Rawalpindi, Pakistan
[d] Advanced Materials Research Center (AMREC), SIRIM Berhad, Kulim Hi-Tech Park, Kedah, Malaysia

ARTICLE INFO

Article history:
Received 14 February 2009
Received in revised form 18 March 2009
Accepted 3 July 2009
Available online 10 July 2009

PACS:
68.55.A-
68.55.J-
68.35.Ct
68.37.Hk

Keywords:
TiN
Surface roughness
Process parameters
Macro-droplets

ABSTRACT

Titanium nitride which is widely used as a hard coating material was coated on tool steel, by physical vapor deposition method. Surface roughness was investigated as a function of deposition rate, substrate bias and temperature, nitrogen flow rate and metal ion etching. The study showed that increase in surface roughness mainly depends on the condition of sample preparation, surface treatment, macro-droplets, pitting defects, rise in compressive stress at higher coating thickness, growth defects and to a lesser extent selection of surface under testing. It was observed that chromium ion etching significantly reduced the surface roughness compared to titanium ion etching.

1. Introduction

Titanium nitride (TiN) thin films deposited by PVD technology have a wide range of commercial application such as cutting tools and artificial jewelry. In all such applications the smoothness of the finished surface is of prime importance. In this context, the terms surface finish and surface roughness are used very widely in industry and are generally used to quantify the smoothness of a surface finish.

Surface finish could be specified through different parameters. According to Lou et al. [1], roughness average (*Ra*) and root-mean-square (rms) roughness (*Rq*) are the most widely used surface parameters in industry. More recently, however other parameters like surface roughness of the maximum profile height (*Rp*) and surface roughness of the maximum profile valley depth (*Rv*) etc., have also been used. In this study the first three parameters (*Ra*, *Rp*, *Rq*) have been used to characterize TiN based hard coatings.

2. Experimental procedure

The objective of the present work is to study the influence of processing parameters on the surface roughness of TiN coatings deposited on D2 tool steel by using cathodic arc physical vapor deposition (CAPVD) technique. In this work, Titanium nitride (TiN) single layer coatings have been deposited in a commercially available HTC 625/2 ARC coating system, detailed description of which is given elsewhere [2]. The deposition parameters studied are given in Tables 1 and 2. A partial pressure of N_2 (% N_2) in gaseous mixture of argon and nitrogen used besides Ar and N_2 gas flow rates. The chamber was evacuated to a pressure of approximately 4×10^{-6} mbar and then back filled with nitrogen gas in the range of $\approx 10^{-3}$ mbar. The complete deposition procedure has been described earlier [2]. Surface roughness of uncoated and coated samples was measured using a surface roughness tester (Model-Surftest SJ-301, Mitutoyo) and an optical microscope was used to see the surface of polished uncoated samples. Macro-droplets, re-sputtered particles and the resulting pits and the growth defects were studied using Field Emission Scanning Electron Microscope (FE-SEM).

3. Results and discussion

The variation in the surface roughness for both uncoated and coated samples was studied. In all cases, the average surface roughness was measured at five different locations and results were averaged. The baseline roughness of the D2 steel substrate

* Corresponding author.
E-mail address: mubarak74@comsats.edu.pk (M. Ali).

1567-1739/$ - see front matter © 2009 Elsevier B.V. All rights reserved.
doi:10.1016/j.cap.2009.07.007

exists mainly because of the migration of tiny particles of SiC emery paper into the sample during metallography process. Again, these tiny particles of SiC increase the surface roughness of TiN-coated samples. Uniformity in surface roughness within the sample is affected due to large sample dimension. Removal of the substrate material in the form of tiny particles during metallographic process also contributes to its roughness.

The most commonly used parameters for characterizing the roughness of PVD coatings is the roughness average (*Ra*) [3,4], also known as Arithmetic Average (AA). In the present study *Ra*, average maximum height of the roughness profile (*Rz*) and root-mean-square (rms) roughness (*Rq*) are calculated; a detailed description of which are given elsewhere [1,5].

Surface roughness parameters of uncoated and TiN-coated D2 tool steel are reported in Table 3 (SC 5.1–5.5); at various deposition times. The end-process surface roughness (*Ra*) of the bottom specimens, measured with a surface roughness tester, was about 0.03 μm. The surface roughness of the cathodic arc TiN-coated D2 tool steel, defined by the *Ra* parameter, at various deposition rates is within 0.62–0.21 μm range and is significantly higher than in the case of the uncoated polished sample. The main limitation of *Ra* is that it gives no indication of the surface texture, namely macro-droplets and pitting defects that can have a significant influence on the performance of the coating, particularly for wear resistance applications [6].

It can be seen from the data tabulated in Table 3 shows that by decreasing the deposition time from 120 to 30 min, the values of surface roughness parameters *Ra*, *Rz* and *Rq* are gradually reduced. This is mainly because of reduction in coating thickness and hence in the growth defects. In Fig. 1, which displays the surface profile for coating deposited for 90 min, the peaks above average centre line (*Ra*) are because of the macro-droplets, whereas the peaks below this line are because of the hollow spots and pitting defects, from where the macro-droplets re-sputtered during the coating stage. Additionally, the coatings deposited at higher deposition time (higher coating thickness) were mainly associated with higher compressive stress and more surface defects that becomes a significant factor to enhance the surface roughness of coated tool.

Surface roughness parameters of TiN-coated D2 tool steel are reported in Table 3 (SC 6.1–6.5); at various substrate biases. The maximum *Ra* value was noted for coatings deposited at zero bias voltage, whereas, the minimum value was given by the coating deposited at −150 V. A significant decrease in surface roughness was thus noted by increasing the substrate bias from 0 to −150 V, which is a consequence of increased substrate temperature, re-sputtering of titanium particles, and reduction in both the size and amount of the macro-droplets. Moreover, the decrease in coating thickness causes a decrease in the compressive stresses. An increase in the substrate bias enhances re-sputtering of Ti particles and hence decreases the film roughness; and vice versa.

The coating deposited at 450 °C (SC 7.1) showed higher value (*Ra* = 0.41 μm) compared to the coating deposited at 150 °C (SC 7.2), (*Ra* = 0.37 μm). This shows that increasing the substrate temperature resulted in the smoothening out the film morphology. This is an interesting observation and requires further investigation by the authors.

Surface roughness parameters of TiN-coated D2 tool steel are reported in Table 3 (SC 8.1–8.3); at various nitrogen gas flow rates. The roughness of the cathodic arc TiN-coated tool steel defined by the *Ra* parameter is within the 0.37–0.27 μm range and is significantly higher than for the case of the uncoated polished sample.

Table 2
Deposition parameters of TiN on D2 tool steel.

Sample code (SC)	Ar gas flow rate (sccm)	Pre-bias (−V)	Etching time (min)	Bias voltage (−V)	Substrate temperature (°C)	N_2 flow rate (sccm)	Coating time (min)
9.1	0	0	0	50	300	250	90.00
9.2	50	1000	Ti for 8	50	300	250	90.00
9.3	50	1000	Ti for 16	50	300	250	90.00
9.4	50	1000	Cr for 8	50	300	250	90.00
9.5	50	1000	Cr for 16	50	300	250	90.00

Table 3
Surface roughness of TiN coatings measured via surface roughness tester.

Sample code (SC)	D2 tool steel (ISO, GAUSS)			Sample code (SC)	D2 tool steel (ISO, GAUSS)		
	Ra (μm)	*Rz* (μm)	*Rq* (μm)		*Ra* (μm)	*Rz* (μm)	*Rq* (μm)
Uncoated	0.03	0.26	0.04	7.1	0.41	3.93	0.60
5.1	0.62	7.31	0.94	7.2	0.37	4.13	0.55
5.2	0.40	4.59	0.63	8.1	0.37	4.39	0.58
5.3	0.28	3.50	0.45	8.2	0.27	3.26	0.44
5.4	0.28	3.43	0.44	8.3	0.31	4.16	0.49
5.5	0.21	3.18	0.38	9.1	0.15	2.72	0.29
6.1	0.55	4.54	0.78	9.2	0.27	3.72	0.45
6.2	0.37	3.99	0.54	9.3	0.33	4.59	0.50
6.3	0.45	4.78	0.68	9.4	0.19	3.46	0.31
6.4	0.22	3.38	0.41	9.5	0.20	3.49	0.32
6.5	0.22	3.27	0.41				

Table 1
Deposition parameters of TiN on D2 tool steel.

Sample code (SC)	Argon gas flow rate (sccm)	Etching time (min)	Bias voltage (−V)	Substrate temperature (°C)	Nitrogen gas flow rate (sccm)	Deposition time (min)	Cathode current (A)
5.1	50	5	50	300	250	120.00	100
5.2	50	5	50	300	250	90.00	100
5.3	50	5	50	300	250	60.00	100
5.4	50	5	50	300	250	45.00	100
5.5	50	5	50	300	250	30.00	100
6.1	50	5	0	300	250	90.00	100
6.2	50	5	25	300	250	90.00	100
6.3	50	5	75	300	250	90.00	100
6.4	50	5	100	300	250	90.00	100
6.5	50	5	150	300	250	90.00	100
7.1	50	5	50	450	250	90.00	100
7.2	50	5	50	150	250	90.00	100
8.1	50	5	50	300	100	60.00	100
8.2	50	5	50	300	200	60.00	100
8.3	50	5	50	300	300	60.00	100

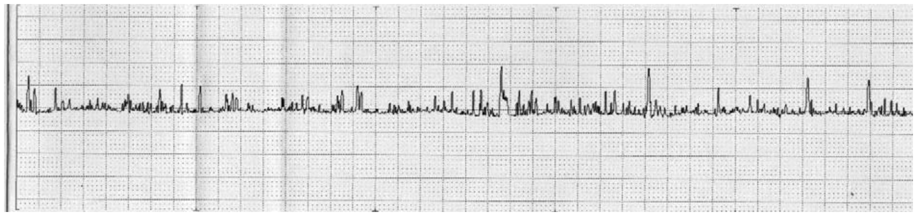

Fig. 1. Surface profile of TiN coating at thickness of ≈6.7 μm measured using a surface roughness tester (EVA-L = 4 mm, λc = 0.8 mm × 5).

The surface roughness increase resulting from the deposition of the TiN coatings could be attributed to the CAPVD process character and explained as following:

1. Occurrence of the characteristic macro-droplets during etching stage at all N_2 gas flow rates.
2. Deposition of pure titanium droplets, originating from the sputtered target during the coating process, bigger in both size and number at N_2 gas flow rate of 100 sccm compared to 200 and 300 sccm.
3. Pits developing due to the titanium macro-droplets, which leave the substrate surface, immediately after termination of the coating process at all N_2 gas flow rates.
4. The surface of the coatings demonstrates inhomogeneities connected with shaped occurrences of the macro-droplets, which re-sputtered during the coating process.
5. Growth defects with an increased effect at N_2 gas flow rate of 300 sccm, originating probably from their sputtering when they hit the substrate surface during the coating process and is due to loss of ion bombardment energy, because of the high pressures of nitrogen gas used.

The maximum *Ra* value (0.37 μm) was noted for coating deposited at N_2 gas flow rate of 100 sccm, whereas, the minimum value was found at 200 sccm (0.27 μm). An increase in *Ra* value (0.31 μm) at 300 sccm is due to rise in growth defects. These results are more encouraging than those of Harris et al. [6] who found the roughness of the TiN coatings on HSS twist Drill, increased from 0.104 to 0.116 μm as the chamber pressure decreased from 1.2 to 0.1 Pa. Similarly, Golombek et al. [7] obtained measured surface roughness *Ra*, parameter value of 0.59 μm, TiN-coated cement carbides using CAPVD technique.

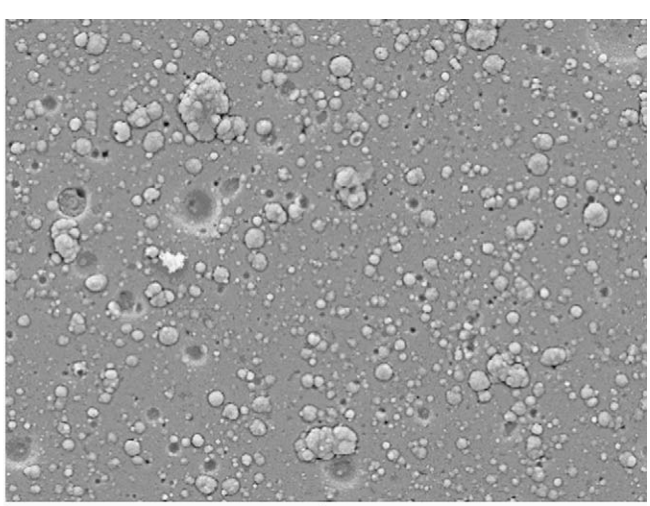

Fig. 2. SEM surface micrograph of TiN coating showing macro-droplets and pitting defects at N_2 gas flow rate of 200 sccm.

The results of quantitative surface roughness parameters of TiN-coated D2 tool steel are reported in Table 3 (SC 9.1–9.5); at various metal ion etching rates. The roughness of TiN-coated tool steel coupons defined by *Ra* parameter is within the 0.15–0.33 μm range and is significantly higher than in the case of the uncoated polished sample. The maximum *Ra* value (0.33 μm) was noted for coating where the Ti ion etching for 16 min. The surface roughness increase during etching and coating stages can be attributed, mainly due to the following factors:

1. Occurrence of the characteristic macro-droplets during etching stage (except zero ion etching and less in the case of Cr ion etching).
2. Deposition of the pure titanium droplets, bigger in both size and number in the case of Ti ion etching and increased by increasing Ti ion etching.
3. The surfaces of the coatings demonstrate inhomogeneities connected with shaped occurrences of the macro-droplets, which re-sputtered during coating process, which is more pronounced for Ti ion etching.

Cr ion etching minimizes the surface roughness of the subsequent coatings [8,9] and similar results can be seen in the present work. By increasing the Cr ion etching time from 8 to 16 min, a slight increase in surface roughness is noted as can be seen in Table 3.

The coating surface demonstrated non-homogeneities connected with shaped occurrences of the droplet shaped and elongated micro-particles, originating probably by their sputtering when they hit the substrate surface during coating deposition process, and can be seen in Fig. 2. Fig. 2 shows hollow spots and pitting defects, considered detrimental factures of CAPVD Technique [10,11].

4. Conclusions

From the present work, it can be concluded that surface roughness mainly depends on nature of substrate and its surface, deposition time, coating thickness, substrate temperature and bias voltage, whether metal ion etching has been performed or not. The surface roughness increase, resulting from deposition of the TiN coatings should be attributed to the cathodic arc PVD process character and occurrence of the characteristic macro-droplets. Again, the variation in surface roughness is due to deposition of the pure titanium droplet(s) originating from the target, and pits developing due to the titanium micro-particles dropping out, immediately after the coating deposition process is completed. Chromium ion etching decreases significantly the surface roughness compared to titanium ion etching in TiN coatings. By increasing titanium ion etching, an increase in surface roughness was recorded.

Acknowledgements

One of the authors, Mubarak Ali, would like to thank the Government of Malaysia, for the award of a scholarship for PhD studies and AMREC, SIRIM Berhad for the access to the experimental facilities for this research.

References

[1] M.S. Lou, J.C. Chen, C.M. Li, Surface roughness prediction technique for CNC end-milling, J. Ind. Technol. 15 (1) (1998–1999) 1–6.
[2] M. Ali, E. Hamzah, M.R.M. Toff, A.H. Hashim, The effect of nitrogen gas flow rate on the properties of TiN-coated HSS using cathodic arc evaporation PVD technique, Surf. Rev. Lett. 12 (4) (2005) 631–643.
[3] C.J. Tavares, L. Rebouta, E. Alves, A. Cavaleiro, J.P. Rivière, A. Declemy, A structural and mechanical analysis on PVD-grown (Ti, Al)N/Mo multilayers, Thin Solid Films 377–378 (2000) 425–429.
[4] P.E. Hovsepian, D.B. Lewis, W.-D. Münz, A. Rouzaud, P. Juliet, Chromium nitride/niobium nitride superlattice coatings deposited by combined cathodic-arc/unbalanced magnetron technique, Surf. Coat. Technol. 116–119 (1999) 727–734.
[5] M. Ali, E. Hamzah, M.R.M. Toff, Friction coefficient and surface roughness of TiN-coated HSS deposited using cathodic arc evaporation PVD technique, Ind. Lubr. Tribol. 60 (3) (2008) 121–130.
[6] S.G. Harris, E.D. Doyle, Y.-C. Wong, P.R. Munroe, J.M. Cairney, J.M. Long, Reducing the macroparticle content of cathodic arc evaporation TiN coatings, Surf. Coat. Technol. 183 (2004) 283–294.
[7] K. Golombek, L.A. Dobrzański, M. Soković, Properties of the wear resistant coatings deposited on the cemented carbides substrates in the cathodic arc evaporation process, J. Mater. Process. Technol. 157–158 (2004) 341–347.
[8] W.-D. Münz, I.J. Smith, D.B. Lewis, S. Creasey, Droplet formation on steel substrates during cathodic steered arc metal ion etching, Vacuum 48 (5) (1997) 473–481.
[9] M. Ali, E. Hamzah, M.R.M. Toff, Study of macrodroplet and growth mechanisms with and without ion etchings on the properties of TiN coatings deposited on HSS using cathodic arc physical vapour deposition technique, Mat. Sci. Eng A 474 (1–2) (2008) 236–242.
[10] A. Matthews, A.R. Lefkow, Problems in the physical vapor deposition of titanium nitride, Thin Solid Films 126 (1985) 283–291.
[11] G. Disatnic, R.L. Boxman, S. Goldsmith, Characteristics of macroparticle emission from a high-current-density multi-cathode spot vacuum arc, IEEE Trans. Plasma Sci. PS-15 (1987) 520–523.