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Tribological Characterization of TiN Coatings prepared by Sputtering

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Abstract

The aim of this paper is to study tribological properties of titanium nitride (TiN) coating deposited on various substrates by DC magnetron sputtering technique. The DC power of metallic titanium target was varied from 200 to 350 W at an increment of 50W. The effect of power variation on structure and surface morphology was characterized by X-ray diffraction (XRD) and field emission scanning electron microscopy (FE-SEM). The tribological properties of coating were investigated using pin on disk apparatus. TiN coating exhibited a higher wear resistance and low coefficient of friction compared to uncoated substrates of mild steel, aluminum and brass.

Key Word: TiN; DC magnetron sputtering; Tribology; Pin on disk

Nomenclature

\begin{tabular}{|l|l|}
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PVD & Physical Vapor Deposition \\
Pa & Pascal \\
sccm & Standard Cubic Centimetre per minute \\
\(\lambda\) & Wave Length \\
\(\mu m\) & Micro Meter \\
\(\phi\) & Diameter \\
COF & Coefficient of Friction \\
\hline
\end{tabular}
1. Introduction

Titanium nitride thin films deposited by PVD methods have been proven as an extremely useful coating on high Speed Steel (HSS) cutting tools, forming tools and as a hard wear and corrosion resistant decorative overcoat for watch cases, bracelets and eyeglass frames. Among many PVD methods, film deposition utilizing ion plating effect has many successful industrial applications. Most of these techniques operate with an evaporation source like an electron beam gun or a hollow cathode gun [1-4]. Another very successful approach of introducing evaporation methods can be seen in various types of arc sources which have been developed since the early 1980’s [5-7]. Various publications have described the advantages of TiN coatings produced by using these sources. The most consistent results exist in the evaluation of the performance of drills coated with TiN films [8,9].

Titanium nitride (TiN) films possessing excellent mechanical properties, low electrical resistivity (18-25 μΩ-cm, better than that of Ti, 40-50 μΩ-cm), high chemical and thermal stability, and interesting optical properties (colors varying from gold to dark brown) [10,11]. It has been applied in areas such as abrasion resistant coatings on tool steels, decorative coatings in architecture, diffusion barrier layers in semiconductor devices, and flat panel displays [12-14]. TiN coatings are widely used for the protection of tools. These coatings generally provide high hardness, low coefficient of friction, good corrosion, oxidation and wear resistance [15, 16]. In addition, the bright golden color of TiN makes it attractive for decoration applications [17]. A most recent application makes use of their biocompatibility and hemocompatibility [18,19] as they have been integrated as surface layers within orthopaedic prostheses and cardiac valves [20,21].

The studies on electrical and mechanical properties of TiN coatings are most common as observed from the literatures. So the objective of this research work is to study tribological properties of TiN coatings. TiN coatings are deposited on various substrates at various power values of 200W, 250W, 300W and 350W and its effect on structural and tribological properties is reported in this paper.

2. Experimental details

A 2” diameter Ti target (99.999% purity, 50.8mm × 6.5mm) was mechanically clamped to the magnetron cathode of the sputtering system. Titanium Nitride (TiN) coatings were deposited by using DC magnetron sputtering set-up (Excel Instruments, India) and inserting argon and nitrogen gases into the sputtering chamber. The flow of gases was kept constant and controlled by Mass Flow controller (MFC), (ALICAT instruments, USA). The substrates were ultrasonically cleaned for 15minutes using methanol and dried at room temperature prior to deposition. Substrates were mounted on the sample holder and a base pressure of 4×10⁻⁴Pa was obtained by a turbo molecular pump backed by a rotary pump. After sputter cleaning, argon and nitrogen gas flow rate was adjusted to 12sccm and 08sccm respectively and sputtering pressure of ~ 1.75Pa was maintained during deposition. In this way, films were obtained at four different DC power values of 200W, 250W, 300W and 350W represented by samples TiN200, TiN250, TiN300 and TiN350 respectively. The deposition conditions are summarized in Table 1.

<table>
<thead>
<tr>
<th>Process parameters and their corresponding values</th>
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<tr>
<td><strong>Target</strong></td>
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<tr>
<td><strong>Substrate</strong></td>
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<td><strong>Pin Materials and Diameter</strong></td>
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<td><strong>Target-Substrate Distance</strong></td>
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<tr>
<td><strong>Base pressure of Chamber</strong></td>
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<tr>
<td><strong>Working pressure</strong></td>
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<td><strong>D.C Power</strong></td>
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<td><strong>Gas ratio (Ar:N₂ in sccm)</strong></td>
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<td><strong>Substrate Temperature</strong></td>
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Crystallographic analyses of the deposited films were done by XRD (Model: Bruker D8 Advance X-ray diffractometer). The surface morphology of TiN coatings were analyzed using the Nova Nano FEG-SEM 450 field emission scanning electron microscopy (FE-SEM) configured with an EDAX Inc, US Analysis system.
Pin-on-disc tribometer (DUCOM) was used to study tribological properties of TiN coatings by conducting dry sliding wear tests. TiN coatings were deposited on pins of different materials such as a mild steel (MS, ϕ3mm), aluminium (Al, ϕ4mm) and brass (ϕ6mm) and were tested against a disc made of hardened ground steel (EN-32, hardness 72HRC, surface roughness Ra=0.07µm). The specimen was held stationary and the disc was rotated while a normal force was applied through a lever mechanism. During the test, frictional force was measured by the transducer mounted on the loading arm. The tests were conducted with sliding velocity (1.05m/s, 2.10m/s and 3.14 m/s) and normal load (10N, 15N and 20N). The present study examines the influence of applied load on the dry sliding wear of TiN-coated Al, MS and Brass pin against hardened ground steel disc, at a varied sliding speed and load.

3. Results and discussions

The XRD graphs of TiN coatings deposited at different DC power values of 200W, 250W, 300W and 350W is shown in Fig. 1. When the power is 200W the intensity of (111) peak is low for TiN coatings. With the increment of 50W power at value of 250W, intensity of (111) peak for TiN coating increases and evolution of (200) and (220) textures for TiN coatings is observed. When the power is 300W, maximum intensity of (111) peak for TiN coatings is observed and less intense (200) and (220) peaks are also present. When the power is 350W, only (111) peak of TiN having very low intensity is observed.

![XRD patterns of TiN coatings deposited at different DC Power](image)

Fig. 1: XRD patterns of TiN coatings deposited at different DC Power

Scherrer formula[22] was used to calculate the average crystallite size (D)of the samples as represented in equation (1): 

$$D = \frac{0.9\lambda}{\beta \cos \theta_B} \quad \ldots \ldots (1)$$

where \(\lambda\), \(\theta_B\) and \(\beta\) are the X-ray wavelength (1.54056Å), Bragg diffraction angle and full width at half maximum (FWHM) of the most dominant peak, respectively. The average crystallite size as calculated by Scherrer formula increases from 32nm to 55nm with increase in DC power from 200W to 300W. The (111) peak of TiN films becomes narrow and intense with increase in power, which results in larger grain size with increase of the power which is also reflected in SEM micrographs as shown in Fig. 2.

The surface topography of TiN coatings was investigated by scanning electron microscopy. The micrographs of TiN coatings deposited at different power 200W and 300W are depicted in Fig. 2. It was noticed that as the
proportion of power increases, surface morphology of TiN coatings changes and increase in average crystallite size is observed.

![SEM images of TiN coatings deposited at (a) 200W and (b) 300W](image)

**Fig. 2: SEM images of TiN coatings deposited at (a) 200W and (b) 300W**

Effect of power on tribological properties was examined using pin on disc tribometer under varying load and rpm conditions. Fig. 3 depicts wear and coefficient of friction (COF) of uncoated and coated aluminum pin by TiN coatings. From Fig. 3a it is clearly observed that TiN300 sample had high wear rate compared to TiN200 at different values of rpm (250rpm,500rpm,750 rpm) and normal load (10N, 15N, 20N). The COF values for TiN200 sample was lower as compared to TiN300 sample as shown in Fig. 3b.

![Graph showing effect of power on uncoated and coated TiN aluminum pins on (a) wear (b) COF](image)

**Fig. 3: Effect of power on uncoated and coated TiN aluminum pins on (a) wear (b) COF**

![Graph showing effect of power on uncoated and coated TiN brass pins on (a) wear (b) COF](image)

**Fig. 4: Effect of power on uncoated and coated TiN brass pins on (a) wear (b) COF**

The effect of power on uncoated and coated TiN brass pins on wear and COF are shown in Fig. 4a and 4b respectively. It was observed that TiN300 sample had maximum wear rate at rpm value of 250rpm and normal load value of 20N and minimum values at rpm value of 500rpm and normal load value of 15N. The maximum wear rate for TiN200 sample was observed at rpm value of 750rpm and normal load value of 10N load whereas for other conditions it had negligible difference.
The COF values for TiN200 and TiN300 samples as shown in Fig. 4b at different conditions exhibited similar trend between their values at a specific condition. The maximum COF for TiN300 sample was observed at at rpm value of 250rpm and normal load value of 20N and minimum values at rpm value of 500rpm and normal load value of 15N. The maximum COF values for TiN200 sample was observed at rpm value of 750rpm and normal load value of 10N load whereas for other conditions it had insignificant variance.

![Fig. 5: Effect of power on uncoated and coated TiN mild steel (MS) pins on (a) wear and (b) COF](image)

Wear and COF values for uncoated and coated mild steel (MS) pin with TiN coating are shown in Fig. 5a and 5b respectively. The wear rate for TiN300 sample shows almost similar values for all conditions. TiN200 sample shows minimum values at rpm value of 750rpm and normal load value of 10N. The maximum COF for TiN300 sample was observed at rpm value of 750rpm and normal load value of 10N and minimum values at rpm value of 500rpm and normal load value of 15N. TiN200 sample shows slight variation in their respective COF values at different conditions giving minimum values at rpm value of 750rpm and normal load value of 10N.

4. Conclusion

The effects of power on structural and tribological properties of TiN coatings produced by DC magnetron sputtering have been investigated. XRD graphs indicate that deposited TiN coatings are crystalline till power value of 300W exhibiting various textures of TiN and gets converted into amorphous structure at power of 350W. TiN coatings have high wear resistance and low COF compared to uncoated pins of mild steel, aluminium and brass. TiN coatings deposited at 200W power shows minimum value of wear and COF.

5. Acknowledgement

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6. References