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MECHANICAL AND TRIBOLOGICAL PROPERTIES OF NANOLAYERED TiAlN/TiSiN COATING

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Abstract: *Ceramic hard coatings, such as TiN, TiAlN, and TiSiN have been widely used to improve surface properties of mechanical components. For highly demanding tribological applications, such as high speed machining without lubrication, ceramic coating should be characterized by high hardness, appropriate toughness, high thermal stability, and oxidation resistance, which can be challenging to achieve in one coating. In order to meet these challenges, TiAlN and TiSiN layers were deposited alternatively to produce nanolayered TiAlN/TiSiN coating. Industrial magnetron sputtering unit with two TiAl and two TiSi targets was used for coating preparation. Single-layer TiAlN coating was additionally deposited for comparison. Mechanical properties were measured by nanoindentation technique. Ball-on-plate test (with alumina ball) was carried out in air to evaluate coating tribological properties. Tribo-tracks were studied by scanning electron microscopy, stylus profilometry and energy dispersive X-ray spectroscopy. Similar values of friction coefficient of around 0.85 were measured for both coatings. On the other hand, wear rate of nanolayered TiAlN/TiSiN coating of 9.1 × 10^{-6} $mm^3N^{-1}m^{-1}$ was significantly lower from ~14 × 10^{-6} $mm^3N^{-1}m^{-1}$ as measured for TiAlN coating. Lower ball wear rate was also found for the nanolayered coating. Higher wear resistance of the nanolayered coating is attributed to its better mechanical properties. This coating exhibits hardness of 38.9 GPa, while for single-layer TiAlN coating hardness of 25.2 GPa was measured. Nanolayered TiAlN/TiSiN coating is also characterized by high value of H^3/E^{*2} ratio of 0.85 GPa, which is much higher from 0.12 GPa as calculated for TiAlN coating. Recently, this ratio was recognized as the indicator of the wear resistance of hard coatings, with higher values of this ratio corresponding to higher resistance to crack formation and propagation.*

Keywords: *nanolayered coating, TiAlN, TiAlN/TiSiN, magnetron sputtering, H^3/E^{*2} ratio, wear, friction.*

1. INTRODUCTION

Hard thin coatings have been successfully used for increasing the lifetime and performances of different types of tools and mechanical parts for more than four decades. Early hard coatings, such as TiN which are characterized by hardness of around 27 GPa [1] played an important role in first decades of protection of tool surfaces. However, TiN oxides already at 500 °C [2], which is low for highly demanding tribological applications, such as high speed machining without lubrication. Properties of TiN coatings can be greatly enhanced by addition of Al which results in formation of TiAlN films, which are characterized by high hardness (≈ 30 GPa), temperature stability up to 900 °C, and high oxidation resistance [3].

Nowadays, the majority of research is dedicated to nanocomposite and nanolayered coatings. TiSiN is one of largely studied nanocomposite coatings [4-10]. It is composed of TiN nanocrystals surrounded by Si_3N_4 tissue, and is characterized by superhardness (> 40 GPa), high oxidation resistance and thermal stability up to 1100 °C [11]. Nanolayered coatings consist of few nm thin layers of two or more materials and are characterized by higher hardness [12-14], toughness [12,15] and oxidation resistance from constituting layers [16]. Some of material combinations used for preparation of nanolayered coatings are TiAlN/Si_3N_4 [12], CrAlN/SiN_x [13], TiAlN/CrAlN [14], AlTiN/CrN [16] and others.

In this research, TiAlN and TiSiN layers were deposited alternatively to produce a nanolayered TiAlN/TiSiN coating. Such coating has the potential application in dry high speed milling of hardened materials [17]. Mechanical and tribological properties of this and TiAlN coating are presented in the article.

2. EXPERIMENTAL DETAILS

Both coatings were deposited in an industrial unit (CC800/9) equipped with four magnetron sources (two TiAl and two TiSi). During preparation of the nanolayered TiAlN/TiSiN coating all four targets were active, while only TiAl targets were used for the deposition of TiAlN coating. The paragraph indentation is to be 5 mm. Coatings were deposited on AISI D2 cold work tool steel. More information about coating preparation can be found in the reference [18].

Mechanical properties were measured by a Fischerscope H100C indenter equipped with Vickers indenter. The maximum penetration depth was kept bellow one tenth of the coating thickness. Oliver and Phar method [19] was applied to calculate hardness (H) and elastic modulus (E). Ball-on-plate test was carried out in air to evaluate coating tribological properties. Alumina ball with diameter of 6 mm was used as a counter-body. Normal load of 5 N, linear speed of 5 cm/s and stroke length of 5 mm were used during all tribology tests. Number of cycles was varied between 1000 and 3000. Cross sectional measurements of the wear tracks were made by 2D stylus profilometry, while 3D profilometry was used for studying the surface topography prior to wear tests. Morphology and elemental analysis of tribo tracks were studied by scanning electron microscopy and energy dispersive X-ray spectroscopy, respectively.

3. RESULTS AND DISCUSSION

Mechanical properties of studied coatings are presented in Table 1. Nanolayered coating is characterized by higher hardness, lower modulus of elasticity and higher resistance to plastic deformation (H^3/E^{*2}). Different mechanical properties of these coatings can be attributed to different architecture and microstructure. While TiAlN coating grows in columnar fashion, nanolayered coating is dense and composed of nanocrystals which size is around 5 nm. At low indentation loads, TiAlN coating can easily deform by dislocation motion, while dislocation activity is almost completely reduced in 5 nm sized crystals [18].

Table 1. Mechanical properties of TiAlN and TiAlN/TiSiN coating.

	TiAlN	TiAlN/TiSiN
H [GPa]	25.2	38.9
E [GPa]	372.0	247.6
H^3/E^{*2} [GPa]	0.12	0.85

Images of surface topography are presented in Figure 1. Nanolayered coating is considerably smoother than single layer TiAlN coating ($Ra_{TiAlN/TiSiN}$ = 30 nm, Ra_{TiAlN} = 66 nm). Surface of TiAlN coating is characterized by greater density of defects which are typical for PVD grown coatings [20]. Lower number of such defects in nanolayered coating can be attributed to the interrupted growth due to alternative deposition of different materials [21,22].

Evolution of friction coefficient with number of tribo-test cycles is presented in Figure 2. Friction coefficient of both coatings increases rapidly in first 50 cycles and reaches value of around 0.5. After small decrease, friction coefficient continues to grow and for TiAlN coating reaches steady value of around 0.8 after 2000 cycles. Steady state for TiAlN/TiSiN coating was reached close to 3000 cycles, with the friction coefficient around 0.9.

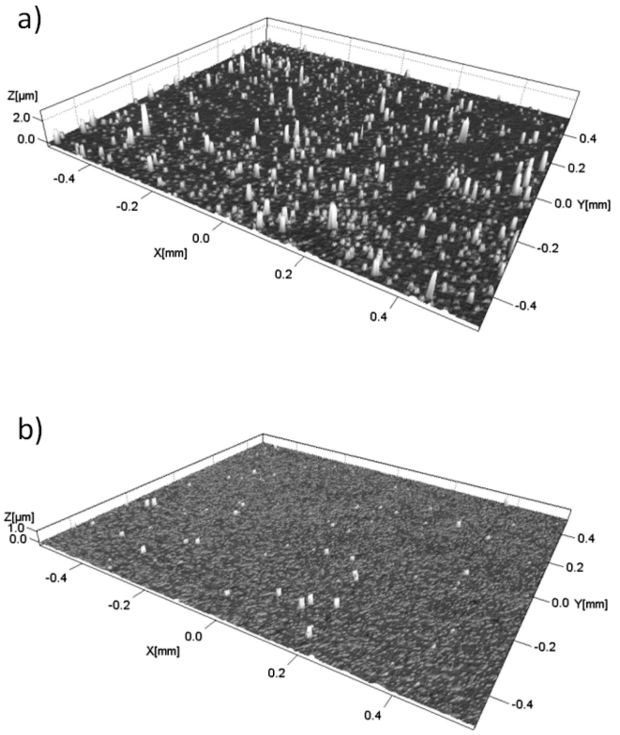


Figure 1. 3D surface topography of: (a) TiAlN and (b) TiAlN/TiSiN coating

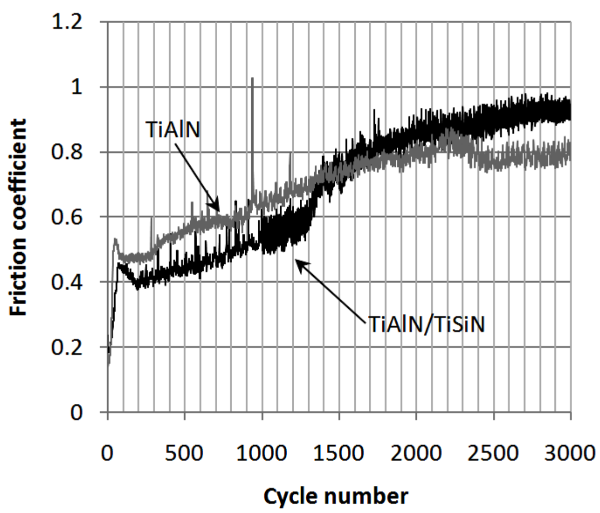


Figure 2. Friction curves of TiAlN and TiAlN/TiSiN coating

In order to calculate the wear loss of coatings, 2D profiles of formed tribo tracks are acquired. Profiles of wear tracks of test with different number of cycles are presented in Figure 3. It can be seen that neither coating was completely wear-off after 3000 cycles. Depth and especially width were larger for single layer coating. For this coating average value of wear rate was ~ 14 × 10^{-6} $mm^3N^{-1}m^{-1}$ which is higher from 9.1 × 10^{-6} $mm^3N^{-1}m^{-1}$ as measured for nanolayered coating (Fig. 4).

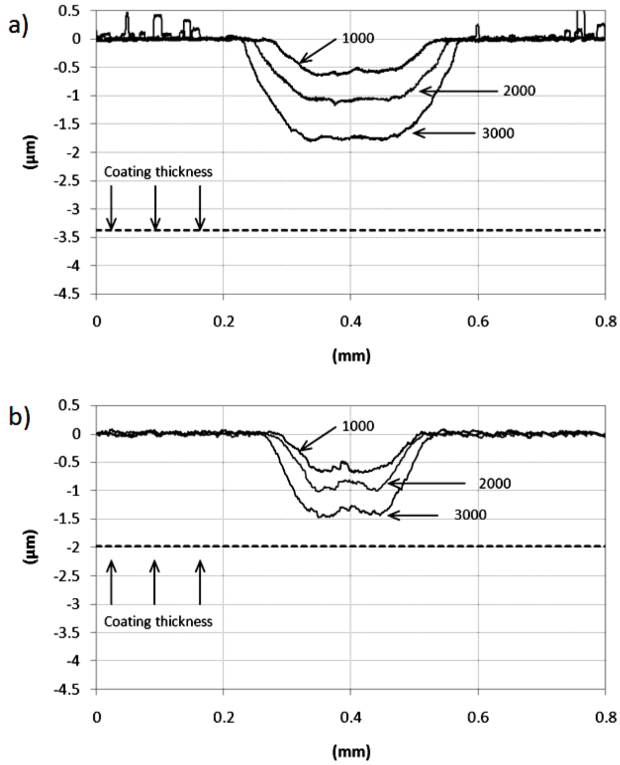


Figure 3. 2D wear track profiles of: (a) TiAlN and (b) TiAlN/TiSiN coating

Values of wear rate and friction coefficient averaged for different number of cycles are presented in Figure 4. Coatings are characterized by coefficient of friction between 0.8 and 0.9 which is typical for hard coatings composed of Ti, Al, Si and N sliding against Al_2O_3 [22-24].

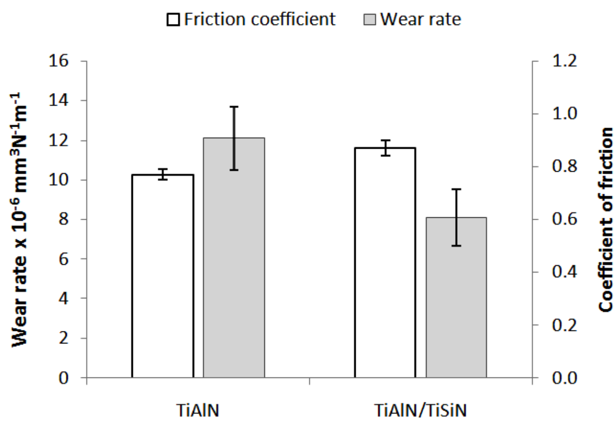
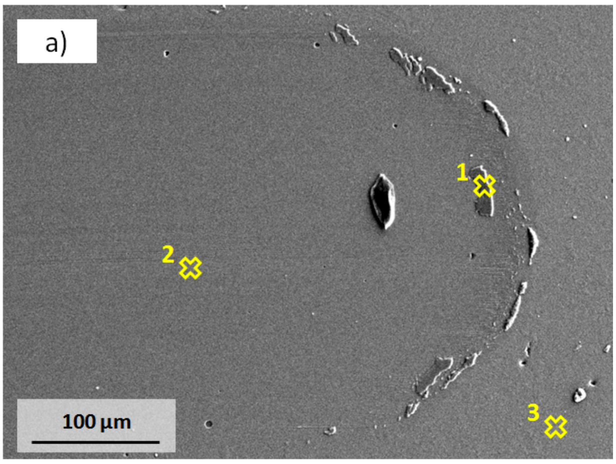
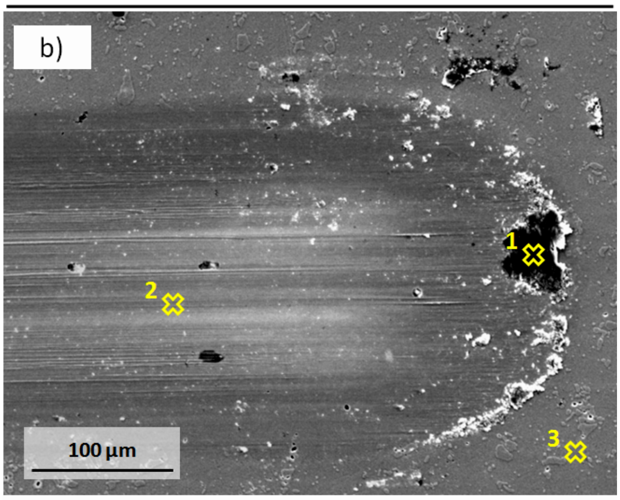


Figure 4. Wear rate and friction coefficient of TiAlN and TiAlN/TiSiN coating

In order to better understand tribological behaviour of studied coatings, wear tracks were analyzed by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). SEM images of wear tracks generated after 2000 cycles are presented in Figure 5. These images show that wear tracks are relatively smooth and clean, with low number of wear debris. Wear debris are mostly found at the end and less at the edges of wear tracks.



Point	Atomic percent (at.%)				
	O	N	Al	Ti	Fe
1	51.66	19.31	17.13	11.9	
2		51.93	26.27	21.29	0.5
3		51.05	26.33	22.62	



Point	Atomic percent (at.%)							
	O	N	Al	Si	Ti	V	Cr	Fe
1	58.4	7.08	8.82	3.07	22.2			0.4
2		34.9	11.4	4.96	23.8	1.2	8.18	15.6
3		35.9	16.1	5.87	40.9			1.23

Figure 5. SEM images of: (a) TiAlN and (b) TiAlN/TiSiN coating. Results of EDS analysis are presented bellow images. EDS spectra were acquired at places marked with a cross

EDS analysis reveals that there is around 50 at. % of oxygen inside wear debris, while no oxygen was detected in and around wear tracks. Since amount of oxygen and aluminium inside wear debris are not typical for stoichiometric Al_2O_3, it can be assumed that these debris are oxides formed during tribo test. Oxides are formed due to heating induced by friction [25,26]. According to elemental analysis, TiO_2 [27], Al_2O_3 [28], and smaller amount of SiO_2 [29] could be formed. High hardness, i.e. high shear strength of studied coatings, along with the presence of hard TiO_2 and Al_2O_3 oxides are key factors leading to high friction in tribological contact of these coatings and Al_2O_3 ball.

From SEM and EDS analysis of wear tracks it can be concluded that coatings are worn by mild abrasion and tribochemical mechanism. Lower values of wear rates of nanolayered coating can be attributed to its higher hardness and higher resistance to crack propagation. In TiAlN coating, which is characterized by columnar structure [18], cracks can easily form and propagate fast between columnar grains [30]. Contrarily, in nanostructured and nanolayered coatings cracks have to bend around nanograins, and at the layer interfaces which is positive because large amount of deformation energy is required for crack propagation [31,32]. Recently H^3/E^{*2} ratio has been recognized as the indicator of resistance to wear [33]. Higher wear resistance of nanolayered coating can be also attributed to higher values of this parameter (see Table 1). In addition, higher amount of surface defects found in TiAlN coating could also lead to higher wear rates of this coating [34].

4. CONCLUSION

Mechanical and tribological properties of single layer TiAlN and nanolayered TiAlN/TiSiN coating are presented in this study. It has been shown that special nanostructure provides higher hardness as a consequence of hindering of dislocation motion, higher resistance for crack generation and propagation, and smoother surface topography. As a result, nanolayered coating exhibited significantly higher wear resistance from the single layer TiAlN. Mechanisms of mild abrasion and tribochemical wear were found for both coatings. High coating hardness and formation of hard oxides were key factors leading to high friction in tribological contact of these coatings and Al_2O_3 ball.

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