

Structure and Tribomechanical Properties of TiZrSiN Nanostructured Thin Films Deposited by Reactive Magnetron Sputtering

V.V. Pilko*, F.F. Komarov

A.N. Sevchenko Institute of Applied Physics Problems, Kurchatov St. 7, 220045, Minsk, Belarus

* Corresponding author: Tel.: +7 017 212 68 12. E-mail: pilkowladimir@gmail.com

Abstract

For the first time, the structure and tribomechanical properties of TiZrSiN nanostructured thin films deposited by reactive magnetron sputtering have been investigated as a function of nitrogen content. Sputtering was performed using two targets of different composition: $\text{Ti}_{0.56}\text{Zr}_{0.36}\text{Si}_{0.08}$ and $\text{Ti}_{0.36}\text{Zr}_{0.56}\text{Si}_{0.08}$ at. %. Three various types of TiZrSiN nanocomposite, formed with nitrogen deficient, nearly stoichiometric, and over stoichiometric nitrogen concentrations have been studied. Structural properties and composition of coatings were analyzed using SEM, EDX, XRD and RBS techniques. Tribomechanical properties were investigated by means of indentation, using Oliver and Pharr method, and scratch tests. The results showed that the tribomechanical properties are closely correlated with both the compositional and the structural modifications of TiZrSiN coatings induced by the addition of nitrogen in different concentrations. Thus, depending on coating composition and nitrogen content the deposited films exhibit hardness values ranged from 16 to 32 GPa. The results are intensively discussed and suggestions, explaining the influence of nitrogen concentration on structural and tribomechanical properties are made.

Keywords

Ternary nitrides; nanocomposites; coatings; thin films; magnetron sputtering; tribomechanical properties.

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1. Introduction

Transition metal nitride (TMN) films fabricated by physical vapor deposition have attracted a considerable amount of attention because of their exceptional properties [1]. TiN films, which have been broadly researched, are nowadays widely used in industry of cutting tools and as a material in prosthetics manufacture. Although the application of TiN coatings is very successful until now, its further demands, like decreasing of average friction coefficient and wear resistance in cutting processes, are motive force for further development and investigation of TiN films [2]. At present, a great attention is paid to introducing some additional elements into binary films in order to enhance the hardness and tribological properties of the films. According to Ref [3, 4], the incorporation of Si into the TMN films generally leads to the formation of nanocomposite TM-SiN films, in which TMN nanocrystallites are surrounded by a thin amorphous

Si_3N_4 layer. In recent years, the study of TiSiN films, in which Si atoms were dissolved into interstitial sites of TiN lattice or formed TiN/ Si_3N_4 nanocomposite films, shows excellent mechanical properties, such as high hardness and oxidation resistance [5, 6] that are usually strongly correlated with coating structure.

The development of sophisticated local analysis techniques such as high-resolution electron microscopy was a prerequisite for the understanding of microstructure evolution. This technique allows the nanoscale characterization of thin films as shown in Ref [7]. With the implementation of X-ray diffraction, new insights into the microstructure of thin nanocrystalline films have been gained, revealing them to be extremely complex regarding crystallographic texture and crystallite size [8].

As thin films are often subjected to severe loading conditions, the establishment of correlations between microstructure and mechanical properties, especially the hardness, is of vital importance [12, 13]. Conventional hardness measurements using

nanoindentation devices are surface sensitive; therefore, approaches to determine hardness-depth profiles have been developed. Knowledge about the local variation of microstructure and hardness in a film, grown by reactive magnetron deposition, can be essential for its further improvement. It is believable that TiZrSiN films can exhibit good mechanical properties and fine wear resistance as is the case for the similar films, such as MoAlSiN [14]. However, information available in the literature on the microstructure, mechanical and tribological properties of TiZrSiN films is very scarce [15, 16] and mostly devoted to their thermal and phase stability. The observed in present paper hardness variations are related to SEM investigations and X-ray diffraction data, providing local information on crystallographic texture and grain size. These results contribute to the basic understanding of hardness evolution of nanocrystalline TiZrSiN films with the varying growth conditions. In our study, Zr and Si atoms were incorporated into TiN film simultaneously using reactive magnetron sputtering, and for the first time, the effects of nitrogen content on the composition, microstructure, hardness, and tribological properties of TiZrSiN films were studied and discussed.

2. Experimental

Three different series of TiZrSiN films were deposited on polished silicon (111) wafers, stainless steel and hard alloy substrates by magnetron sputtering deposition technique in a reactive Ar–N₂ mixture atmosphere (99.999 % purity for each gas). The substrates were degreased in acetone and ethanol before deposition. Two composite targets of Ti_{0.56}Zr_{0.36}Si_{0.08} and Ti_{0.36}Zr_{0.56}Si_{0.08} at. % (Ø110 × 10 mm) were used. Both targets were pre-sputtered for 10 min to remove the surface oxide at a pressure of 6.0×10^{-4} Pa. The magnetron sputtering system was equipped with a planar magnetron source, the substrate-target distances were fixed at 100 mm. The substrates are mounted on a rotary sample holder manipulator to insure film homogeneity. The deposition chamber is connected to a turbo molecular pump through an adaptive pressure controller valve, which allows fixing the final total pressure in the sputtering chamber. Flow regulators are fitted for controlling independently the sputtering Ar and the reactive N₂ gases. For the first series (denoted as series A), a high DC power of 400 W was applied to the target. Before the deposition of the films, the residual pressure in the sputtering chamber was typically lower than 10^{-3} Pa. The substrate temperature was kept

constant at 300 K, negative bias of 90 V was applied to the substrates, the total working pressure, $P_{\text{tot}} = (P_{\text{Ar}} + P_{\text{N}_2})$, was fixed at 0.4 Pa. The N₂/Ar flow ratio was initially varied and made up the value of N₂/Ar = 2/10. Under these conditions, TiZrSiN samples with nitrogen deficient films were obtained. For the second and third film series (series B and C) depositions were performed in a mixed (Ar + N₂) atmosphere. During the deposition, the total pressure P_{tot} was kept constant at 0.4 Pa. The series B and C have been deposited using N₂/Ar flow ratios of 4/10 and N₂/Ar = 6/10, respectively, leading to TiZrSiN films which referred as nearly-stoichiometric films or series B and over-stoichiometric films denoted as series C. XRD analysis of as-deposited samples was employed for structural identification using a D8 Bruker AXS X-ray diffractometer operating in Bragg–Brentano configuration and equipped with CuK_α wavelength (0.15418 nm) and an energy dispersive Si(Li) detector (Sol-X detector) defined with a 0.2 mm opening angle slit. The grain size was evaluated according to the broadening of the (111) diffraction line of c-(Ti,Zr)N using the Scherrer equation, i.e. ignoring in a first approximation the contribution of microstrains. Morphologies and thickness of the TiZrSiN coatings were observed by transmission and scanning electron microscopies (SEM, Hitachi 800, Japan). Microindentation (Shimadzu Dynamic Ultra Micro Hardness Tester, Japan, applied with a Berkovich indenter) was performed to determine the coatings' hardness. The penetration depth was controlled at a level of about 10 % of the coatings thickness in order to minimize the influence of substrates. The measurements were performed with a thermal drift of less than 50 pm/s. After achieving contact between indenter and sample surface, the force was continuously raised slightly above 50 mN, after this the force was held for 5 s. Subsequently, the force was reduced to zero only interrupted by a step at 10 % of maximum force to measure thermal drift.

Composition and depth distribution of the elements in the films were studied by Rutherford backscattering spectroscopy (RBS) of helium ions using a Van de Graaff accelerator HVEE AN-2500. The energy of the analyzing ⁴He⁺ beam was 1.4 MeV, and the scattering angle was 170°. Due to low sensitivity of RBS technique to light impurities, EDX measurements were performed with the Zeiss Neon 40 Esb cross-beam microscope at the base pressure $\sim 1 \times 10^{-3}$ Pa at 5 keV, with the Oxford Instruments EDS 7424 detector. The tribological properties of the films were evaluated using a conventional disk-on-plate tribometer under a normal load of 0.2 N with a 60 mm

diameter disk made of HG012 alloy as a counterpart material. Three tests were performed for each sample, the sliding speed was 0.1 m/s. All the tests were conducted in air with a relative humidity of ~50 %.

3. Results and discussion

3.1. Elemental and phase composition

Fig. 1 depicts the RBS spectra of TiZrSiN coatings deposited at different conditions. To obtain the profiles of atomic concentrations of heavy elements the SIMNRA [17] simulation was performed.

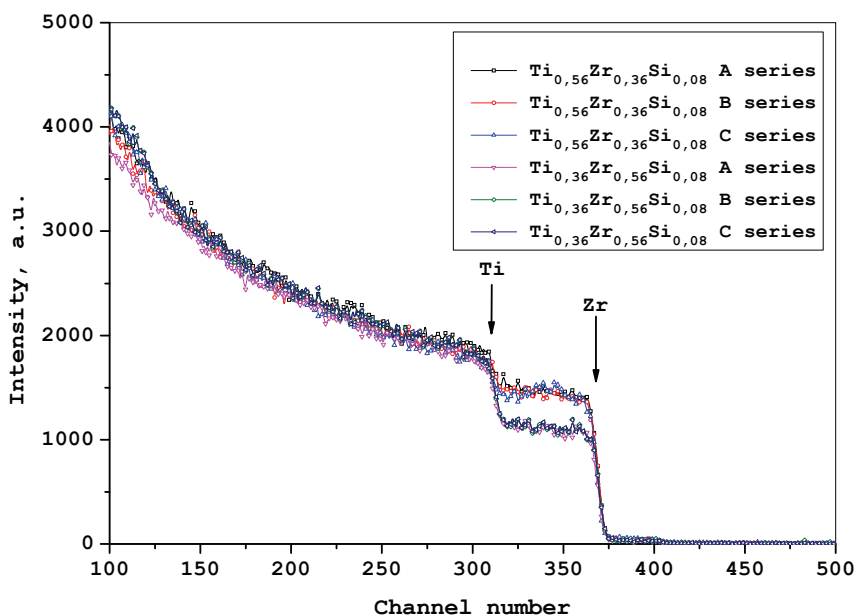


Fig. 1. The RBS spectra from a 1400 keV 4He bombardment of the TiZrSiN coatings with varying nitrogen content

RBS cross-sections for the Ti, Zr and Si and non-RBS cross sections for nitrogen elastic backscattering were used to simulate the spectrum. The difficulties to quantify nitrogen concentrations from the results of RBS technique due to its small sensitivity comparing to heavy elements were resolved by means of EDX analysis. EDX measurements of the deposited films indicates the atomic contents as listed in Table 1.

As shown in Table 1, oxygen content in these structures was presented in insignificant amounts. The Ti, Zr and Si atomic concentrations were deduced from the RBS simulation. The results of mathematical fittings of RBS spectra illustrate the homogeneous depth distributions of Ti, Zr, and Si atoms in these films. Also, from the RBS spectra it was evaluated that a concentration ratio of Ti/Zr in coatings was varied from that in the targets and corresponded 1/2 for $\text{Ti}_{0.36}\text{Zr}_{0.56}\text{Si}_{0.08}$ target and 2/1 for $\text{Ti}_{0.56}\text{Zr}_{0.36}\text{Si}_{0.08}$ one while Si concentration remains the same at the whole analyzing depth. This effect can be related to the preferential sputtering of targets during film deposition and is discussed thoroughly in Refs. [18, 19]. These results were confirmed by the EDX values shown in Table 1. The uncertainty of compositional analysis was estimated at a level of 7 %. RBS data showed that the thickness of TiZrSiN films exceeded $1 \times 10^{19} \text{ at./cm}^2$.

Table 1

Atomic content of TiZrSiN films via EDX analysis

Target composition	Deposition conditions	Atomic concentration, at. %				
		Zr	Ti	Si	N	O
$\text{Ti}_{0.36}\text{Zr}_{0.56}\text{Si}_{0.08}$	A series	36.8	18.7	7.8	34.0	2.3
	B series	29.3	15.2	7.5	46.6	1.3
	C series	22.9	10.2	8.3	57.0	1.6
$\text{Ti}_{0.56}\text{Zr}_{0.36}\text{Si}_{0.08}$	A series	20.6	41.0	7.4	29.0	2.1
	B series	16.3	33.1	8.2	41.4	1.0
	C series	10.6	21.5	7.7	58.6	1.6

Fig. 2 shows the XRD data illustrating the phase composition of TiZrSiN films. For the TiZrSiN coatings under discussion, all the observed XRD lines correspond to a (Ti,Zr)N solid solution with fcc structure. The crystallites exhibit preferred orientations, that strongly depend on depositing conditions. This behaviour is typically observed for TiZrN films deposited by magnetron sputtering [15].

Comparing the XRD patterns presented in Fig. 1, it was revealed that the increasing of Zr component content in TiZrSiN films leads to a shift of the diffraction peaks of the c-(Ti,Zr)N solid solution to lower diffraction angles, i.e. to a lower interplanar spacing. This can be attributed to the substitution of Ti atoms with Zr of bigger radius in the metal sub-lattice [18]. The lattice parameter of the c-(Ti,Zr)N solid solution is increases from the value of 0.4417 to 0.4529 nm with the increasing of Zr concentration, which confirmed the formation of a c-(Ti,Zr)N solid solution with prevalence of Zr atoms. Similar effect can be caused by residual stresses in obtained coatings [19] as soon as the deposition was held at room temperature and under different partial pressures of nitrogen. However, in this case we can expect different shifts for each depositing condition. Nevertheless, as it can be clearly seen in Fig. 1, especially for (Ti,Zr)N peaks of (111) orientation, the shifts are the same for every depositing condition.

An evolution of phase composition of the as-deposited films was revealed. The observed broadening and asymmetry of peaks profile of films with deficient and nearly stoichiometric contents of nitrogen and a significant increase of preferred (200) – orientation crystallites peaks for the overstoichiometric films can be related with a concentration of defects and nitrogen content that affect structure and phase state of TiZrSiN films as it is discussed in Ref [18]. It is necessary to keep the ratio between the metallic elements and nitrogen concentrations at the stoichiometric level to stabilize the crystalline cubic structure of films. In a case of nitrogen lack, an additional amorphous phase α -Zr(Ti)N can be formed and the structure of these films resembles that of a nanocomposite. The texture evolution is commonly

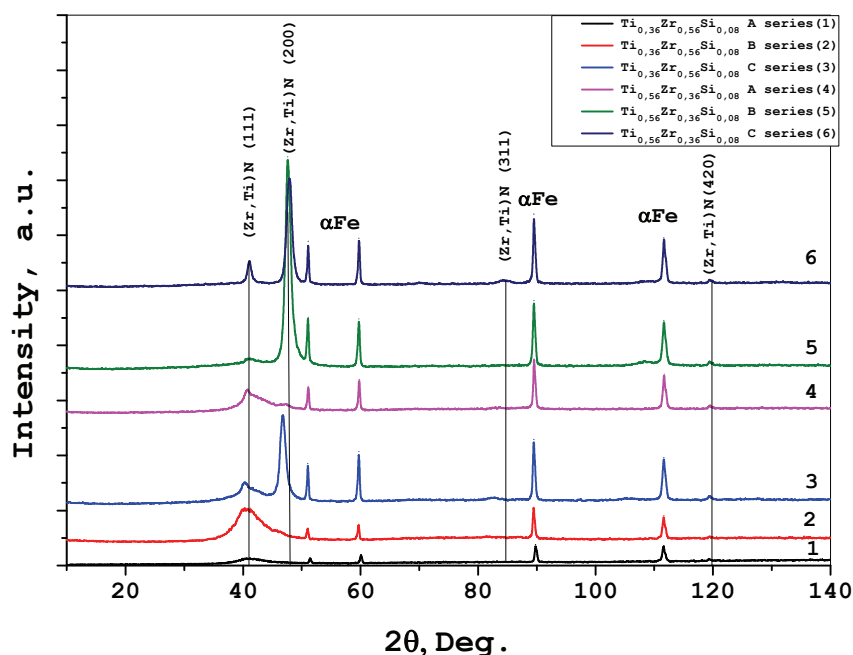


Fig. 2. XRD patterns of as-deposited TiZrSiN films formed by reactive magnetron sputtering of targets with different composition: with deficient (A), stoichiometric (B) and overstoichiometric (C) concentrations of nitrogen

ascribed to the surface energy [19]. Thompson proposed that the preferred orientation of transition metal nitride films associates with the competition between the surface energy and strain energy. During deposition process of the TiZrSiN coatings, the substrate bias was applied inducing the high energy bombardment of growing films by particles, which is proportional to bias. In this case, the strain energy has a dominant contribution to the total free energy minimization for the films and the preferred orientation corresponds to the plane with the lowest surface energy, i.e. (111). With increment of nitrogen partial pressure, we can expect attenuation of particle bombardment effect and thus, surface energy prevails that leads to the preferred orientation (200) as it is now corresponds to the plane with the lowest surface energy [20].

The average grain sizes of the coatings were estimated by the Debye – Scherrer formula, using the full width at half maximum (FWHM) value of the (111) XRD diffraction peak. The calculated grain sizes of TiZrSiN coatings versus nitrogen content in the coatings are plotted in Fig. 3. It can be seen that the grain size increases from (12.7 ± 3.9) nm up to (32.2 ± 7.1) nm for $\text{Ti}_{0.36}\text{Zr}_{0.56}\text{Si}_{0.08}$ coatings and from (15.2 ± 4.6) nm up to (41.5 ± 5.3) for $\text{Ti}_{0.56}\text{Zr}_{0.36}\text{Si}_{0.08}$ films with increasing of nitrogen content.

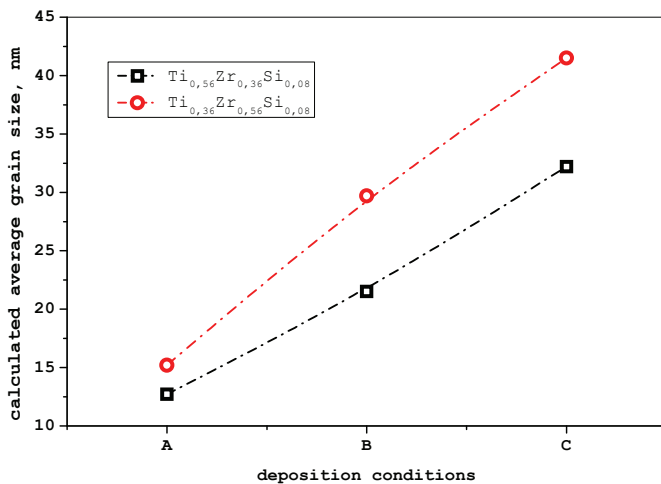


Fig. 3. The average grain sizes of TiZrSiN coatings versus nitrogen content

The calculated values are in a good agreement to those obtained directly from SEM images of the coatings that are shown below. During deposition of TiZrSiN coatings, the increasing of nitrogen flow leads to a decrement of deposition rate. Lower deposition rate means less sputtered atoms arriving at the surface

of the substrates per unit of time. Consequently, less nucleation sites are provided and this results in the grain size coarsening. Fig. 4 shows the cross-sectional SEM and TEM images of $\text{Ti}_{0.36}\text{Zr}_{0.56}\text{Si}_{0.08}$ coatings with varying nitrogen content. The same structure behavior with slight differences in grain sizes is observed for $\text{Ti}_{0.56}\text{Zr}_{0.36}\text{Si}_{0.08}$ coatings (not presented).

Since the ratio of deposition temperature T_s (300 K) to ZrN melting point T_m (2980 K) or to the same parameter of TiN (3200 K) is approximately 0.1, the microstructure of TiZrSiN coatings is expected to be corresponding to Zone T according to the structure zone model in Ref [21]. Indeed, the SEM cross-sectional morphologies of TiZrSiN coatings deposited under deficient and stoichiometric conditions (A and B series in Fig. 4) are in a good agreement with the primary feature of Zone T structure. As the nitrogen concentration exceeds nearly stoichiometric value, there are observed drastic changes in the dimensions and geometric shape of the structure. The size of crystallites rises with increasing of nitrogen content as it can be seen in Fig. 3, which is in accordance with the average grain size obtained by calculations.

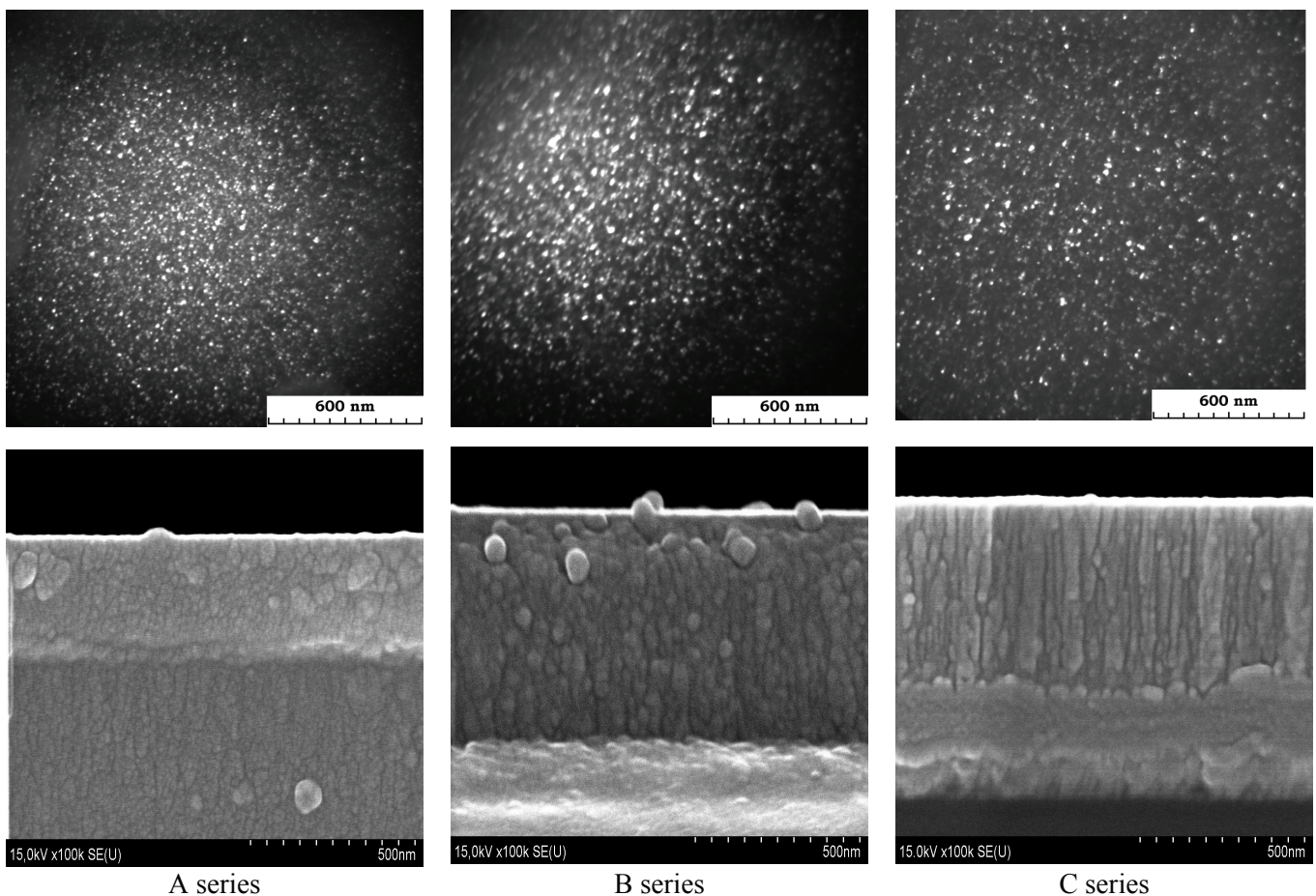


Fig. 4. The cross-sectional SEM and TEM images of the $\text{Ti}_{0.36}\text{Zr}_{0.56}\text{Si}_{0.08}$ coatings with varying nitrogen content

3.2. Mechanical properties

Fig. 5 shows the evolution of the friction coefficients as a function of the sliding time. The value of friction coefficient varied in the range of 0.2 – 0.6 at the steady state. A slight diminution of the friction coefficient was observed (tracks Ti++A and Zr++A) for films deposited with deficient of nitrogen content. As reviewed in Ref [22], this slight reduction in terms of the friction coefficient could be explained by the formation of the self-lubricant tribolayers. In these particular nanocomposites, SiO₂ and/or Si(OH)₂ could form during wear tests at humid environments [23] as soon as due to a lack of nitrogen not entire silicon will form a-Si₃N₄ phase.

On the contrary, a slight growth and fluctuation of friction coefficient values are observed during the tribological tests (tracks Ti++B, Zr++B, Ti++C, Zr++C). This behavior could be related to the debris produced during the tribological tests. These debris is supposed to remain in the local regions of the wear track, hence causing the variation in friction coefficient [24]. A higher friction coefficient of coatings deposited at so-called “over – stoichiometry” conditions and comparable to those for stainless steel samples is usually ascribed to high debris generation on the wear track during disc-on-plate tests. Similarly, the wear behavior was correlated with the friction coefficient evolution.

As reviewed by Leyland and Matthews [25, 26], the wear behavior is usually determined by the H/E ratio. In this study, the dimensionless parameter was calculated for all samples. In fact, with increase of nitrogen content, the decreasing H/E parameter from

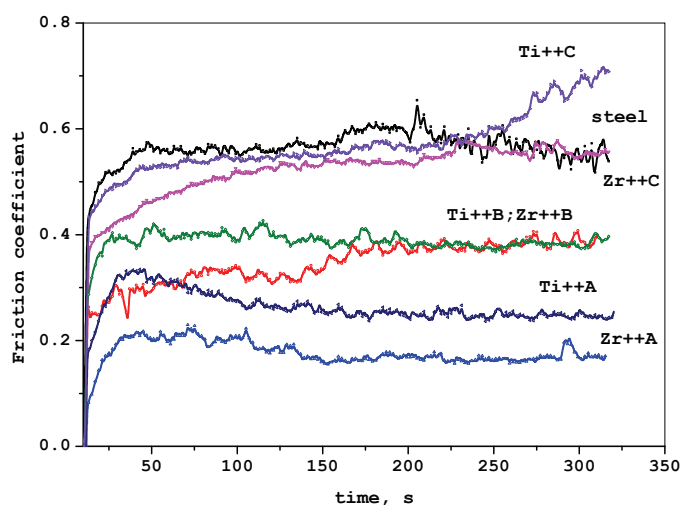


Fig. 5. Friction coefficient values versus wear time measured for TiZrSiN films obtained by means of reactive magnetron sputtering on the steel (AISI 321) substrate

(0.095 ± 0.002) to (0.069 ± 0.001) for Ti_{0.56}Zr_{0.36}Si_{0.08} coatings and from (0.077 ± 0.0017) to (0.053 ± 0.002) for Ti_{0.36}Zr_{0.56}Si_{0.08} films was obtained. These values are in a reasonable agreement with the apparent increasing of friction coefficient shown in Fig. 5.

The load-displacement curves of TiZrSiN coatings are plotted in Fig. 6. The indentation hardness for Ti_{0.56}Zr_{0.36}Si_{0.08} (Ti_{0.36}Zr_{0.56}Si_{0.08}) coatings first increases from (14.7 ± 0.8) GPa (21.6 ± 0.5 GPa) for the nitrogen deficient films to (18.4 ± 0.5) GPa (32.3 ± 0.5) GPa for nearly – stoichiometric films, and it is followed with a decrement to (16.9 ± 0.5) GPa (19.4 ± 0.7) GPa for over – stoichiometric films. According to the Hall–Petch effect in nanostructured materials, the hardness of coatings increases with decreasing grain size. Therefore, the hardness improvement should be attributed to both the solid solution strengthening (for Zr – prevailing coatings) and Hall–Petch effect. Considering a previous study of nanostructured coatings [27], lower hardness of films with finer grains (deficient of nitrogen) comparing to

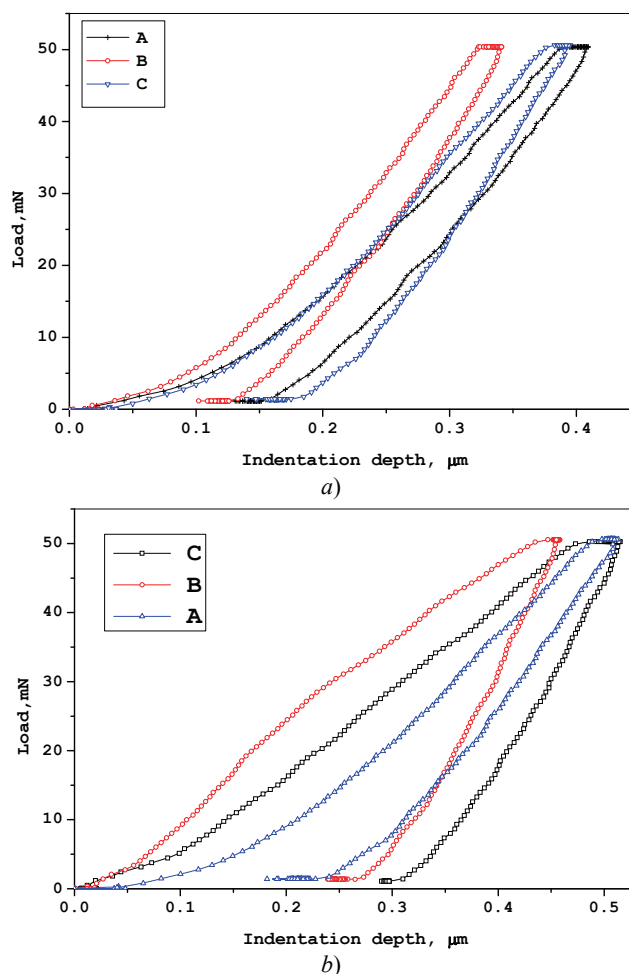


Fig. 6. Comparing the load – displacement curves from microindentation measurements for the TiZrSiN coatings: a – Ti_{0.36}Zr_{0.56}Si_{0.08}; b – Ti_{0.56}Zr_{0.36}Si_{0.08}

that with coarser ones (nearly – stoichiometric) can be attributed to deviations from grain boundary hardening described by the Hall-Petch relationship which is based on the dislocation pile-up mechanism: so-called inverse Hall-Petch effect [28]. Considering oxidation effects, a low hardness of coatings with deficiency of nitrogen may be related to the minor traces of surface oxides formation, which means reducing of nitrogen content diminishes the chemical stability of coatings. More surface oxides would be expected to form on the TiZrSiN coatings with lower nitrogen content, which will produce more detrimental effects on the hardness.

Conclusion

TiZrSiN coatings were deposited by magnetron sputtering with variation of reactive gas partial pressure using two composite targets of $\text{Ti}_{0.56}\text{Zr}_{0.36}\text{Si}_{0.08}$ and $\text{Ti}_{0.36}\text{Zr}_{0.56}\text{Si}_{0.08}$ at. %. Mechanical properties of the sputtered nanocrystalline TiZrSiN thin films have been investigated by microindentation and structure related alterations have been observed. The local growth conditions, adjusted by the different nitrogen partial pressure are identified as mainly determining the hardness due to the resulting variation in grain size and primary nanocrystallite orientation. Increasing nitrogen content during deposition process leads to the evolution of solid solution grains sizes from highly distorted small crystals to well-developed coarsened columnar grains and the structures from (100) to (200) preferred orientation. A moderate nitrogen addition strongly improves the hardness of the TiZrSiN coatings due to both the solid solution strengthening and Hall-Petch effect. The highest recorded hardness of 32.3 GPa is achieved for the coatings with prevailing of Zr atoms content at so-called “nearly – stoichiometric” conditions. Further nitrogen addition drastically deteriorates mechanical properties of the coatings. The observed findings confirm the general notion of a growth-related hardness variation in single-phase nanocrystalline thin films.

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