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PROPERTIES OF TITANIUM BASED HARD COATINGS DEPOSITED BY THE CATHODIC ARC METHOD

II. MECHANICAL, ANTICORROSIVE AND WEAR RESISTANCE CHARACTERISTICS

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Ti based hard coatings (TiN, TiC, Ti(C,N)), single or multilayer structured, were deposited on different types of substrates (plain carbon steel, stainless steel, high-speed steel, cemented carbide) in a cathodic arc device. Vickers microhardness measurements, Calotest procedure, scratch tests, corrosion, milling and drilling tests were used to analyze some mechanical, anticorrosion and wear resistance characteristics of the films. The study of the influence of the main deposition parameters (reactive gas pressure, substrate bias, arc current) on the film properties was carried out in order to optimize the reactive cathodic arc deposition process.

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1. Introduction

In the first part of this paper [1], an analysis was performed on surface chemistry, chemical composition, phase composition and texture of Ti based hard coatings prepared by the cathodic arc technique.

The aim of this work is to determine mechanical, anticorrosive and wear resistance properties of single and multilayer TiN, TiC and Ti(C,N) hard coatings deposited on different substrates (various types of steels, cemented carbides) under several deposition conditions. These investigations were carried out in order to select a suitable set of process parameters for arc deposition of hard coatings intended to be used for industrial applications.

2. Experimental details

The deposition setup and the variation range of the deposition parameters were presented in Part I of the paper [1]. TiN, TiC, and Ti(C,N) single layers were deposited on plain carbon steel, stainless steel, high-speed steel (HSS) and cemented carbide. The multilayer coatings consisted of 4 distinct layers: TiC / $TiC_{0.7}N_{0.3}$ / $TiC_{0.2}N_{0.8}$ / TiN, where TiC was the underlayer.

For both Ti(C,N) and multilayer coatings, substrate bias and arc current values were V_s = 225 V and I_a = 90 A, respectively.

Microhardness (Vickers) measurements on coatings deposited on HSS and cemented carbide substrates were performed using a Wolpert microhardness tester (20 g load). Film thickness was determined both by optical microscope examination of the cross section through the coating and by Calotest procedure. Scratch tests under standard conditions (indenter – 0.2 mm radius diamond tip, load - continuous increase from 0 to 100 N, scratching speed – 10 mm / min, scratching distance –

10 mm) [2], [3] were undertaken to determine the coating adhesion, using a laboratory apparatus. The critical load (L_C) values were determined by optical microscopy (L_C is defined as the load where film flaking starts).

Corrosion resistance was investigated by an electrochemical test, using a PHOM 4 pH/mV-meter. The electrolyte was salt water (50g NaCl in 1l water) and the test duration was 180 min. Corrosion behavior was appreciated by measuring the changes of the free corrosion potential with time.

Wear resistance of the coatings was evaluated by milling and drilling experiments. Milling tests were performed dry, under the following conditions: tool – milling cutter of 12 mm diameter and 24 teeth, made of P30 cemented carbide (87.5% WC, 5% TiC, 7.5% Co), coating - TiN, axial feed – 12.5 mm/min, pitch feed – 0.7×10^{-3} mm, depth of cut – 0.5 mm, rotating speed - 950 rot / min, workpiece - OL 37 carbon steel plate ($150 \times 25 \times 2$ mm). Milling performance of the coated mills was appreciated in terms of the wear width and the axial force. The tests were interrupted periodically to examine the worn specimens with an optical microscope. The axial force was measured using a resistive strain transducer with a Hotinger type strain gauge. For the drilling experiments, testing conditions were: tool – 8 mm HSS drill, coating - TiN, TiC and Ti (C,N) (single and multilayer structure), rotating speed - 900 rot / min, axial feed – 0.1 mm / rot, workpiece - K 41 carbon steel plate with Brinell hardness of about 210 daN / mm^2, hole depth – 45 mm.

3. Results and discussion

3.3 Mechanical characteristics

3.3.1 Microhardness

TiN coatings

The dependence of the Vickers microhardness ($HV_{0.02}$) on the main deposition parameters (N_2 pressure, substrate bias and arc current) is shown in Figs. 1-3. The microhardness increases with the substrate bias increase, whereas the discharge current variation has only a slight influence on the microhardness. It can also be seen that over a large range of the deposition parameters ($p_{N2} = 2 \times 10^{-2}$ - 5×10^{-1} Pa, Ia = 60 – 130 A and V_s = 50 – 225 V), the microhardness variation is not important (< 200 $HV_{0.02}$).

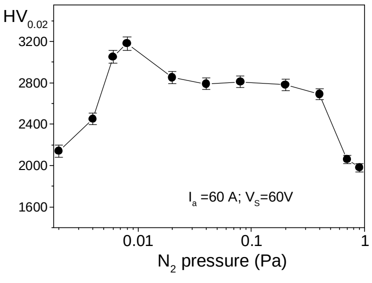


Fig. 1. TiN microhardness vs. N_2 pressure.

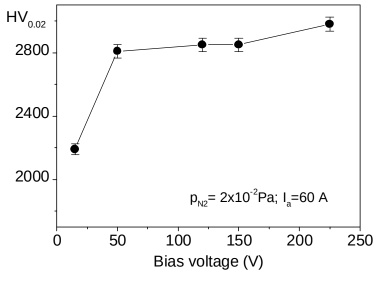


Fig. 2. TiN microhardness vs. substrate bias.

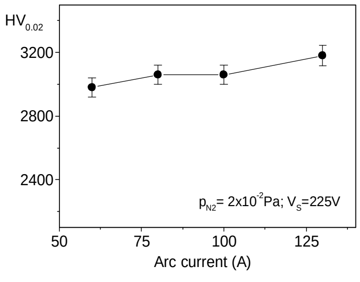


Fig. 3. TiN microhardness vs. arc current.

TiC coatings

Fig. 4 shows the variation in the Vickers microhardness of TiC films as a function of CH_4 pressure. Unlike the case of TiN coatings, the microhardness of TiC films showed a marked dependence on the reactive gas pressure. The optimum CH_4 pressure was in a narrow range, from about 7×10^{-2} to

10^{-1}Pa, for which microhardness values exceeding 4000 $HV_{0.02}$ were found. The measurements also showed that a variation of the substrate bias in the range 50 – 225 V did not result in a change of the film microhardness.

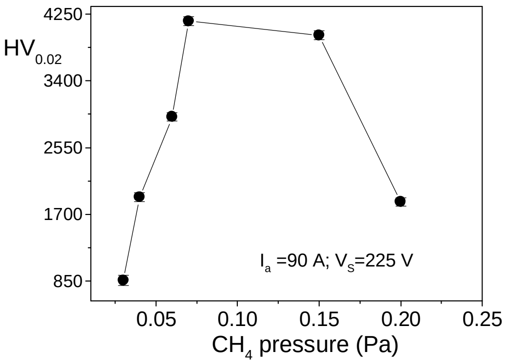


Fig. 4. TiC microhardness vs. CH_4 pressure.

Ti (C,N) and multilayer coatings

Microhardnesses for Ti(C,N) and multilayer coatings are presented in Table 1.

One can see that microhardness of Ti (C,N) film increases with the C/N ratio. Both Ti(C,N) and multilayer coatings exhibit microhardness values between those obtained for TiN and TiC layers.

Table 1. Microhardness $HV_{0,02}$ of Ti (C,N) and multilayer coatings (V_s= 225 V; I_a= 90 A).

Coating	$TiC_{0.2}N_{0.8}$	$TiC_{0.7}N_{0.3}$	Multilayer
$HV_{0.02}$	2960	3280	3060

3.3.2 Adhesion

The experiments carried out showed that the adhesion mainly depends on the substrate characteristics (material, surface quality, hardness) and on the film properties (thickness, microhardness), as it was already reported (e.g. [2], [3]). The critical load increases with the film microhardness and thickness and with the substrate hardness.

For TiN coatings, the highest values of the critical load (≥ 70 N) were obtained for films deposited on cemented carbide or HSS substrates with good quality surfaces. For example, for a TiN layer of 2.8 μm thickness and $HV_{0.02}$ = 2850 deposited on a compact and polished P30 substrate ($HV_{0.1}$ = 1640), critical loads of about 75 N were measured. These values, compared with the data in the literature, indicate an excellent adhesion that assures a good behavior of various coated tools and parts in industrial applications. The coatings deposited on bad quality cemented carbide substrates (rough surface, inhomogeneous structure with pores and voids) exhibited a relatively poor adhesion. In these cases, the critical load did not exceed 10 – 25 N.

For TiC, Ti(C,N) and multilayer coatings, the adhesion test were carried out on films (~3 μm thickness) deposited on HSS substrates. Critical loads for Ti(C,N) were close, but somewhat lower, to those measured for TiN (Lc = 55-65 N). A worse adhesion was observed for TiC coatings, for which critical loads of 25 – 40 N were found. In this case, the films were relatively brittle, probably due to the hydrogen incorporation in the film structure and to their high microhardness [4], usually associated with high tensile stress

3.3.3 Film thickness and deposition rate

The coating thickness and the deposition rate of the TiN coatings were measured on samples prepared under different deposition conditions.

The experimental results are summarized in Table 2. The deposition rate does not depend on the nitrogen pressure or on the substrate bias, but linearly increases (from 0.11 to 0.21 μm/min) with the arc current. This shows that the deposition rate mainly depends on the Ti vapor pressure, which rises with the increase of the arc current.

Table 2. Coating thickness and deposition rate for TiN coating P_{N2} - nitrogen pressure; V_s – substrate bias voltage; I_A - arc current; t - deposition time.

Sample	Deposition parameters				Thickness	Deposition rate
	P_{N2} (Pa)	V_s(V)	Ia(A)	t(min)	(μm)	(μm/min)
1	2×10^{-2}	120	60	15	1.50	0.10
2	1×10^{-1}	120	60	15	1.65	0.11
3	2×10^{-2}	15	60	15	1.65	0.11
4	2×10^{-2}	225	60	15	1.60	0.11
5	2×10^{-2}	120	90	15	2.25	0.15
6	2×10^{-2}	120	130	15	3.15	0.21

3.4 Corrosion resistance

Evolution in time of the free corrosion potential for TiN coatings prepared on stainless steel substrates under different deposition conditions is shown in Fig. 5. For comparison, the corrosion behavior of an uncoated sample is also given.

For the uncoated sample the corrosion process is active during the entire test time. For the coated samples, a better corrosion behavior was found. A short active region is followed by a passivation, which is stabilized at different potential values depending on the deposition conditions. A lower stabilization potential indicates a better corrosion resistance. The coatings deposited at different N_2 pressures (2×10^{-2} and 8×10^{-2} Pa) do not exhibit significant differences in their corrosion resistance. This fact is in agreement with the above experimental results which showed that a N_2 pressure variation over a relative wide range (from about 2×10^{-2} to 5×10^{-1}Pa) has only a slight influence on the TiN film characteristics (stoichiometry, phase composition, microhardness, adhesion). It can also be seen that an increase of the substrate bias (from 120 to 225 V, samples 2 and 4) results in a better corrosion resistance. This may be understood as a consequence of the ion bombardment on the substrate. An increase of the ion energy leads to an improvement of the film compactness and to a decrease of the density of microdoplets and voids. The experiments also showed that a better corrosion behavior was obtained for the films deposited on substrates with a smoother surface.

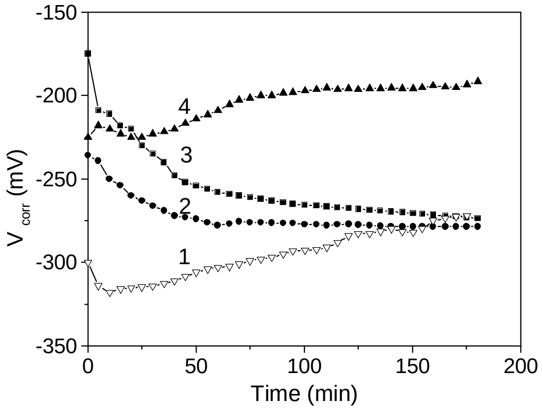


Fig. 5. Time dependence of the free corrosion potential V_{corr} (I_a = 60 A) Curves: **1**- uncoated sample; **2** - $p_{N2} = 2 \times 10^{-2}$Pa, V_s= 120 V; **3** - $p_{N2} = 8 \times 10^{-2}$Pa, V_s= 120 V; **4** - $p_{N2} = 2 \times 10^{-2}$Pa, V_s= 225 V

3.5. Wear resistance

Milling tests

Figs. 6 and 7 illustrate the wear behavior of a TiN coated mill (deposition conditions: $p_{N2} = 10^{-1}$ Pa, I_a = 90 A, V_s = 225 V) in comparison with that of an uncoated one (the axial force and the flank wear as a function of the milling distance are showed).

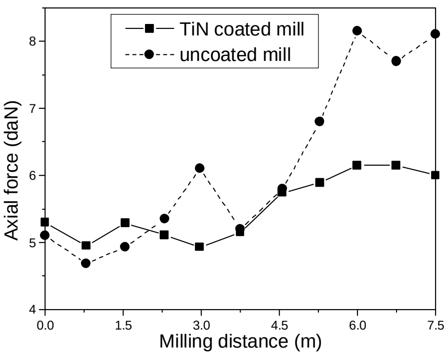


Fig. 6. Axial force vs. milling distance.

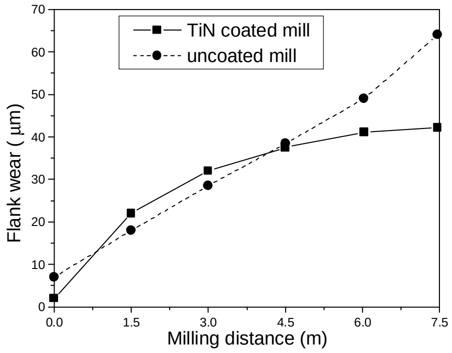


Fig. 7. Flank wear vs. milling distance.

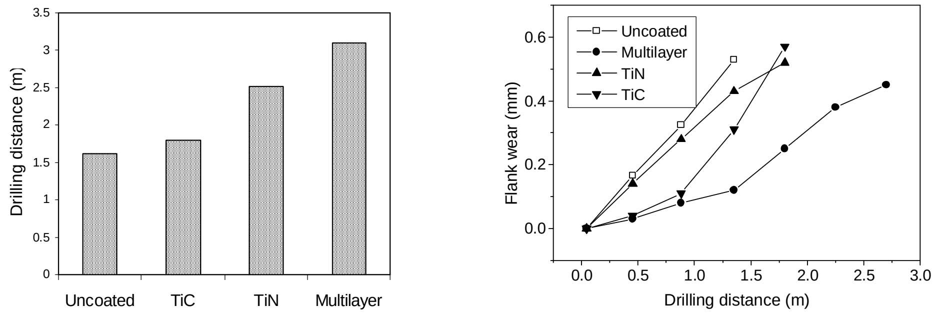
From the microscope examination of the worn mills and from the data in Figs. 6 and 7, the following conclusions may be drawn:

- As a result of the TiN deposition, the face width is reduced (from 6.4 to 1.8 µm). The coating thickness is almost uniform all over the tooth faces (about 2 µm).
- The wear behaviors of the coated and uncoated tools are similar in the first stage of the milling process (for a milling distance < 4.5 m, both the wear width and the axial force have close values). In the next stage, the wear behavior is superior for the coated tool, as a result of a lower friction between the chips and the coating and of a lower welding tendency of the chips on the rake face (the differences between the coated and the uncoated mills become more pronounced with the increasing milling time).
- As compared to the uncoated tools, the variations of the axial force during the milling process are significantly reduced for the coated mills.

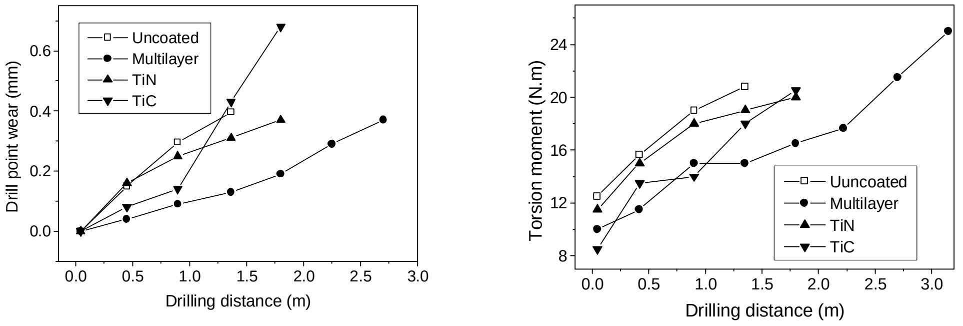
Drilling tests

The drilling performance of TiN, TiC and multilayer coated 8mm HSS drills was analyzed for drilling distance, flank wear, drill point wear and torsion moment (Fig. 8, where deposition conditions are also given). For each coating, the drilling test was stopped when the torsion moment was two times greater than the initial one. This corresponds to a flank wear of about 0.6 mm, which was considered as a criterion for the drill failure.

As it results from the figures, the TiC coating leads only to a slight improvement of the drilling performance. This behavior is in agreement with the results reported by other authors [4]-[6], who showed that TiC coated drills have rather poor results in machining steel workpieces due to excessive chipping and to chemical interaction between TiC and steel at relatively high temperatures. A better wear resistance was found for TiN coated drills, for which an increase of the drilling distance of about 55 % in comparison with that of the uncoated drill was obtained. The best performance was found for the multilayer coating. An increase of the tool life with more than 90 % and a significant decrease of the flank wear, drill point wear and torsion moment were measured.



a) Drilling distance vs. coating type. b) Torsion moment vs. drilling distance.



c) Drill point wear vs. drilling distance. d) Flank wear vs. drilling distance.

Fig. 8. Relative drilling performance of TiN, TiC and multilayer coatings (deposition conditions: $p_{gas} = 2 \times 10^{-1}$Pa; V_s= 225 V; t = 60 min)

While evaluating these results, one has to keep in mind that drilling performance strongly depends on the tool type, testing conditions and workpiece material. Further experiments are needed to choose the most adequate coating for a certain application and to select the optimum deposition conditions.

4. Conclusions

Research carried out in this paper has proved that mono and multilayer Ti based hard coatings of excellent quality can be successfully prepared by cathodic arc deposition method. The analysis of the influence of the main deposition parameters (reactive gas pressure, substrate bias, arc current) on the film characteristics (microhardness, adhesion, deposition rate, wear and corrosion resistance) allows to select the optimum conditions for preparing Ti based hard coatings suitable for a wide range of applications. It is interesting to note that the properties of TiN coatings do not significantly change for a variation of the deposition conditions over a relatively wide range (nitrogen pressure: $2 \times 10^{-2} - 5 \times 0^{-1}$ Pa, substrate bias: 60 –225 V) and this fact is beneficial in the industrial applications of this deposition technique. Mechanical properties of TiC and Ti(C,N) coatings are mainly influenced by the change in reactive gas pressure.

Wear and corrosion tests have shown that the coatings taken into account could improve the performance and extend the lifetime of various tools and parts. As compared to TiN and TiC monolayers, the multilayer coatings (TiC / Ti(C,N) / TiN) exhibited superior wear resistance for coated HSS drills.

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