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INDEX

Characteristic of AlN Layer Deposited by d.c. Magnetron Sputtering on AISI 410 Steel

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***Abstract*--** **The aim of this study is to investigate the effect of AlN sputtering with d.c. magnetron sputtering on surface roughness, specific wear and corrosion rate of the AISI 410 steel. AlN thin film was deposited on AISI 410 steel substrates using d.c. magnetron sputtering in discharging a mixture of N_2 and Ar gas. The influence of deposition time on the surface roughness, hardness, specific wear for the obtained films was evaluated by means of surfcorder, microhardness testing, and wear testing. The investigations of the electrochemical corrosion behavior of the samples were carried out using EG&G 273 Potentiostat/Galvanostat in three electrodes chamber. As the result, the surface treatments with d.c. magnetron sputtering methods increase hardness, but decrease corrosion rate and specific wear rate. Changes in wear rate and corrosion rate could be related to the surface roughness.**

***Index Term*--** **AlN thin film, d.c. magnetron sputtering, corrosion rate, specific wear rate.**

I. INTRODUCTION

Martensitic stainless steels are widely used in engineering applications where good resistance is a key performance indicator in addition to the corrosion resistance shown by stainless steels. Such applications include turbine blade, tools, bearings and in surgery, for example bone saws for orthopaedic surgery [1-2]. However the performance of these steels is reduced due to simultaneous effect of wear and corrosion mechanisms [2-3]. The martensitic stainless steels grade, in particular AISI 410 is widely used in chemical industry, and in medical instruments as cutting and non cutting instrument [4]. Many attempts have been made in the past few decades to modify the surface and in general improve their tribological properties [5]. Corrosion resistance properties of martensitic stainless steel was lower compared to austenitic and feritic stainless steels [6]. Wear resistance properties of martensitic stainless steel was low, so it was necessary to improve in many efforts to improve the life time [7]. An ideal tool must have a high ductility and a maximum possible resistance to wear in working conditions. Normally such a combination is impossible to obtain. Thus there have been various attempts made to solve, at least partly, the problem by creating layer structures using, among others, methods of thermo chemical treatment, by making composite materials and single layer using CVD and PVD methods [8].

Hard coatings of the metal nitrides increase life of the elements coated by them. Also their abrasion wear is less in contacted with hard materials, and they are more resistant to chemically active environment compared with the uncoated metal materials. Deposition of hard coatings (transition metal nitrides, carbides, or oxides) on surface by PVD and CVD processes features one of the most intensely developed directions in improvement working properties of the materials [9]. Transition metal nitrides are extensively used in various scientific and industrial applications, due to their substantial hardness, chemical stability, high melting point, high wear resistant. Especially, they are widely used in various engineering applications such as cutting tools, aeronautic, automotives [10].

PVD uses lower processing temperatures compared to CVD. High residual compression stress and fine grains originated by the PVD process usually improve the hardness and toughness [11]. AlN film deposited by PVD produces polycrystalline films, smooth, uniformed layer, and short processing times and no heating of the substrate [12]. The aim of the present investigation was to study the roughness, hardness, corrosion rate, and specific wear properties AlN deposited by d.c. magnetron sputtering on AISI 410 steel.

II. EXPERIMENTAL PROCEDURE

2.1. Materials

The alloy studied here was AISI 410 with the following chemical composition (wt. %): C, 0.12; Cr, 12.83; Mn, 0.43; Si, 0.34; Ni, 0.21; Fe, balance. The sample consisted a disks of 14 mm in diameter and 3 mm in thickness. The samples surfaces were fine-ground using 200, 400, 600, 800, 1000, and 1200 grit SiC paper and finally it were polished with autosol. The substrates were cleaned in an ultrasonic bath cleaner using acetone.

2.2. Sputtering process

Sputtering process was carried out in 285 mm diameter and 200 mm height stainless steel vacuum Balzers using d.c. magnetron sputtering. The samples were placed on the sample holder having as an anode with the respect to the chamber wall. The vacuum chamber was evacuated to base pressure of 7.6 x 10^{-2} torr and high purity argon was introduced at pressure 7 x 10^{-2} torr. Next, the target was sputtered-cleaned at 300 V, sputter current 80 mA for 10 min and then nitrogen (99.96% pure) was introduced and the working gas mixture was adjusted to total pressure 0.6 x 10^{-2} torr. The substrates were not heated externally. The substrates temperature were fixed at 250 ^{o}C, assumed to result from plasma heating due to ion bombardment. Purities of Al target was 99.999% with 50 mm in diameter and 3 mm in thickness, having as a cathode. A schematic diagram of the d.c. magnetron sputtering system used in our experiments is shown in Fig. 1.

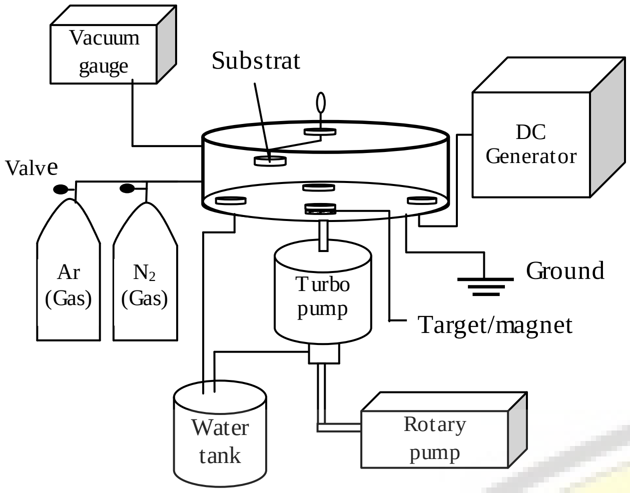

Fig. 1. Schematic diagram of the d.c. magnetron sputtering

2.3. Hardness Test

Microhardness measurement were performed using Vickers hardness testing machine (Buehler USA) with diamond indentor using a load of 10 g and indentation time of 10 seconds. The Vickers hardness number can be determined using following expression [13]:

$$HVN = 1.8544 \frac{F}{d^2} \qquad (1)$$

where :

F = load (kg)

d = indentation diameter (mm)

2.4. Wear Test

An Ogoshi OAT-U abrasion wear testing machine was used to investigate the wear behavior. In the present work the specimen was abraded with rotating disk. The test specimens with a dimension of 40 x 50 mm were prepared for abrasion wear testing. The schematic diagram of the wear testing is shown in Fig. 2. The rotating disk with dimension 2r in diameter, and B in thickness. Abrasion depth can be expressed by h, groove length b, and abraded volume W, load P = 2.12 kg, speed 0.244 m/s, abrasion time 10 s, and sliding distance l_o = 2.44 m. The specific wear rate can be determined in the following expression [14-15]:

$$W_s = \frac{1{,}5\, W_0}{P_0\, l_0} \qquad (2)$$

where :

W_s = specific wear rate (mm^3/kgf. m)

W_o = the amount abraded volume (mm^3)

P_o = normal load (kgf)

l_o = sliding distance (m)

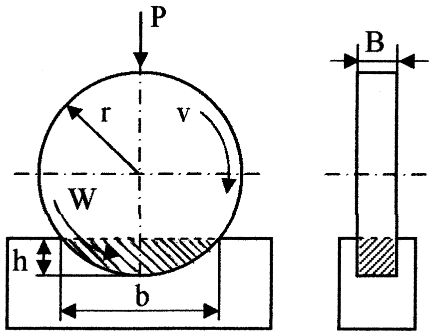

Fig. 2. Schematic diagram of the wear testing

2.5. Corrosion testing

Investigation of the electrochemical corrosion behavior of the samples was done in EG&G 273 Potentiostat/Galvanostat, using the conventional three-electrodes technique. The potential was referred to a saturated calomel electrode (SCE), using a 0.9% NaCl solution. The testing temperature was done at 25 ^{o}C and corrosion rate r mils per year (mpy) was calculated using following expression [16]:

$$r = 0.129 \frac{i_{corr}}{\rho} EW \qquad (3)$$

where :

i_{corr} = corrosion current ($\mu A/cm^2$)

ρ = density (g/cm^3)

EW = ekuivalent weight (dimensionless)

The equivalent weight was calculated by equivalent numbers, N_{EQ}, that is EW = $(N_{EQ})^{-1}$

$$N_{EQ} = \sum\left(\frac{f_i}{a_i/n_i}\right) = \sum\left(\frac{f_i\, n_i}{a_i}\right)$$

where :

f_i = mass fraction

n_i = electron valensi

a_i = atom weight

III. RESULTS

Fig. 3 shows the anodic potentiodynamic polarization curves of the untreated and treated AlN deposited by d.c. magnetron sputtering in 0.9% NaCl solution. The untreated steel did not have a passivation region, instead the current density increase at high rate immediately after the corrosion potential under the testing conditions. The polarization curve of the AISI 410 steel was considerably modified after sputtering AlN (Fig. 3). The current density (i_{corr}) was decreased from 1.81 μm for the untreated sample to 1.69, 1.72, 1.74, respectively for the untreated samples. Among all the samples, sample with deposition time 70 minutes had the highest current density, and its corrosion rate was higher than the untreated steel. The corrosion rate of the sputtering AlN decreases in deposition time 40, 50, 60 minutes, but tend to increase in each time deposition. The corrosion has the highest value at deposition time 70 minutes, higher than untreated specimen.

Microscopic observation of the sputtering AlN indicates that the sample has undergone change as shown in

Fig. 4. The sample without treatment has shown pitting corrosion with potential corrosion value -341.04 mV and pitting corrosion is still occurred on the sample treated of AlN coating.

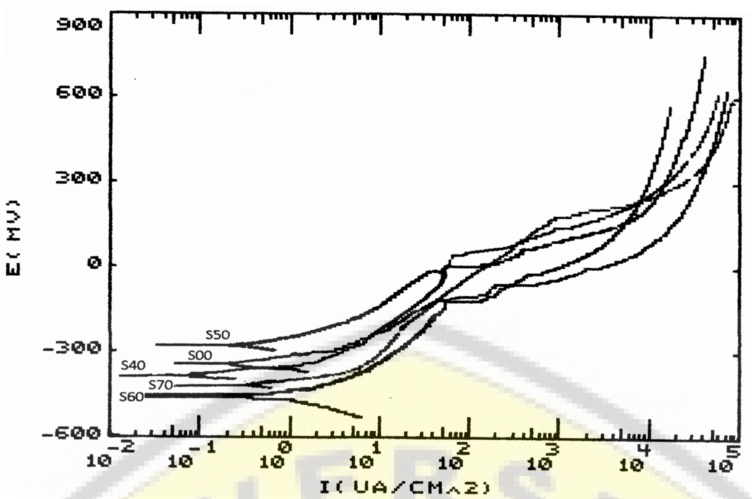

Fig. 3. Potentiodynamic polarization curves of the AlN sputtering deposited by d.c. magnetron sputtering in 0.9% NaCl solution. a). S00 = untreated specimens; b). S40 = sputtering 40 minutes; c). S50 = sputtering 50 minutes; S60 = sputtering 60 minutes; S70 = sputtering 70 minutes

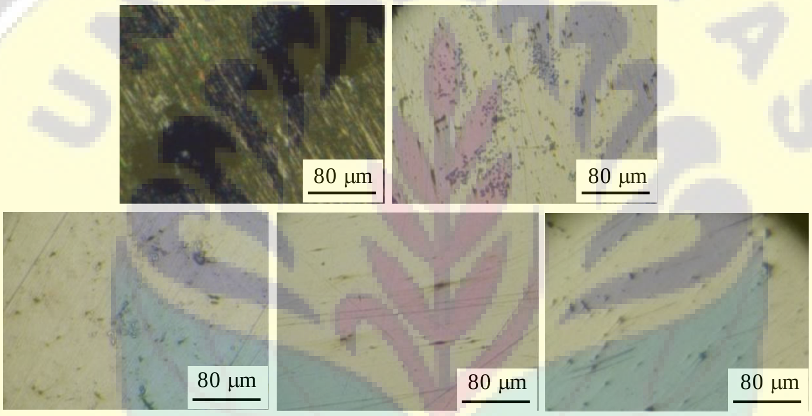

Fig. 4. Microscopic observation on the surface after corrosion test in 0.9% NaCl solution. a) raw material; b) 40 minutes of sputtering; c) 50 minutes of sputtering; d) 60 minutes of sputtering; e) 70 minutes of sputtering

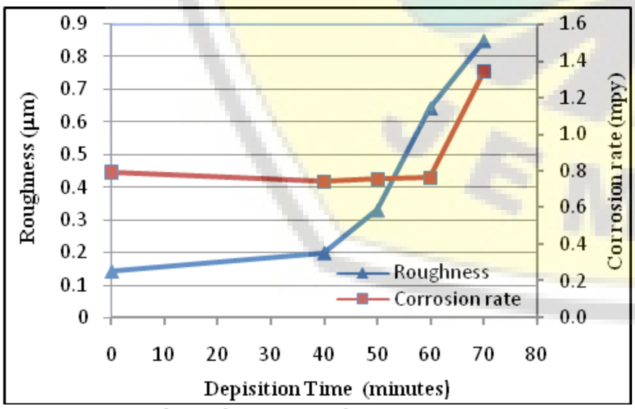

Fig. 5. Correlation between roughness versus corrosion rate

Fig.5 shows the correlation between the surface roughness and corrosion rate as a function of deposition time. As the deposition time increase, the surface roughness of the AlN coating gradually increase from 0.072 of the untreated specimen to 0.198, 0.329, 0.642 and 0.849 µm for the sample with sputtering time of 40, 50, 60 and to 70 minutes.

Evaluation of the corrosion testing indicates that the AlN deposition with d.c. magnetron sputtering has shown current density i_{corr} changes. The corrosion rate of the AlN deposition decreases for deposition time 40 minutes and then increase again with an increase sputtering time deposition of 50, 60 and 70 minutes. The corrosion rate and surface roughness has the same tendency to increase at time variation.

The microhardness and specific wear relation is plotted against the time deposition as shown in Fig. 6. The sputtering AlN with d.c. magnetron sputtering can increase microhardness AISI 410 steel. The microhardness value has a maximum of 60 minutes deposition time. The specific wear of the sputtering AlN deposition decrease with an increase deposition time. As the deposition time increased, the specific wear of the AlN coatings specimens gradually decrease from 0,923 mm^3/kg.m of untreated specimens, and it reach minimum values of approximately 0,475 mm^3/kg.m at the deposition time of 50 minutes, and then steeply increased again with further increase of deposition time.

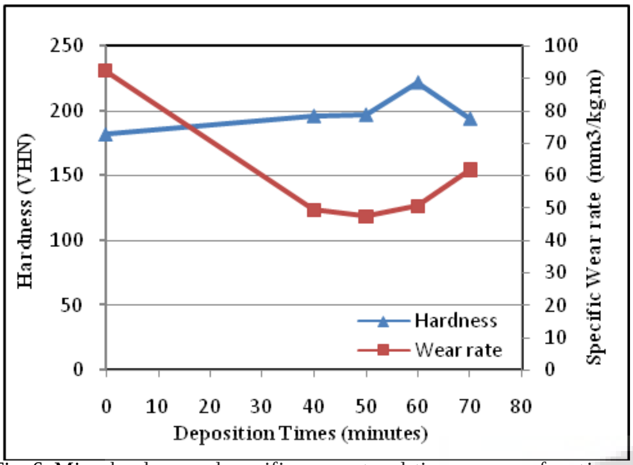

Fig. 6. Microhardness and specific wear rate relation as a function of deposition time.

The roughness and specific wear rate relation are plotted against the deposition time as shown in Fig. 7. As the time deposition increase, the roughness of the AlN coatings increase gradually from 0,141 μm of untreated specimens, and steeply increase again with further increase of deposition time.

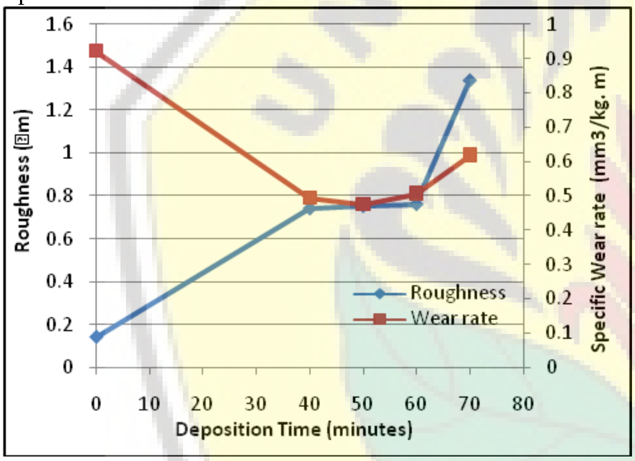

Fig. 7. Specific wear rate and surface roughness relation as a function deposition time.

Fig. 8 shows SEM-EDAX observation for the cross section AlN sputtering AISI 410 sample for 60 minutes sputtering time. The concentration of Al and N atoms are plotted as a line scan from surface layer to the base material.

The Al and N atoms are maximums near to the surface and reduce very sharply inside the base metal. Fig 9 shows atomic content of Al and N atom. It seems that the atoms Al and N can diffuse into base metal.

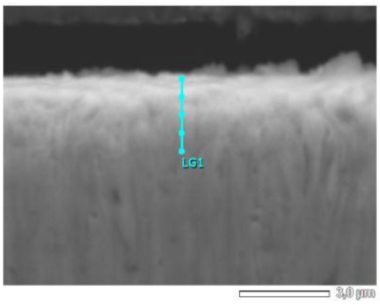

Fig. 8. SEM-EDAX micrograph of cross section of the AISI 410 sample treated for 60 minutes sputtering AlN

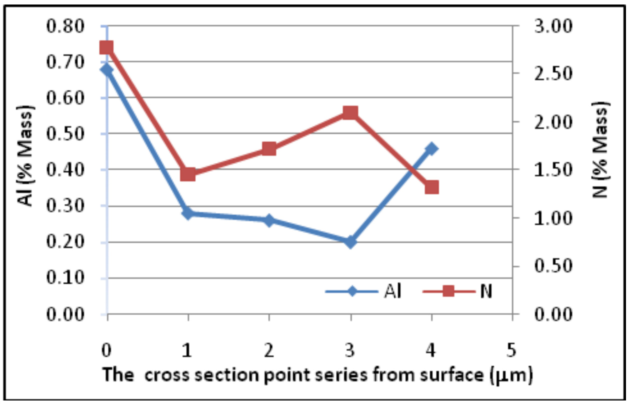

Fig. 9. Aluminium and Nitrogen concentration profiles across the cross-section of AISI 410 sample treated for 60 minutes sputtering AlN

IV. DISCUSSION

Based on the results of the roughness test, the AISI 410 steel substrate without coating shows the roughness of 0.141 μm. The deposition of the AlN sputtering with d.c. magnetron sputtering onto the specimens causes increase the roughness. The surface roughness steeply increased with further increase of time deposition [8] [17].

It was found out, as a result of the electrochemical corrosion investigations that the corrosion rate with potentiodynamic polarizations in 0.9% NaCl solution, the corrosion rate of the AlN coating can decrease corrosion rate the AISI 410 steels untreated. The corrosion rate increases with an increase in the roughness. The deposition of the coatings causes porosity and perforations so it caused increasing galvanic corrosion [18-19]. The surface morphology shows the pitting corrosion (Fig. 4).

It was found out, as a result of the electrochemical investigation, that the lower current density i_{corr}. This may be explained by the fact that sputtering with d.c. magnetron sputtering AlN gives better possibility to prevent corrosion causes, like scratches or crevices. Small pores and cracks in the coatings and the difference defect and failures occurring in single layer in the deposition process may be masked by the successively deposited coatings layer [9]. There for the corrosion agent path is extended or blocked.

The lowest value of wear rate does not occur on the sample with the highest hardness of the sample. There is no correlation between hardness and wear [20]. The wear rate value is more affected by the growing of surface roughness, it is caused the surface coefficient of friction. Surface roughness causes the increasing contact stress, because the surface area of contact is not homogeneous, so the friction will be increase.

The discussion and Fig. 5 qualitatively explained that the corrosion resistance of AISI 410 steels can be improved by AlN thin film deposited by d.c. magnetron sputtering. However, the detailed corrosion mechanisms are still not clear at this stage due to many unknown factors affecting corrosion of sputtered AlN AISI 410 steels. Further studies have to be performed to characterize the phase configuration, microstructure, residual stress, and grain size. It is also necessary to investigate the corrosion behavior of each surface roughness and the N/Al atomic ratio and their contributions to the overall corrosion resistance of the AlN layer deposited by d.c. magnetron sputtering on AISI 410 steels.

V. CONCLUSION

The thin film deposition of aluminium nitride by d.c. magnetron sputtering technique has been carried out successively. The composition of the coatings with AlN sputtering with d.c. magnetron sputtering causes the increase of surface roughness Ra = 0.198 – 0.849 μm in comparison with the AISI 410 steels substrate for the deposition of coatings. The corrosion rate of AlN coating decrease 0.792 mpy of untreated specimen and it reach minimum values of 0.741 mpy of sputtering AlN 40 min. The specific wear rate of the AlN coating decreases from 0.923 mm^3/kg.m for untreated specimen and it reaches minimum values of 0.475 mm^3/kg.m for sputtering time of 50 min. There is no direct correlation between the surface micro-hardness and the wear behavior. The corrosion rate and specific wear rate are influenced by surface roughness. It was observed that Al and N atoms diffuse into base metal.

The deposition of the coatings with AlN thin film deposited by d.c. magnetron sputtering causes the increase of the roughness and the friction coefficient. These effects are not expected in corrosion behavior and specific wear rate.

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