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Structure and Properties of Thin Layer – Substrate on High Speed Steel with Different Thickness of TiN

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Abstract

The main application of TiN coating deposited on metallic materials by Arc – PVD method is to increase the wear resistance. The properties of TiN coating are related to the properties of the substrate, technological parameters, microstructure, thickness, adhesion and hardness of the film. This paper presents the results of mechanical property measurement of 2 μm and 4 μm thick TiN coatings deposited on high-speed steel. The hardness was measured by nanoindentor and the adhesion by scratch test. The microstructure of TiN coating was evaluated by SEM microscopy.

Key words: TiN coating, PVD technology, hardness, scratch test

Introduction

In the recent years, technology of deposition of thin film layers on the surface of steel parts has been developed considerably. This development enables to apply the coated materials in the field of mechanical and electrical engineering and also for decorative purposes. The methods of application of thin coatings include physical vapour deposition of thin layers (PVD) based on the conversion of solid metal to a gaseous phase and its condensation on the substrate surface[1]. The PVD processes take place in a vacuum chamber at a pressure of 10^0 – 10^5 Pa, depending on the methods used. The functionality and reliability of thin layers are affected by the microstructure and properties of the substrate, technological parameters of the deposition process, micro structure of the coating, its chemical and phase composition, thickness, hardness and adhesion to the substrate[2].

Materials and experimental procedure

A thin coating was deposited on the high-speed steel substrate which was processed thermally resulting in the hardness of 61 HRC. The microstructure of the steel consisting of tempered martensite and carbides is illustrated in Figure 1. The shape and dimensions of specimens are shown in Figure 2. Chemical composition of steel is summarised in Table 1.

Figure 1 Microstructure of etched high – speed steel after heat treatment

Surface of steel specimens was polished before coating. The final stage of the substrate surface preparation took place in a coating apparatus. Bombardment by ions of an inert gas Ar at a pressure of 10^3 Pa released impurities from the treated surface and resulted in its activation and heating to the required temperature.

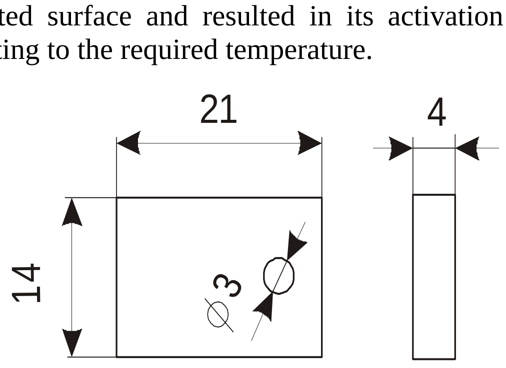

Figure 2 Shape and dimension of coated samples. 1000x.

Table 1 Chemical composition of high – speed steel

Steel	Actual chemical composition [%]								
	C	Mn	Si	Cr	Mo	V	W	P	S
19 830	0.86	0.24	0.14	3.96	5.15	1.88	6.4	0.003	0.011
Chemical composition according to STN 419 830 [%]									
19 830	0.8 – 0.9	max 0.45	max 0.45	3.8 – 4.6	4.5 – 5.5	1.5 – 2.2	5.5 – 7.0	max 0.035	max 0.035

Table 2 Technological parameters of PVD process

			Technological parameters			
			Substrate temperature [^oC]	Nitrogen pressure [Pa]	Accelerating voltage [V]	Coating period [min]
TiN	2	19 830	450	2	300	20
TiN	4	19 830	450	2	300	40

The thin coating was deposited with the arc evaporation method (cathodic arc). This coating method is based on the release of particles from titanium cathode by an electric arc and their bias voltage acceleration toward the substrate surface ; in the course of its motion and on the surface of the substrate titanium reacts with the supplied nitrogen to TiN. This coating method was used to produce 2 μm and 4 μm TiN layers and the layer thickness was controlled by the time of coating. Technological parameters of the deposition process are presented in Table 2.

The microstructure of specimens coated with TiN coating was investigated by light microscopy analysis. An instrumental method was used to determine the hardness of the TiN coatings having thickness 2 μm and 4 μm. This method is based on monitoring of the indentation force and the respective depth of indentation. The test provides a set of data which can be used to determine the relationship between the loading force F and the indentation depth h (indentation curve), Figure 3, and subsequently the hardness. When determining this parameter it is important to eliminate the effect of substrate properties on the measured values of microhardness of the thin coatings(3).

Microhardness of specimens used in our study was measured by an apparatus Shimadzu DUH 202. The scratch test was employed to determine adhesion of the TiN coating to the steel substrate while the loading force on a diamond tip indenter ranged between 10 to 50 N, with 10 N increments, Figure 4.

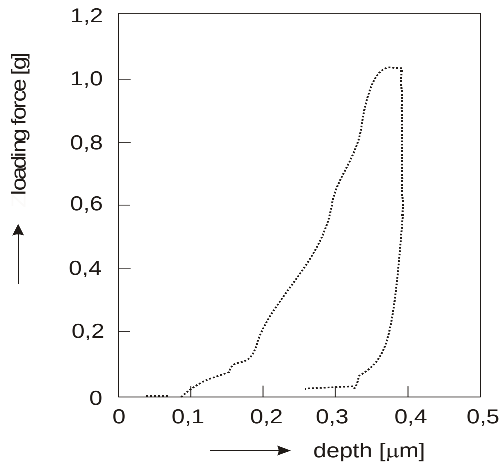

Figure 3 Indentation curve

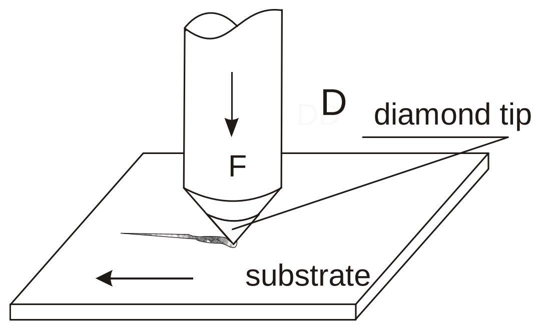

Figure 4 Scratch test

This test allowed us to measure the magnitude of a critical load F_{NC} which results in detachment of the coating. Therefore the critical load F_{NC} defines strength of adhesion of the coating to the substrate.

Results and Discussion

Morphology of the specimen surface with TiN coating deposited on polished steel surface is shown in Figure 5. Unevenly distributed particles and wells can be seen on the coating surface. Characteristic appearance of TiN can be observed on vertical sections in Figure 6, prepared by standard metallographic procedure. The coating presented in Figure 6 is compact and uniform and copies almost exactly the substrate surface. In some places (indicated by arrows in Figure 6b), the coating is thinner or completely absent. This involves predominantly the places in which coarse carbide particles were eliminated after thermal processing.

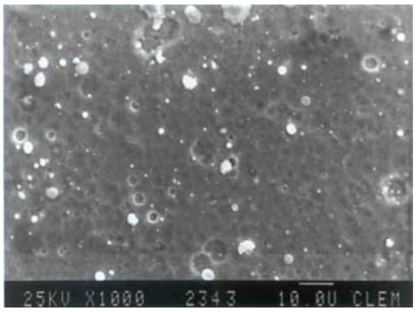

Figure 5 Detail of the surface of a TiN coating with particles and wells, SEM

Figure 6a TiN coating of thickness 2 μm deposited on polished high - speed steel substrate. Magn. 1000x, etched

Detailed observation of the fracture surface by scanning electron microscopy, Figure 7, revealed the presence of microparticles that had been retained in the coating layer in the course of its deposition. The presence of such particles is related to respective deposition technology. During the reactive arc evaporation, the cathode surface melts locally at the site of cathode spot[4] which results not only in evaporation of individual Ti atoms but also in release of larger clusters of atoms, the so-called microdroplets

Figure 6b TiN coating of thickness 2 μm deposited on polished and etched high – speed steel substrate and showing some coating defects. 1000x

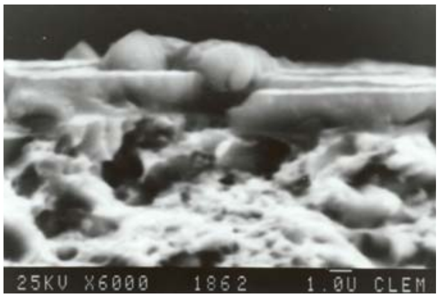

Figure 7 Detail of coating microparticles

These microdroplets are then integrated in the condensing layer, Figure 5. Furthermore, in the process of deposition, the high-energy (10 - 100 eV) ions emitted by the cathode are capable of forcing atoms out of the substrate surface. After termination of the process, unevenly distributed particles and wells remain on the surface of the coated system, as seen on Figure 5. All the mentioned phenomena affected the morphology of the final TiN coating. While the roughness of the substrate surface, represented by the average value of mean arithmetic deviation R_a was initially 0.08 μm, it increased in the course of deposition to 0.29 μm and 0.32 μm on the thinner and the thicker coated layers, respectively.

EDX analysis revealed that the particles consist of titanium droplets which, during the arc evaporation process, did not manage to react with the working gas to nitride, Figure 8. The presence of Ti particles was also confirmed by Ballo[4].

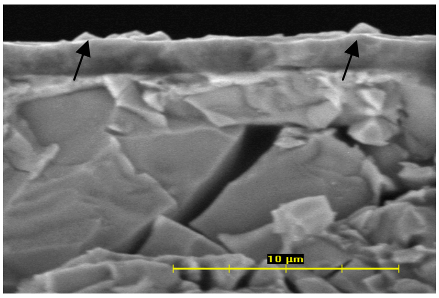

Figure 8 Spectrum of titanium microparticle, SEM

The data from the scanning electron microscope in Figure 9 shows the structure of the TiN coating deposited on polished steel substrate. Structural orientation of this coating is ambiguous. Similar to Figure 7, Ti particles integrated in the coating structure are also visible.

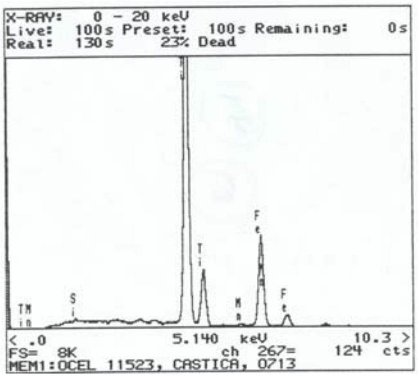

Figure 9 Structure of the TiN coating of thickness 4 µm deposited on polished high – speed steel with some particles visible in the coating, SEM

Microhardness is one of important mechanical properties which characterise the system thin coat – steel surface. Essentially, it involves the resistance of the material to plastic deformation produced by applying load to an indenter[3]. Table 3 shows the values of microhardness of the coated specimens. The microhardness of steel 19 830 is included for comparison.

Table 3 Values of microhardness DHV

	Substrate	Coated system TiN - substrate	
Sample	19 830	TiN 2 µm	TiN 4 µm
DHV	925	4561	6914

The microhardness of coatings of both thicknesses was by an order of magnitude higher than that of the original substrate. The highest value (6914 DHV) was measured in the thicker coating. One of the criteria determining the coating quality is its adhesion to the substrate. The scratch test was used to determine the critical force F_{NC} needed for detachment of the deposited coating. The result obtained for the specimen with 2 µm TiN coating is shown in Figure 10a. Detachment of the coating required critical force F_{NC} = 40N, which was associated with the appearance of visible light substrate zones in the central part of the indentation. Coatings with such strength of adhesion ensure partial resistance of coated materials when exposed to mechanical stress[4]. The strength of adhesion between the 4 µm coating and polished substrate is documented in Figure10b.

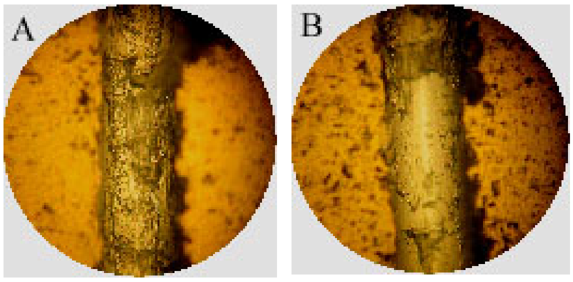

Figure 10 Scratch test: a) failure of 2 µm TiN on steel 19 830 at F_{NC} = 40 N, b) failure of 4 µm TiN on steel 19 830 at F_{NC} = 50 N

At F_{NC} = 50 N no adhesion of coating deposited to steel 19 830 was ensured. According to Ballo[4]. such coating provides no sufficient protection of parts against the mechanical stress.

Conclusion

1. Changes in the micro-geometry of the TiN coating surface are related to technological conditions of its formation. The existence of Ti microdroplets and wells on the coating surface affect the values of mean arithmetic deviation of R_a profile.
2. The presence of carbide particles on the substrate surface impairs deposition of the TiN coating.
3. The hardness of the deposited TiN coating is at least twice as high with both its thicknesses than the hardness of the original steel substrate. The highest hardness 6 914 DHV was measured for the 4 µm coating deposited on polished steel surface.

4. The coating with a thickness of 4 μm, deposited on steel 19 830 with adhesion strength of 50 N renders sufficient protection to substrate exposed to mechanical stress.

References

(1) Hagarová, M. 2003. Thesis, Faculty of Metallurgy, Technical University in Košice,

(2) Jakubéczyová, D and Fáberová, M. 2003. Možnosti povrchovej úpravy P/M nástrojových ocelí. *Metal* : 1 – 7

(3) ISO/DIS 14577, 2001. *Metallic materials – Instrumented indentation test for hardness and materials parameters,*

(4) Ballo, V. 1998. Doctoral, Thesis, Faculty of Metallurgy, Technical University in Košice,