Properties of Nanosontructured Ti-Zr-N Thin Films Prepared by Pulsed Magnetron Sputtering: Applied Power Effect

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Abstract-The effectivness of applied power on structure and tribomechanical properties of Ti-Zr-N films, deposited on AISI304 stainless steel substrate, by pulsed magnetron sputtering PMS technique was investigated. The composition and Ti-Zr-N films properties were varied as the applied power changing from 125 W up to 225 W at a fixed deposition time of 90 min and constant nitrogen / argon gas ratio of 20%. XRD analysis reveals that, formation of FCC structure Ti-Zr-N as a solid solution phase and TiN as a chemical compound phase. The dominant phase Ti-Zr-N shows a preferred orientation in (111) orientation. It was obtained that all Ti-Zr-N coatings have a nanostructured appearance. The microhardness of the thin films is increased up to 1000 HV0.025 which approximately represents 4-folds as that of AISI304 stainless steel substrate. Furthermore, the rate of wear has been improved from 66.64 mm³/Nm for AISI 304 substrate to 15.75 mm³/Nm after depositing Zr-N. Over and above, the coefficient of friction (C.O.F) is decreased from nearly 0.69 for the austenitic substrate to nearly 0.16 after coating. Finally, the corrosion performance of Ti-Zr-N films is improved about 1000 times as that of AISI304 substrate.

Keywords—PMS, Nanostructured Ti-Zr-N films, Tribomechanical properties, AISI304substrate

I. INTRODUCTION

The industrial tools have considerable improvement as a reason of advance enhancement in protective coating processes [1, 2]. Transition metal nitrides (TMN) based thin films belong to the improvment assembly of the service quality for industrial machinery and automobile parts [3]. It was noticed during the last decades that a large concern for studying Ti-Zr-N and Ti-Al-N coatings owing to having a good tribomechanical properties such as low (C.O.F) and good corrosion performance compared to TiN [4, 5]. coatings with Ti–Zr–N were fabricated by various physical vapor depositions (PVD) such as cathodic arc deposition [6], arc ion plating [7], evaporation by electron beam [8] and PMS [9].

Moreover, PMS is largely employed for industrial application owing to several advantages like low target poisoning; which preventing arcing and increasing the rates of deposition [10, 11].

Little studies elsewhere [12, 13] gived that the applied power has a significant factor to control the tribomechanical properties of Ti-Zr-N coating. In This research, further investigation of the effectivness of applied power on the structure, mechanical, tribological of Ti-Zr-N films deposited on AISI304 substrate has been considered.

II. EXPERIMENTAL

A. Sample Preparation

AISI304 stainless steel $(10 \times 20 \times 1 \text{mm})$ was used in this study as a substrate material. The chemical composition of AISI304 in wt% is: 0.075 C, 1.2 Mn, 19.1 Cr, 8.5 Ni, 0.5 Si and Fe balance. The substrates were grinded and polished by typical metallographic way. Then, the substrates were ultrasonically cleaned in methanol for 10 min then put in the chamber of deposition.

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B. Deposition Conditions

The schematic design of the system as in Fig. 1. The AISI304 substrates were mounted on a 50mm diameter sample holder, which is centered in the front of three guns in the chamber of deposition. A high purity (99.99%) target 50mm in diameter with a composition of 90% Ti and 10% Zr was used. The distance between the target and the sample holder located at 30mm. After inserting the substrates into the PMS chamber, the system was evacuated by two stages; rotary and turbo pumps from atmospheric pressure to a base pressure of approximately 5x10-6mbar.



Fig. 1 Schematic diagram of the pulsed magnetron sputtering system

Pure argon plasma sputtering (150 W and 100 KHz) was applied on the TiZr target for 3min to eliminate the oxide layer effect, sputter the localized states of the grown nitrides and to eliminate the sharp surface defects that accountable for sparking effects. After that, the deposition of the Ti-Zr-N coatings was carried out in a mixture of 80% Ar and 20% N2 under a constant total working gas pressure of 5×10^{-3} mbar. The applied power was varied to 7 levels from 125 W to 225 W. A chrome-alumel thermocouple was, placed close to the surface of a blank sample, used to measure the sample temperature during the PMS process. The measured sample temperature has a range from 35° C to 40° C. After the deposition process was completed, the chamber is permitted to cool down before venting to atmospheric pressure.

C. Characterization And Testing The Ti-Zr-N Coatings

Crystal structure of the substrates after and before coating was analyzed by X-ray diffraction using (Philips-PW1710 diffractometer) with Cu K α radiation of $\lambda = 1.541838$ Å. The scans were obtained with a 0.02° step size in a range from 20° to 100°. The morphology of substrates surface after and before coating was obtained by Olympus BX51 optical microscope. Vickers microhardness measurement was measured by a Leitz Durimet microhardness tester equipped with static load of 25gmf. The Vickers microhardness tester was accredited depending on the ISO/IEC 17025:2005 requirements. The roughness of the samples surfaces was measured by a Form Talysurf 50 which was accredited depending on the requirements of ISO/IEC 17025:2005. The contact angle (C.A) of water and surface energy were detected by Phoenix 300 (C.A) Analyzer manufactured by S.E.O Co. Ltd, at room temperature. Phoenix 300 utilized a precision camera and advanced PC technology to capture the static droplet image and determine the (C.A) by Sessile Drop method. The (C.O.F) was performed at a mean sliding speed of 10 mm/s with a normal load of 1N. A 3mm tungsten carbide (WC) ball was used as counterpart material without lubrication at room temperature in air atmosphere with humidity of 35% - 40%. Wear measurements were conducted depending on ASTM G133-10 standard test method. Wear track were measured by Form Talysurf50. The rates of wear were obtained by equation K = V/SF where V is the wear volume in mm3, S

is the total sliding distance in m and F is the applied load in N. The electrochemical corrosion experiments were done by Gill AC instrument in 5% H2SO4+ 0.05 KSCN solution using the potential dynamic technique at $28^{\circ}C \pm 3^{\circ}C$ temperature and humidity of $38\% \pm 5\%$. The using of H2SO4 solution is to obtain a strong corrosive medium which can corrode different metals in a uniform corrosion mode. The exposed effective area was fixed at 0.36 cm2. In this study, the corrosion test was made using three-electrodes; silver-silver chloride saturated electrode as a reference electrode, platinum as a counter electrode and the investigated sample as a working electrode. The potential–current corrosion curve of all samples is recorded with a potential scan rate of 1 mV/s.

III. RESULTS AND DISCUSSION

A. XRD Analysis

The X-ray diffraction patterns of AISI304 substrate and that coated with Ti–Zr–N films were obtained at different applied powers are presented in Fig. 2. The substrate spectrum is correlated to γ -phase austenitic stainless steel. After coating, the γ -austenite phase shows lower intensities which denote that the deposition thickness is lower than the depth of incident X-ray penetration.



Fig. 2 XRD of AISI 304 and Ti-Zr-N thin film samples at different applied powers

The XRD data point that the main phases observed after coating are a solid solution Ti-Zr-N phase and a chemical compound phase of TiN. The Ti-Zr-N has a crystallite structure with a face centered cubic lattices. The massive intensity of Ti-Zr-N (111) and TiN (200) points that the expansion orientation of Ti-Zr-N and TiN is mainly due to planes (111) and (200), respectively.

Fig. 3. demonstrates the relative intensity ratios of Ti-Zr-N (111) and TiN (200) planes at different applied power. As the applied power increases up to 175 W, the Ti-Zr-N (111) intensity increases to a maximum value. The increase of Ti-Zr-N (111) intensity because of the unity of energetic nitrogen ions. As the applied power increases, the impinging energy of nitrogen ions increases which promote the physical and chemical reaction between the nitrogen species and the surface of Ti-Zr target. With further raise in the applied power, the Ti-Zr-N (111) intensity plane decreases as a result of growing of Ti-Zr-N (311) plane.



Fig. 3 Intensity ratio of (Ti, Zr) N (111) and TiN (200) phases at different applied power

The Ti-Zr-N (111) plane is the hardest orientation in transition metal nitrides because of the reflection peak positions of this plane are found around the central point between the TiN (111) peak and the ZrN (111) peak [14-17]. The Ti-Zr-N phase with the same preferred orientation (111) has been detected in similar coatings which were coated by other PVD techniques [14, 15, 18]. Moreover, the lattice parameters of TiN and Ti-Zr-N phases were calculated and are establish to be 4.24 and 4.27, respectively. A typical value of lattice parameter for TiN (4.24) was detected elsewhere [18]. The increase in the lattice parameter of Ti-Zr-N coatings compared to that of TiN is ascribed to the substitution of Ti with Zr in the TiN lattice [18, 19]. Debye Scherrer Eq. (1) was used to obtain the crystallite size of the Ti-Zr-N (111) plane at different applied power [20].

Crystallite Size =
$$\frac{k\lambda}{(FWHM)\cos\theta}$$
 (1)

Where k is a Scherrer factor (0.8-1.0), λ is the wavelength of incident radiation, θ (rad) is the Bragg angle of the reflection and FWHM is the full width at half maxima.



Fig. 4 Crystallite size of the Ti-Zr-N thin film at different applied power

Fig.4 demonstrates the crystallite size of the Ti-Zr-N (111) compound at different applied power. For 125 W applied power the energy of ion transfer is enough for the nucleation and more growth of Ti-Zr-N (111) compound and lower crystallite size. With increasing applied power to 175 W, an energy amount is transferred to the surface of the targt, which leads to an additional increase of Ti-Zr-N (111) intensity and more decrease of crystallite size. With further increase of the power up to 225 W, newly formed compounds and the earlier crystallite of produced phases are able to absorb more energy, which causes the crystallite size of Ti-Zr-N (111) to increase [21].

B. Coating Parameters

The variation of coating parameters as at different applied power are utilized in Fig. 5. The coating thickness is determined as five values average taken along the step surface between the coated and uncoated sample. The deposition rate is utilized using the formula of t/s, where t is the average thickness of the coated layer in nm, and s is the total deposition time in sec. It is noticed that the thickness and the deposition rate increase continuously with increasing the applied power. K. Kusaka et al. [22] reported that as the applied power increases, the energetic target atoms which sputtered increase and the film thickness and deposition rate increase.



Fig. 5 Film thickness and deposition rate of Ti-Zr-N coatings at different applied power

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C. Microhardness

Fig.6 illustrates the surface microhardness variation of the Ti-Zr-N thin film samples at different applied power. It was noticed that, the hardness value significantly increases to a maximum value 1098.8 HV as the applied power is increased to175 W which represents nearly 4.5 folds the hardness of the substrate. After that, the hardness gradually decreases up to 635.7HV with more increase in the applied power up to 225 W.



different applied power

The enhancement in hardness is mostly attributed to the formation Ti-Zr-N phase owing to a solid solution strengthening mechanism that provides an energy barrier to the dislocations all around the crystals that have distortion of the periodic lattice caused by the add atoms of Ti and Zr during deposition [17]. The higher hardness was obtained at the smallest crystallite size at 175 W applied power as in Fig. 4. Moreover, transition metal nitride (111) is the hardest orientation owing to the truth of Schmid factor is zero on all slip systems [17].



Fig. 7 The water (C.A) and surface energy of the Ti-Zr-N coatings at different applied power

D. Surface Energy And Surface Roughness

Fig. 7 presents the water (C.A) and surface energy of Ti-Zr-N thin film samples at different applied power. It appears that the (C.A) decreases from 79.3° for AISI304 substrate to 33.7° for Ti-Zr-N film coated at 225 W applied power. Elsewhere [23] reported that low (C.A) and high surface energy refers to the high surface wettability and vice versa which is agreed well with the present study. On the other hand, the surface energy increases from 31.2 mN/m for AISI304 substrate to 75.9 mN/m for Ti-Zr-N thin film prepared at 225 W owing to formation of TiN and Ti-Zr-N hard phases which in turn increases the surface strengthening.

The parameters of roughness is obtained from the mean height of peaks and valleys on the surface. Fig. 8 illustrates the Ti-Zr-N roughness at different applied power. It is noticed that the Ra is gradually raised with the raise in applied power. The raise in surface roughness maybe owing to ion bombardment at the plasma glow discharge [24].



Fig. 8 Ra of the Ti-Zr-N coatings at different applied power

Beside the Ra parameter, there are other roughness parameters such as maximum profile peak heights (Rp), maximum profile valley depths (Rv) and (Rz) is the sum of (Rp) and (Rv) [25]. The Rp/Rz is an significant ratio for depicting the surface form. When the ratio above 0.5 the surface has sharp peaks and when the ratio under 0.5 the surface has rounded peaks [25]. Rounded peaks favor to spread out the liquids on the surface. Using this assumption the effect of Rp/Rz ratio on the surface wettability can be investigated. According to this assumption and the results reported in Table 1, the ratio values of (Rp/Rz) of all TiZrN surface samples are under 0.5 and therefore the surface exhibit rounded peaks and this leads to higher surface wettability.

TABLE I
ΓΗΕ CORRELATION BETWEEN Rp/Rz RATIO AND THE SURFACE WETTABILITY
OF THE Ti-Zr-N SAMPLES AT APPLIED POWER

Samples	$R_v(\mu m)$	$R_z (\mu m)$	$R_p(\mu m)$	R_p/R_z
125 W	0.14414	0.2327	0.0886	0.38
150 W	0.13074	0.2328	0.1021	0.44
162 W	0.2732	0.5103	0.2097	0.41
175 W	0.34594	0.64002	0.29408	0.45
187 W	0.5791	0.7201	0.3089	0.43
200 W	0.55944	0.9061	0.34666	0.38
225 W	0.9545	0.9381	0.28194	0.3

E. Wear And Friction Coefficient Measurements

Fig.9 represents the optical micrograph of wear track of the substrates after and before coatings. Moreover, Fig. 10 shows the track width the wear for the AISI304 and Ti-Zr-N films deposited for different applied powers.



Fig. 9 Optical micrograph of the wear track of AISI 304 and Ti-Zr-N thin film samples

It has been observed that the track widths of the wear for all coated samples are narrower than that of the uncoated sample, signifying the enhancement in the wear resistance. The sample was prepared at 125 W, has the highest wear track width owing to the lack of hard phase Ti-Zr-N (111) and the existence of TiN phase, which possess low wear performance and high (C.O.F) of (0.6 to 0.8) [26]. Moreover, the track width of the wear 3

decreases as the applied power increases up to 175W, then it increases with more increase in the applied power. The enhancement in the wear is recognized to the surface strengthening resulting from the increment of Zr to TiN.



Fig. 10 The wear track width of the AISI 304 and Ti-Zr-N thin film samples at different applied power

Formation of hard phases Ti-Zr-N possess excellent wear resistance, owing to the structure of the stable nitride layers in the film, and enlarged the hardness by solid solution strengthening [27]. It was recorded that TiN coatings have low wear performance and high (C.O.F) of (0.6-0.8) [26] compared to high wear performance and low (C.O.F) of (0.25-0.3) for Ti-Zr-N coatings [28].

Fig. 11 characterizes the (C.O.F) of the AISI304 and Ti-Zr-N films at different applied power. From this figure, the (C.O.F) decreases from nearly 0.69 for AISI304 substrate to nearly 0.16 for the Ti-Zr-N sample prepared at 175 W. At this applied power, the Ti-Zr-N coating sample exhibit best low (C.O.F) nearly 4.3 times lower than of the substrates before coating. The improvement in the wear performance of Ti-Zr-N coatings can be interpreted to that nitrides of metals from the fourth group such as Ti and Zr of the periodic table gives less wear resistance due to chemical compounds with high atomic binding energy between atoms provide high wear performance [29].



From the above results, one can correlate well between the behavior of the wear performance and hardness of Ti-Zr-N films with the relative intensity of Ti-Zr-N (111) plane. On the other hand, the (C.O.F) of Ti-Zr-N films correlates well with the intensity of Ti-Zr-N (111) plane.

Fig.12 noticed the relationship between the (C.O.F) and the sliding distance for the AISI304 and Ti-Zr-N films. It appears that, the sample prepared at applied power 175 W is the best performance of (C.O.F). This sample is recorded no brittle failure during the wear test and it shows approximately (C.O.F) of 0.16. Fig.13 represents the wear rate of the Ti-Zr-N thin film samples at different applied power. One can notice that, the wear rate decreases as the applied power increases to reach minimum value at the specimen prepared at 175 W, after that it increases with more raise of the applied power. From this result, the applied power enhance the wear resistance.



Fig. 12 The (C.O.F) as a function of sliding distance for of AISI 304 and Ti-Zr-N thin film samples



Fig. 13 The Wear rate of AISI 304 and Ti-Zr-N thin film at different applied power

F. Corrosion Performance

The typical potentiodynamic polarization curves for uncoated AISI304 and coated substrates with Ti–Zr–N films immersed in 0.5 M H₂SO₄+0.05M KSCN solution as in Fig. 14. The average values of the corrosion parameter values are calculated from the polarization curves for specimens and are recorded in Table 2. The results show that, the AISI304 uncoated substrate shows the highest corrosion potential (Ecorr = 202.6 mV) and corrosion current (Icorr = 53.317 x 10^{-3} mA/cm²) in comparison with the associated values for the coated substrates with Ti-Zr-N. This result indicates that the Ti-Zr-N coatings deposited on AISI304 improves the corrosion performance of the AISI 304 substrate. Similar results was noticed elsewhere [13]. The reduce of the anodic current of the coated substrates was owing to that Ti and Zr ions will react with nitrogen and deposited onto AISI304 substrate by PMS technique cause high dense with compact grain distribution, this dense in coat protects the corrosive ions penetration through surfaces in coated layers [30].



Fig. 14 The potentiodynamic polarization curves of AISI 304 and Ti-Zr-N thin film samples

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TABLE II CORROSION DATA OF AISI 304 AND Ti-Zr-N FILM SAMPLES IMMERSED IN 0.5M H_2SO_4 +0.05M KSCN SOLUTION

Power (W)	I_{corr} (x10 ⁻³ mA/cm ²)	E _{corr} (mv)	Corrosion Rate (x10 ⁻⁴ mm/year)
AISI 304	53.317	202.6	57.209141
125 W	11.85	-306.35	12.71505
150 W	2.2516	-270	2.4159668
162 W	1.5325	-316	1.6443725
175 W	0.49	-237.35	0.52577
187 W	1.6	-266.12	1.7168
200 W	9.0963	-315.5	9.7603299
225 W	0.4585	84.751	0.4919705

From the table 2, one can see that the corrosion rate decreases from 57.209×10^{-4} mm/year to 0.526×10^{-4} mm/year for the sample that was coated for applied power of 175 W. This can be revered to the raise of the relative intensity of hard (Ti, Zr) N (111) plane with increasing the applied power up to 175 W as in Fig. 3. From the results in table2 and Fig.14 one can conclude that, the corrosion performance inhanced for the Ti-Zr-N coated substrate in comparison with that of AISI304 uncoated substrate.

IV. CONCLUSION

1- Nanostructure of Ti-Zr-N thin films was utilized by PMS technique at different applied powers.

2- Structural composition, hardness and tribological performance of the Ti-Zr-N coatings are widely varied with changing the applied power.

3- Applied power at 175W produces the lowest value of crystallite size which contains mainly Ti-Zr-N phase with high hardness, wear resistance, corrosion resistance and surface energy; and lower (C.O.F).

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Interest

- 1. Powder metallurgy processing.
- 2. Functional Graded Materials (FGMs).
- 3. Smart Materials, Shape Memory Alloys (SMAs).
- 4. Wood Plastic Composites.
- 5. Thin Films Coating.

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