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EFFECT OF DEPOSITION PARAMETERS ON MICROSTRUCTURE AND TRIBOLOGICAL PROPERTIES OF HARD CA-PVD MULTI-COMPONENT TiAlCrN AND TiAlCrCN COATINGS

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Multicomponent TiAlCrN and TiAlCrCN coatings were deposited using vacuum arc evaporation technique on AISI 430 stainless steel. The influence of working gas pressure, C/N ratio, bias voltage on the structure and tribomechanical properties of the obtained coatings has been studied. The surface morphology, chemical compound of the coatings obtained under the various conditions has been analyzed by SEM with EDX, XRD, and XRF analysis. The dry wear pad-on-disc tests against 100Cr6 counterbody at 20 N load have been carried out. It was established that Vickers micro-hardness was varied from 26 to 41 GPa depending on deposition parameters.

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INTRODUCTION

Hard binary, ternary and multicomponent films based on transition nitrides and carbides obtained by physical vapor deposition (PVD) have been extensively studied in recent years [1, 2]. Among them TiN, TiCN, CrN, TiAlN, and TiAlCrN hard films are well known for their high hardness, good adhesion, wear and corrosion resistance and they are frequently used as protective layers on the surface of cutting and stamping tools, as well as on various mechanical components and bearing parts [3-6]. The most common TiN coating is widely used on the surface of cutting tool but it oxidizes rapidly to TiO_2 when the temperature exceeds approximately 550°C and starts delaminated [7].

TiAlN coatings outperform TiN, TiC, CrN, and Mo_2N coatings by resistance to temperature oxidation, keeping its characteristics up to 925ºC [8]. They have low friction coefficient and high hardness and are widely used in the field of high speed cutting. These properties were attributed to a stable aluminum oxide layer formed at the working surface [9]. The Al_2O_3 acts also as the dry lubrication preventing the adhesive interaction of the coating with the processed material, providing low friction in a contacting area. As a result, the impact and thermal loads tend to decrease, improving the wear resistance.

The formation of TiAlN coatings showed that due to the introduction of new elements forming multi-element nitrides by adding Si or Cr to this system makes it possible to significantly improve their mechanical characteristics and oxidation resistance by achieving a finer nanostructure and the formation of composites with high strength [10].

The TiAlCrN coated tool showed excellent performance during the dry turning 20CrMo steel in the field of cutting force, cutting temperature, surface roughness, tool life and tool wear [11]. Compared with uncoated tool, the main cutting force obtained with TiAlCrN coated tool decreased 5...18 % [12].

The quaternary compound of TiAlCrN superhard coating material was deposited on tungsten carbide (WC-Co) substrates using cathodic arc deposition techniques at the deposition temperature of 623 K. The maximum hardness and elastic modulus values as measured by nanoindentation tests are at 53 and 887 GPa, respectively [13].

Kovalev et al [14] studied nanostructured (Al,Ti)N and (TiAlCr)N PVD coatings. It is found out that grain sizes for such coatings reach 5…20 nm. Cutting tests were conducted when turning hardened tool steel H13 (HRC 50 52) and aerospace materials.

However, there are few studies on the structure and wear mechanisms of TiAlCrCN coatings.

Surface hardness of the Ti-Al-Cr-C-N and Ti-Al-Cr-Si-C-N coated samples obtained by reactive reactive cathodic arc conditions was respectively obtained 8.7 and 10.8 GPa compared to 2.16 GPa for the un-coated substrate [15].

Development of the new nanostructured coating with high hardness (40 GPa) and thermal stability (> 1200°C) is one of the most important problem of the modern material science.

The main objective of this research work was to establish a relationship between the deposition parameters (working pressure and bias voltage, C/N ratio) and the formed structural state on the tribomechanical properties of deposited TiAlCrN and TiAlCrCN coatings.

1. EXPERIMENTAL SETUP

The TiAlCrCN and TiAlCrN coatings were obtained on AISI 430 SS in "Bulat" type facility [16]. The alloyed cathode Ti(36 wt.%)Al(2.6 wt.%)Cr was used. Before deposition, the substrates were cleaned in an ultrasonic bath with alcohol for 10 min. The C/N pressure in the deposition process was about $1.5\cdot10^{-2}$ Torr. For CN coatings the N_2 and C_4H_{10} gas mixture was used. The deposition rate was ~ 30 µm/h. The deposition parameters are summarized in Table 1.

Table 1

Deposition parameters

№	Coating	C/N ratio, at.%	Bias voltage	P, C/N, Torr	h, µm
S1	TiAlCr(CN)	14/86	-50	$1.5 \cdot 10^{-2}$	11
S2	TiAlCr(CN)	12/ 88	-100	$1.5 \cdot 10^{-2}$	15
S3	TiAlCr(CN)	14/ 86	Floating	$1.5 \cdot 10^{-2}$	9.4
S4	TiAlCrN	100 N	-100	$1.5 \cdot 10^{-2}$	11

The surface topography of the coatings was studied by using JEOL JSM-6390LV scanning electron microscope (SEM) with an accelerating voltage of 20 kV, chemical composition was examined using energy-dispersive X-ray analysis (EDX). X-ray diffraction (XRD) analysis were undertaken using a DRON-3M device, under Cu–Kα radiation, made monochromatic by highly oriented (002) pyrolytic graphite (HOPG) in the diffracted beam. The XRD line scans were done in the θ...2θ scanning mode, where the incident angle θ and the diffracted angle 2θ are scanned simultaneously. Energy-dispersive spectrometer SPRUT-K was used for X-ray fluorescent analysis. Film thickness was determined by XRF examinations and presented in Table 1. The dry friction and wear tests were carried out on a block-on-disc wear testing machine of CMT-1 type. The rotating counter face was made of 100Cr6 steel disc. The rotation frequency of the rollers (counter face) was 500 min^{-1} and the load was 20 N.

2. RESULTS AND DISCUSSION

The view of the obtained coatings on AISI 430 SS samples is presented in Fig. 1. The S1 and S2 coatings are of metallic color, whereas S3 is black and S4 is bronze one.

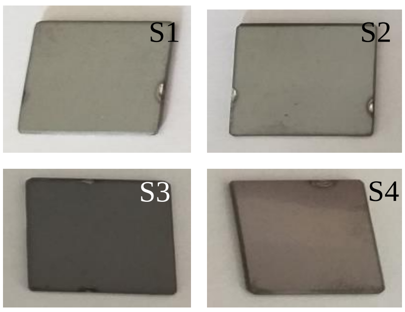

Fig. 1. Color change of the coatings due to deposition parameters

The XRD patterns for S1-S4 coatings are presented in Fig. 2. The main features of the XRD patterns for S1, S2, S4 samples are the presence of TiN and AlTi crystalline phases. Additional TiO phase was revealed for S3 type sample only. In accordance with FWHM calculations for all samples, the grain size of TiN phase was varied from 10...15 and 30...45 nm for AlTi phase, confirming formation of nano-crystalline structure. The metastable cubic ($Ti_{1-x}Al_x$)N phase may also exist which decomposes at elevated temperatures forming extremely small-scale formations of cubic TiN and cubic AlN domains, before transforming into thermodynamically stable phases (i.e. cubic TiN of the NaCl type and AlN of the ZnS-wurtzite type).

The surface morphology of the S1-S4 type's coatings and typical EDX spectra are shown in Figs. 3, 4.

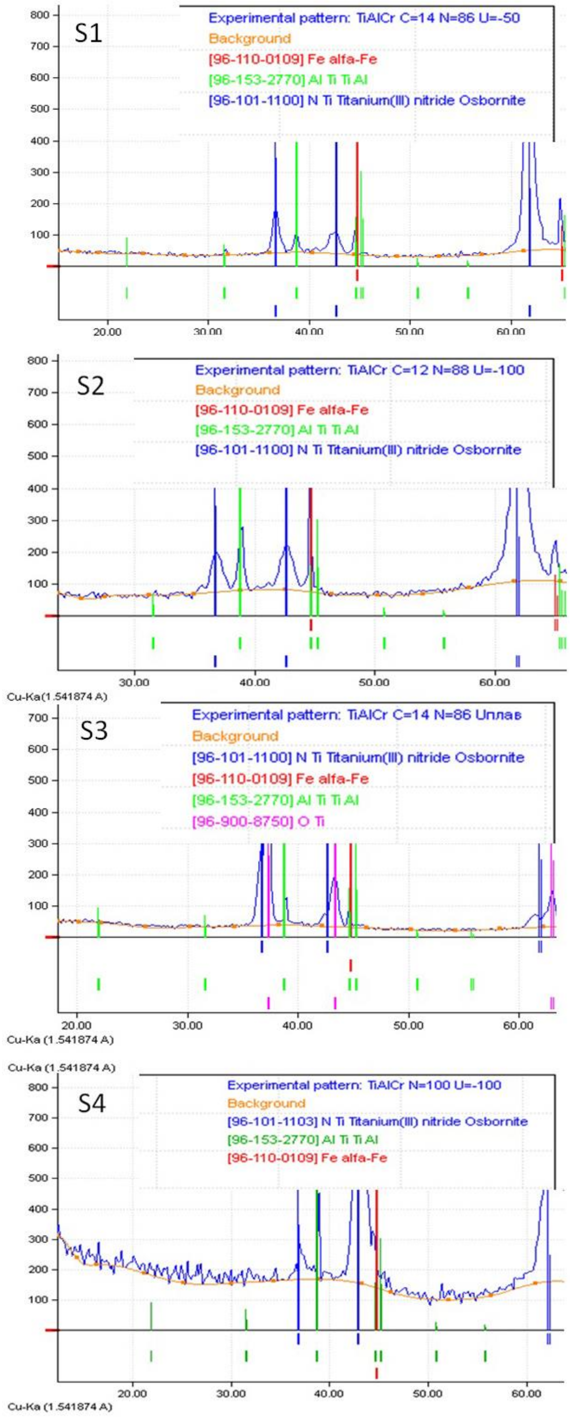

Fig. 2. XRD patterns for S1-S4 type coatings

After tribological tests, the results of weight wear loss and friction coefficients were obtained (Tables 2, 3). Table 3 presents the values of the initial microhardness and after the wear tests. The minimal value of microhardness was monitored for S1 sample

26.7 GPa, whereas the maximal comprised 41 GPa for S4 sample of TiAlCrN-type coating. The formation of oxide with high ionic potentials (TiO in S3 sample) is expected to operate as additional lubricious compounds.

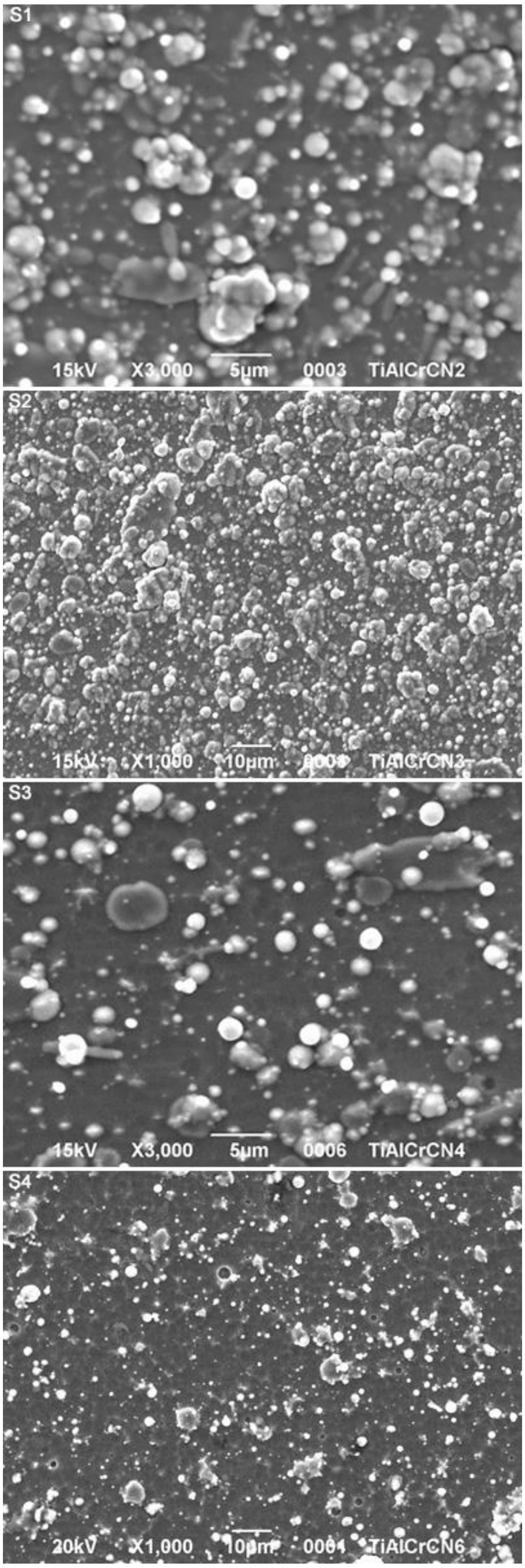

Fig. 3. SEM images for S1-S4 type coatings

We also assume that the formation of cubic TiN and AlTi phases, the construction of coherent boundaries with a cubic $(Ti_{1-x}Al_x)N$ matrix, leads to an increase in hardness, providing additional obstacles to the movement of dislocations. The refinement of the grain structure to the nanoscale level, which occurs during the formation and decomposition of a supersaturated solid solution, was noted during the preparation of a Ti-Cr-Al-N coating in [17].

When using the method of ion-plasma vacuum-arc deposition, nano-structuring is facilitated by a high degree of plasma ionization which can enhance the average energy of the deposited particles. It stimulates the processes of ordering in the coating increasing the mobility of ad-atoms on the surface of the growing film.

Minimal coefficient of friction (COF) 0.78 was revealed for S1 type coating and the maximal 0.95 for S2.

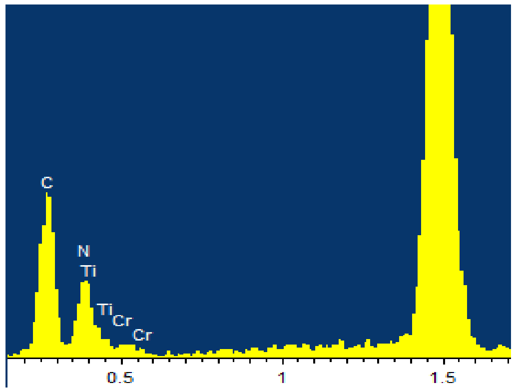

Fig. 4. Typical EDX spectra for S1-S3 type coating

Table 2

Weight wear loss

№	Weight loss, g	
	sample	roller
S1	0.0	+0.0002
S2	0.0	0.0
S3	+0.0002	-0.0006
S4	-0.0001	-0.0010
AISI 430	0.0	-0.0005

Table 3

The results of tribological tests

№	Vickers Microhardness		Friction moment	COF
	Initial	After wear tests	N × m	
S1	26.7	1.6	3.5	0.78
S2	36.5	4.7	4.5	0.95
S3	28.5	1.3	4.0	0.89
S4	41.0	3.3	4.0	0.89
AISI430	1.2	1.5	2.0	0.44

CONCLUSIONS

Multicomponent vacuum arc coatings based on TiAlCrCN and TiAlCrN have high mechanical properties with hardness variation from 27 to 43 GPa. Such high hardness of nanostructured coatings can be attributed to covalent nature, solution hardening and small crystallite size, as well as the influence of applied negative bias potential.

The structure of the coatings in all cases is formed by two nano-crystalline TiN and AlTi phases with the average size of crystallites of ~ 10…15, and 30…45 nm, respectively.

Additional TiO phase was found in TiAlCrCN coating obtained under floating potential.

The TiAlCrCN coating obtained under bias voltage of -50 V at a pressure of $1.5 \cdot 10^{-2}$ Torr has the lowest fiction coefficient and better tribological properties.

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ВЛИЯНИЕ ПАРАМЕТРОВ ОСАЖДЕНИЯ НА МИКРОСТРУКТУРУ И ТРИБОЛОГИЧЕСКИЕ СВОЙСТВА МНОГОКОМПОНЕНТНЫХ ВАКУУМНО-ДУГОВЫХ ПОКРЫТИЙ TiAlCrN И TiAlCrCN

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Многокомпонентные покрытия TiAlCrN и TiAlCrCN были нанесены методом вакуумно-дугового осаждения на нержавеющую сталь AISI 430. Изучены влияния давления рабочего газа, отношения C/N, напряжения смещения на структуру и трибологические свойства полученных покрытий. Морфология поверхности, химический и фазовый составы покрытий были исследованы с помощью SEM с EDX-, XRD- и XRF-анализов. Были проведены трибологические испытания покрытий в условиях сухого трения по схеме “диск-колодка” с контртелом из стали 100Cr6 при нагрузке 20 Н. Установлено, что микротвердость покрытий варьируется от 26 до 41 ГПа в зависимости от параметров осаждения.

ВПЛИВ ПАРАМЕТРІВ ОСАДЖЕННЯ НА МІКРОСТРУКТУРУ І ТРІБОЛОГІЧНІ ВЛАСТИВОСТІ БАГАТОКОМПОНЕНТНИХ ВАКУУМНО-ДУГОВИХ ПОКРИТТІВ TiAlCrN І TiAlCrCN

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Багатокомпонентні покриття TiAlCrN і TiAlCrCN були нанесені методом вакуумно-дугового осадження на нержавіючу сталь AISI 430. Вивчено вплив тиску робочого газу, відношення C/N, напруги зсуву на структуру і трібологічні властивості отриманих покриттів. Морфологія поверхні, хімічний і фазовий склади покриттів були досліджені за допомогою SEM з EDX-, XRD- і XRF-аналізів. Були проведені трибологічні випробування покриттів в умовах сухого тертя за схемою "диск-колодка" з контртілом зі сталі 100Cr6 при навантаженні 20 Н. Встановлено, що мікротвердість покриттів варіюється від 26 до 41 ГПа в залежності від параметрів осадження.