



Characterization of TiN, CrN and (Ti, Cr) N Coatings Deposited by Cathodic ARC Evaporation

M. Akbarzadeh*, A. Shafyei, H. R. Salimijazi

Department of Materials Engineering, Isfahan University of Technology, Isfahan, Iran

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ABSTRACT

In this investigation, physical vapor deposited Ti-Cr-N coatings were coated on tool steel substrates using reactive arc evaporation. Microstructure and mechanical properties of coatings such as roughness, thickness, phase composition, hardness, Young's modulus and coefficient of friction were studied. Phase compositions were investigated by X-ray diffraction method. Surface microstructure and morphology were studied using scanning electron microscope (SEM) and coating compositions were determined by energy dispersive spectroscopy (EDS). Mechanical properties were measured by nano indentation. The friction behavior of the coatings were investigated using ball-on-disc tests under normal loads of 7 N. The results showed that (Ti, Cr)N and TiN coatings consisted of only one cubic phase solid solution, while CrN coatings consisted of hexagonal Cr₂N and cubic CrN phases. Average grain size of the coatings was found to be in the range of 14 to 20 nm. Mechanical behavior of the (Ti, Cr)N coatings was related to the Ti content, i.e. hardness decreased with decreasing Ti content.

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1. INTRODUCTION

The life time and wear resistance of different instruments and industrial parts could be improved by deposition of a hard thin film coating on the surface [1]. Titanium nitride thin films deposited by PVD technology have a wide range of commercial applications such as cutting tools and artificial jewelry because of their wear-resistant characteristics and gold-colored appearance [2, 3]. TiN coating has the NaCl-type crystal structure. The advantages of TiN coatings include high hardness, high chemical stability, and excellent adhesion to substrates. They are widely used on molds, punches, cutting tools, machine parts for motors and biomedical prostheses [4]. TiN coatings are often used for their excellent corrosion protection and surface finish, and high resistance to wear with low coefficient of friction [5-7]. A serious weakness of TiN coatings is their narrow oxidation resistance at

temperatures above 400°C, where a TiO₂ layer is formed and therefore it suffers from lower thermal stability [8].

On the other hand, CrN coating are widely used for their lower coefficient of friction, and excellent oxidation and corrosion resistances, higher ductility and fracture toughness, and comparable adherence. Therefore, in many cases it has replaced to TiN [8-11].

CrN coatings have received more attention in last two decades because they could be deposited at lower temperatures compared to TiN coatings. Maximum service temperature for TiN and CrN coating is 500 and 750°C, respectively. Also, it has noted in literatures that the oxidation resistance of CrN as compared to TiN is almost 1000 times higher [8, 12, 13]. Investigation showed that CrN coatings with optimum thickness work much better than TiN coating under the same operation conditions. The multi-component coatings usually have good physical and mechanical properties and are widely used in various industries [14]. Properties of these coatings, such as friction coefficient, wear rate, hardness and toughness may be controlled by the selection of the elements and/or phases forming, their chemical composition and/or volume fractions, and deposition

*Corresponding Author Email: m.akbarzade@ma.iut.ac.ir (M. Akbarzadeh)

process parameter. These physical quantities are usually influenced by chemical composition, phase content, and microstructure, which are dependent on the deposition conditions and surface preparation procedure. In the recent innovative multi-component coatings such as e.g. Ti(C,N), (Ti, Al)N, (Ti, Si)N, (Ti, B)N, (Ti, Ag)N, (Ti, Zr)N, (Ti, Nb)N, (Ti, Al, V)N and (Ti, Al, V, Cr, Mo,)N, these properties have been much improved [15-21]. An increasing amount of attention is now focused on ternary Ti-Cr-N coatings. Researchers have reported that new ternary Ti-Cr-N coatings have high-temperature strength compared to CrN and TiN coatings [13, 22-28]. However, most of these have scarcely been investigated. The goal of the presented work is to study the CrN, TiN and (Ti, Cr)N PVD coatings deposited by cathodic arc evaporation.

2. EXPERIMENTAL

Substrates 5 mm in thickness were cut from a 50 mm diameter AISI D6 steel rod (composition 2.13% C, 0.25% Si, 11.50% Cr, 3.00% Mn, 0.70% W, balance Fe). Samples were annealed at 980°C followed by quenching in water and tempered at 180°C. The hardness of the samples was around 55 Rc. They were then polished to 0.1 μm using standard metallographic preparation techniques. After surface preparation, samples were degreased in acetone and subsequently in a methanol solution, and then sputter cleaned for 20min with argon at 1Pa pressure, while the substrate was biased at 2000 V to remove the surface oxide layer under the pressure of 10⁻³ Pa. The specimens were coated using cathodic arc evaporation (CAE). (Ti, Cr)N coatings were deposited with two evaporation sources. Initially, the chamber was evacuated to a pressure of 10⁻⁶ mbar with a diffusion pump before introducing a reactive gas of Cr (99.8% in purity) and Ti (99.6% in purity) target cathodes into a reactive nitrogen atmosphere at a nitrogen pressure of 2 Pa at bias voltages of -1000 V. The distance from the target source to the substrate was 200mm. Working pressure of the chamber was 10-3mbar. The coating process was carried out for 1h. During the ion etching and deposition, the substrates were rotated in a planet-sun fashion. Substrates were coated at 2Pa nitrogen pressures by using -1000 (Ti, Cr)N; while coatings were deposited with two evaporation sources. (Ti, Cr)N coatings were deposited under evaporation currents of 80A and 300A. Moreover, two monolayer TiN and CrN coatings were coated under the same conditions, while Cr and Ti evaporation current of 300A was used as references.

The surface roughness was measured using stylus type (Talysurf Taylor Hobson) instruments. The composition of the coatings was measured using energy dispersive spectroscopy (EDS). The average surface

roughness was measured at five different locations and the average of these five results was reported.

X-ray diffraction (XRD) technique (Philips PW-3710 diffractometer) using Cu Kα radiation in the 2θ range from 10 to 100° was employed to evaluate the crystal structure, phase contents and crystallographic orientations of the coatings. In order to determine the crystallographic structure and phase compositions of the coatings, XRD scans were performed and compared to the Powder Diffraction Files (JCPDS's) published by the International Centre for Diffraction Data (ICDD). The crystallite size and micro-strain was evaluated from the XRD patterns based on the Scherrer formula [29, 30]:

$$\beta \cos \theta = \frac{K \lambda}{d} \quad (1)$$

where β is the line broadening at half the maximum intensity (FWHM) in radians, θ and λ the wavelength and the diffraction angle and d crystallite size.

The hardness and Young's modulus were measured using nano indentation test (CSM) which was developed by Oliver and Pharr [31]. Maximum indentation depth, load and max loading and unloading rate were 320 nm, 3000 μN 60.00mN/min, respectively. Six indentations on each coating were applied and the average value was presented.

Friction tests were performed using a ball-on-disk machine. This equipment is controlled by its PC software, which allows observing the evolution of the friction coefficient. The WC-6% Co ball (10mm diameter) was loaded against the rotating sample with a normal load of 7 N at room temperature, while the sliding speed was 0.081m/s.

3. RESULTS AND DISCUSSIONS

The summary of deposition parameters, chemical composition, and thickness of coatings is tabulated in Table 1. The deposition rate is directly related to the evaporator current. The maximum vaporization rate of material is relative to the vapor pressure and could be calculated using the Hertz-Knudsen equation:

$$\frac{dN_e}{dt} = a_v (2\pi mKT)^{-0.5} (P^* - P) A_s \quad (2)$$

where dN is the number of evaporating atoms per cm² of the surface area, p* the vapour pressure of the material at temperature T, m the mass of the vaporized species, Pp pressure of the vapor above the surface, K Boltzmann's constant, T absolute temperature and C a constant that depends on the rotational degrees of freedom in the liquid and the vapor. According to Hertz-Knudsen formula and because of the lower vapor pressure of chromium compared to titanium, TiN coating is thinner than CrN coatings at the same coating conditions. Moreover, (Ti, Cr)N coatings had the highest thickness due to using two target sources.

Figure 1 shows the optical microscope images of the coating. Micro-droplets produced by cathodic arc evaporation on the coating surface can be observed. The number of micro droplets formed on the CrN surface is much more than that on the TiN coating. This could be due to the rapidly melting of the source material by the arc. Investigators have suggested that melting and boiling points of target materials are the most important factors in the size and number of micro droplets. They are formed during arc evaporation and contain materials which are emitted from the source surfaces. Figure 2 shows SEM images of the micro-droplet on the (Ti, Cr)N coatings surface. The micro droplets can be found both on the coating surface and in the body of the arc deposited coatings. The diameter of the droplets was about 0.5 μm with a few larger droplets (about 2 μm). One of the main disadvantages of the CAV technique is the formation of a large quantity of micro droplets on the surface of the coatings that adhere to the substrate and roughen the surface of the coatings [32].

Surface roughness of the coatings is presented in Figure 3. The high surface roughness could be attributed to the nature of the CAV technique and occurrence of the characteristic macro-droplets [2].

The XRD patterns of the coatings are shown in Figure 4. Coatings were highly textured with a preferential orientation in a direction of (200). Coatings deposited by various PVD processes often exhibit certain preferred orientations, which are heavily affected by the process parameters. Since, the thicknesses of coatings are less than 5 microns, some peaks from the substrate can be detected as well. The XRD patterns of (Ti, Cr)N, TiN and CrN coatings are similar to each other due to similarity in their lattice parameters and crystallographic structures. CrN samples represent two stable modifications of chromium nitride: hexagonal Cr_2N and cubic CrN. Whereas, TiN is stable over a broad range, while CrN that has a very narrow composition range, needs to use a very high partial nitrogen pressure during deposition [33, 34]. Therefore, hexagonal Cr_2N could be easily observed under stoichiometric nitrogen conditions, Cr_2N diffraction peaks could be detected in the CrN and (Ti, Cr)N coatings.

Vetter et al. [35] used both single source and multi-source cathodic arc deposition at substrate temperatures of up to 560°C and observed two phases, cubic (Ti, Cr)N and β -hexagonal $(\text{Cr,Ti})_2\text{N}$. Nainapampil et al. [36] and Sang et al. [37] deposited (Ti, Cr)N by cathodic arc evaporation method and a closed field unbalanced magnetron sputtering method with separate Ti and Cr targets, respectively. They observed a mixture of TiN and CrN phases in $(\text{Ti}_{1-x}\text{Cr}_x)\text{N}$ coatings. Hones et al. [38] and Zeng et al. [27] deposited (Ti, Cr)N by reactive magnetron sputtering. Also, Lee et al. [37] deposited (Ti, Cr)N by ion-plating, using different ratios of chromium and titanium. They observed only one

solid solution cubic phase. Hiroyuki et al. [39] results were in direct contrast to the study by Nainapampil et al. [40].

According to the XRD pattern, the (Ti, Cr)N coatings produced in this study possessed only one solid solution cubic phase. As seen in Figure 4(b), peak of (220) in (Ti, Cr)N coatings shifted toward higher diffraction angles with decreasing the titanium content. CrN has a smaller lattice parameter than TiN and this leads to a smaller lattice parameter for the (Ti, Cr)N coatings with high Cr content.

The grain size of the coatings are estimated based on the Scherrer formula from the X-ray diffraction patterns of (220) reflexes and are presented in Figure 5. According to Lin et al. [41], there is a straight line relation between the grain size of the PVD coating thickness. In this study, grain size increased from 14 to 19nm as the coating thickness increased from 2 to 4 μm .

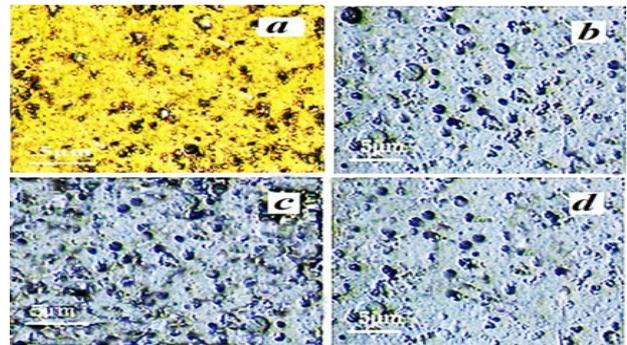


Figure 1. Optical microscope images of the surface of coatings deposited under condition of a, b, c, and d.

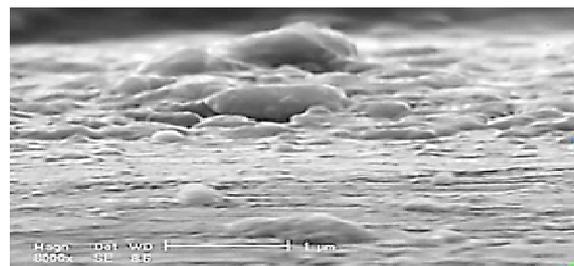


Figure 2. SEM image of micro droplets formed on the (Ti, Cr)N coatings surface

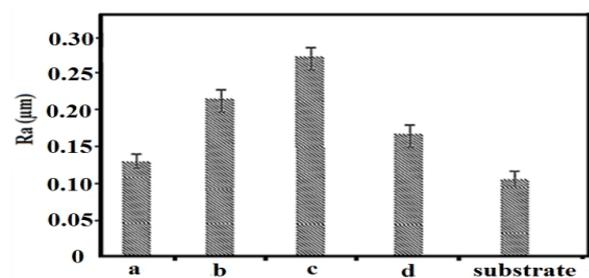


Figure 3. Surface roughness of PVD coatings.

TABLE 1. Deposition conditions and coating composition

Sample	Cr evaporator current (A)	Ti evaporator current (A)	Thickness (μm)	Deposition rate (μm. min ⁻¹)	Cr Content (Cr/(Cr + Ti))	Ti Content (Ti/(Cr + Ti))
a	0	300	2.1±0.1	0.035	0	1
b	80	300	2.8±0.4	0.041	0.45	0.65
c	300	80	4.5±0.0	0.075	0.73	0.27
d	300	0	2.8±0.1	0.046	1	0

TABLE 2. Results of the nano indentation test.

Sample	Hardness (MPa)	Young's modulus (GPa)
a	28469	360
b	25992	415
c	22662	373
d	17690	253

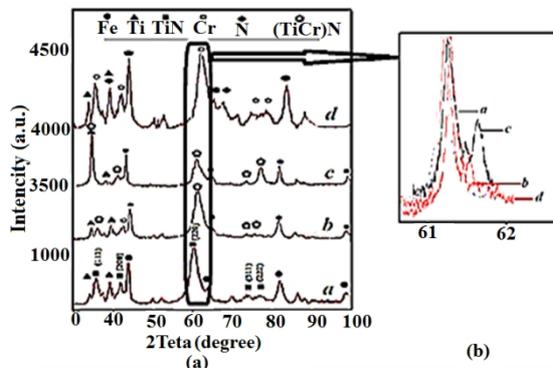


Figure 4. XRD patterns of coatings.

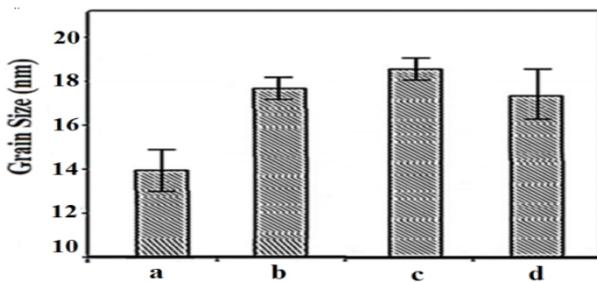


Figure 5. The calculated grain size of coatings

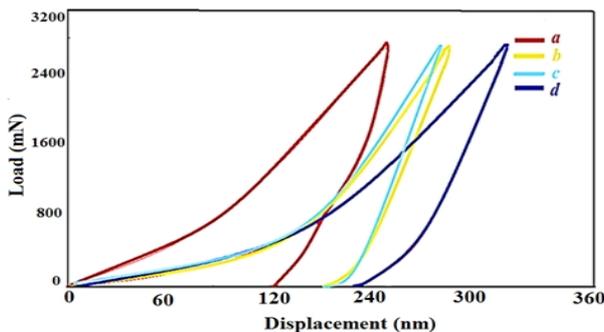


Figure 6. A typical Load–displacement curves of coatings

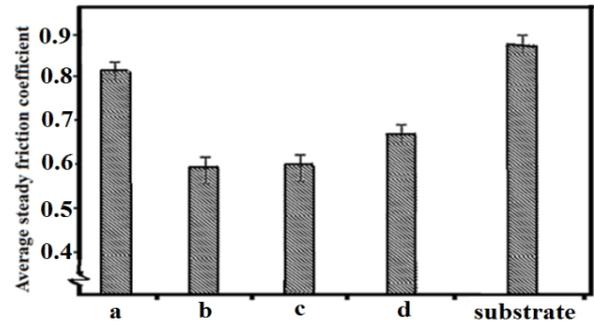


Figure 7. Average steady friction coefficient of coatings and substrate.

Figure 6 shows the typical load–displacement curves for coatings. The calculated hardness and Young's modulus and stiffness are presented in Table 2.

The highest hardness was related to TiN, while the lowest was related to CrN coating. Results indicated that the Young's modulus of (Ti·Cr) N coatings was higher than CrN and TiN coatings. The mechanical behavior of the (Ti, Cr) N coatings was related to the Ti content. The hardness decreased with decreasing the Ti content. It has been reported that compressive stresses and grain size have a great effect on the mechanical properties of coatings [42]. The coatings clearly follow the Hall–Petch relationship (HPR):

$$H_v = H_0 + Kd^{-\frac{1}{2}} \tag{3}$$

where, H_v is hardness, H_0 the term depending on the hardness of the individual grains, K a constant defining the influence of the grain boundaries and d the grain size. This relationship is based on the observation that grain boundaries impede dislocation movement and that the number of dislocations within a grain have an effect on how easily dislocations can traverse grain boundaries and travel from grain to grain. The dependence of Ti contents on the hardness in the Ti–Cr–N coatings is similar to that obtained by Vishnyakov et al. [34].

The variation of the steady state friction coefficient of the coatings and substrate is presented in Figure 7. Due to the weak adhesion of the micro droplets to the surface of coatings, they may be removed from the surface in the early stages of the wear test. Then, due to the self lubrication properties of the coating, the friction coefficient decreased. Among the various depositions, (Ti, Cr) N coatings showed the lowest steady state friction coefficient.

4. CONCLUSION

In the current study CrN, TiN and (Ti, Cr)N coatings were deposited on quenched and tempered tool steel substrates by using a cathodic arc evaporation method. The physical and mechanical behaviors of the coatings were investigated. The (Ti, Cr)N and TiN coatings possessed only one solid solution cubic phase, while CrN coatings contained hexagonal Cr₂N and Cubic CrN phases. All coatings showed preferred crystal orientation (220). The average grain size of the coatings was found in the range of 14 to 20nm. The highest hardness was obtained in TiN coatings, although the hardness of (Ti, Cr)N coatings was also higher than CrN coatings. The mechanical behavior of the (Ti, Cr)N coatings was related to the Ti content. The hardness decreased with decreasing the Ti content. Results indicated that the Young's modulus of (Ti, Cr)N coatings were higher than CrN and TiN. (Ti, Cr)N coatings showed the lowest steady state friction coefficient. According to the above mentioned results, it could be concluded that in many cases (Ti, Cr)N can be replaced by TiN and CrN coatings.

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M. Akbarzadeh, A. Shafyei, H. R. Salimijazi

Department of Materials Engineering, Isfahan University of Technology, Isfahan, Iran

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Physical and Mechanical Properties

Ti-Cr-N Coating

در این پژوهش پوشش‌های $(Ti, Cr)N$ ، توسط روش رسوب فیزیکی بخار با استفاده از قوس کاتدی بر روی فولاد ابزار رسوب گردید. خواص مکانیکی و ریزساختاری پوشش‌ها همچون زبری، ضخامت، ترکیب شیمیایی، فازهای تشکیل دهنده، سختی، مدول یانگ و زبری مورد مطالعه قرار گرفته است. شناسایی فازهای موجود در پوشش توسط روش پراش اشعه ایکس (XRD) انجام شد. همچنین، از میکروسکوپ الکترونی روبشی (SEM) و آنالیز تفکیک انرژی (EDX) جهت مطالعه ریز ساختار و مورفولوژی رشد و ترکیب پوشش استفاده گردید. بررسی خواص مکانیکی توسط آزمون نانو فرورونده صورت پذیرفت. رفتار اصطکاکی پوشش توسط آزمون سایش لغزشی گلوله بر روی دیسک با استفاده از گلوله‌ای از جنس کاربرد تنگستن در بارهای اعمالی ۷ نیوتن انجام گرفت. نتایج نشان داد در حالی که پوشش‌های TiN و $(TiCr)N$ دارای ساختار تک فاز مکعبی هستند، پوشش CrN از دوفاز هگزاگونال Cr_2N و مکعبی CrN تشکیل شده است. پوشش‌ها دارای اندازه دانه ۱۴ تا ۲۰ نانومتر می‌باشند. خواص مکانیکی در پوشش‌های CrN ، $(TiCr)N$ و TiN به ترتیب افزایش می‌یابد. رفتار مکانیکی پوشش $(Ti, Cr)N$ وابسته به نسبت تیتانیوم بوده، به طوری که با افزایش آن، خواص ذکر شده بهبود می‌یابند.

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