Properties of thin coating deposited on tool steel after different technological conditions of physical vapour deposition process

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Received 30 June 2006, received in revised form 25 February 2008, accepted 26 February 2008

Abstract

Microhardness and scratch adhesion testing are two basic techniques, which are often used for determination of mechanical properties. The microhardness of coatings was assessed by nanoindentor. The adhesion of the coating to the substrate was measured by scratch test. The thin, TiAlN-based coatings were deposited by PVD (Physical Vapour Deposition) process with different values of accelerating bias voltage of substrate. Structure of the coating was determined by SEM microscopy.

Key words: coating, PVD process, microhardness, adhesion

1. Introduction

Modern progressive cutting tools are characterized by application of hard coatings on its surface, which guarantee good abrasion resistance. The main function of coatings is to increase the surface hardness and wear resistance and consequently to improve lifetime of tools.

To realize these goals it is necessary to study the complex of physical and chemical processes on the tools surface [1].

The method of Physical Vapour Deposition (PVD) belongs to the technologies of thin coating deposited on tools, the parts exposed to mechanical stress, as well as working objects. PVD processes allow: to form new coatings with precisely defined properties, to develop new and applied technological operations of surface treatment, to use coating for conventional material and thereby ensure qualitative increase of its advanced properties, to realize new progressive engineering equipment and systems [2].

Thin coatings give a new feature to the substrate surface and consequently change the properties of coated parts. The functionality and reliability of thin layers are affected by the structure and mechanical properties of the substrate, technological parameters of the deposition process, structure of the coating, its chemical and phase composition, thickness, hardness, and adhesion to the substrate [3].

Evaluation of some properties of the system thin layer-substrate needs specific methods and procedures. The most important mechanical properties from the point of view of this application are hardness and adhesion of thin coating to substrate. Microhardness measurement is the basic mechanical test for coated materials, investigating material resistance against local plastic deformation that appears during penetrating indentor to the material system [4]. The most commonly used methods for the testing of the adhesive-cohesive behaviour of hard coating-substrate systems are indentation methods. These methods are based on measuring stresses necessary for breaking the bond between the coating and the substrate during stating pressing of the indentor or during a scratch [5].

2. Material and experimental procedure

Specimens of tool steel were used for experiment. The hardness of steel after applied heat treatment was 59 HRC. The structure of the steel consists of martensite and carbides, as is shown in Fig. 1.

The chemical composition of studied materials is

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Table 1. Chemical composition of STN 419 436 steel

Steel	Actual chemical composition (wt.%)						
	C	Mn	Si	P	S	Cr	Ni
19 436	2.024	0.33	0.28	0.018	0.016	11.40	0.13
	Chemical composition according to STN 419 436 (wt.%)						
19 436	1.8–2.05	0.2–0.45	0.2–0.45	max 0.03	max 0.035	11.0–12.5	max 0.50

Table 2. Technological parameters of PVD process

Coating	Coating thickness (μm)	Substrate – steel	Technological parameters			
			Substrate temperature (℃)	Nitrogen pressure (Pa)	Accelerating voltage (V)	Coating period (min)
TiAlN (300, 2)	2	19 436	500	0.5	300	20
TiAlN (300, 4)	4	19 436	500	0.5	300	40
TiAlN (100, 2)	2	19 436	500	0.5	100	20
TiAlN (100, 4)	4	19 436	500	0.5	100	40

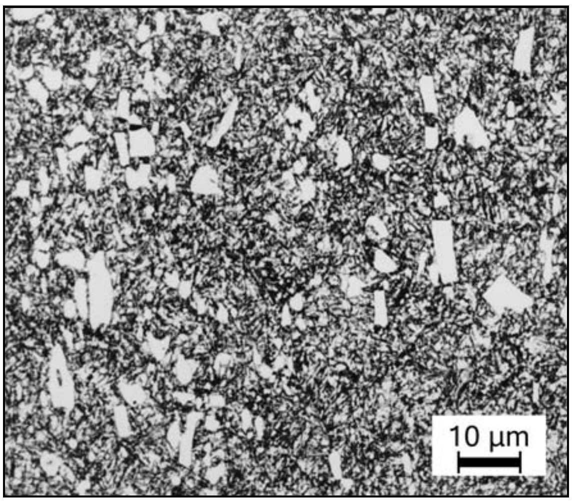

Fig. 1. Structure of steel after heat treatment.

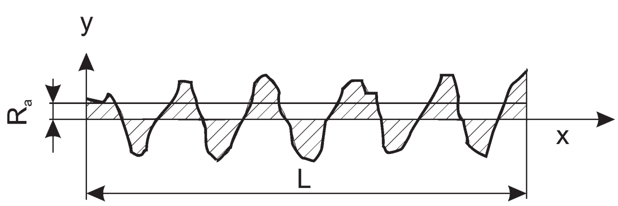

Fig. 2. The mean arithmetic deviation R_a.

stated in Table 1 in comparison with the composition according to STN 419 830 standards.

Surface of steel specimens was polished before coating. The roughness of the substrate surface, represented by the average value of mean arithmetic deviation R_a, Fig. 2, was measured by Taylor/Hobson – Surtronic 3+ equipment according to EN ISO 4287.

The method based on reactive cathode arc evaporation was used for the coating deposition, which belongs to PVD methods of thin layer deposition, Fig. 3 [2].

There is material of coating locally steamed off and ionized in arc discharge at the pressure of 5×10^{-1} Pa in the deposition process. The cathode, which is TiAl alloy, was kept in the process compact, only a very small local part in position of cathode spot of diameter 0.1 mm was melted. Before the deposition process of thin layer on the substrate, the surface was cleaned up in the coating equipment using argon at the pressure of 10^3 Pa. By cleaning process the surface was going to be active. The substrate was heated up by ions evaporated from the material of cathode and accelerated to the substrate. The reactive gas – nitrogen was added in the coating chamber, due to the formation of chemical compound on the substrate. This coating method was used to produce 2 μm and 4 μm TiAlN coating and the coating thickness was controlled by coating time. Technological parameters of the deposition process are presented in Table 2.

An instrumental method was used to determine the hardness of the TiN coatings of thickness 2 μm and 4 μm. This method is based on monitoring the indentation force and the depth of indentation. The test provides a set of data which can be applied for determination of the relationship between the loading force F and the indentation depth h (indentation curve),

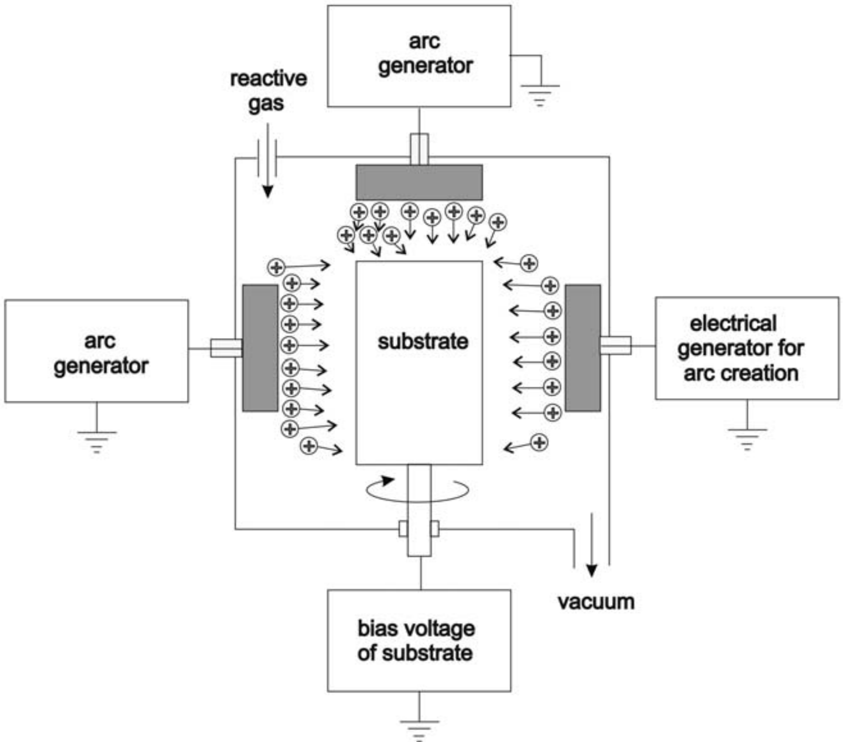

Fig. 3. Scheme of equipment for arc evaporation method [2].

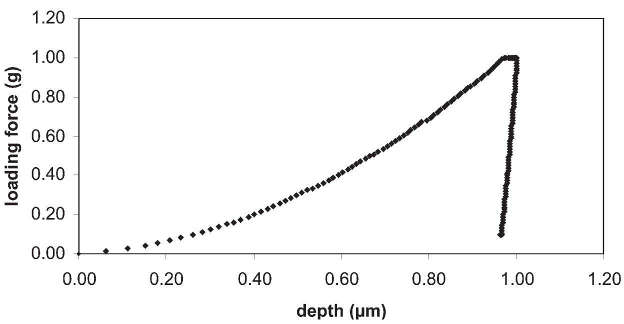

Fig. 4. Indentation curve [6].

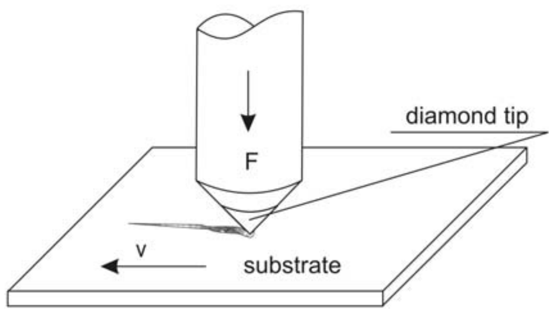

Fig. 5. Scratch test [2].

Fig. 4, and subsequently the hardness [6]. When determining this parameter, it is important to eliminate the effect of substrate properties on the measured values of microhardness of the thin coatings [7]. Microhardness of specimens used in our study was measured by a Shimadzu DUH 202 apparatus at loading level of 5 g, loading speed $1.35\,g\,s^{-1}$ and 10 s hold.

A scratch test was employed to determine adhesive-cohesive behaviour of the TiAlN coating to the steel substrate where the loading force on a diamond tip indenter was in the range between 1 to 80 N. The table movement was ensured by constant speed of $10\,mm\,min^{-1}$. Figures 5 and 6 show a principle of the indentation test for the determining adhesion and a graphic record of the scratch test [8]. This test allowed measuring 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.

The structure of coatings was studied by the Quanta 400 scanning electron microscope at accelerated voltage of 15 kV.

3. Experimental results and discussion

Morphology of the specimen surface with TiAlN coating deposited on polished steel surface is shown in Fig. 7. Unevenly distributed particles and wells could be seen on the coating surface. Compar-

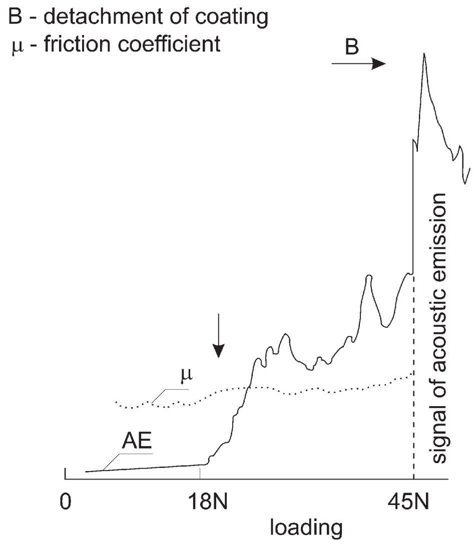

Fig. 6. The graphical record of scratch test [8].

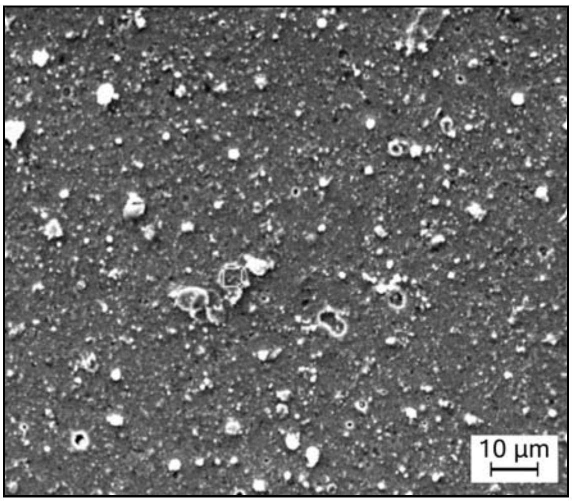

Fig. 7. Detail of the surface of a TiN coating with particles and wells, SEM.

Table 3. Chemical composition of microparticle

Element	wt.%	at.%
C K	08.23	14.38
N K	32.98	49.40
O K	03.14	04.12
AlK	22.73	17.68
TiK	32.92	14.42

ison of obtained values of micro-geometry measurements on the substrate and coating surface found out that these increased for both coating thick-

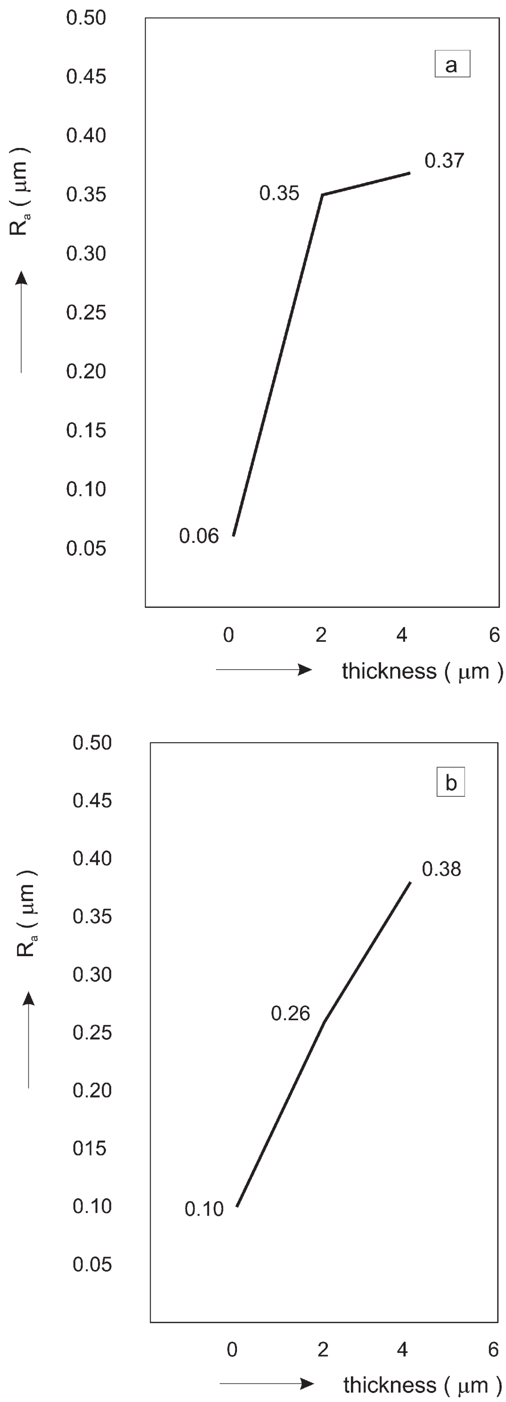

Fig. 8. The value of R_a of a TiAlN coating deposited on the steel substrate at the bias voltage: a) 300 V, b) 100 V.

nesses. The measured values of R_a are given in Fig. 8a,b.

During the reactive arc evaporation process, cathode surface was locally melted at the site of cathode spot [2] which results not only in evaporation of individual Ti and Al atoms but also in release of larger clusters of atoms, the so-called microdroplets. These

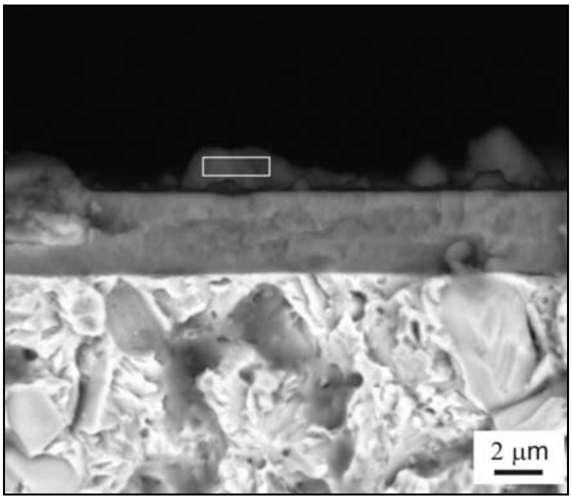

Fig. 9. Microparticles in TiAlN coating, SEM.

Table 4. Chemical composition of coating, SEM

Element	wt.%	at.%
C K	07.01	14.40
N K	17.61	31.01
O K	04.98	07.67
MgK	00.74	00.76
AlK	25.63	23.43
SiK	00.48	00.42
S K	00.39	00.30
TiK	40.25	20.73
FeK	02.90	01.28

microdroplets are then integrated in the condensing layer. In addition to that, in the deposition process, 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, Fig. 7. All the mentioned phenomena affected morphology of the final TiAlN coating.

Figure 9 shows microparticles which were after deposition process inbuilt into the coating layer. The EDX analysis shows that the studied particles are on the basis of Ti, Al and N (spectrum was obtained from the selected microparticle which is marked by rectangle, Fig. 9). The EDX spectrum is given in Fig. 10. Results of the quantitative EDX analysis of this microparticle are presented in Table 3.

The structure of thin coatings is a result of the formation of nuclei of phases and of their growth [8]. The structure of TiAlN coating by using SEM, Fig. 11, is composed by columnar crystals. The structure of thin coating is composed by columnar crystals. Figure 12 shows the chemical composition of studied coating. Obtained results of the quantitative EDX analysis of this coating are stated in Table 4. The major portion has Ti, Al and N, which compose the main compound of thin coating.

Microhardness is one of important mechanical

Table 5. Values of microhardness DHV

Sample	Coated system TiAlN-substrate			
	TiAlN (300, 2)	TiAlN (300, 4)	TiAlN (100, 2)	TiAlN (100, 4)
DHV	2670	2850	2800	2920

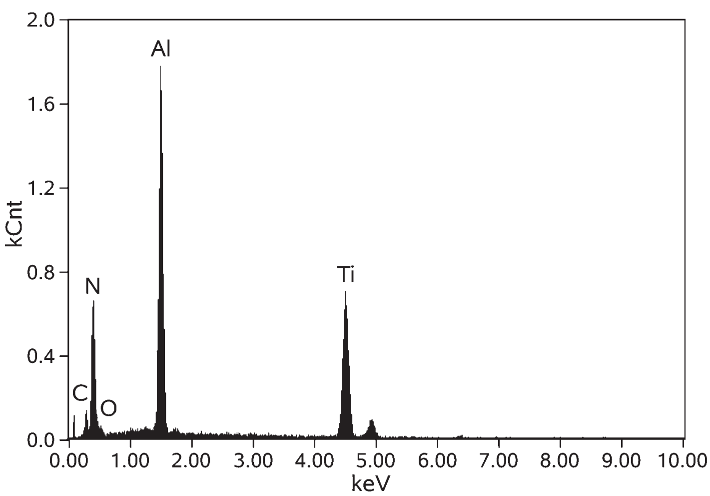

Fig. 10. Spectrum of microparticle, SEM.

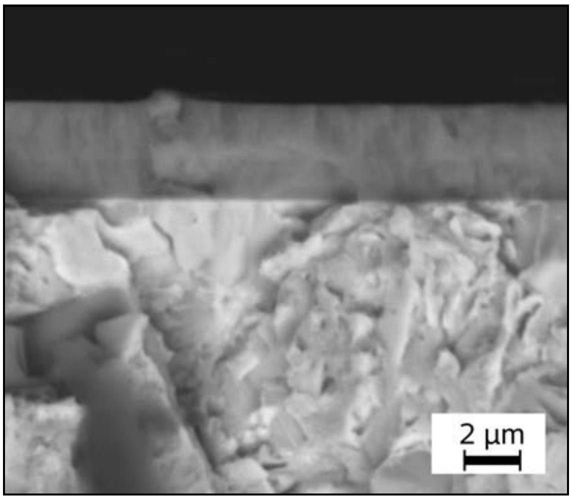

Fig. 11. Structure of TiAlN coating, SEM.

properties which characterize the system thin coating--steel surface. Essentially, it involves resistance of the material to plastic deformation produced by applying load to an indentor [4]. Table 5 shows the values of microhardness (DHV) of coated specimens. The higher values of hardness were measured for 4 μm thin coating deposited at both bias voltage of substrate.

For evaluation of adhesive forces in the system thin coating-basic material is in practice used most often the scratch test. The obtained values of critical forces which represented degree of coating adhesion to substrate are summarized in Table 6.

Figures 13 and 14 show the plots of scratch test where it is possible to observe the increase of acoustic signal and friction coefficient with the specimen loading increasing.

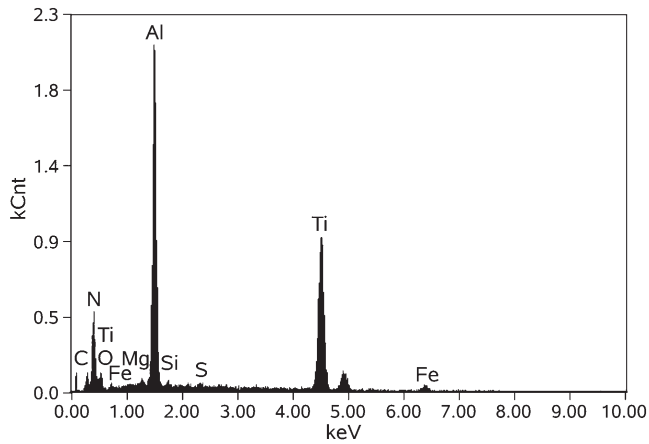

Fig. 12. EDX analysis of TiAlN coating, SEM.

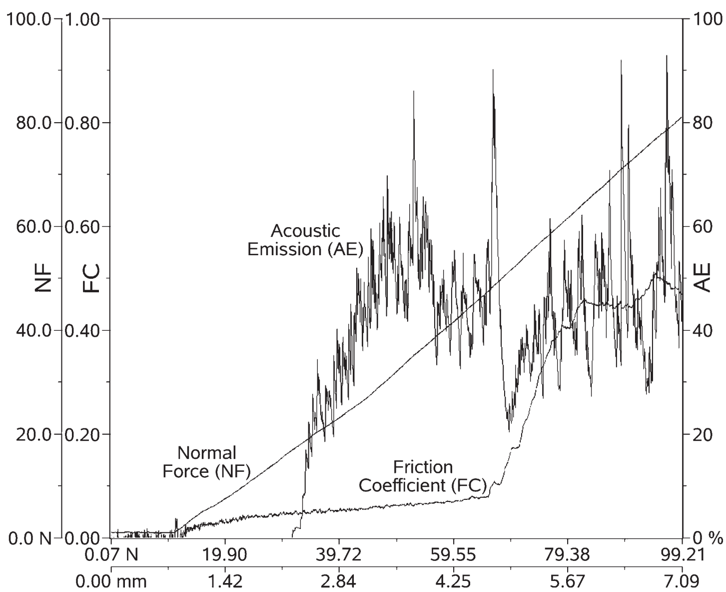

Fig. 13. Diagram of scratch test for 4 μm TiAlN thin film coated on steel substrate at bias voltage of 300 V.

Table 6. The obtained values of critical forces (N)

Sample	Coated system TiAlN-substrate			
	TiAlN (300, 2)	TiAlN (300, 4)	TiAlN (100, 2)	TiAlN (100, 4)
F_{NC} (N)	43	56	50	60

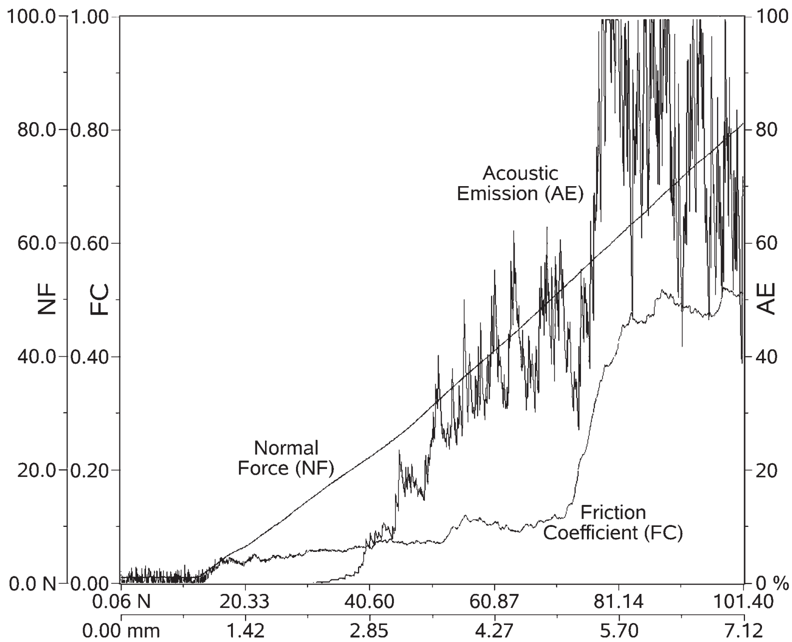

Fig. 14. Diagram of scratch test for 4 μm TiAlN thin film coated on steel substrate at bias voltage of 100 V.

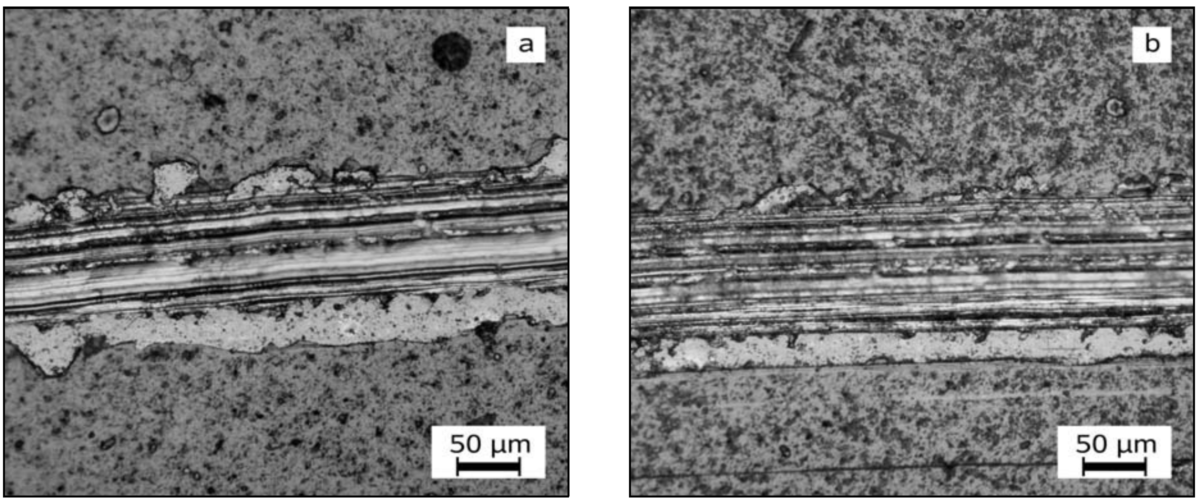

Fig. 15. Scratch test: a) the failure of 4 μm TiAlN thin film coated on steel substrate at bias voltage of 300 V and F_{NC} = 56 N; b) the failure of 4 μm TiAlN thin film coated on steel substrate at bias voltage of 100 V and F_{NC} = 60 N.

The adhesion strength between the 4 μm coating and polished substrate after different bias voltage is documented in Fig. 15a,b. According to [9], coating with $F_{NC} = 50$ N already provides sufficient protection of structural parts against the mechanical loading.

4. Conclusion

On the basis of realized experimental programme and achieved results it can be concluded:

1. The technological conditions of coating depos-

ition process have a remarkable influence on microgeometry of coating surface. The presence of microdrops and cavities on the coating surface has direct influence on the increase of mean arithmetic deviation R_a.

2. Higher values of hardness were measured for coatings of thickness 4 μm deposited at both bias voltage of substrate.

3. Scratch test showed that the best adhesion to substrate was obtained for 4 μm TiAlN thin film coated on substrate at bias 100 V, whereas measured critical force F_{NC} was equal 60 N.

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