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Role of substrate and deposition conditions on the texture evolution of titanium nitride thin film on bare and plasma-nitrided high-speed steel

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Abstract

Titanium nitride (TiN) films are prepared by direct current (DC) magnetron sputtering of a titanium (Ti) target in an argon (Ar) + nitrogen (N_2) atmosphere on bare and pulsed DC plasma-nitrided high-speed steel (AISI M2) substrate. One set of coating is deposited using different N_2 partial pressures keeping the working pressure fixed, and it leads to variable film thickness. Another set of coating of equal thickness is prepared at the same working pressure with variable substrate biasing voltage. The detailed structure of the coatings is analyzed by X-ray diffraction study. The texture coefficient (T_c) determined from X-ray diffraction studies at various deposition conditions gives information about crystallographic orientation. It is found that the same deposition condition leads to different orientation of TiN coatings on bare AISI M2 and plasma-nitrided AISI M2 substrate. The results are discussed in terms of energy minimization and substrate-induced preferred orientation.

Keywords: Magnetron sputtering; Argon nitrogen plasma; X-ray diffraction study; Texture coefficient

Introduction

Titanium nitride (TiN) has been widely used as a coating for tool steels since the last few decades. TiN coating on a cutting tool increases the tool life due to its high hardness and adhesion, good ductility, excellent lubricity, high chemical stability, and tough resistance to wear, corrosion, and temperature [1]. TiN coating by magnetron sputtering has developed rapidly over the last decade in such a way that it has become an established process of choice for the deposition of a wide range of industrially important coatings. Due to their unsuitability for use with many substrate materials such as low alloy steel and titanium alloys and lack of load-bearing support provided by the substrate, these coatings have certain limitations. To address these problems and to extend the commercial viability of advanced PVD processes, duplex processes have been developed [2]. Physical vapor deposition treatment of pre-nitrided steel is a good example of a duplex process. Components treated in this way exhibit low wear characteristics of the ceramic coating, combined with the high load-bearing capacity and high fatigue strength characteristics of the nitrided layer. Also, it has been reported by Gredic et al. that the plasma nitriding prior to coating deposition strongly improves the adhesion properties of the hard coating [3]. It is important to note that most of the TiN coatings are reported to exhibit a preferential crystallographic orientation [4-6] which affects the mechanical behavior of the coatings. The preferential orientation depends on various process parameters and on the nature of the substrate surface [7].

Although the effects of some major process parameters to determine the preferred orientation of TiN thin film are available in the literature, at present, not much of systematically examined effects of process parameters on TiN texture on plasma-nitrided high-speed steel (AISI M2) are available. This paper reports thoroughly on the difference in preferred orientation observed in direct current (DC)-sputtered TiN coatings deposited under various conditions onto bare AISI M2 and plasma-nitrided AISI M2 substrate. Process parameters like bias voltage and nitrogen partial pressure are considered. In this study, AISI M2 high-speed steel (HSS) (0.86% C, 6.0% W, 5.0% Mo, 4.1% Cr, 1.9% V, and 0.5% Co, in wt.%) is selected as the substrate material because it is mostly used as a cutting tool material.

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Experimental methods

Disc-shaped samples of 20 mm in diameter and 4 mm in thickness, made from AISI M2 HSS, were prepared to mirror polish finish by standard metallographic method and cleaned with acetone before processing. Plasma nitriding process and TiN coating deposition were done in separate systems. The schematic diagram of the plasma nitriding system used in this study is given elsewhere [8]. Initially, the samples were placed on the sample holder which was negatively biased. The vacuum chamber was evacuated to 2 Pa. The glow discharge was generated using a DC pulsed power supply with a gas mixture composition of 80% N_2-20% H_2. The working pressure of 500 Pa and a voltage of 550 V were maintained during the process. The samples were kept at a temperature of 500°C with an accuracy of ±10°C, which was read on a temperature indicator using a J-type thermocouple. After 24 h, AISI M2 samples were cooled with nitrogen gas and latter taken out from the chamber when the samples attained room temperature. Figure 1 shows the schematic diagram of a homemade magnetron sputtering system for the deposition of titanium nitride. The discharge chamber was evacuated by a diffusion pump supported by a rotary pump, and the base pressure was maintained below 2×10^{-3} Pa. Argon gas was used for the plasma ignition, and the operating pressure was held at 3×10^{-1} Pa. The water-cooled titanium target with a diameter of 70 mm was made as cathode of the magnetron sputtering system. The stainless steel chamber with a diameter of 45 cm and length of 50 cm was made as anode in the steady state. The magnetic field strength at the centre of the cathode reached 600 G. In this study, one set of TiN coatings of thickness 2.5 μm was produced as a function of biasing voltage (0 to −100 V) keeping N_2 partial pressure of 3×10^{-2} Pa at a substrate temperature of 200°C. Another set of TiN coatings of thickness (2.90 to 1.87 μm) was produced as a function of N_2 partial pressure (1×10^{-2} to 8×10^{-2} Pa). The working pressure at 3×10^{-1} Pa and input power of 300 W were fixed during the deposition of both sets of samples for 45 min. To clean the substrate surface prior to the deposition and improve film's adhesion, the bare and plasma-nitrided substrates were pre-treated by Ar plasma in a sputtering chamber for 10 min with a bipolar power supply bias of −300 V.

Phase composition of all the untreated and treated samples was studied by X-ray diffraction (XRD) using a XRD3000PTS diffractometer (GE Inspection Technologies GmbH, Ahrensburg, Germany) with the CuKα radiation (λ = 1.5406 Å) in the Bragg-Brentano configuration operated at 40 keV and 50 mA. XRD patterns were recorded with step durations of 4 s at each step in the angular range of 30° to 90°. Information regarding the preferred orientation of the different planes was obtained by measuring the intensities of the diffraction peaks corresponding to the various planes. The texture coefficient T_c is defined as [9]

$$T_{\mathrm{c}} = [nI_m(hkl)/I_0(hkl)]/\sum(I_m(hkl)/I_0(hkl)) \qquad (1)$$

where $I_m(hkl)$ is the measured intensity of reflection from a given (hkl) plane and $I_0(hkl)$ is the relative intensity of reflection from the same plane as indicated in a standard sample. For random distribution, $T_c = 1$. The thickness of the deposited films was determined by properly calibrated quartz crystal thickness monitor model no. DTM-101 provided by Hind HiVac, Bangalore, India. The results were counterchecked by SEM under

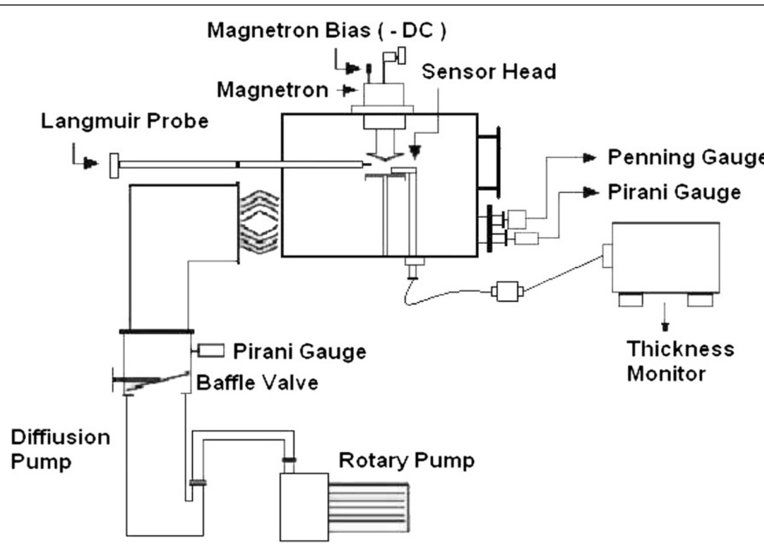

Figure 1 Schematic diagram of the experimental device.

different conditions of deposition on Si(111) wafer. The obtained thickness values from SEM and measured values from quartz thickness monitor show a discrepancy of 5%. A single cylindrical probe with a length of 3.0 mm and a diameter of 0.1 mm was installed in the discharge chamber. The probe measurement was carried out at the discharge center in the downstream region of 70 mm from the cathode, i.e., 10 mm from the substrate. Plasma parameters such as electron temperature (T_e in eV), electron density (N_e), and ion density (N_i) were measured with the probe in practically magnetic field-free region. We have measured the Langmuir probe measurement near the substrate plane, keeping in mind of the fact that the electron and ion density in the vicinity of the substrate can significantly affect the physical properties of deposited film.

Results and discussion

Study of plasma parameters as a function of nitrogen partial pressure and substrate biasing voltage

A study of basic plasma parameters such as electron density (N_e), electron temperature (T_e), and ion density (N_i) is essential to understand the effect of different plasma parameters on the texture growth of TiN thin film. A single Langmuir probe was used to calculate T_e and N_e at various deposition parameters. Figure 2 shows the evaluation of T_e and N_e as a function of nitrogen partial pressure and substrate biasing condition. As seen from Figure 2a with the increase in N_2 partial pressure (P_{N_2}), the electron density value drops gradually from 9.4×10^{14} to 5.93×10^{14} m^{-3}. With the increase in substrate biasing voltage from 0 to −100 V, the electron density value changes from 7.70×10^{14} to 9.80×10^{14} m^{-3} (Figure 2b). Since in the ohmic heating regime there exists an inverse relation [10] between N_e and T_e, the gradual increase of T_e upon nitrogen addition is observed. The total ion density is expected to follow the similar behavior as that of electron density if one refers to the quasi-neutrality property of plasma. Since Ar/N_2 plasma used in this work contains various ion species (Ar^+, N^+, N_2^+, N_3^+, etc.) as well as Ti ions, they all contribute toward total ion density evaluation. Increasing N_2 partial pressure at constant working pressure leads to the increase of nitrogenic ion (N^+, N_2^+, N_3^+) and lowers titanium and argon ion flux [11]. From the Langmuir probe *I-V* characteristics, the mean ion mass at each discharge condition is calculated, and using the values of mean ion mass [12], the contribution of Ar^+ ions and nitrogenic ions in the discharge is presented in Figure 3. It is clear from the figure that, with the

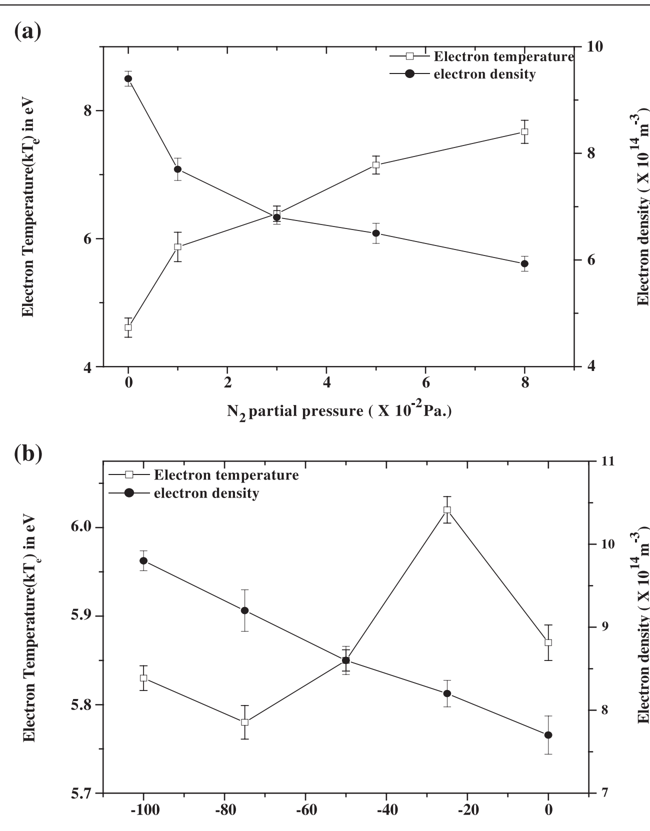

Figure 2 Variation of N_e and T_e with (a) N_2 partial pressure and (b) substrate biasing voltage.

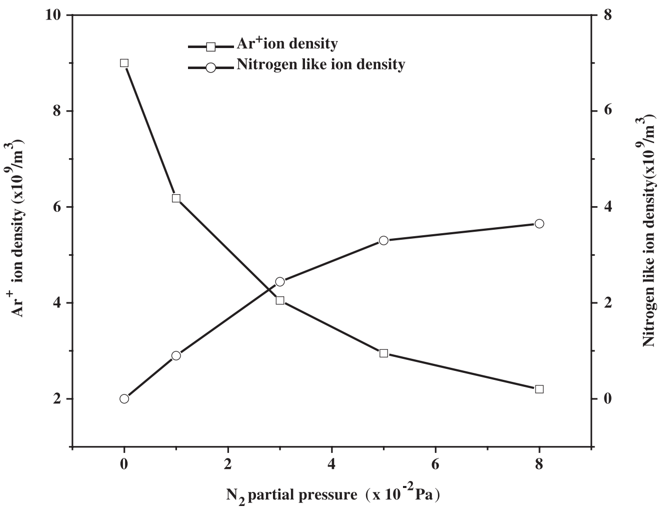

Figure 3 Effect of nitrogen partial pressure on density of Ar^+ and nitrogen-like ions.

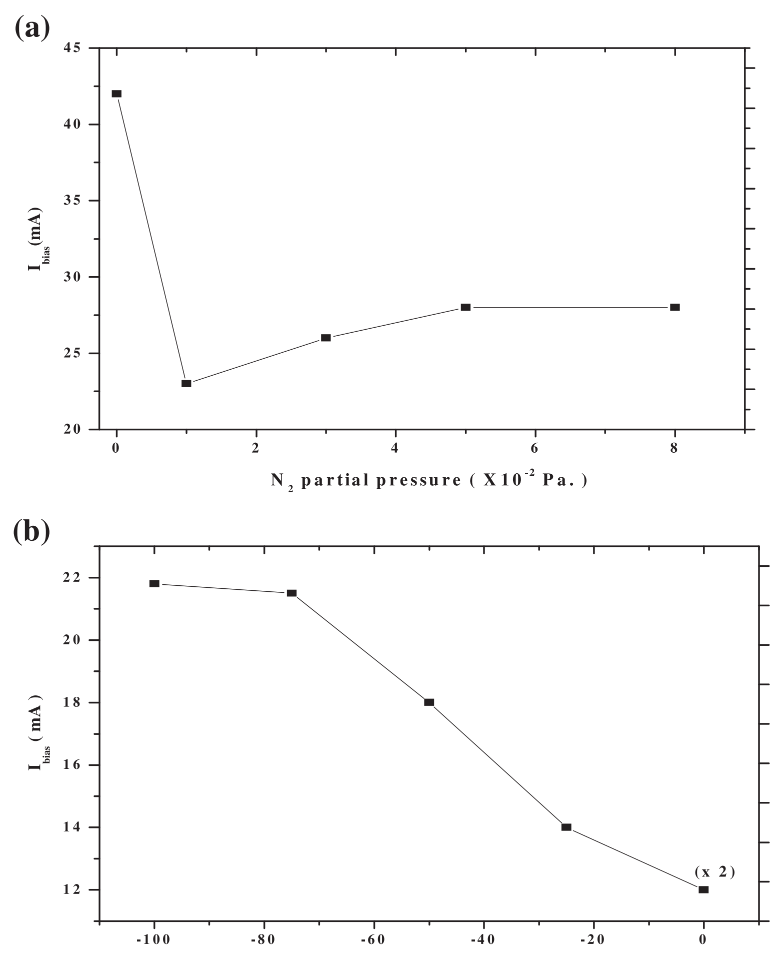

Figure 4 I_{bias} as a function of (a) N_2 partial pressure and (b) substrate biasing voltage.

addition of nitrogen in argon plasma, a gradual decrease of Ar^+ ion and corresponding increase of nitrogenic ions are observed. Also, the fact of addition of N_2 at constant working pressure resulting lower neutral argon density aids to this observation. The substrate current which is nothing but the ion current flowing to the substrate can give a qualitative picture if not quantitative of the different kinds of ionization processes happening inside the plasma. It is interesting to find that the magnitude of discharge current (which is the sum of the electron current and ion current flowing to the target of the magnetron) also decreases with the addition of nitrogen in the discharge. Thus, with the introduction of nitrogen, there is a reduction of ion density in the discharge, and as such, a fall of substrate current is observed. With further addition of nitrogen in the discharge, a slight variation of substrate current is found (Figure 4a). During the TiN deposition, nitrogen, argon, and Ti ions simultaneously bombard growing TiN films. The sheath electric field accelerating the ions to the substrate [13] scales linearly with increasing substrate biasing voltage. As such, the gradual increase of substrate biasing current as a function of substrate biasing voltage is observed (Figure 4b), and it indeed signify enhanced degree of ion bombardment during TiN growth.

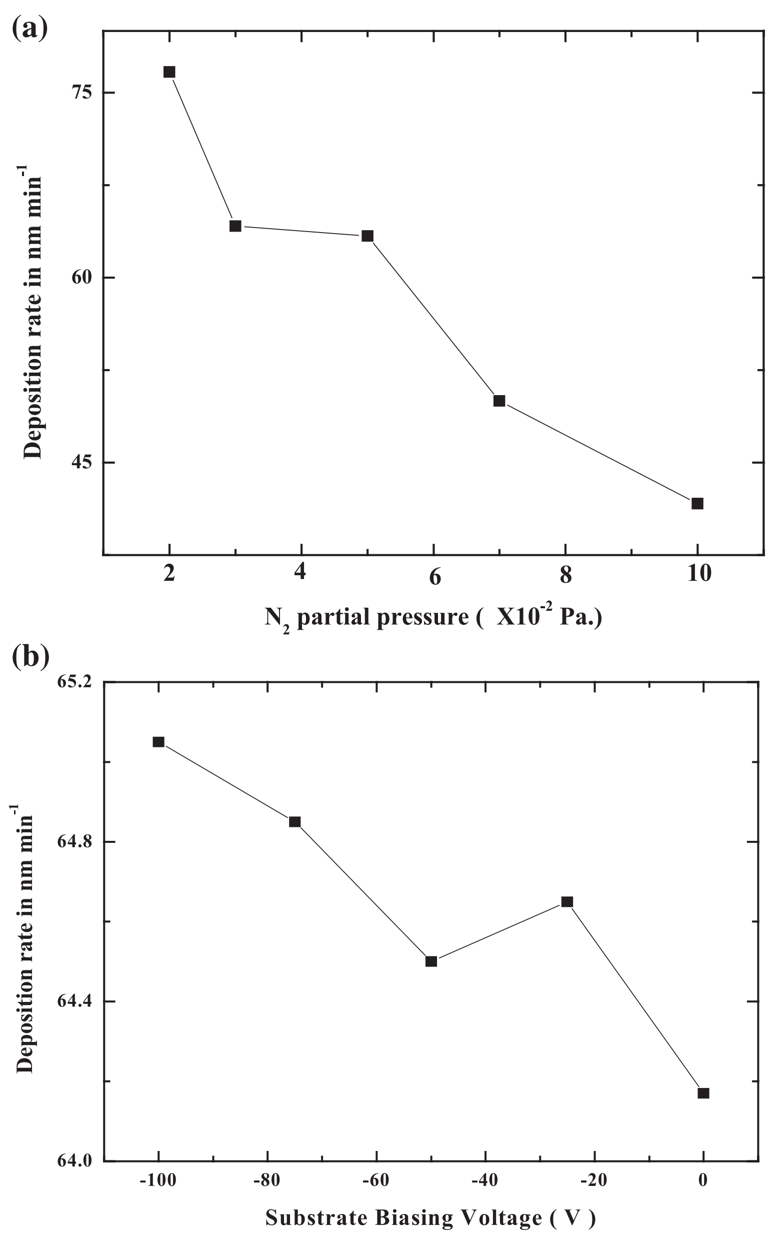

Figure 5 Deposition rate as a function of (a) N_2 partial pressure and (b) substrate biasing voltage.

Effect of process parameters on TiN deposition rate

Figure 5 shows sputtering rate as a function of both nitrogen partial pressure and substrate biasing voltage. The deposition rate decreases with increasing nitrogen content in the discharge (Figure 5a). As the deposition rate is closely related to the number of Ti species which arrives at the substrate surface, lower deposition rate represents that fewer Ti reach the substrate The sputtering yield of titanium to Ar^+ ions is greater than any nitrogenic ions. As with increasing nitrogen partial pressure, the density of Ar^+ ions decreases, as such decrease in TiN deposition rate is observed in this investigation. From Figure 5b, it is clear that the modulation of sputtering rate on substrate biasing voltage is not as strong as its modulation on N_2 partial pressure. Increasing the substrate biasing voltage only slightly increases the sputtering rate from 64.17 to 65.05 nm min^{-1}. The increase of sheath electric field accelerating ions to the substrate is responsible for the observed improvement in deposition rate.

XRD studies of the deposited thin films

The phase composition and texture in polycrystalline TiN thin film deposited onto bare and plasma-nitrided AISI M2 high-speed steel is analyzed by X-ray diffractometer. Figure 6 shows X-ray spectra for TiN coating on (a) bare AISI M2 sample and (b) plasma-nitrided AISI M2 substrate at different nitrogen partial pressures. The coatings deposited onto the abovementioned substrates show the presence of (111), (200), and (220) TiN diffraction peaks and α Fe(110) peak on bare sample. All iron nitride phases like ε $Fe_{2\text{-}3}$ N and γ′ Fe_4N can be observed

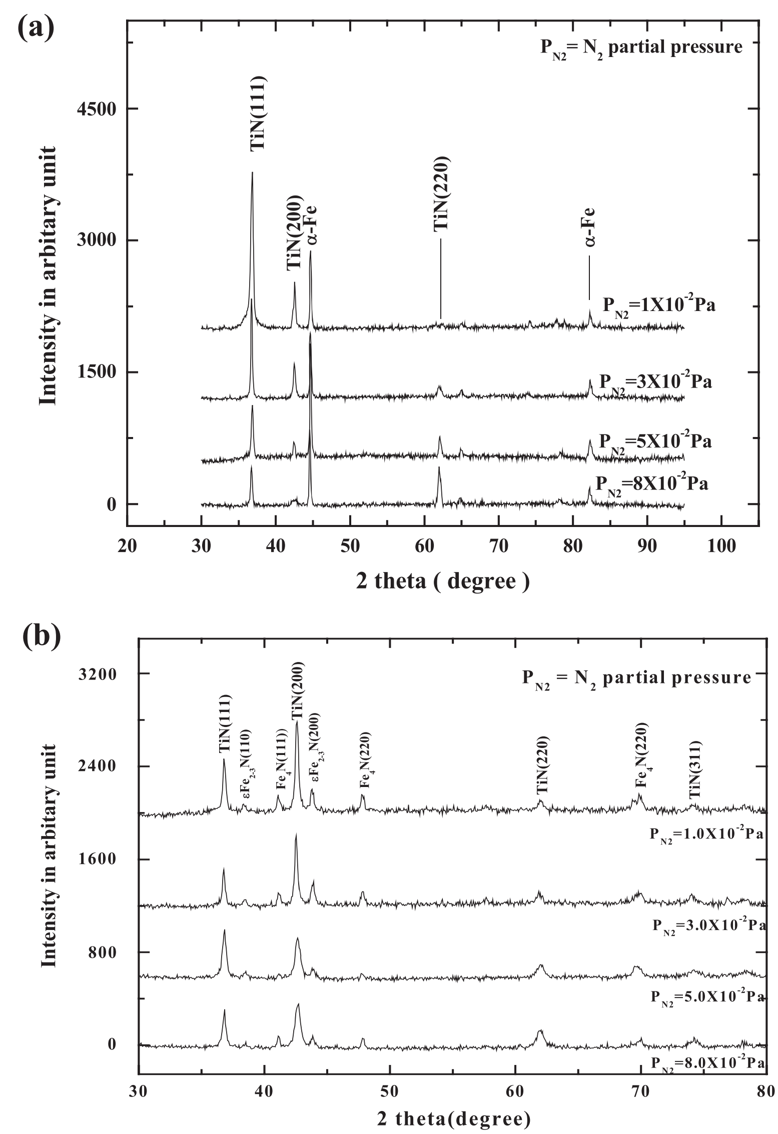

Figure 6 Variation of XRD of TiN with nitrogen partial pressure. (a) Bare substrate and **(b)** plasma-nitrided substrate.

even after deposition of TiN coating onto plasma-nitrided substrate. It is clear from Figure 6 that the orientation of TiN is clearly influenced by deposition and substrate conditions. On bare samples, a gradual decrease of TiN (111) and TiN (200) peak intensity and corresponding increase in TiN (220) peak intensity as a function of increasing nitrogen partial pressure is observed. On plasma-nitrided samples, the relative intensity of diffraction peak is different than bare samples, showing a gradual increase of intensity of TiN (220) and TiN (200) peaks. The X-ray diffractrograms of TiN films deposited at 3×10^{-1} Pa on bare and plasma-nitrided AISI M2 high-speed steel substrates at varying DC bias voltages (0 to −100 V) are also studied in the present investigation (Figure 7). At 0-V substrate biasing voltage, (111), (200), and (220) TiN peaks are present on bare substrate. With the application of negative bias voltage, the intensity of those peaks changes. When biasing voltage reached −50 V, (200) and (220) intensity becomes minimal, and film orientation changes to (111) completely. On the other hand, on plasma-nitrided substrate, a gradual increase of TiN (200) peak intensity compared to TiN (111) peak is observed with increasing substrate biasing voltage. From the observed intensity of diffraction planes, it is possible to calculate the texture coefficients (T_c) of a particular diffraction plane using Equation 1. The value of T_c conveniently determines the degree of preferred orientation or texture of diffraction planes. In Figure 8, the texture coefficients (T_c) for various TiN diffraction peaks are plotted as a function of N_2 partial pressure. On bare substrate, a preferred orientation of (111) is

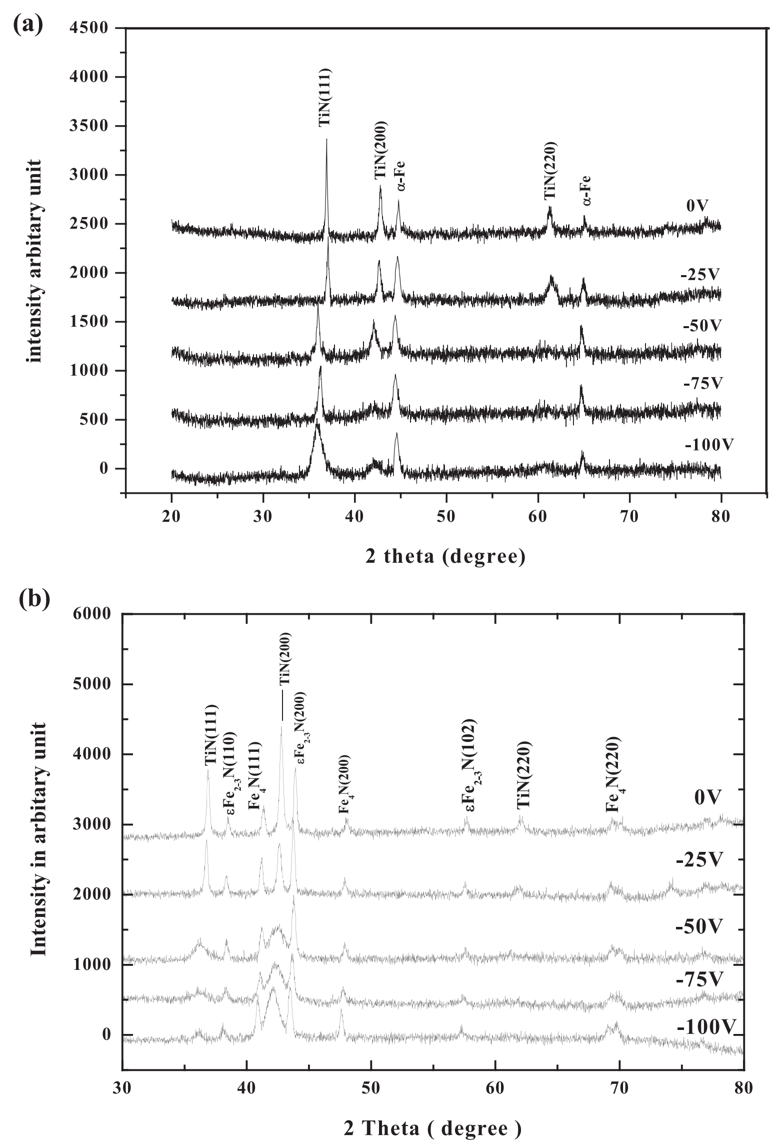

Figure 7 Variation of XRD of TiN with substrate biasing voltage. **(a)** Bare substrate and **(b)** plasma-nitrided substrate.

observed up to thickness 2.15 μm (corresponds to N_2 partial pressure 5×10^{-2} Pa) and changes to (220) orientation above this value. However, texture growth is different in the case of plasma-nitrided high-speed steel. Here, the (200) preferred orientation is observed up to thickness 2.8 μm (corresponds to 3.0×10^{-2} Pa N_2 partial pressure) and changes to (111) preferred orientation above this range. Evaluation of T_c for TiN diffraction peaks deposited at various substrate biasing conditions is shown in Figure 9. It is seen that with increasing biasing voltage, the texture coefficient of TiN (111) peak increases for bare substrate. On the other hand after −25 V, the texture coefficient of TiN (200) peak increases while a gradual decrease is observed for TiN (111) peak for plasma-nitrided AISI M2 substrate.

Effect of deposition parameters on the texture growth of TiN films

The preferred orientation of the films can be explained in terms of the energy W_{hkl} which is the sum of the surface energy (S_{hkl}) and the strain energy (U_{hkl}). Pelleg et al. [14] observed critical thickness above which strain energy is the driving force in determining the most preferred orientation of TiN film. Since all our deposits have thickness above the critical limit, hence strain energy is responsible in determining the preferred orientation. It should be noted that the surface energy is independent of film thickness, whereas the strain energy increases linearly with the thickness of the film [15]. A (111) preferred orientation is the result of relieving

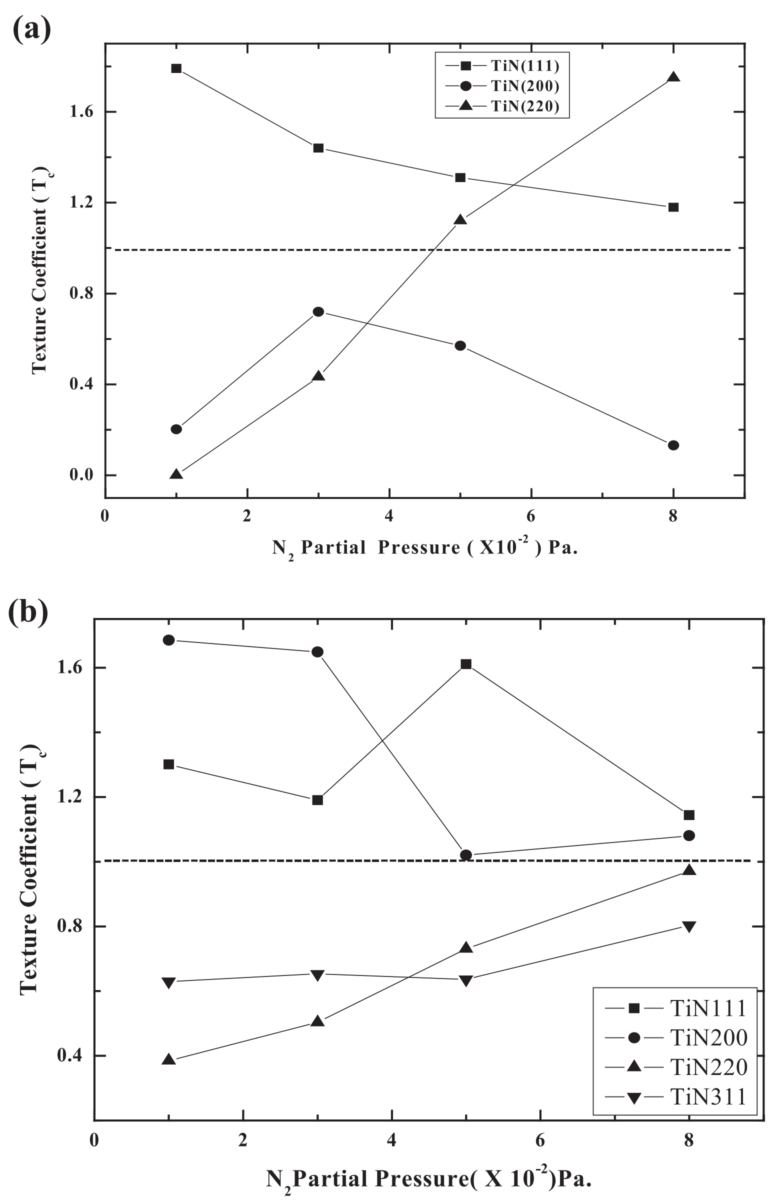

Figure 8 Variation of T_c of TiN planes with nitrogen partial pressure. (a) Bare substrate and **(b)** plasma-nitrided substrate.

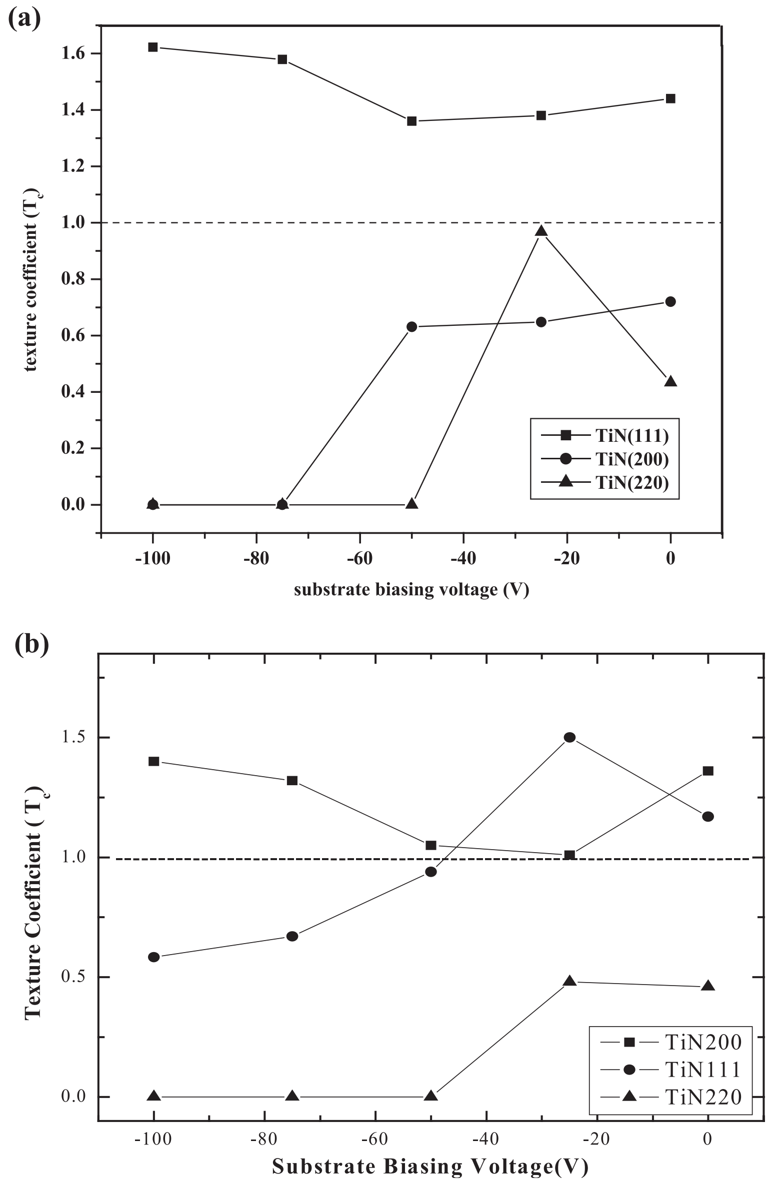

Figure 9 Variation of T_c of TiN planes with substrate biasing voltage. **(a)** Bare substrate and **(b)** plasma-nitrided substrate.

strain energy [16]. The thickness of deposited TiN coatings decreases at higher partial pressure of nitrogen. The decrease of coating thickness results in lowering of strain accumulation and hence decrease of TiN (111) texture coefficient, and corresponding increase of TiN (220) texture coefficient is observed (Figure 8a) on bare AISI M2 substrate. In comparison with other studies, where the growth of TiN coatings at low nitrogen pressure generally exhibited a strong (111) orientation while it changes to (200) direction with increasing fraction of nitrogen in the discharge [17-19], however, the thickness effect is found to be more pronounced in determining TiN texture growth on bare substrate in our study. Increasing substrate biasing voltage leads to higher degree of ion bombardment on

Table 1 Inter-atomic spacing between TiN planes and γ′ Fe_4N planes

Phases	γ′ Fe_4N	TiN
	d (nm)	*d* (nm)
(111)	*0.2191*	0.2440
(200)	0.1901	*0.2141*
(220)	0.1351	0.1487

Note the similarity of calculated values (italics) for inter-planar distances of γ′ Fe_4N (111) plane and TiN (200) plane.

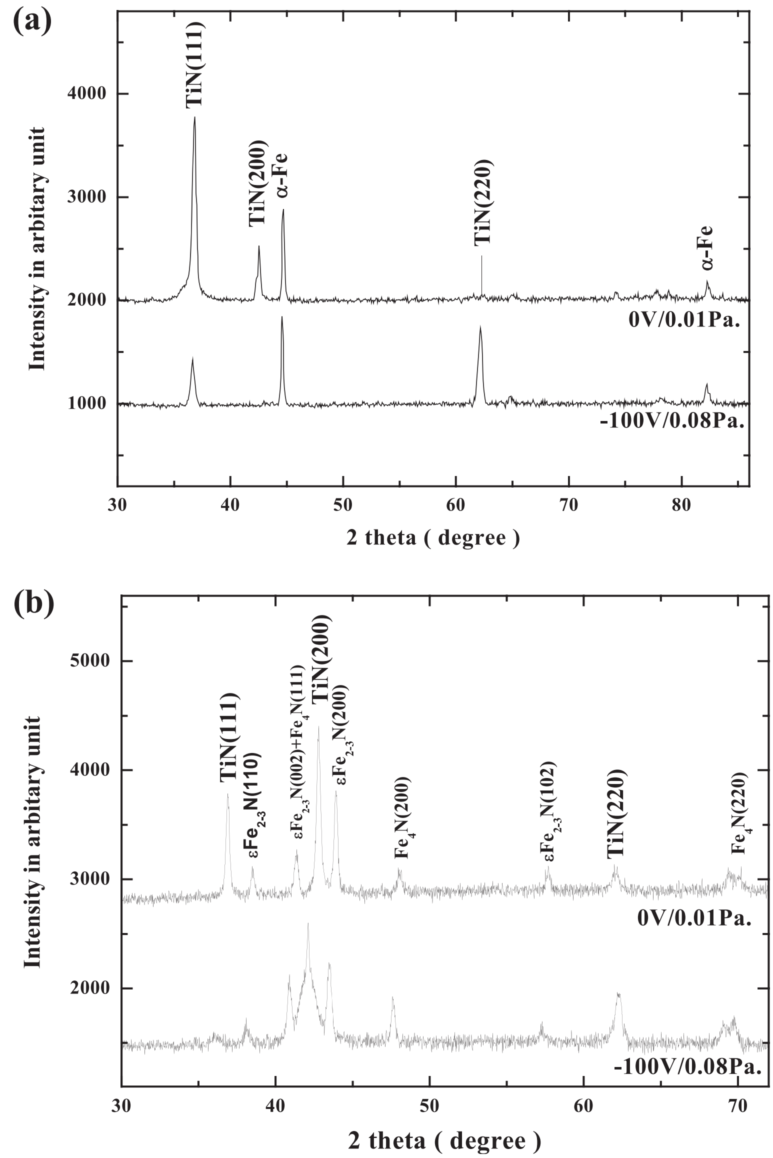

Figure 10 Variation of XRD of TiN. (a) Bare substrate and **(b)** plasma-nitrided substrate at extreme ion bombardment condition.

growing TiN thin film. The surface bombardment of gradually energetic particles increases the strain accumulation in films as a function of biasing voltage. In that case, (111) preferred orientation is expected. At comparatively large substrate biasing voltage, a change of TiN texture from (111) to (200) orientation corresponding to that of the lowest surface free energy [20-22] is reported in the literature. In our study, it is observed that the degree of preferred orientation of TiN (111) diffraction plane on bare substrate increases with increasing substrate biasing voltage (Figure 9a) within the range considered. However, the same deposition conditions lead to different preferred orientations of TiN diffraction planes on plasma-nitrided substrate. A (200) orientation is particularly preferred at lower nitrogen partial pressures and almost all of the

Table 2 Texture coefficient and preferred orientation of TiN planes

Substrate type	Deposition condition	Value of T_c for TiN planes			Preferred orientation
		(111)	(200)	(220)	
Bare substrate	0 V/0.01 Pa	1.79	0.203	0	(111)
	−100 V/0.08 Pa	0.532	0	1.460	(220)
Plasma-nitrided substrate	0 V/0.01 Pa	1.17	1.36	0.46	(200)
	−100 V/0.08 Pa	0.21	1.59	0.49	(200)

substrate biasing conditions. Thus, minimization of strain energy cannot explain the texture of TiN films on plasma-nitrided substrate, and as such, we have to look for the substrate-induced texture growth in this case.

Effect of substrate on the texture growth of TiN films

Very few reports in the literature are available discussing the contribution of substrate surface structure [23] on the preferential growth of TiN thin film. It can be seen from the XRD analysis that near-surface crystallography affects the orientation of TiN film subsequently grown on them. The XRD analysis of the plasma-nitrided samples shows the presence of γ′ Fe_4N and ε $Fe_{2\text{-}3}$ N diffraction peak leading to the formation of compound layer on the surface of the substrate. The inter-atomic spacings of γ′ Fe_4N planes of plasma-nitrided substrate are found to be very similar to TiN diffraction planes. Earlier studies [3,24-26] of TiN growth on plasma-nitrided (less nitrogen-more hydrogen containing DC pulsed nitriding plasma) substrate have reported the preferred orientation of TiN (220) plane. However, in order to explain the preferential growth of TiN (200) peak on plasma-nitrided substrate in our study, we have compared the inter-planar distances (d) of γ′ Fe_4N planes of plasma-nitrided surface with TiN diffraction planes on bare substrate and are listed in Table 1. Both TiN (200) and γ′ Fe_4N (111) planes are closed pack. Also, from Table 1, it is clear that the inter-atomic spacing between TiN (200) planes and γ′ Fe_4N (111) planes are very similar, i.e., an atomic mismatch of only 0.23%. Thus, interfacial strain at the substrate (111)/coating (200) interface would be very low [27]. This factor may be responsible for higher degree of preferred orientation of TiN (200) plane on plasma-nitrided substrate at lower nitrogen partial pressure (Figure 8b) and higher substrate biasing voltages (Figure 9b). Since different iron nitride phases are present on plasma-nitrided surface, additional investigations of the coating/substrate interface using HR SEM and TEM are indeed needed to confirm the mentioned fact which is beyond the scope of the present study.

TiN coatings at selected deposition conditions

From Figure 4, it is clear that increasing nitrogen partial pressure and substrate biasing voltage leads to higher degree of ion bombardment during deposition. Bombardment at high nitrogen partial pressure $P_{\mathrm{N}_2} = 0.08$ Pa and high biasing voltage $V_{\mathrm{b}} = -100$ V; −100 V/0.08 Pa is the strongest, while 0 V/0.01 Pa is the weakest. A comparison of XRD pattern of TiN film of equal thickness (2.5 μm) deposited at these two extreme conditions is done and is given in Figure 10.

Table 2 illustrates the combined effect of deposition parameters on TiN texture. The preferred orientations of TiN film changes from (111) to (220) with increasing ion bombardment for bare substrate. It is due to the fact that under a sufficiently high degree of ion bombardment, planes with more open direction will survive [28], and orientation results with this direction. As (220) direction is more open than (111) direction, preferred orientation is observed in this direction. However, on plasma-nitrided high-speed steel, TiN has (200) preferred orientation regardless of bombardment condition. A decrease of (111) texture coefficient and corresponding increase in (220) are observed in this case.

Conclusions

The present investigation reports the growth of TiN texture on bare and plasma-nitrided AISI M2 high-speed steel. While there are many reports available in the literature regarding the growth of TiN as a function of deposition parameters on Si and bare substrate, very few investigations are available regarding the effect of substrate such as plasma nitrided on the growth of TiN texture. In this study, the occurrence of TiN (220) plane at high nitrogen partial pressure condition is attributed to lowering of strain energy with film thickness while (111) plane preferentially grows at lower nitrogen pressure on bare AISI M2 substrate. As strain energy is a dominating factor in determining TiN growth in this study, a gradual increase of (111) texture coefficient with increasing substrate biasing voltage is observed on bare AISI M2 substrate. The substrate-induced texture growth of TiN is clearly visible on plasma-nitrided high-speed steel. At low nitrogen partial pressure and most of the substrate biasing conditions, a TiN (200) orientation is preferred on plasma-nitrided substrate. The matching of inter-planar distances of TiN (200) plane with γ′ Fe_4N (111) plane of plasma-nitrided substrate is believed to be a factor responsible for this preferred orientation.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

PS carried out all the experiments and calculations, designed the study, and drafted the manuscript. BKS and SM provided guidance at various stages of the study and reviewed the manuscript. RR and AJ helped in the XRD analysis. All authors read and approved the final manuscript.

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