

# Mechanical and Tribological Characteristics of Hard Multilayer Coatings Deposited by Arc Cathodic Technique

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## Abstract

*Mechanical and tribological properties of the alternate TiN/TiAlN, ZrN/TiAlN, TiSiN/Ti and TiSiN/Cu multilayer coatings prepared by the cathodic arc technique were investigated. The microhardness, thickness, adhesion, friction coefficient and wear resistance measurements were carried out. The properties of these multilayer's were compared with those of TiAlN, ZrN and TiSiN monolayer's.*

**Keywords:** *Multilayer coatings, cathodic arc method, tribological performance*

## Introduction

Multilayer structures have been used for many years in the coating technology for improving the performance of hard coatings for various machining applications [1]. Due to the requirements for the materials used as protective coatings such a high hardness, good adhesion at the substrate, low reactivity and friction with the materials in contact, an optimum solution seems to be a multilayer coating.

This coating structure consists of an inner layer with a good adhesion, one or more intermediate layers having high hardness, strength and toughness and an outer layer with a low reactivity and friction coefficient [2].

In recent years, nanometer scale multilayer and hard coatings called heterostructures proved to be very attractive for wear-corrosion protection of materials. This paper reports the results of the investigation of alternate TiN/TiAlN, Zr/TiAlN, TiSiN/Ti and TiSiN/Cu multilayer coatings prepared by the cathodic arc method. The films were analyzed in terms of mechanical characteristics and tribological performance.

## Experimental

The hard multilayer coatings were prepared in a cathodic arc deposition unit [3]. The deposition chamber was equipped with two cathodes made of Ti and TiAl alloy, Zr and TiAl alloy, respectively Ti and TiSi alloy.

The coatings were deposited on high speed steel. Specimens to be coated were ultrasonically cleaned with trichloroethylene and mounted on a rotating holder inside the chamber.

The multilayer structures were prepared by using two rotating shutters placed in front of each cathode. The shutters were periodically open and close so that Ti, TiAl, Zr, TiSi atoms were alternatively introduced in the reactive atmosphere for a certain duration.

Vickers microhardness HV was measured at an applied load of 0,1 N. Scratch tests under standard conditions were undertaken to determine the coating adhesion. The critical load  $L_c$ , was measured by optical microscopy.

Film thickness was evaluated by microscope examination of the cross section through the coating. The bilayer period was calculated from the overall thickness of the multilayer and the number of the bilayer.

Tribological performance of the coatings (sliding friction coefficient and wear) was investigated by using a testing apparatus mainly consisted of a coated sample pressed on a rotated steel disc for TiN/TiAlN, ZrN/TiAlN, coatings and a CSM tribometer for TiSiN/Ti and TiSiN/Cu films.

## Results and Discussion

### TiN/TiAlN Multilayer Coatings

The main process parameters both for TiAlN single layer and for the TiN/TiAlN multilayer's with various bilayer periods were as follows: reactive atmosphere  $-N_2$ , gas flow rate 150sccm, arc current 90 and 100 A at the TiAl and Ti cathode respectively, substrate bias 220V, deposition time 60 min, deposition rates of TiN and TiAlN layers 0,9nm/s. The overall thickness of the coatings was controlled to be of about 3,5 $\mu$ m.

The main mechanical properties of the coatings (microhardness, adhesion, bilayer period) are summarized in Table 1.

**Table 1.** Mechanical characteristics

Coating	HV <sub>0.015</sub> (GPa)	$L_c$ (N)	$\Lambda$ (nm)
TiN	21.8	52	-
TiAlN	23.3	47	-
TiN/TiAlN-12	26.2	46	600
TiN/TiAlN-360	29.1	49	20
TiN/TiAlN-720	29.4	48	10
TiN/TiAlN-1500	23.5	47	4.5

The TiAlN films had higher microhardness than that of the TiN.

For the multilayer's, the microhardness values first increase (from 26.2 to 29.4GPa) with the decreasing bilayer period (from 600 to 10nm), followed by a decrease to 23.5GPa for  $\Lambda = 4.5nm$ . TiN single layer coatings exhibited the best adhesion ( $L_c=52N$ ).

The wear behavior of the coatings can be examined in Figs.1 and 2 [4], where dependences of the friction coefficient and of the worn layer thickness on sliding distance  $L_c$  are illustrated. In the first stage of the test ( $L < 100\text{m}$ ), a marked increase of the wear with the sliding distance can be observed.

This can be understood as a consequence of the decrease of the surface roughness occurring at the beginning of the wear process. In the following stage, the friction coefficients of the coatings remain constant, whereas the worn layer thickness increases, with a slope depending on the coating type. The best wear resistance and the lowest friction coefficient were measured for the TiN/TiAlN-720 multilayer ( $\Lambda = 10\text{nm}$ ).

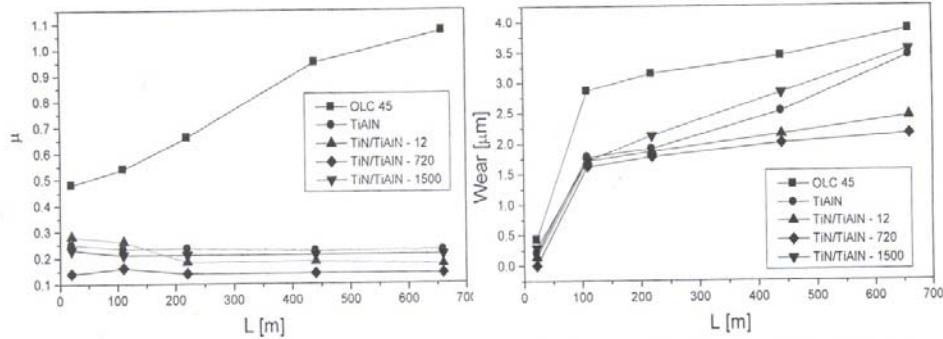


Fig. 1. Friction coefficient vs. sliding distance

Fig. 2. Worn layer thickness vs. sliding distance

### ZrN/TiAlN Multilayer Coatings

The main process parameters both for the TiAlN and ZrN monolayer's and for the ZrN/TiAlN multilayer's were as follows: reactive atmosphere  $\text{-N}_2$ , gas flow rate  $\text{-50 sccm}$ , arc current  $\text{-90}$  and  $\text{130 A}$  at the TiAl and Zr cathode, respectively, substrate bias  $\text{100 V}$ .

The deposition rates of ZrN and TiAlN layers were chosen to be of  $\text{1.6}$  and  $\text{0.6 nm}$ , so that the ratio of the thickness of the ZrN and TiAlN individual layers in a bilayer structure was of  $\text{2.7}$ . The overall coating thickness was  $\text{3.9 } \mu\text{m}$ .

The main mechanical characteristics of the coatings are summarized in Table 2, where  $\Lambda$  is bilayer period,  $\text{HV}_{0.010}$  microhardness,  $L_c$  critical load and  $D_r$  deposition rate. The microhardness and the critical load were measured for films deposited on high-speed steel substrates.

Table 2. Mechanical characteristics

Coating	$\Lambda$ (nm)	$\text{HV}_{0.010}$ (GPa)	$L_c$ (N)	$D_r$ (nm/s)
ZrN	-	26.1	43	1.6
TiAlN	-	21.2	54	0.6
ZrN/TiAlN-360	22	24.8	46	1.
ZrN/TiAlN-720	11	28.3	45	1.1
ZrN/TiAlN-1440	5.5	28.3	48	1.1

The values of the friction coefficient measured for a sliding distance of  $\text{100 m}$  are listed in Table 3.

Table 3. Friction coefficient of the coatings

Coating	Sample uncoated	ZrN	TiAlN	ZrN/TiAlN-720	ZrN/TiAlN-1440
$\mu$	0.9	0.225	0.675	0.325	0.450

The  $\mu$  lowest values were found for the ZrN film (0.225), followed by the super lattices (0.325-0.450) and the TiAlN coating (0.675)

The wear behavior of the various coatings in the salt water can be examined in figure 3, where the depths of the worn traces after 100m sliding distance are illustrated [5].

As compared to the uncoated samples, all films exhibited superior wear behavior under corrosive conditions. The best wear resistance was found for the super lattices with  $\Lambda$  of about 5nm.

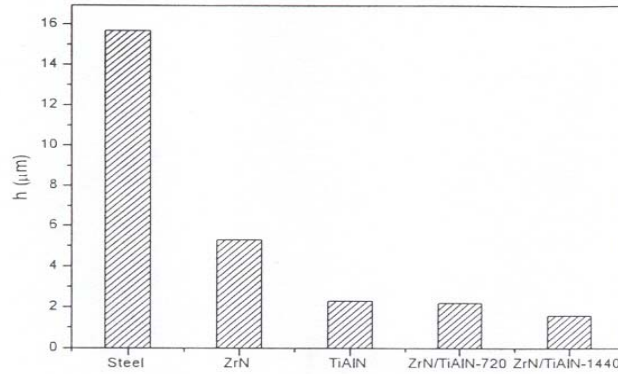


Fig. 3. Depth h of the worn trace

#### TiSiN/Ti and TiSiN/Cu Multilayer Coatings

The main deposition parameters were as follows: reactive atmosphere  $-N_2$ , gas flow rate 50sccm, arc current 90A for TiSi and Ti cathode and 60 A for Cu cathode, substrate bias 100V, deposition time 60 min. The coating characteristics are presented in the Table 4.

Table 4. The coating characteristics

Coating	Coating characteristics				
	n	$D_{Me}(nm)$	$D_N(nm)$	$\Lambda (\mu m)$	$HV_{0.015}$
TiSiN	1	-	-	2.3	2910
TiSiN/Cu-9	9	120	250	1.8	2330
TiSiN/Cu-360	360	3	6.3	1.7	2330
TiN	1	-	-	2.7	2330
TiN/Cu-9	9	42	300	1.7	1940
TiN/Cu-360	360	7.5	7.5	1.6	1910
TiSiN/TiSi-9	9	470	470	4.2	1960
TiSiN/TiSi-360	360	12	12	4.3	1460

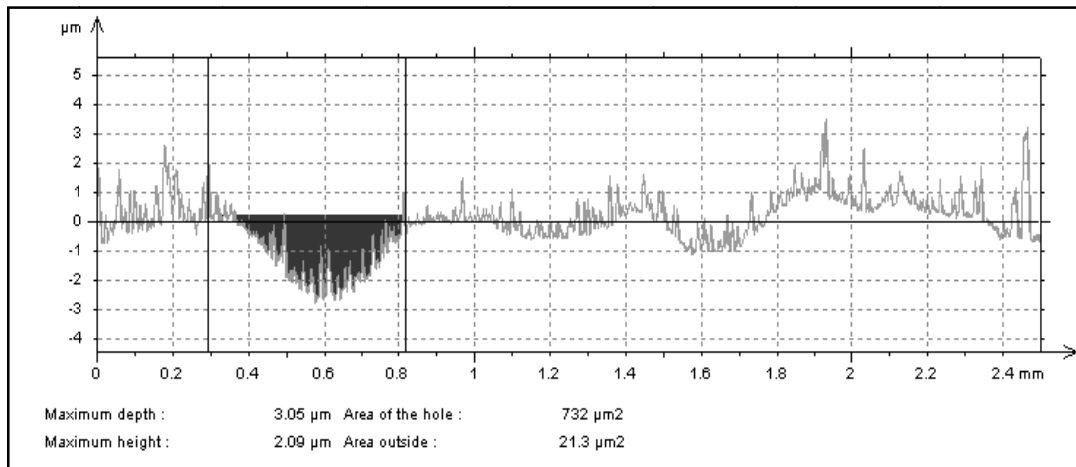
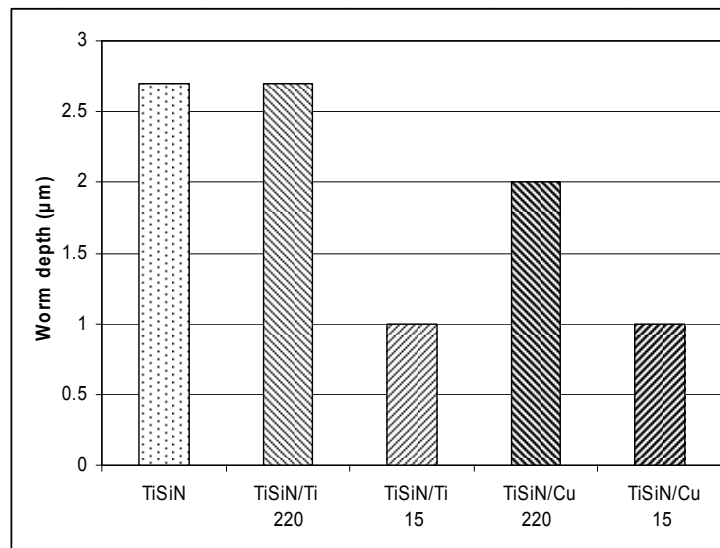
Where n is the number of monolayer's,  $D_{Me}$  thickness of an individual metal or alloy layer,  $D_N$  thickness of an individual nitrate monolayer and  $\Lambda$  overall coating thickness.

The values of the friction coefficient measured with CSM microtribometer are presented in the Table 5. The testing conditions were as follows: normal load 1N, sapphire ball with 6 mm diameter, dry friction in air, relative humidity 30% temperature 25 °C.

**Table 5.** Friction coefficient of the coatings

Coating	Friction coefficient $\mu$
TiSiN	0.141
TiSiN/Cu-9	0.156
TiSiN/Cu-360	0.135
TiN	0.145
TiN/Cu-9	0.130
TiN/Cu-360	0.150
TiSiN/TiSi-9	0.107
TiSiN/TiSi-360	0.239

The lowest  $\mu$  value was obtained for the TiSiN/TiSi-9(0.107). The TiSiN/TiSi-360 film has the highest  $\mu$  value (0.239). The wear behavior of the coatings was evaluated by measuring the depth of the worn trace. The surface profilogram of the TiSiN/TiSi-9 coating is represented in the figure 4. The wear of various coatings is shown in figure 5 [6].

**Fig. 4.** Surface profilogram of TiSiN/TiSi-9 coating**Fig. 5.** Wear of TiSiN, TiSiN/Ti and TiSiN/Cu coatings tested against 57HRC steel ball

The best wear resistance was found for the super lattices of both coatings TiSiN/Ti and TiSiN/Cu with the low  $\Lambda$  values.

## Conclusions

The multilayer hard coatings which were investigated exhibited the best mechanical properties and tribological performance (the lowest friction coefficient and the best wear resistance). We can say that these coatings would be a promising approach for machining applications, particularly to be used as protective coatings against wear and corrosion.

## References

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## Caracteristicile mecanice și tribologice ale filmelor dure multistrat depuse prin metoda arcului catodic

### Rezumat

*In această lucrare sunt prezentate rezultatele cercetărilor privind proprietățile mecanice și tribologice ale filmelor dure multistrat TiN/TiAlN, ZrN/TiAlN, TiSiN/Ti și TiSiN/Cu depuse prin tehnica arcului catodic. Au fost realizate măsurători de microduritate, grosime de strat, aderență, coeficienți de frecare și rezistență la uzură ale straturilor enumerate mai sus. Proprietățile acestor straturi au fost comparate cu proprietățile monostraturilor TiAlN, ZrN și TiSiN.*