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CHARACTERISTICS OF TIN COATINGS DEPOSITED BY PVD AND IBAD ON NITRIDED STEELES

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Abstract

The paper discusses microstructure, mechanical and physical testing with several examples of advanced coating systems. Another development for use with the plasma nitriding system is surface deposition after the nitriding treatment. This allows nitriding to be carried out in the plasma nitriding chamber followed by a surface deposition treatment, such as depositing titanium nitride. TiN was deposited with classics BALZER PVD equipment. The other samples were produced with IBAD technology in DANFYSIK chamber. The properties and quality of ion beam assisted deposition (IBAD) coatings depend on many process parameters, such as the ion energy, ion to atom arrival ratio, angle of ion incidence, pressure, temperature, etc. X-ray diffraction studies were undertaken in an attempt to determine the phases present, and perhaps an estimate of grain size from line broadening. The coating morphology was evaluated (with SEM), using the well-known structure zone model of Thornton. The present coating method can produce dance structures, high hardness and the high critical load values can be achieved.

1. INTRODUCTION

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].

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 IBAD coatings were deposited with ion beam bombardment in a DANFYSIK 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.

Specimen was made of high-speed steel and bearing steel in plate 5 mm thick and 25 mm diameter. The specimens were first austenized, quenched and than tempered to the final hardness of 810 HV.

The techniques utilized for morphology and microstructure studies are light optical microscopy (LOM) and scanning electron microscopy (SEM). The composition of the films (nitrogen to metal ratio) was determined by energy-depressive X-ray analysis (EDAX). The Vickers michrohardness of the coating is measured in the as deposited state

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^o min^{-1}.

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

3. RESULTS AND DISCUSSION

The result from scanning electron microscopy shows a columnar structure reaching from the substrate to the coating surface (Fig. 1).

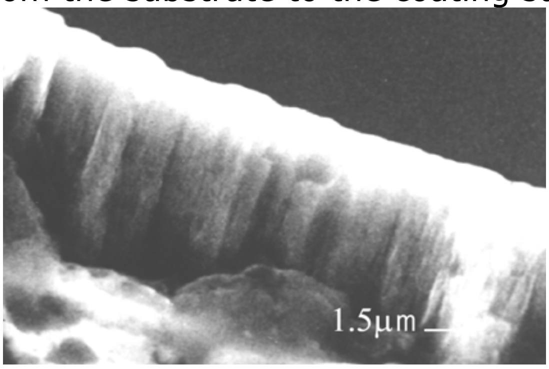


FIGURE 1. SEM of coating cross-section TiN(PVD)

The films are very dense. The interface indicates very good coating to substrate adhesion. It was found that the plasma-nitriding process enhanced the coating to substrates adhesion. In some places of hard coatings cohesive failure of the coating and the delimitation of the coating was observed (Fig. 2).

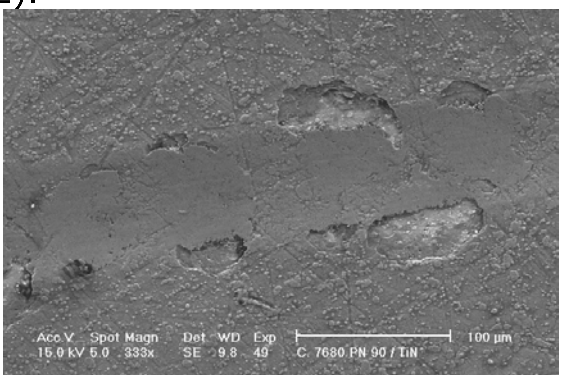


FIGURE 2. SEM morphology of scratch test: pn/TiN(PVD).

The hardness values are also shown to demonstrate the increase in surface hardness due to nitriding at low pressure and coating. The materials under study are TiN coatings with a thickness of approximately

3μm deposited by PVD and 1μm deposited by IBAD. From table 1, it is obvious that, the similar hardness was measured with 30g. With smaller load indentation, 5g, TiN(IBAD) coating show, irrespective of the coating thickness, the greatest increase in hardness (5200$HV_{0.005}$). The evolution of film composition versus type of coating is shown in table 2.

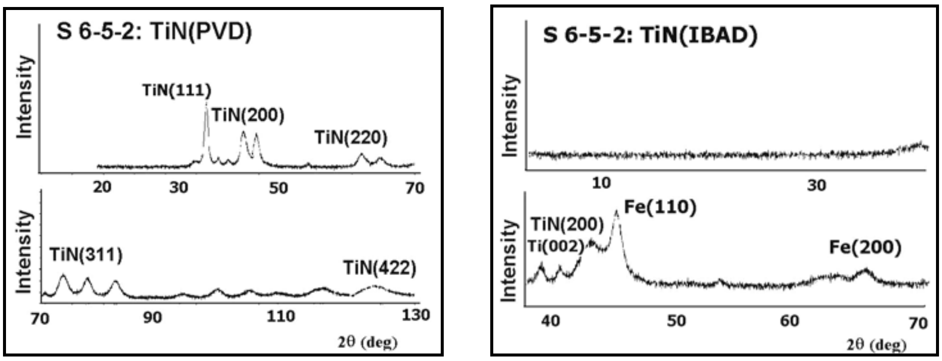
TABLE 1. Surface microhardness $HV_{0.03}$ (GPa).

steel	uncoated	TiN(PVD)	(TiN)IBAD	pn60/(TiN)IBAD
S 6-5-2	8.30	28.75	28.50	30.10

TABLE 2. Atomic ratio N/Ti in coating.

	Type of coating	Ratio N/Ti (atomic)
1	TiN(PVD)	0.98
2	Pn/TiN(PVD)	0.93
3	TiN(IBAD)	1.00

Compared with the corresponding XRD patterns shown in Fig.3, TiN film grown under ion beam assisted deposition has a (200) preferred orientation and TiN deposited by PVD has a (111) preferred orientation.



a) b)

FIGURE 3. X-ray diffraction spectrum of a)TiN(PVD) and b)TiN(IBAD).

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.

XRD analysis revealed the presence of only one phase, δ-TiN, and is no other phases, such as Ti_2N, could be found. These results are in agreement with those obtained in other paper [3]. The interplanar spacing d_{hkl}, determined from XRD spectra have values higher than those from randomly oriented strain-free powder samples. This indicates that films were in compressive stress.

The coating morphology was evaluated using the well-known structure zone model of Thornton. All observed morphologies are believed to be from region of zone I (PVD) and from the border of region zone T (IBAD). It has been suggested [4], that the transition from open porous coatings with low microhardness and rough surface, often in tensile stress to dense coatings films with greater microhardness, smooth surface occurs at a well-defined critical energy delivered to the growing film.

The width of column is derived from the width of the diffraction peaks (λ=0.154nm, θ=62.5^{o} i β=0.056rad), and it is 70 nm.

The simplest form of X-ray diffraction (XRD) characterization of thin film microstructure is Cohen-Wagner plot. A typical result for compact film is shown in Fig. 4.

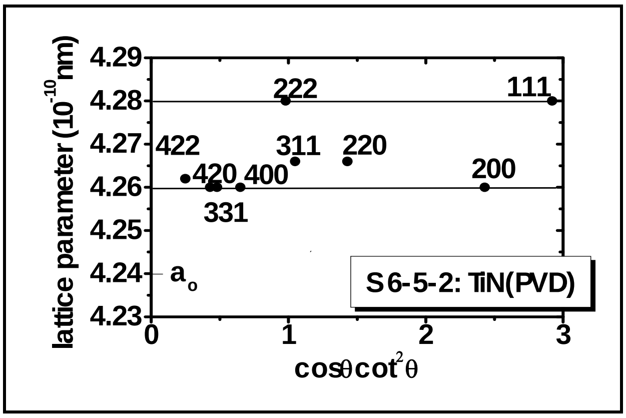


FIGURE 4. The Cohen-Wagner plot, lattice parameters a_{hkl} vs.cosθcot$^2\theta$, for dense TiN (a_{hhh}>>a_{hoo})

The anisotropy of lattice parameters, a_{hhh}>>a_{hoo}, is characteristic for compact film.

4. CONCLUSION

It was concluded that formation of plasma nitrided layer at low pressure, beneath a hard coating, is important in determining the use of hard coating. An excellent coating to substrate adhesion and greater hardness was found to be significant factor influencing the use of plasma nitrided at low pressure.

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

The IBAD process leads generally to significant changes in coating properties. These improved properties are a consequence of the dense microcrystalline structure.

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REFERENCES

[1.] K. Mogensen, N. Thomsen, S. Esikilden, C. Mathiasen and J.Bottiger, *Surface and Coatings Technology* 99, 1998, pp140-146.
[2.] W. Ensinger, *Surface and Coatings Technology* 99 (1998) pp1-13.
[3.] K. H. Bäther, U. Herrmann, A. Schröer, *Surface and Coating Technology,* 74-75, 1995, pp793-801.
[4.] L. Combadiere, J. Machet, *Surface and Coating Technology,* 88, 1996, pp 28-37.