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RESEARCH

The Modification of Duplex Coatings With Nitrogen Ion Implantation

In the present investigation the wear resistance of duplex coated and surface modified with ion implantation was studied under dry sliding condition. A typical duplex process involves plasma nitriding and the coating treatment of steels. The PVD and IBAD coating under investigation was TiN. Ion implantation was provided with N ions. The three basic points that are considered fundamental to studies of friction are the surface area and nature of the intimate asperity contacts, the surface adhesion and shear strength, and the nature of deformation and energy dissipation occurring at the asperity junctions. The optimization procedure for coated parts could be more effective, knowing more about the fundamental physical and mechanical properties of a coating, their interdependence and their influence on the wear behaviour. Wear resistance and exchanges of friction coefficient was measured with on line test using special designed tribology equipment. Following the tests, the wear zone morphology and characteristics of surface layer structure as well as important properties were investigated A variety of analytic techniques were used for characterization, such as scratch test, calo test, SEM, AFM, XRD and EDAX. Finally, the results were correlated with properties determined from mechanical and tribological characterization. Based at all results the correlation between the mechanical properties, the surface structure and tribological characteristics were explained.

Keywords: PVD, IBAD, duplex, superhard, ion implantation, nanohardness

1. INTRODUCTION

The various deposition techniques of coatings and process parameters differ widely in morphology and microstructure, in phases, grain size, texture, defects, impurity content, state of stress, and their mechanical and tribological properties.

Ion bombardment during physical vapour deposition (IBAD), has more independent parameters than classic plasma based technique (PVD). The film deposition process exerts a number of effects such as crystallographic orientation, morphology, topography, densification of the films [1]. However, the adhesion, structure and durability of coatings on various substrates can be substantially improved by irradiating the substrates and the condensing film with ions and energetic neutrals in the energy range of several electron volts [2].One of the most widely used physical vapour deposited coatings for engineering components is TiN.

It is well-known that the mechanical properties of the substrate and interface play critical role in determining system performance. The adhesion strength transmits the surface loads into the component and takes up the stresses that arise because of the different properties of coating and substrate. The nitrided layers is a key issue in duplex coating processing [3]. Since the preferred orientation in TiN thin films has a significant effect on their properties, the study of preferred orientation and the variation of the preferred orientation with the experimental conditions are important for the application of TiN. Ion beam assisted deposition (IBAD), can be used to control the preferred orientation and the properties of the TiN film. IBAD has more independent parameters than a PVD technique and it is possible to study the effect of deposition parameters on the structure of grown films.

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The tribological performance of a component is governed by its design, environment, contact conditions and the materials of which it is composed. In order to understand, explain or predict the performance of a given component, one must naturally possess all relevant information about these parameters.

Tribological characteristics of a material are determined by properties related to its sliding against any other material. The progressive loss of substance from the operating surface of a body can result from mechanical interaction at and/or a chemical reaction induced between two contacting surfaces. Dynamic friction is defined as the resistance to a relative motion of contacting bodies and can be quantitatively represented as the coefficient of friction [4].

2. EXPERIMENTAL

The substrate material used was high speed steel type S 6-5-2. Prior to deposition the substrate was mechanically polished to a surface roughness of 0.12 μm (R_a). The PVD treatment was performed in a Balzers Sputron installation with rotating specimen.

The coatings were deposited with ion beam bombardment in a DANFYSIC machine. The base pressure in the vacuum chamber was 10^{-4} Pa for all experiments. System has Kaufman-type ion source, sample holder and quartz crystal thickness monitor. A pure titanium intermediate layer with a thickness of about 50nm has been deposited first for all the coatings to enhance the interfacial adhesion to the substrates.

Tribo-tests were performed on a CSM tribometer with a ball-on-disc configuration at 0.02m/s sliding speed and 1N normal load. The tribological behaviour of the coatings is scrutinized in conjunction with the detailed examinations of the mechanical properties and microstructure

The composition of the films (nitrogen to metal ratio) was determined by energy-dispersive X-ray analysis (EDAX). The Vickers michrohardness of the coating is measured in the as-deposited-state. Tests were carried out using a CSM tester.

The determination of phases was realized by X-ray diffraction using PHILIPS APD 1700 X-ray diffractometer. The X-ray sources were from CuKα with wavelength of 15.443 nm (40kV, 40mA) at speed 0.9° min^{-1}.

The adhesion of TiN films was characterized by the scratch-test method. We used scratch tester equipment with an acoustic sensor (CSEM-REVETEST). The acoustic emission recorded enabled us to highlight the scaling or the decohesion of the deposition film. The results confirmed by optical microscopy and scanning electron microscope

The film morphology was measured with a scanning electron microscope (PHILIPS XL SERIES 300).

3. RESULTS AND DISCUSSION

Vickers microhardness measurements on substrates are given in Table 1.

Table 1. Surface microhardness ($HV_{0.03}$)

	Unit	pn/ IBAD	PVD	pn/ PVD/II	Fused Silica
Average	Vickers	2007,3	3028,2	3927,1	943,1
StDev	Vickers	190,4	409,9	157,5	40,4

The materials under study are TiN coatings with a thickness of approximately 3μm deposited by PVD and 1μm deposited by IBAD. From table 2, it is obvious that, the high hardness was measured on sample with duplex coating and additional ion implantation. With smaller load indentation, TiN(IBAD) coating show, irrespective of the coating thickness, the greatest increase in hardness.

The nanoindentation elastic modulus was calculated using the Oliver–Pharr data analysis procedure. The individual values of E are the different for all measurements, figure 1.

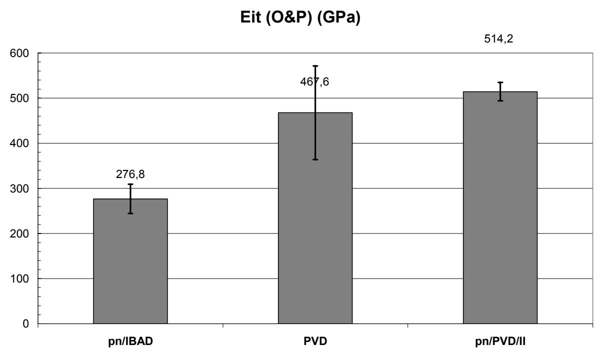

Figure 1. Young's elastic module.

The individual values of E are the different for all measurements. The errors related to the measurements and estimations were different and for duplex coating with ion implantation is less than 4%. Good agreement could be achieved between the E_c values and nanohardness.

For each measurement, the penetration (Pd), the residual penetration (Rd), the acoustic emission (AE) and the frictional force are recorded versus the normal load. The breakdown of the coatings was determined both by AE signal analysis and optical and scanning electron microscopy. AE permits an earlier detection, because the shear stress is a maximum at certain depth beneath the surface, where a subsurface crack starts. Critical loads are presents in Figure 3. The critical load Lc1 corresponds to the load inducing the first crack on the coating. No cracks were observed on sample 1.The critical load Lc2 corresponds to the load inducing the partial delamination of the coating. The critical load Lc3 corresponds to the load inducing the full delamination of the coating. AE permits an earlier detection, because the shear stress is a maximum at certain depth beneath the surface, where a subsurface crack starts (PVD).

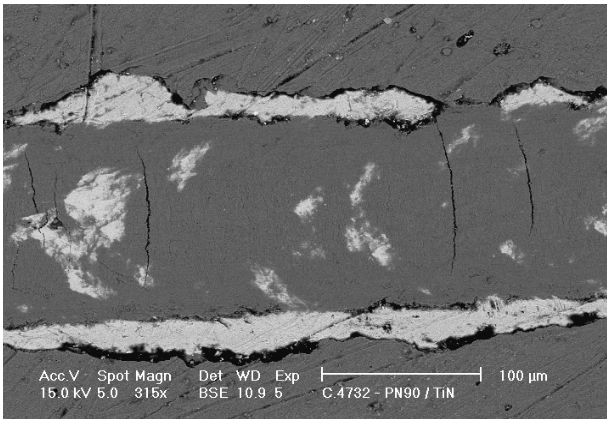

Figure 2. Delamination of TiN(PVD) coating observed by SEM (BSE).

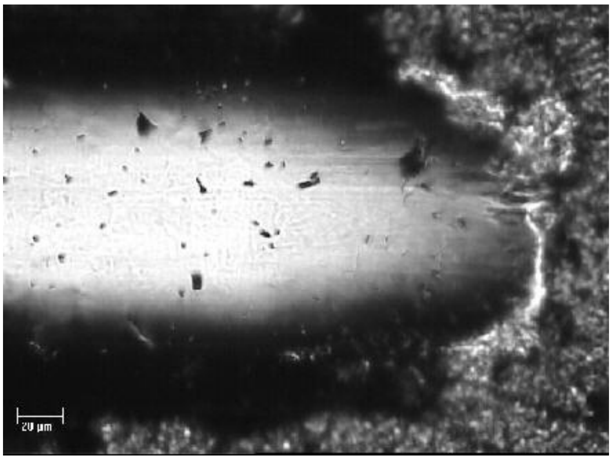

Figure 3. SEM photograph showing the sctrach channel of TiN(IBAD).

The values for critical loads L_c are in general smaller for coatings deposited by PVD. Figure 2 presents the SEM photograph of a scratch channel of sample with film TiN(PVD) and figure 3 with TiN(IBAD).

As known the sliding process is accompanied with the thermal effects. The significant part of the mechanical energy is converted into thermal energy. However , this thermal energy is inappropriate and the converted heat would be able to consider as a waste heat with negative environmental consequences. The friction coefficient of coated sample (TiN-IBAD) is presented in Figure 4.

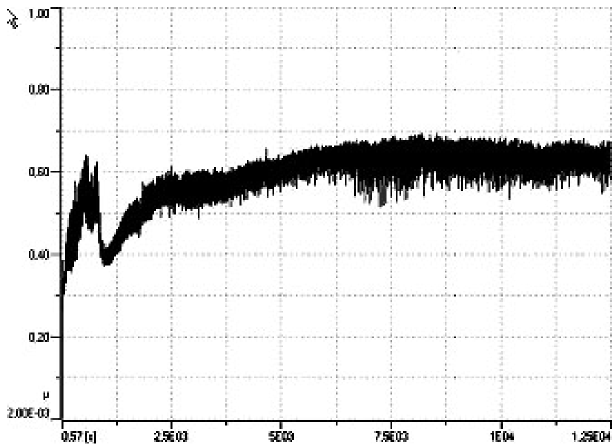

Figure 4. Friction coefficient of TiN(IBAD).

The wear rate of the hard coatings significantly decreases with increase in hardness and especially in H/E ratio.

X-ray diffractogram for the coating is shown in Figure 5.

Compared with the corresponding XRD patterns shown in Figs 5, TiN film grown under ion beam assisted deposition has a (200) preferred orientation and TiN deposited by PVD has a (111) preferred orientation. During TiN film growth, titanium atoms are firstly piled, as compact as possible, depending on the local conditions. The nitrogen atoms occupy the octahedric sites in varying number according to the energy that these atoms possess to cross the potential barriers created by the surrounding titanium anions. The formation of (111) preferred orientation has its origin in a kinetically controlled growth. The (111) plane is the most closely packed and exhibits the lowest surface energy.

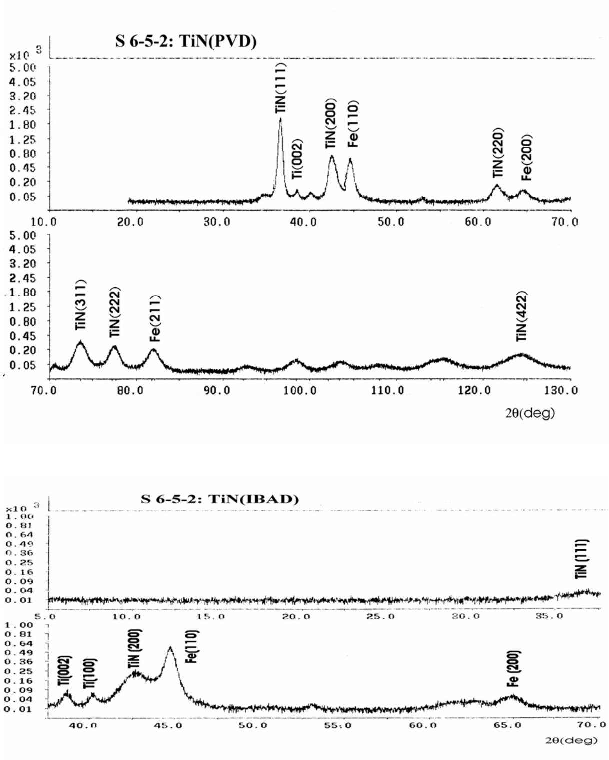

Figure 5. X-ray diffraction spectrums of a)TiN(PVD) and b)TiN(IBAD).

XRD analysis revealed the presence of only one phase, δ-TiN, and there are no evidence for other phases, such as Ti_2N, could be found. The ε-Ti_2N do not lead to a improvement in the tribological behaviour [5].

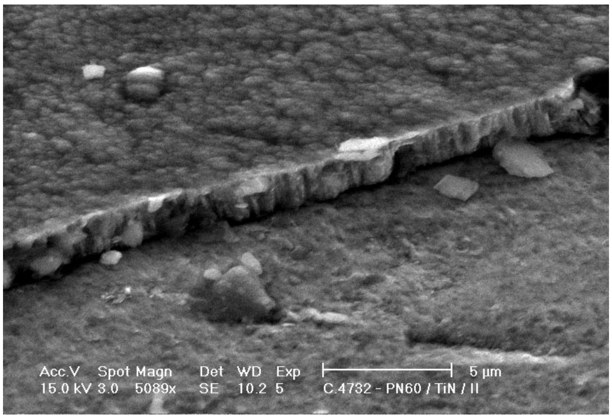

Figure 6. Surface morphologies of pn/TiN(PVD)/II.

The coating morphology was evaluated using the well-known structure zone model of Thornton. All observed morpholgies are believed to be from region of zone I (PVD) and from the border of region zone T (IBAD). It has been suggested , that the transition from open porous coatings with low microhardness and rough surface, often in tensil stress to dense coatings films with greater micrihardness, smooth surface occures at a well defined critical energy delivered to the growing film.

The microstructure of the TiN film, Fig. 6, shows columns in the film.

4. CONCLUSIONS

Deposition of TiN on plasma nitrided steel by IBAD process was successfully used to produce a hard and wear-resistant surface.

It is shown that TiN (IBAD) coatings have a high wear resistance and low friction coefficient. The IBAD process reduces significantly the friction coefficients and improves the wear resistance.

The microhardness, the coating to substrate adhesion and morphology of the hard plasma nitrided with deposited TiN(IBAD) coating and additional ion bombardment obtained offer the better wear resistance of the former coatings.

The above findings show that deposition process and the resulting coating properties depend strongly on the additional ion bombardment.

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