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THE INVESTIGATION OF WEAR PROPERTIES OF TIN COATINGS ON THE H13 TOOL STEEL UNDER DIFFERENT DEPOSITION PARAMETERS

ABSTRACT

The present paper reports the influence of working pressure and nitrogen flow rate on the mechanical and wear properties of TiN thin films deposited by DC reactive magnetron sputtering with RF bias voltage. The process of PVD coating is conducted in a nitrogen-argon gaseous mixture at a temperature of 150°C and a DC power of 200W for 90 min. The micro hardness and the wear resistance of the TiN are determined prior to and after the PVD coating. The wear volumes of samples were evaluated using optical profilometre. Worn surfaces were observed using scanning electron microscopy. It was observed that coated samples showed considerable higher mechanical properties and wear resistance compare to uncoated samples. Abrasive wear mechanism was observed on the surface of worn surfaces.

Keywords: PVD, Wear, Coating, Working Pressure, TiN

1. INTRODUCTION

TiN coatings are used in many industrial areas such as aerospace application, chemical industrial, medical applications, cutting tool materials [1 and 3]. The properties of coatings such as hardness, scratch resistance and wear resistance depend on the micro structure of coatings. A lot of deposition parameters such as sputtering pressure, bias voltage, substrate temperature, sputtering yield can affect the structure of coatings. Various deposition methods have been used to coat TiN on the surface of different materials [1, 4 and 8]. Among them magnetron sputtering is widely used because of its many advantages over the other methods such as low levels of impurities, easy control of deposition rate relatively low cost and so on [9 and 10]. Although, considerable number of researches have been conducted to enhance the properties of TiN coatings, there are not enough study the effect of RF (Radio Frequency) power, working pressure and nitrogen partial pressure on the properties of TiN coatings.

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2. RESEARCH SIGNIFICANCE

In this study, it was investigated the effect of RF bias voltage, working pressure and Nitrogen partial pressure on the properties of TiN coatings using magnetron sputtering method.

3. EXPERIMENTAL METHOD

AISI H13 hot work steel was used as the substrate material. Before deposition process, the surfaces of substrate materials with 25mm diameter and 5mm thickness were grounded with SiC papers with 2000 grit and then polished with Al_2O_3 suspension. After these processes, all samples were ultrasonically cleaned in ethanol path. The RF/DC magnetron sputtering deposition system was used to coat the substrate surfaces as shown in Figure 1.

Figure 1. Magnetron sputtering system

DC power was applied to magnetron and RF power was applied to substrate. A pure titanium disk (99.99%) was used as a target and N_2 was as a reactive gas, Argon was used as a sputtering gas. The reason why use the DC reactive sputtering from elemental targets related with different factors such as; it is possible to produce compound film with controllable stoichiometry, high purity films can be produced, the range of the applied power can be extended without the fear of being cracked and deposition can be done at temperature less than 300°C [10]. The samples were put inside the chamber and substrates were heated to 150°C. All deposition processes were performed at the same substrate temperature. The other deposition parameters are given in Table 1. These parameters were selected according to literature and previous laboratory experimental studies.

Table 1. Deposition parameters of TiN coating

Sample Name	Working Pressure (mtor)	DC power	RF Power	Gas Flow Rate Argon (sccm)	Gas Flow Rate Nitrogen(sccm)
a	3	200	50	50	1
b	3	200	50	50	2
c	5	200	50	50	1

The surface and cross section morphology were observed by field emission scanning electron microscopy (FESEM). The chemical compositions of coatings were determined by energy dispersive spectroscopy (EDS). The micro hardness Vickers technic was used to determine hardness values of coatings. The hardness parameters were

245.3mN indentation load and 10S dwell time. The wear properties of coatings were evaluated by ball on disc tribometre. The wear experiment parameters are given in Table 2.

Table 2. Wear experiment parameters

Normal Load (N)	Sliding Speed (mm/s)	Cycle	Counter Body	Time (s)
2	60	2000	Al_2O_3	1047

The wear volumes of samples were measured with optical profilometer (Nanofocus, Germany). The worn surfaces were characterized with field emission scanning electron microscopy.

4. RESULTS AND DISCUSSION

4.1. Surface and Cross Section Analysis

The surface morphology of coatings was observed by scanning electron microscopy. Figure 2 shows the surface photos of TiN coatings.

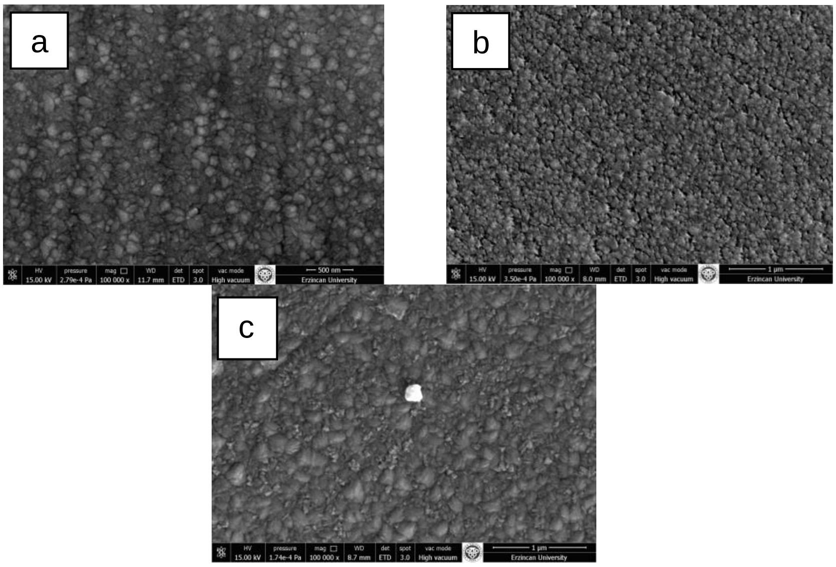

Figure 2. The surface morphology of coatings

In order to measure the thickness of TiN coatings, all samples were cut form uncoated side. And then fractured using hummer after keeping inside the nitrogen bath. The thicknesses of coatings are given in Table 3. The fractured cross sections were given in Figures 3

Table 3. The thicknesses of coatings

Sample	Coating Layer Thickness (nm)
a	998.36
b	960.2
c	941.6

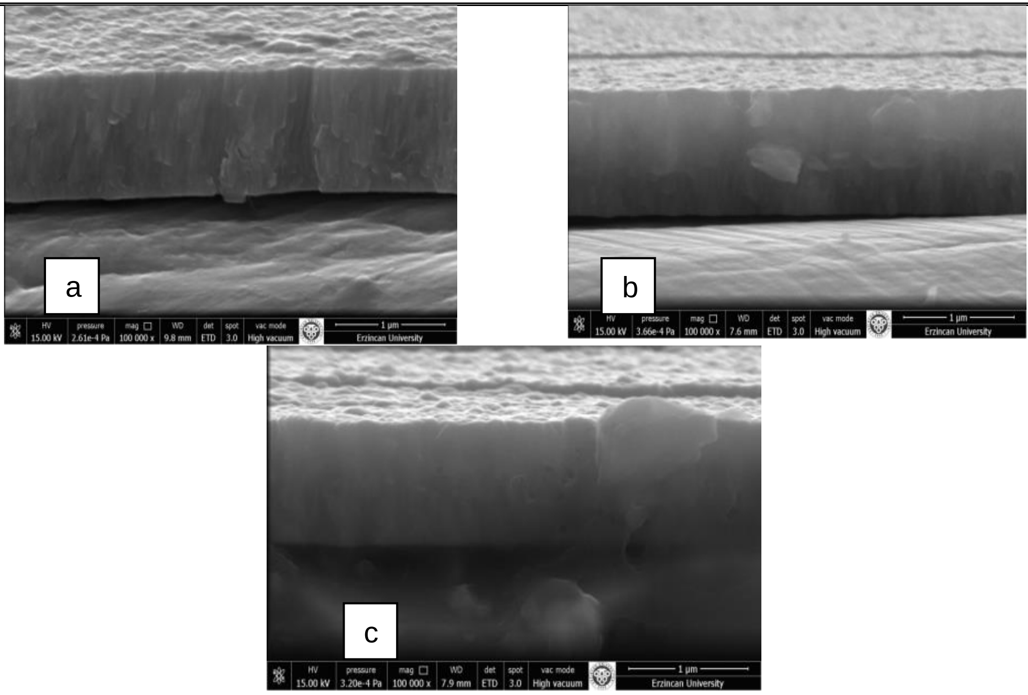

Figure 3. The cross-section photos of TiN coatings

All coatings showed columnar morphology as shown in Figure 3. The thicknesses of a, b and c coatings are 998.36nm, 960.2nm and 941.6nm respectively. For coatings a and b, the decrease in the thickness is related with the deposition parameters. As it can be seen in Table 1, b coating has higher N_2 flow rate ratio. For this reason, it can be expected that poisoning of the target surface is higher in this deposition conditions. And this resulted decreased in deposition rate. For this reason, thickness of sample b is lower than sample a. In the case of sample c, it has the highest working pressure. Therefore, sputtered target atoms are colliding with the ions inside the plasma. It gives result that lower number of atoms are reaching to substrate surface. This is decreasing deposition rate and thickness of coatings. The XRD graphs of TiN coatings deposited at different deposition parameters are given in Figure 4.

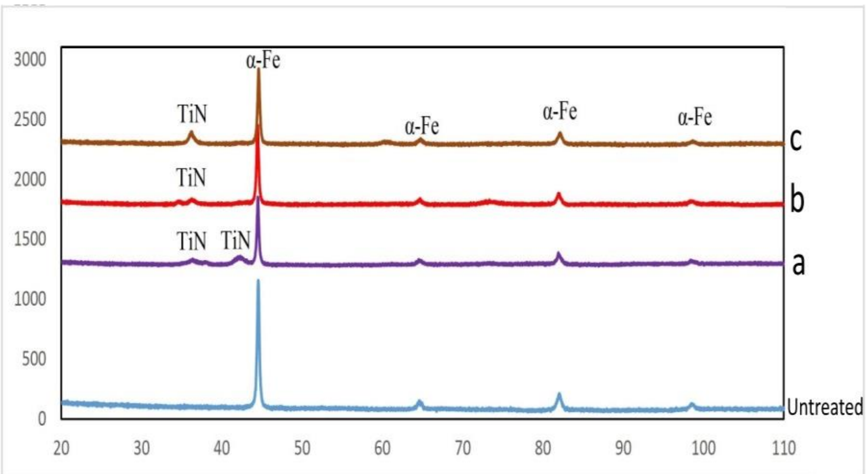

Figure 4. The XRD pattern of coated and uncoated samples

4.2. Hardness Properties

The hardness values of coatings were measured with micro hardness tester. The hardness results of coatings are given in Table 4.

Table 4. Hardness values of coating

	Hardness of Vickers ($HV_{245,3}$)
a	751.4
b	717.1
c	739.3

The highest hardness was attained in sample a with lowest working pressure. The highest hardness of Sample a is related with its dense structure. The lowest hardness was attained in Sample b with highest nitrogen flow rate.

4.3. Wear Properties

The wear properties of TiN coated AISI H13 steel were measured with ball on disc tribometre. The friction coefficients of coatings are given in Figure 5.

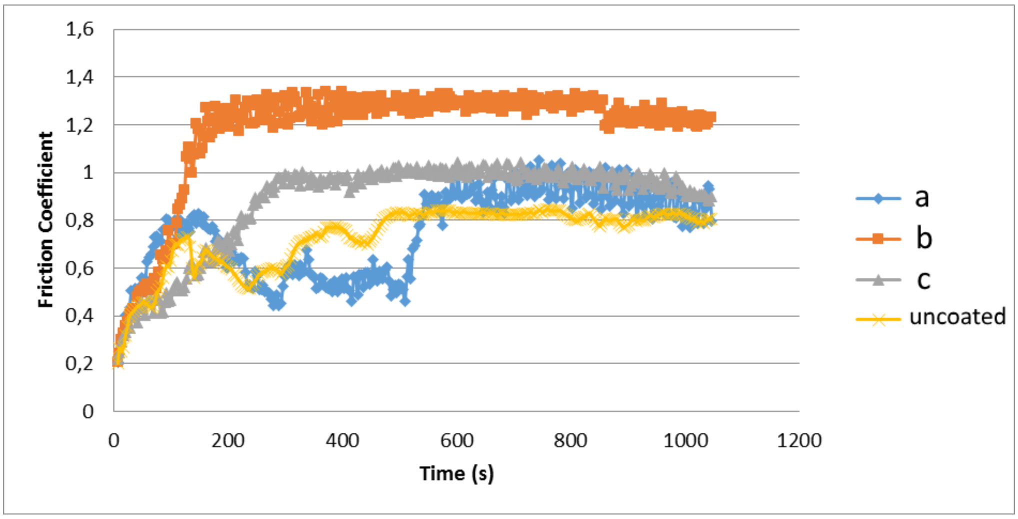

Figure 5. The friction coefficient of coatings

The friction coefficient figure of coatings consists of two sections. At early stage of experiments is running in stage (between zero and 600 seconds) and the rest is steady state stage. The wear amounts of samples were measure by optical profilometer. The wear amount values are given in Table 5. Figure 6 shows the optical profilometer surface photos of TiN coatings.

Table 5. The wear amount of TiN coated AISI H13 Steels

	Wear Volume (μm^3)
Uncoated H13 Steel	25.328256
a	1.059525
b	8.057339
c	3.377225

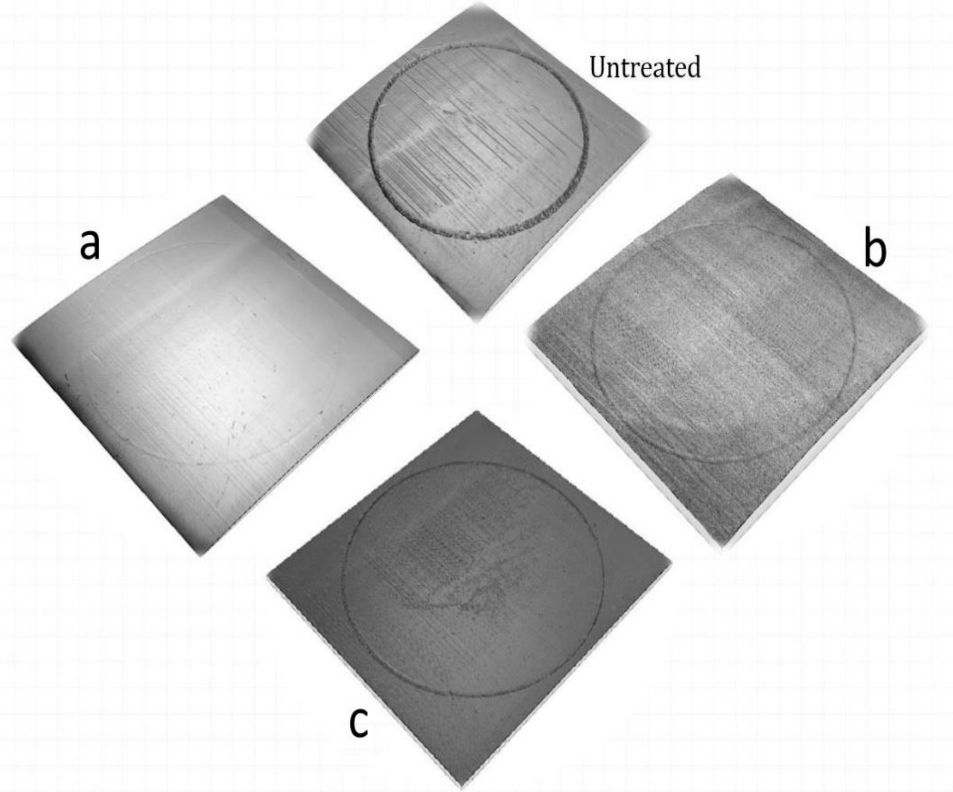

Figure 6. Optical profilometer photos of TiN

All coatings showed higher wear resistance than uncoated samples. Application coatings enhanced wear resistance properties of AISI H13 steel. The lowest wear amount was attained from Sample a. The highest wear amount was measured from Sample b. It can be seen in Table 5 that increasing RF power increased the wear resistance of TiN coating. Increasing RF bias voltage enhanced the density of film and reduce possible defects inside the coatings [1]. The SEM and EDS analysis photos of worn surfaces of samples are given in Figure 7.

Figure 7(a) shows the SEM image of sample a which consists of smooth surface and thin scratches. This is because of high value of hardness and small grain structure of this coating. Figure 7(b) shows severe abrasive wear surface for sample b. Coating was partly removed from substrate as shown in EDS photos. This can be related with lover hardness value of Sample b. Figure 7(c) shows deep grooves and delamination of coating from substrate for sample c. EDS analysis photos confirm the delamination of coatings. Overall abrasive wear was the main wear mechanism for all samples.

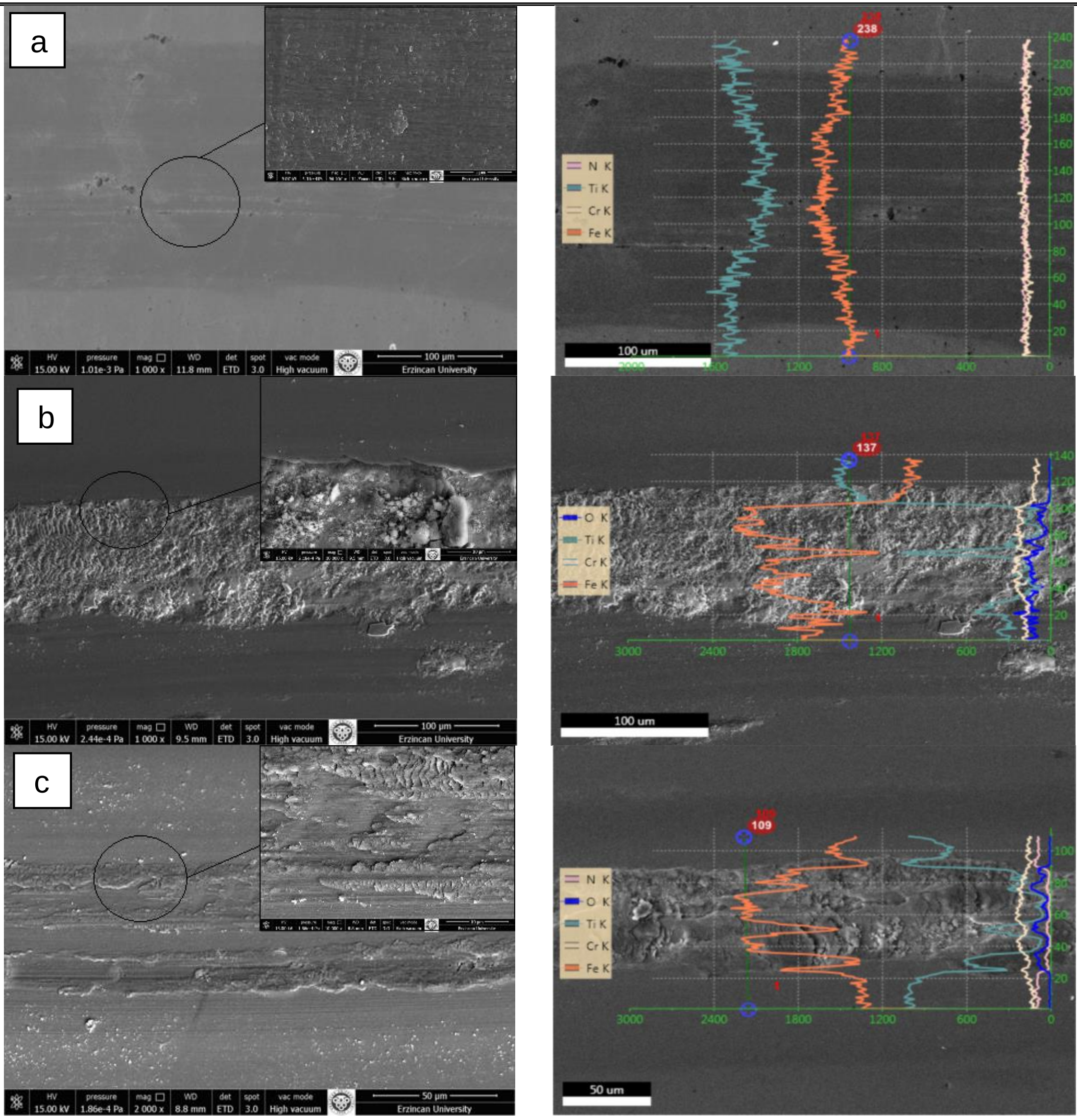

Figure 7. The worn surfaces of samples

5. CONCLUSIONS

In this study, The Effect of Working Pressure and Nitrogen Partial Pressure on the Mechanical and Wear Performance of TiN Coatings were investigated. Following conclusions were attained,

- The highest micro hardness was attained with 3 mtor working pressure, 200W DC power, 50W RF power and 1 sccm nitrogen flow rate
- Application of TiN coatings increased wear resistance of AISI H13 steel when compare with uncoated one.
- Highest wear resistance was attained with the lowest working pressure and nitrogen flow rate conditions
- There is correlation between hardness of coating and wear resistance. The sample which has highest hardness (sample a) also showed highest wear resistance. The samples which have lowest hardness (samples b) also showed lowest wear resistance.
- Abrasive wear is the main wear character for all coatings. Abrasive wear mechanism increased with decreasing hardness value.

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