

Article

Evaluating the Tribological Properties and Residual Stress of TiCrN Thin Films Deposited by Cathodic-Arc Physical Vapor Deposition Technique

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Abstract: The present study reports the tribological properties and residual stress of titanium chromium nitride (TiCrN) coatings. Thin films of TiCrN were deposited on tungsten carbide substrates at 400 °C in a vacuum of 5×10^{-6} mbar using the cathodic-arc physical vapor deposition technique with chromium variation. X-ray diffraction (XRD) spectroscopy was employed to probe the structures of the deposited thin films. The phase constituent was found to gradually shift from cubic TiN to cubic CrN. Both the hardness and elastic modulus of the sheet changed from 29.7 to 30.9 GPa and 446 to 495 GPa, respectively. The biaxial compressive residual stress after an initial absolute scan in the range of 30–100° was determined using XRD ($d\text{-sin}^2\psi$ method). These mechanical and tribological properties of films were investigated with the help of instrumented nanoindentation and a ball-on-disk tribometer wear test. The wear test indicates that the TiCrN thin film, featuring a Cr/Ti ratio of 0.587, exhibits superior wear resistance and maximum compressive residual stress in comparison to other thin films.

Keywords: titanium chromium nitride (TiCrN) thin film; cathodic-arc physical vapor deposition (CAPVD); tribological properties; nanoindentation; residual stress



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1. Introduction

To keep up with economic, technological, and environmental concerns, modern equipment must be constructed with ever-increasing performance requirements, frequently pushing components to their utmost limits [1]. Tribological deficiencies, such as lubrication breakdown, excessive wear, and tribo-corrosion, can be greatly exacerbated by the growing need for performance [2]. This can result in unnecessary operational costs, decreased efficiency, and premature failure [3,4]. Because tribological processes arise from the interaction of two or more bodies in relative motion in a given environment, surface engineering can be employed to provide surfaces with the high performance they need for challenging operational situations [5,6]. Physical vapor deposition (PVD) and chemical vapor deposition (CVD) technologies have allowed for novel approaches to the production of tribological coatings [7–10]. Evaporation is at the core of PVD processes; there are a variety of ways to evaporate the source material. Hard, dense, and thick protective coatings are often deposited using the cathodic-arc deposition (CAD) process, in which the target material (cathode) is vaporized using an electric arc. The necessity to create dense and adherent ceramic coatings at moderate substrate temperatures (200–600 °C) motivated the development of PVD technology. The PVD coatings made of transition metal nitrides and carbon nitrides, such as TiN, CrN, TiCrN, and TiCN, are on the upswing because of their

excellent resistance to corrosion and wear [11–16]. Hard coatings have been frequently used to protect the working surfaces of tools, extending their service life, improving their efficiency, and boosting their productivity.

By altering material properties like hardness, toughness, corrosion resistance, and residual coating stresses, structural parameters like density, crystallinity, and grain size have a significant impact on performance in protective coatings for tribological applications [17–19]. It is reported that the coating properties of CrN and M-CrN are directly affected by the hardness of the substrate [20,21]. The incorporation of chromium in the cubic (face centered cubic) TiN structure leads to enhanced thermal stability of the coating [22]. Cr atoms are in crystalline lattice sites substituting Ti atoms to form a solid solution. The Cr content in the TiCrN films influences the microstructure, hardness, and oxidation resistance [22–25]. Coatings of the type (TiCr)N are candidates for the wear protection of tools and machinery parts [26–30]. While the difference in thermal expansion coefficient between the coating and the substrate contributes significantly to the overall residual stress, it has been established that the residual stress in coating systems is also associated to microstructure [31]. Despite the plethora of data available on structure and hardness of TiCrN coatings, relatively few studies have investigated their wear properties. The CAD of TiN thin films has not been the subject of as many investigations as other deposition methods; hence, it is difficult to fully assess the impact of Cr addition on the film's characteristics. For tribological and structural applications, the mechanical properties, wear resistance and residual stress of cathodic-arc PVD (CAPVD)-assisted TiCrN hard thin films have not been studied extensively. Coating parameters like substrate bias, deposition temperature, and cathode currents, etc., can be further optimized for better tribological properties. In depth quantitative and qualitative analysis of toughness for thin coatings can be conducted. Critical thickness of the coating developed can be optimized in the case of thick coatings for improved wear resistance. Specific methods can be adapted to calculate volume loss of the coated substrate during wear tests.

In the present investigation, an effort has been made to investigate the wear and mechanical response as well as the role of residual stress of CAPVD-assisted TiCrN thin films with various chromium contents. The “ $\sin^2\varphi$ ” method is also used to calculate the amount of biaxial residual stress on the coated surface. The mechanical and tribological properties of the films were evaluated using instrumented nanoindentation and a ball-on-disk tribometer wear test. The optimum coating parameters were selected from the as-coated six compositions based on the above-mentioned tests conducted by which thin films were developed. The wear test results demonstrate that the TiCrN thin film with a Cr/Ti ratio of 0.587 exhibits superior wear resistance and has the highest compressive residual stress among the investigated films.

2. Experimental Procedure

TiCrN films were deposited by CAPVD technique using two separate targets, which can produce independent plasma flows. The rotatable substrate holder was placed in the center of the chamber. Hence, for thin coatings of TiCrN, samples of tungsten carbide (WC) ($30 \times 30 \times 5 \text{ mm}^3$) were taken. A flat surface of the substrate was polished up to 1 μm diamond paste to obtain the required surface finish. Prior to coating, substrates were cleaned and conditioned thoroughly through a series of processes. The specimens were ultrasonically cleaned by ethyl alcohol then chemically cleaned for 40 min in the Thermovide V300 Pre-cleaning Unit, maintained at a temperature of 60–70 °C. The plasma was generated from the cathode spot on pure Ti and Cr targets (chamber wall used as anode). The two plasma sources operated simultaneously during the TiCrN film deposition process. The distance between the sample and the rotating cathode was 6.5 cm. Prior to

deposition, the chamber was evacuated to a base pressure of 10^{-6} mbar. The chamber was heated up to 450 °C with a continuous flow of argon of a range of 15–27 sccm. During the deposition of films, nitrogen and argon gases were introduced into the deposition chamber with N₂ partial pressure of 2×10^{-5} mbar. The flow rate of nitrogen gas was 300 sccm. The temperature of the substrate during coating was set at 400 °C. Six sets of experiments were performed by keeping the substrate bias at −50 V. The arc currents for Ti were kept at 175 A and for Cr it varied from 80 A to 175 A. T1, T2, T3, T4, and T5 samples are for the Cr current 80, 100, 125, 150, and 175 A, respectively, keeping constant Ti current at 175 A (Table 1). In the case of the T6 sample, the Cr and Ti current were kept at 175 and 100 A, respectively. During the deposition of films, N₂ was introduced into the deposition vacuum chamber with N₂ partial pressure of 2.1×10^{-5} mbar. All the coatings discussed in this section were deposited to a uniform thickness of 3.2 µm.

Table 1. Coating parameters for TiCrN thin film deposition.

Coating Parameters	TiCrN I (T1)	TiCrN II (T2)	TiCrN III (T3)	TiCrN IV (T4)	TiCrN V (T5)	TiCrN VI (T6)
Ti Current (A)	175	175	175	175	175	100
Cr Current (A)	80	100	125	150	175	175
Cathode Speed (rpm)	11	11	11	11	11	11
Vacuum Pressure (mbar)	10^{-6}	10^{-6}	10^{-6}	10^{-6}	10^{-6}	10^{-6}
Heat Treatment (°C)	450	450	450	450	450	450
Bias Voltage (V)	50	50	50	50	50	50

The coatings were also comprehensively characterized for their phase composition, residual stress, and hardness using X-ray diffraction (XRD) (X'Pert Pro, Panalytical, Malvern, UK) (2θ-glancing angle, Cu-K α , 45 kV) and nanoindentation (NHT, CSM) equipped with Berkovich indenter, respectively. The surface morphology and composition of the coatings were studied using scanning electron microscopy (SEM: HITACHI S-3400 N, Hitachi, Tokyo, Japan) with energy dispersive spectroscopy (EDS: HITACHI S-3400 N), respectively. The hardness results presented are an average of 20 indentations (load 50 mN in sinus mode). The details of the test parameters are listed in Table 2. The instrument was operated in sinus mode and the indentations were made using a sinus frequency of 10 Hz. The hardness and elastic modulus were determined using the Oliver analysis [32–34]. Conventional ball-on-disc equipment was used to measure the coefficient of friction (COF) and to determine the sliding wear rate (WR) of the coatings. ASTM G-99 is followed for the wear test. The stress analysis was carried out using the “sin² ϕ ” method.

Table 2. Parameters used in Nanoindentation.

Mode	Sinus
Delta Slope Contact	80%
Measurement max load	50 mN
Loading rate	100 mN/min
Unloading rate	200 mN/min
Sinus frequency	10 Hz
Sinus amplitude	5 mN
Indenter type	Diamond Berkovich

3. Results and Discussion

3.1. Effect of Test Parameters on TiCrN Thin Film

It can be observed from Figure 1a that the substrate current is linearly proportional to the chromium cathode current at constant coating time and Ti cathode current. Over the entire range of Cr current (80 to 175 A) in the TiCrN coatings, the phase constituent was found to gradually shift from cubic TiN to cubic CrN, as shown in the XRD patterns in Figure 1b. The XRD spectrum was matched with the standard JCPDS files as per the ICDD database and the identified peaks corresponding to cubic TiN (No. 38-1420), cubic CrN (No. 6-2494), and TiCrN (No. 38-1420) are shown in Figure 1b. With the increase in the current value, the lattice constant of TiN is found to change from 4.23 to 4.24 Å. The observed change in lattice constant with the change in current is due to the substitution of “Ti” atoms by Cr atoms in the TiN matrix resulting in the formation of a solid solution TiN(Cr). Similarly, at higher values of current (current > 185 A), the coatings contain a cubic CrN(Ti) phase with partial substitution of Cr atoms with Ti atoms (lattice constant of 4.15 instead of 4.14 for CrN). This observed change in phase constitution from TiN-TiCrN-CrN is expected to change residual stress.

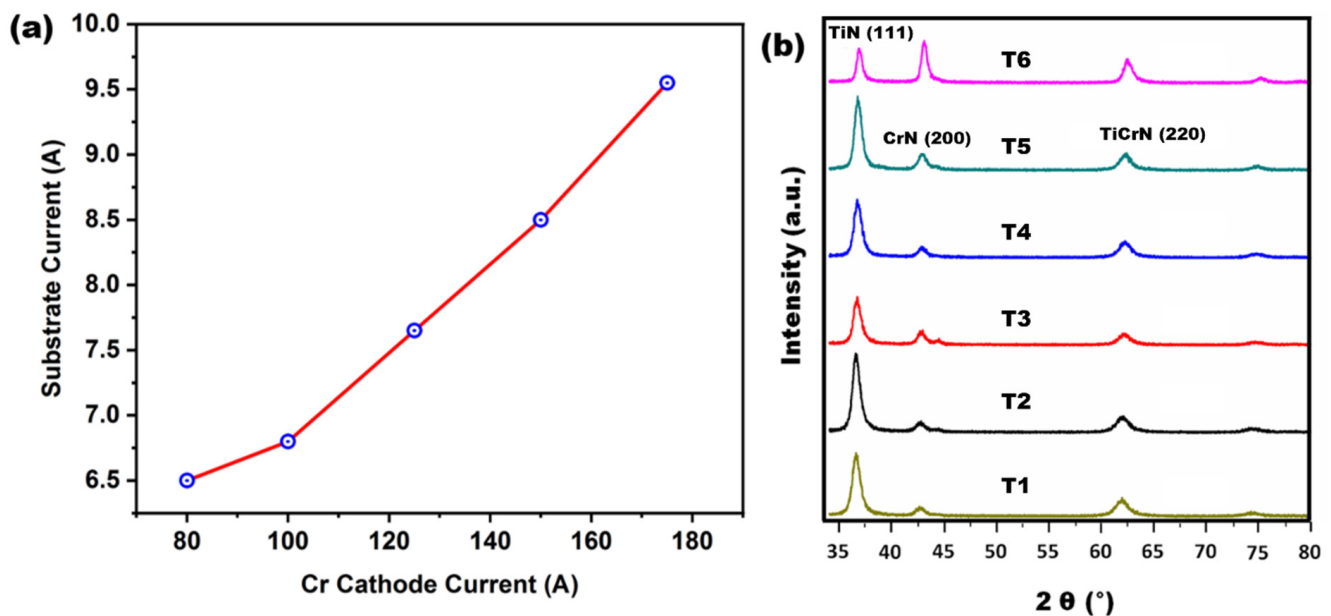


Figure 1. (a) Variation in substrate current at different Cr cathode currents. (b) X-ray diffraction (XRD) spectra of the deposited TiCrN coatings with chromium current variations.

The SEM image shows the microstructure, and the EDS spectra shows the presence of Ti and Cr elements (Figure 2).

Expected coating thickness is calculated by using the empirical relation [15] (Equation (1)),

$$t_{\text{Expected}} = 0.0025 \times I_s \times T_{\text{Depo}} \quad (1)$$

where I_s is the substrate current in ampere. T_{Depo} is the deposition time in minute. t_{Expected} and t_{Actual} are the target thickness and actual measured thickness of the coating, respectively.

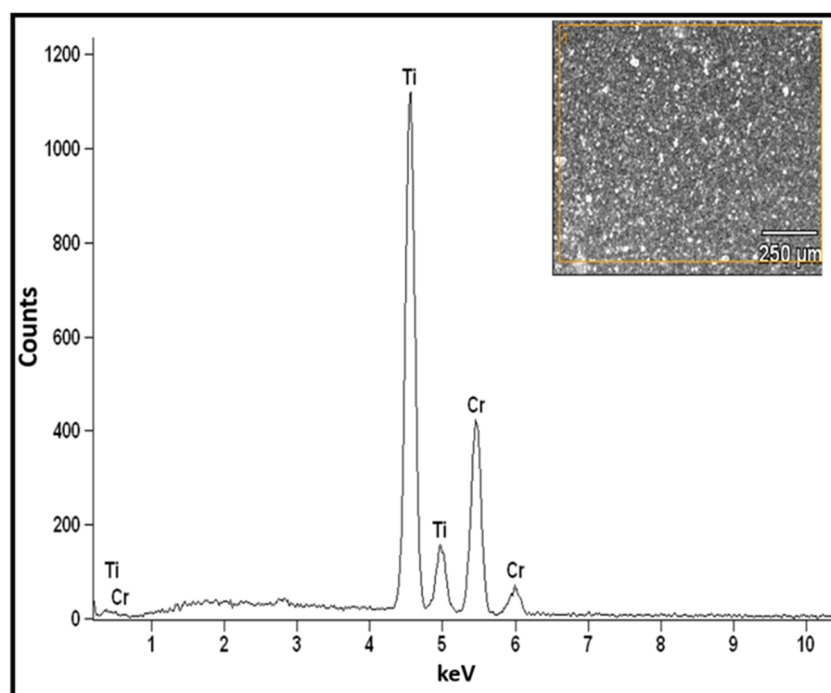


Figure 2. EDS spectra showing titanium and chromium peaks along with SEM image of sample T2.

The film thicknesses of all the deposited coatings were obtained using a Calo tester CT 250 (PLATIT advanced coating systems product) which is shown in Figure 3a,b. The samples were cleaned with an alcohol solution and placed in the sample container appropriately. The Calo tester consists of a holder for the surface to be tested and a steel sphere of a known diameter (30 mm) that is rotated against the surface by a rotating shaft connected to a motor whilst diamond paste (1 μ suspension) is applied to the contact area. The sphere is rotated for a short period (90 s) of time but due to the abrasive nature of the diamond paste, this is sufficient to wear a crater through thin coatings. An optical microscope (Zeiss Axio Cam MRc5) is used to take two measurements after the Calo test across the crater and the coating thickness is calculated using a simple geometrical equation.

$$d = (A^2 - B^2) / 4D$$

where d is the coating thickness and D is the diameter of the sphere ($d \ll D$). The values of A , B , and D can be obtained from the Calo tester as shown in Figure 3c,d.

Table 3 displays the thicknesses of all coatings deposited. The coating thickness was computed using the calotte grinding measuring instrument depicted in Figure 3e. For each coating evaluation, five observations were taken, and the average value was used to determine the coating thickness of each sample. Figure 3f demonstrates that there are minor deviations between the expected and the actual thickness.

Table 3. Dependence of coating thickness on substrate current.

Sample	I_s (A)	$T_{\text{Depo.}}$ (min)	t_{Expected} (μm)	t_{Actual} (μm)
T1	6.5	180	2.925	2.85
T2	6.8	180	3.06	3.10
T3	7.7	180	3.46	3.20
T4	8.6	180	3.87	3.25
T5	9.6	120	2.88	2.60
T6	7.0	180	3.15	3.00

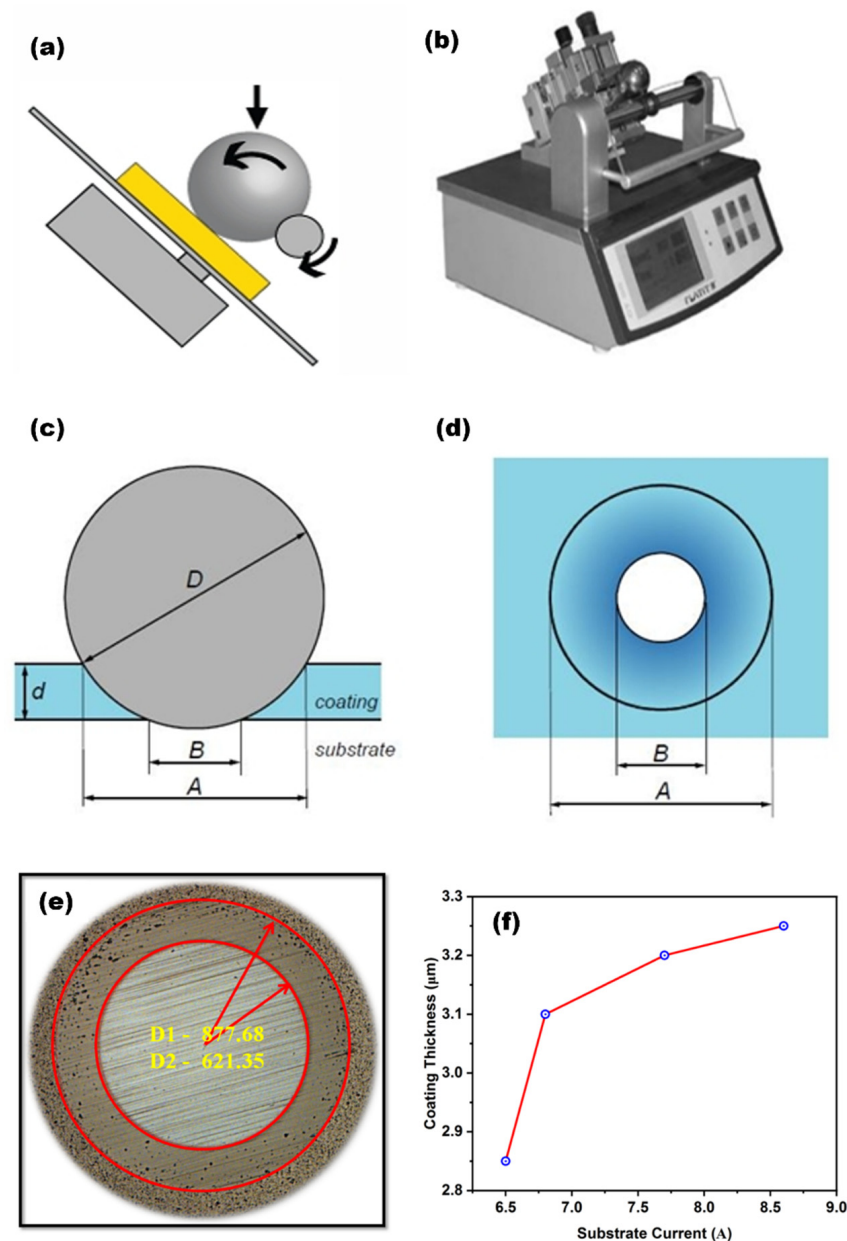


Figure 3. (a) Working principle of a Calo tester. (b) Image of a Calo tester. (c,d) Procedure for thickness measurement of the film using a Calo tester. (e) Image of TiCrN coating thickness measurements using the Calotte grinding measurement tool. (f) Effect of substrate current on coating thickness at fixed coating time and constant Ti current.

3.2. Correlations Between Coating Parameters and Mechanical Properties

Figure 4 illustrates the relationship between the Cr-to-Ti ratio (denoted as Cr/Ti) and the hardness of all the samples. When the current of Cr increases from 80 to 175 A, Cr/Ti also increases from 0.428 to 2.571. The Cr/Ti ratio was determined to be the lowest (0.428, as shown in Table 4) for sample T1, while it was found to be the highest (2.571, as shown in Table 4) for sample T6. On the other hand, there is an opposite trend observed in the hardness of the coating surfaces. The hardness value exhibits variation between 31 and 26 GPa, depending on the chromium current employed. The literature reports the hardness values of binary CrN and TiN coatings to be 18 and 24 GPa, respectively [35,36]. In this case, the highest level of hardness (30.9 GPa, as indicated in Table 4) observed in sample T1 can be attributed to the existence of multiple phases, as identified by the XRD spectra displayed in Figure 1b. The XRD analysis reveals the initial presence of a single-phase

material consisting of titanium nitride (TiN) with Cr substitution. Subsequently, a mixed phase of TiN and chromium nitride (CrN) is observed. Then, in the case of a higher current of Cr, Ti is introduced as a substitution in CrN. The higher concentration of the CrN phase in sample T6 is responsible for the observed lowest hardness value (26 GPa, as shown in Table 4). The reported values of these coatings lie somewhere between the hardness of TiN and CrN, as reported in the literature [37,38]. The XRD spectra, which shows intense peaks corresponding to CrN (see Figure 1b), clearly demonstrates this.

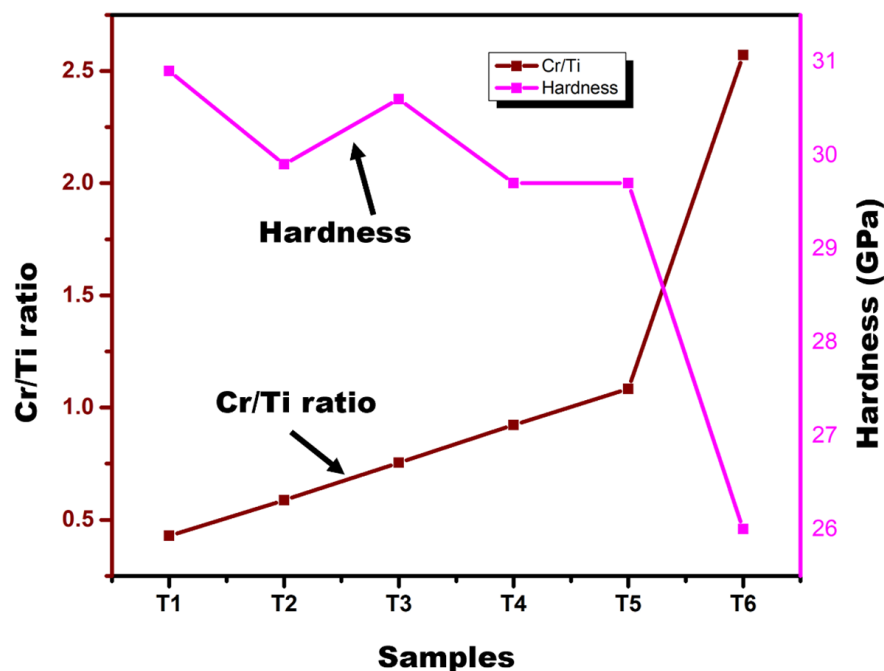


Figure 4. Cr/Ti ratio and hardness of samples.

Table 4. Hardness and elastic modulus at different Cr/Ti ratios.

Sample	Cr/Ti Ratio	Hardness (H) (GPa)	Modulus (E) (GPa)	Plasticity Index (GPa) (H^3/E^2)
T1	0.428	30.9 ± 5.5	495 ± 15.5	0.1216
T2	0.587	29.9 ± 2.7	470 ± 17.3	0.1222
T3	0.754	30.6 ± 6.5	487 ± 22	0.1256
T4	0.923	29.7 ± 2.1	457 ± 20.9	0.1292
T5	1.083	29.7 ± 2.2	446 ± 17.7	0.1357
T6	2.571	26.0 ± 4.0	442 ± 22.5	0.0899

The modulus of elasticity and plasticity index of the samples are displayed in Figure 5. The elastic modulus of sample T1 was determined to be 495 GPa, which represents the highest value observed among all the samples. Conversely, the lowest value of 442 GPa was found in sample T6. The high value of the modulus of elasticity in T1 can be attributed to its higher hardness (30.9 GPa, as shown in Figure 4) in comparison to the other samples. The plasticity index, calculated by dividing the square of hardness by the cubic power of the modulus of elasticity, was observed to be the lowest in sample T6. The reason for this is because the T1 sample has the lowest values of hardness and modulus of elasticity.

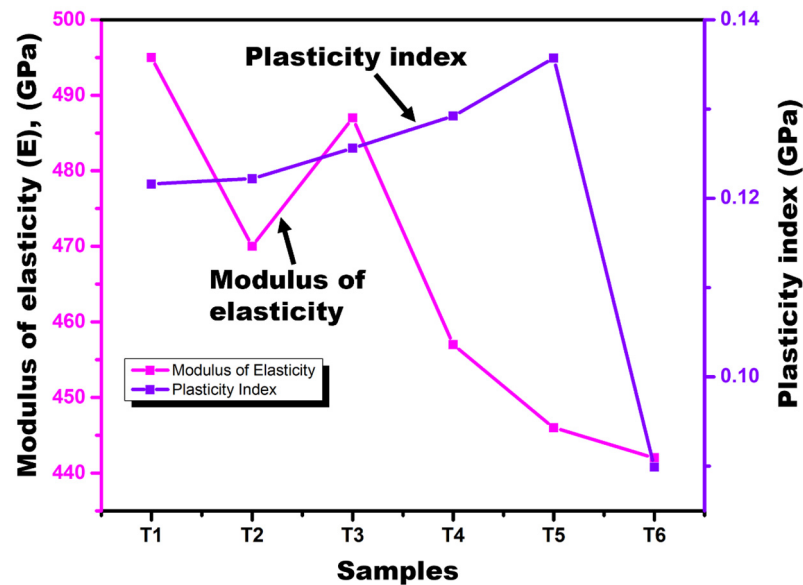


Figure 5. Variation in modulus of elasticity and plasticity index values of samples.

3.3. Tribological Study of TiCrN Thin Films

A conventional ball-on-disk machine was used to measure the COF and the sliding WR of thin films. It was observed that the coating volume loss is negligible as compared to the volume loss of the bare WC ball (counter body) [ASTM G-99]. Therefore, in the present investigation, the volume loss and WR of the bare WC ball were calculated for each set of tests to discover the better coating parameters. The scar is formed at the contact point between the sample and the bare WC ball. For sample T2, scars on both ball and sample are shown in Figures 6a and 6b, respectively. The scar on the ball is like a chord cut on a solid sphere with a diameter of 471.42 μm , schematic is shown in Figure 6c. However, the observed scar on the thin film surface consists of a wear track of 424.24 μm as shown in Figure 6b. Figure 6d depicts the relationship between the COF and sliding distance for each set of coating.

After a sliding distance of 180 m, the COF value reaches its steady state. The image (Figure 6d) indicates that the initial COF is very low, increases slowly, and attains a steady state that is independent of the sliding distance. Special attention was given to evaluating the steady-state COF. The average COF value was found to be less as compared to the steady-state COF value. This may be due to the initial low value of the frictional force. As time progressed, the steady-state COF was achieved keeping the fact in mind that the coating was not completely absent from the substrate. The volume loss and scar depth of the bare WC ball were calculated by using the following formulae (Equations (2) and (3)) and represented in Figure 7.

$$V = \pi \times h \times \frac{(3a^2 + h^2)}{6} \quad (2)$$

$$h = r - \sqrt{(r^2 - a^2)} \quad (3)$$

where V is the volume loss (in mm^3), a is the radius of the scar on the WC ball (in mm), h is scar depth (in mm), and r is the radius of the WC ball (in mm). Wear torque is the product of the applied load on the ball and the sliding distance covered by the ball. Sliding distance and sliding velocity were calculated by using the following relations (Equations (4) and (5)):

$$S = V_s \times T \quad (4)$$

$$V_s = \frac{\pi \times D \times N}{60} \quad (5)$$

where S is the sliding distance (in meters), V_s is sliding velocity (in m/s), T is the test duration for each sample (in sec), D is the track diameter (in meter), and N is the speed of the disc (in rpm).

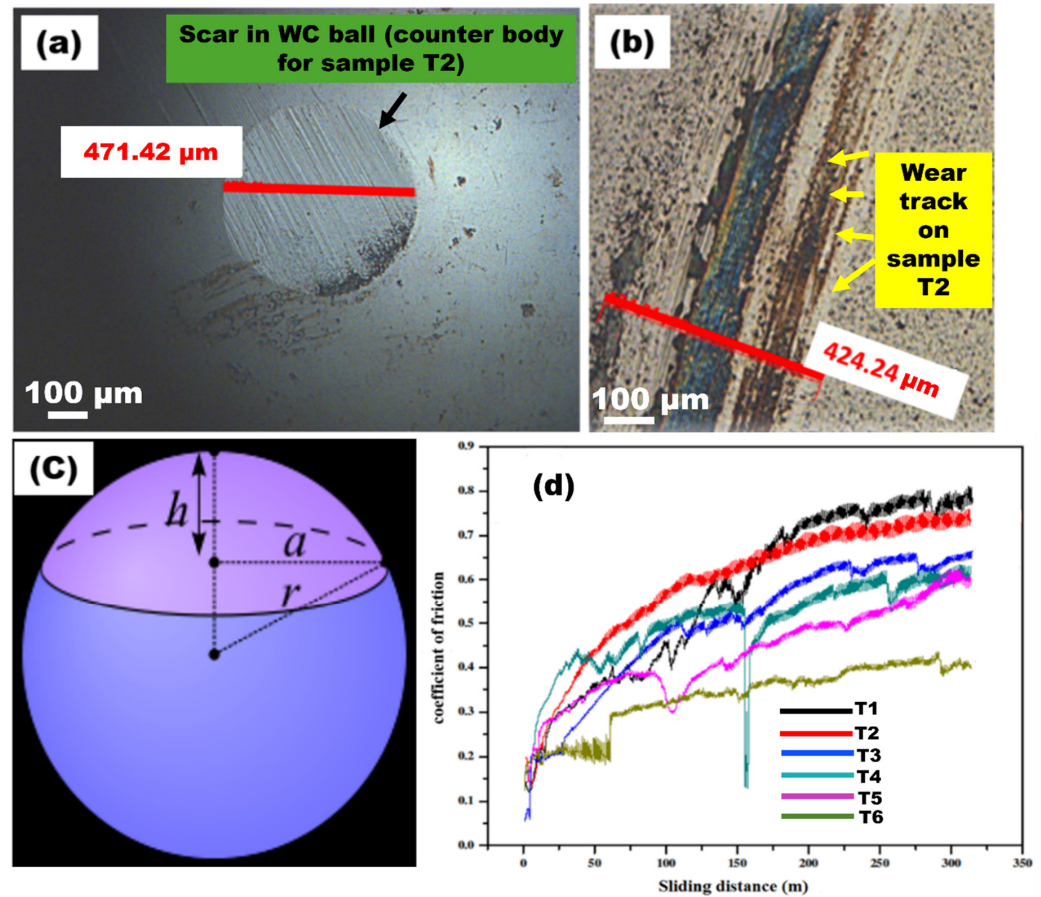


Figure 6. SEM micrographs of the (a) scar on the WC (tungsten carbide) ball (counter body for sample T2) and (b) wear track on the sample T2. (c) Schematic of chord cut on a sphere. (d) Dependence of steady state friction on sliding distance.

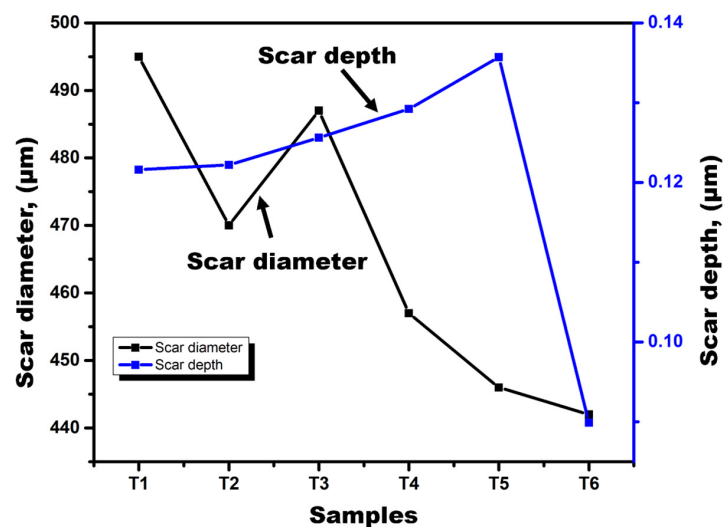


Figure 7. Scar diameter and scar depth of counter body (bare WC ball) used in the wear test for each sample.

The WR of the ball for the respective coated sample was calculated by using the above-mentioned procedure. From Figure 8, it can be inferred that the WR of the counter body (WC ball) is highest ($463 \mu\text{m}^3/\text{Nm}$) for the T2 sample. From the results, it is observed that when there is a higher amount of wear loss on the ball surface, the better is the corresponding coating. It indicates that sample T2 is more resistant to wear than samples with other chromium contents. Consequently, this will be one of the criteria needed for optimizing a single-layer hard TiCrN coating for tribological applications. Figure 8 also presents a relationship between the ratio of Cr to Ti and COF. As the ratio of chromium to titanium increases, the COF is found to decrease. It is important to consider factors like hardness, elastic modulus, toughness, WR, and residual stress when selecting the optimal composition.

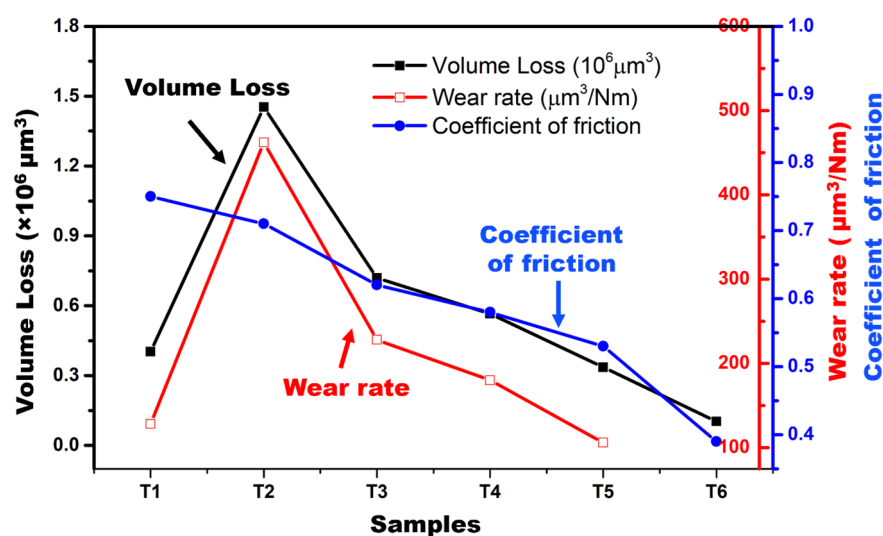


Figure 8. Volume loss, wear rate, and coefficient of friction of counter body (WC ball) for each sample.

3.4. Influence of Cr/Ti Ratio on Residual Stress

Residual stress plays an important role when selecting coating parameters for wear resistance. The “ $\sin^2\varphi$ ” method is used for biaxial stress calculation [39]. The following equation (Equation (6)) is used for stress calculation.

$$\sigma = \left(\frac{E}{1 + \nu} \right) \times \frac{1}{d_o} \times \left(\frac{\partial d}{\partial \sin^2 \varphi} \right) \quad (6)$$

where σ is stress, E is the modulus of elasticity, ν is the Poisson’s ratio, φ is the orientation of the sample, d_o is the intercept from the plot of d versus $\sin^2 \varphi$, and d is the lattice spacing.

The coating has a residual stress that ranges anywhere from 4.8 to 7 GPa (Figure 9). It is possible to draw the conclusion that the nitride film containing Cr/Ti ratio of 0.587 (T2) shows the greatest amount of compressive stress. The higher the magnitude of the compressive stress that the coating is subjected to, the greater its wear resistance [40]. Open microstructures presenting voids will generally have tensile intrinsic residual stresses due to attractive atomic forces acting across the voids [31]. Since attractive atomic forces act through voids in open microstructures, these residual stresses will typically be tensile [31]. However, denser films formed via ion bombardment typically exhibit compressive stresses that are highly dependent on the energy of the impinging ions, which can be controlled via species ionization and substrate bias. Compressive residual stresses (up to 10 GPa) can be produced when particles from a bombardment are implanted in the forming layer and disrupt the lattice. Hard protective thin films typically feature significant compressive stresses, which might be useful for halting fracture processes by closing forming cracks.

Alternatively, delamination of a coating occurs under extreme tensile or compressive loads. Therefore, interface engineering to enhance adhesion is essential under these circumstances.

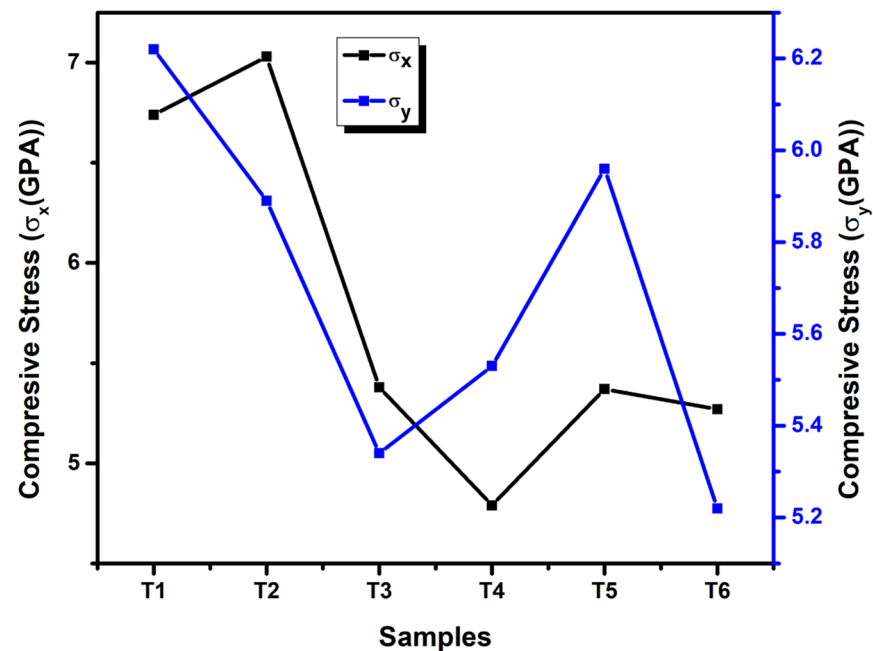


Figure 9. Comparison of compressive stress (σ_x and σ_y) developed in each sample.

4. Conclusions

By altering the cathode current, thin films of TiCrN were effectively deposited on WC substrates, allowing for subsequent tribology experiments to be conducted. The conclusions drawn from this work are as follows.

1. The substrate current is shown to be linearly proportional to the chromium cathode current for a given coating time and Ti cathode current. The XRD patterns reveal that the phase constituents of the TiCrN thin films gradually change from cubic TiN to cubic CrN over the entire range of Cr current (80 to 175 A) in the coatings.
2. Thin films have much higher microhardness than the pure phases. Observations indicate that the high hardness is a result of the two-phase structure.
3. The findings of the wear test indicate that the TiCrN thin film with Cr/Ti ratio of 0.587 exhibits a higher wear resistance than thin films with Cr/Ti ratio of 0.428, 0.754, 0.923, 1.083, and 2.571, as the wear rate of the counterpart (WC ball) is the highest ($463 \mu\text{m}^3/\text{Nm}$) of all coatings. The nitride thin film with Cr/Ti ratio of 0.587 exhibits maximum compressive residual stress.

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