Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 27
ISSUE 1
March
2008

Comparison of the structure, properties and wear resistance of the TiN PVD coatings

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Received 15.01.2008; published in revised form 01.03.2008

Manufacturing and processing

ABSTRACT

Purpose: The aim of the paper was to compare the structure, mechanical propeties, adhesion and wear resistance of the monolayer TiN PVD coatings deposited onto heat treated and plasma nitrided hot work tool steel X37CrMoV5-1.

Design/methodology/approach: The microhardness tests of the PVD coatings were made with use of a dynamic ultra-microhardness tester. The topography of the surface and the structure of the PVD coatings was observed with use of scanning electron microscopy.The evaluation of the adhesion of coatings to the substrate was made with use of the scratch test. The wear and friction tests were performed with use of a pin-on-disc device at the room temperature and at the temperature of 500°C.

Findings: IIn case of the TiN coating deposited onto the nitrided hot work steel X37CrMoV5-1 a very good adhesion to the substrate has been revealed in comparison with the TiN coating deposited onto the heat treated hot work steel. Taking into account the results of measurements, one can state that the lowest wear at certain conditions at both room and elevated temperatures is characteristic for the TiN coating deposited onto plasma nitrided hot work steel type X37CrMoV5-1.

Practical implications: The results of the investigations provide useful information for applying the plasma nitriding and the TiN PVD coating for the improvement of the mechanical properties and wear resistance of tools made from hot work steels.

Originality/value: The paper contributes to better understanding of the adhesion and wear resistance at the elevated temperature up to 500°C of the monolayer TiN PVD coating deposited onto the heat treated and plasma nitrided hot work tool steel.

Keywords: Thin&thick coatings; Plasma nitriding; Mechanical properties; Wear resistance

1. Introduction

Wear of extrusion dies is an important problem for the aluminum hot working industry. The working conditions of the forming dies and punches are very severe: elevated temperatures (~500-600°C), high pressures, oxidating atmospheres and abrasion from hard inclusions. There has been a chieved an important development in the area of surface engineering of these tools, i.e. an important increase the durability of the tools while maintaining the mechanical properties, dimensional tolerance and surface finish of the product. Due to a high working temperature involved the extrusion dies are generally manufactured from hot work tool steels. One of the most popular grades is the X37CrMoV5-1 steel, due to its high tougness and resistance to softening. It has also a reasonable hardness (~ 48HRC) due to tempering at relatively high temperature. These steel need to be surface treated in order to improve the mechanical and tribological properties of the surface and the resistance to thermal crack initiation. Among the surface engineering techniques employed in the last two decades, gas and plasma nitriding, PVD hard coatings deposition and duplex treatments were reported in the literature [1-7] as the most popular.

It has been documented in the literature that PVD hard coatings provide surfaces with low friction and high wear resistance, however in the field of extrusion dies this techniques has several disadvantages. The crucial one is the large difference in hardness between coating and substrate which results in coating failure [8-11]. The plasma nitriding was carried out in the broad range of temperatures range (400-590ºC), which makes it possible to obtain varying growth of the surface layers, depending on the process conditions, and also on the chemical composition of the substrate. Combining the plasma nitriding and the PVD processes in a duplex surface treatment reduces the hardness gradient between the coating and the substrate and results thus in an improved adhesion and an increase of the durability of the tools [12-15].

The aim of this paper was to examine the structure, mechanical properties and wear resistance of the monolayer TiN coatings deposited by means a PVD technique onto heat terated and plasma nitrided X37CrMoV5-1 type hot work tool steel substrate.

2. Materials and research methodology

The monolayer TiN coating was prepared in BALZERS BAI 730 deposition system by ion plating PVD process at 450˚C and deposited onto X37CrMoV5-1 type hot work steel substrate. The samples in the form of disc (diameter 55 mm and thickness 4 mm) were quenched at 1020˚C and tempered at 550˚C to a hardness of 55 HRC. After the heat treatment, the samples were ground and polished and the PVD coating was deposited. After the heat treatment one of the samples was nitrided, the following plasma nitriding (PN) conditions were applied:

- gas composition - 90%N_2+10%H_2,
- surface temperature - 550˚C,
- treatment time - 3h.

After nitiriding the samples were polished to a roughness R_a = 0.08 μm, than the PVD coatings were deposited. The thickness of the TiN coating deposited onto heat treated and plasma nitrited hot work tool steel was 1.91 μm.

Experimental methodology: hardness test, roughness test, scratch test and wear resistance test, made with equipment and parameters described in [8,13,15].

3. Results and discussion

The microhardness tests of the PVD coatings were carried out at 10 mN load, which ensures the limited indenter penetration depth to eliminate the substrate influence. The highest microhardness of 2927 $HV_{0.001}$ is characteristic of the TiN coating deposited onto plasma nitrided steel, and the lowest of 2711 $HV_{0.001}$ of the same coating onto heat treated hot work tool steel.

Roughness of the investigated PVD coatings is within the 0.018–0.319 μm range. The results of these measurements correspond with the metallographic examinations made with use of SEM microscopy. The topography of the coatings influences the roughness, which is characterized by heterogeneity in form of cavities and elementary particles as a low smoothness of the surfaces of the investigated TiN coatings onto plasma nitrided X37CrMoV5-1 type steel.

As a result of the metallographic examination made with use of SEM it has been found out that the morphology of the investigated TiN coatings deposited onto heat treated and plasma nitrided hot work steel X37CrMoV5-1 type is characterized by a significant inhomogeneity due to occurrence of numerous microdroplets on their surface as well as to pits developed by falling out by some of these droplets. The greates number of these defects was observed in case of TiN coating on the heat treated hot work tool steel (Fig.1a) and the smallest one in case of TiN coating on plasma nitrided hot work tool steel (Fig.1b). The results of this investigation fit well the results of roughness measurements as well as the values of the friction coefficient. Metallographic examinations of coatings fractures show that TiN coating on heat treated and plasma nitrided X37CrMoV5-1 steel has a compact columnar structure and a uniform thickness (Fig.2 a, b).

The critical load values L_c, that are characteristic to adhesion of the investigated PVD coatings to the heat treated and nitrided hot work steel are given in Table 1.

Table 1.
Critical loads for TiN coating deposited onto heat treated and plasma nitrided hot work tool steel X37CrMoV5-1

Substrate material/coating type	Type of defect/Force [N]				
	L_c(AE)	L_{c3}	L_{c4}	L_{c5}	$L_c(F_t)$
X37CrMoV5-1+TiN	40.00	21.00	49.00	72.00	78.33
X37CrMoV5-1+PN+TiN	44.00	58.00	73.00	92.00	91.00

It has been found out, on the basis of on the L_c (AE) values as well as on the basis of metallographic examinations of fractures that monolayer TiN coatings have a very good adhesion to the substrate from the nitrided hot work tool steel (Fig.3a), whereas their adhesion to the substrate from the heat treated hot work tool steel is the worst (Fig.3b). The damage of the coatings begins always with the widespread coating spalling on both edges of the originating scratch. The difference consists in the location of these spalling defects. In case of the TiN coating deposited on plasma nitritided hot work tool steel the damage begins at the load value of about 58 N from numerous chips on both the edges of the scratch combined with stretching of its bottom. In case of the TiN coating deposited onto heat treated hot work tool steel the spalling defects begin at the load value of about 21 N, after which flakes and conformal cracks connected with delamination are occuring.

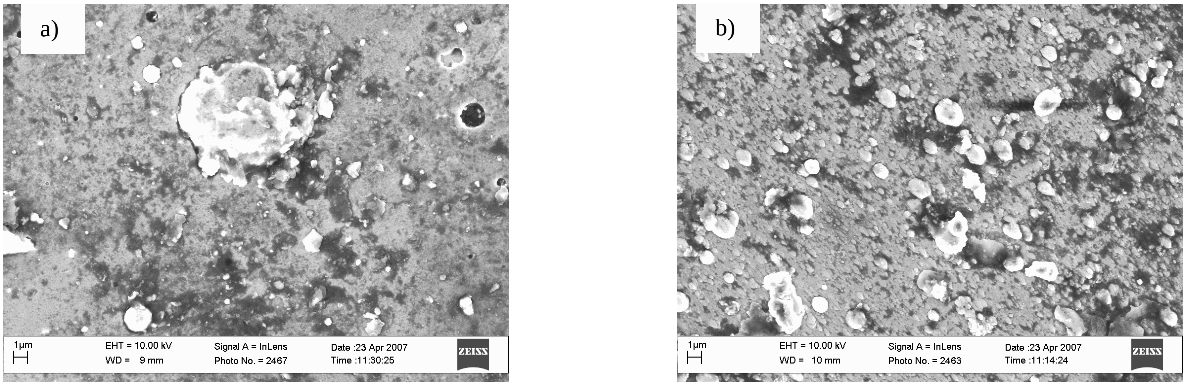

Fig. 1. Topography of the TiN PVD coatings deposited onto hot work tool steel X37CrMoV5-1 substrates: a) after heat treated; b) after plasma nitrided

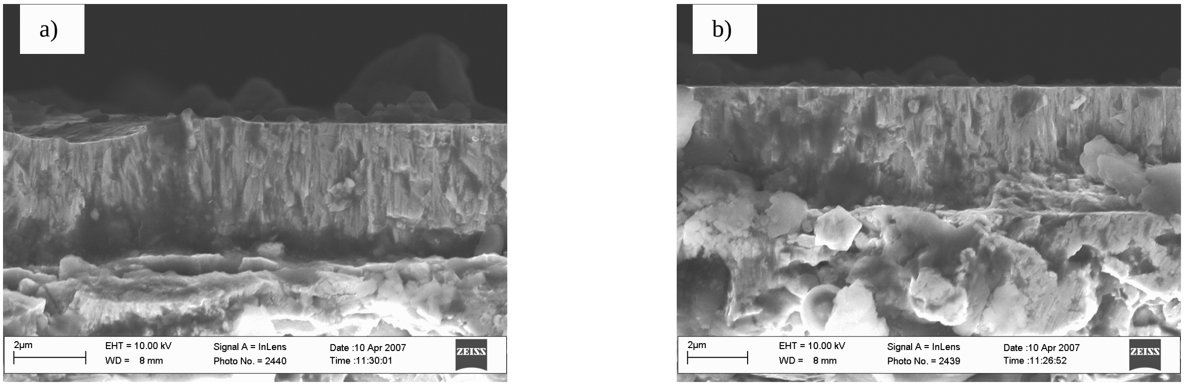

Fig. 2. Fracture of the investigated TiN PVD coatings deposited onto hot work tool steel X37CrMoV5-1 substrates: a) after heat treated; b) after plasma nitrided

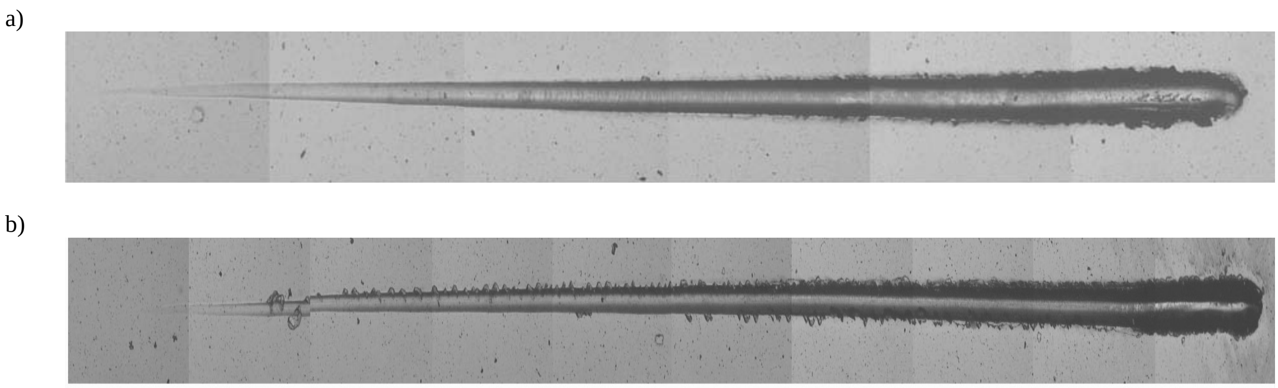

Fig. 3. Scratches with typical delamination of the investigated TiN PVD coatings deposited onto hot work steel X37CrMoV5-1: a) after plasma nitrided; b) after heat treated

The test of the resistance to abrasion with use of pin-on-disc method were carried out in order to determine the functional and working characteristics of the TiN coatings deposited by PVD process onto the heat treated and plasma nitrided hot work tool steel substrate. The investigated test were carried out at room temperature (20˚C) and at the temperature 500˚C. During the test changes of the friction coefficient between the corundum ball and the examined test piece were recorded. Analysis of these changes of friction coefficient makes it possible to state that at the assumed experimental conditions at the room temperature and after 1000 revolutions the friction coefficient attains a value of about 0.5 for the TiN coating deposited onto nitrided hot work steel and to about 0.7 for the same coating on heat treated hot work tool steel at the temperature 500˚C the values of the friction coefficient for the coated test pieces are 0.6 for the TiN coating on nitrided X37CrMoV5-1 steel type and 0.8 for the TiN on heat treated steel. For greater number of revolutions 7500 at room temperature the friction coefficients are changing to a value of 0.6 for the PN + TiN coating and to a value of about 0.7 for the TiN coating on heat treated hot work tool steel.

Table 2.
Comparison of volume of materials removed during tribological wear for 1000 and 7500 revolutions

Substrate material/Coating type	Volume of materials removed V [mm^3]			
	1000 revolutions		7500 revolutions	
	20˚C	500˚C	20˚C	500˚C
X37CrMoV5-1+TiN	0.025	0.193	0.052	0.706
X37CrMoV5-1 +PN+TiN	0.009	0.179	0.032	0.649

One can state on the basis of wear tests, that during the tests at the room temperature both for 1000 and 7500 revolutions the highest resistance to sliding wear was characteristic of the TiN coating on plasma nitrided hot work X37CrMoV5-1 steel grade (Table 2). The change of the number of revolutions from 1000 to 7500 increase the wear volume threefold.

4. Conclusions

The following conclusion have been drawn from the investigations:

- the TiN PVD coating on the plasma nitrided X37CrMoV5-1 hot work tool steel is characteristic of a better adhesion (L_{c3}=58 N) to the substrate material as compared with the adhesion of the same coating on the heat treated steel (L_{c3}=21 N). This difference is due not only to adhesion but also to a thicker interface between the coating and the substrate as well as to 148µm thick nitrided layer of the hardness 1480 $HV_{0.1}$ in the PVD coated substrate,
- improved mechanical properties of the substrate after plasma nitriding reduce cracking, spalling, chipping and delamination, of coatings, contributing to improvement of the adhesion parameters and increasing the resistance to wear,
- the temperature influences the results of friction test. First of all there is a much greater volume wear at the temperature of 500°C. The deposition of TiN coating onto the nitrided tool steel improves its anti-wear properties due the decrease of the friction coefficient.

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