STRUCTURE AND PROPERTIES OF PVD COATINGS DEPOSITED BY ARC AND LARC TECHNOLOGY

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ŠTRUKTÚRA A VLASTNOSTI PVD POVLAKOV DEPONOVANÝCH ARC A LARC TECHNOLÓGIOU

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Abstrakt

Príspevok sa zaoberá stanovením štruktúrnej stavby a vlastností tenkých tvrdých povlakov. Povlaky TiAlN a nc-($Ti_{1-x}Al_x$)N/a-Si_3N_4 (nACo) boli na povrch oceľového substrátu deponované metódou ARC a LARC, ktoré patria k PVD (*Physical Vapour Deposition*) technológiám. Vývoj TiAlN povlakov nezostal len na úrovni jednoduchých systémov. V súčasnosti sa v širokej miere využívajú výhody multivrstvového usporiadania TiAlN povlaku, príp. jeho kombinácia s iným tenkým povlakom a úspešne využívajú aj vysoko tvrdé nanokompozitné povlaky. Nástroje a strojné súčasti s PVD povlakmi musia, počas svojej aplikácie v praxi, spĺňať podmienky zachovania dostatočnej kvality. Pred samotnou depozíciou v povlakovacom zariadení je dôležité pripraviť substrát vhodnou voľbou tepelného spracovania na dostatočnú tvrdosť. Povrch substrátu sa ďalej pripravuje leštením a čistením v acetóne. Ďalšie čistenie, predohrev a aktivácia povrchu substrátu sa realizujú priamo v povlakovacom zariadení. Na stanovenie vlastnosti povlakovaného systému sa používajú metódy, ktoré zohľadňujú skutočnosť, že sa jedná o tenké, tvrdé povlaky hrúbky niekoľko mikrometrov. Na štúdium štruktúrnej stavby bola použitá elektrónová mikroskópia. Na stanovenie mechanických vlastností povlakov bolo použité meranie tvrdosti nanoindentačnou metódou Nanoindentačná metóda umožnila okrem tvrdosti, stanoviť aj modul pružnosti tenkého povlaku pri nízkych zaťaženiach (v mN). Na určenie hrúbky povlaku bol použitý Kalotest. Táto metóda umožňuje stanoviť hrúbku povlaku, a zároveň usporiadanie jednotlivých vrstiev vo viacvrstvovom povlaku. Odolnosť voči opotrebeniu bola testovaná Rockwellovou metódou. Táto metóda je založená na zaťažovaní Rockwell indentora do povrchu vzorky použitím konštantného zaťaženia. Odolnosť voči šíreniu trhlín pozdĺž rozhrania je kritériom adhézie povlaku k substrátu. Na stanovenie kvality povlaku sa využíva HF stupnica. Výsledkom testu bolo stanovenie adhézne – kohéznych vlastností povlakov. Stopy po jednotlivých testoch, boli pozorované mikroskopicky.

Abstract

The paper presents methods for assessment of structure and properties of thin hard coatings. The coatings were TiAlN a nc-($Ti_{1-x}Al_x$)N/a-Si_3N_4 (nACo) were deposited on the steel substrate by ARC and LARC coating technology, which belong to PVD (*Physical Vapour*

Deposition) technologies. The development of TiAlN coatings did not stop at the level of simple systems. Currently the advantages of multilayer arrangement of TiAlN coating or its combination with other thin coatings have been used extensively and the utilization of high hard nanocomposite coatings is successfully. Tools and machine parts protected deposited PVD coatings have to show sufficient quality during their application in practice. Before deposited process of coating by a coating machine the substrate must be adjusted to appropriate hardness by suitable heat treatment. Then the substrate surface is further prepared by polishing and acetone cleaning. Additional cleaning, preheating and activation of substrate surface are accomplished directly by the coating machine. A methods is available for determination of properties of the coated system that consider the fact that they have to deal with thin hard coatings only several micrometers thick. Electron microscopy was used in our study to investigate the structure of coatings. For determination of coating properties was used hardness measurement by nanoindentor. Nanoindentation method allowed us to measure hardness and modulus of elasticity of thin coatings also at low loads (in mN). Thickness measurements were carried out with *Calotest*. This method enables the configuration of particular layers in multilayer coating. The wear resistant of coated materials was tested by Rockwell test. This test is based on forcing the Rockwell indenter into a surface of the specimen using a constant load. The indentation test results in formation and increase of cracks. Resistance to increase of these cracks along the interface is adhesion criterion of coating to the substrate. HF scale used for evaluation of coatings quality. The result of this test was estimate of adhesive – cohesive behaviour coatings. Wear tracks after several tests was evaluated by microscopy.

Keywords: PVD, coating, hardness, Calotest, Rockwell test

1. Introduction

Many working properties of materials are determined by the status and quality of their surfaces. Practical applications of tools and machine parts cause changes in their surface properties resulting in decreased hardness, resistance to wear, dimension stability and overall decrease in reliability and service life. Currently, many surface treatment technologies are available aimed at increasing the surface quality. They include thermal processing techniques, chemical-thermal processing, surface alloying and mechanical processing methods. More advanced surface treatment is based on technologies of physical and chemical deposition of thin coatings. The essence of physical deposition of thin coatings, referred to as PVD *(Physical Vapour Deposition)* processes, is condensation of evaporated coating material on the surface of coated material (substrate) [1]. The layer produced is noted for high hardness, wear resistance and chemical and thermal stability. The ever increasing demands of practice resulted in development of new types of PVD coatings or their potential combinations and arrangements. This involves monolayers, multilayers, gradient or other way structured layers. Proportion of TiAlN and nanocomposite coatings on the cutting tools shows a constant increase. In order to assess the suitability of coatings for concrete applications it is necessary to describe and define their properties. The thickness of PVD coatings is mostly in the micrometre range. To determine mechanical properties, such as hardness, one must use methods ensuring that the values of hardness of the thin coatings are not affected by properties of the substrate. Deposition of coatings onto material surface produces coated systems the characteristics of which are determined by methods describing adhesion-cohesion behaviour of the entire system.

2. Material and experimental procedure

Thin coatings on the steel substrate were prepared by vacuum arc cathodic evaporation by low voltage arc evaporation technology used LISS, a.s. Company. The hardness of high - speed steel S 600 fy Böhler after applied heat treatment was 63 HRC. The structure of steel consists of martensite and carbides [1]. Chemical composition of steel is summarised in Table 1.

Table 1 Chemical composition of steel S 600

Steel	Chemical composition according to Böhler								
S 600	C	Mn	Si	P	S	Cr	Mo	V	W
	0.88	0.35	0.22	0.021	0.011	4.12	4.97	1.77	6.50

TiAlN was deposited by ARC PVD technology. The material is evaporated and concurrently ionized by the arc from electrodes. The ionized material is accelerated towards the substrates by negative bias, which is applied to them. While traveling it ionizes atoms of gas atmosphere (N_2, Ar) as well. The ionized atoms create the actual deposited coating by surface reaction when reaching the substrate. Conventional PVD units are equipped with so-called planar electrodes (they are flat, in a shape of a plate or a small ring), on which excessive and centralized erosion and substantially earlier wear occurs when applying a strong magnetic field. Nanocomposite coating (nACo) nc-($Ti_{1-x}Al_x$)N/a-Si_3N_4 prepared by PVD technology based on low voltage arc and using two cylindrical central cathodes, which were made from Ti and AlSi (LARC - Lateral Rotating Arc – Cathodes). The preparation nanocomposite coatings require an evaporation of a great volume of ionized particles and their large ionization. This condition stands in not only for the electrode material but for the atmosphere particles, too. Both can be achieved by using a relatively strong magnetic field, which in a needed way influences the arc burning [2].

Described techniques ensure their high wear resistance under high - speed machining conditions when cutting tool oxidation wear is dominant. The major cause of the high wear resistance of TiAlN and nc-($Ti_{1-x}Al_x$)N/a-Si_3N_4 coatings during high - speed machining is the formation of the protective alumina films on the cutting tool surface. This film is formed at the surface of TiAlN coating during high – speed machining. The tendency of the work piece material to adhere is reduced [3]. Technological parameters of PVD process in Table 2 are given.

Table 2 Deposition conditions of PVD process

Coating	Technological parameters				
	Substrate - steel	Substrate temperature (°C)	Arc current on the electrode (A)	Bias (V)	Pressure (mbar)
TiAlN	S 600	430	AlTi/180	40	1.0
			Ti/210	160	
nACo		450	AlSi/82	75	1.4
			Ti/100		

The structure of thin coatings was observed by scanning electron microscopy. The structure of thin coatings is determined particularly by technological parameters of the deposition process, namely the substrate temperature, bias on the substrate and pressure of working gas [4]. High hardness is one of the basic parameters of quality of abrasive resistant coated materials. Nanoindentation methods allow measurement of hardness and modulus of elasticity of thin coatings [5]. The thickness of coatings deposited on cutting tools is important for their practical applications as it affects the cutting life of these tools. Calotest enables determination of thin coating thickness and configuration of particular layers in multilayer coating [6]. The Rockwell test is one of the basic and most frequently used tests for determination of adhesion of thin coatings to the surface substrate [1]. The HF scale used for evaluation of coating quality is presented in Fig. 1

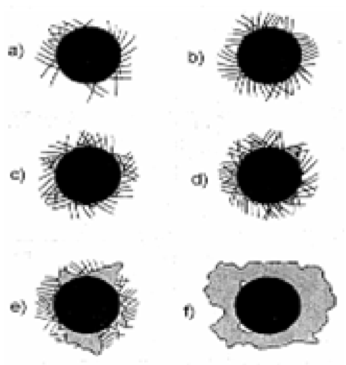

Fig.1 Evaluation of adhesion by the indentation test: a) good adhesion HF1, b) acceptable adhesion HF2, c) decreased adhesion d) decreased adhesion, e) f) insufficient adhesion [1]

3. Experimental results and discussion

Structure of the specimen surface with thin coatings on steel surface and EDX analysis coated specimens are documented in Fig. 2 - 5.

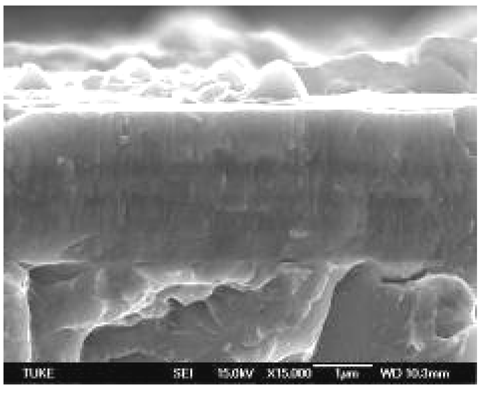

Fig.2 The specimen surface with TiAlN coating, SEM

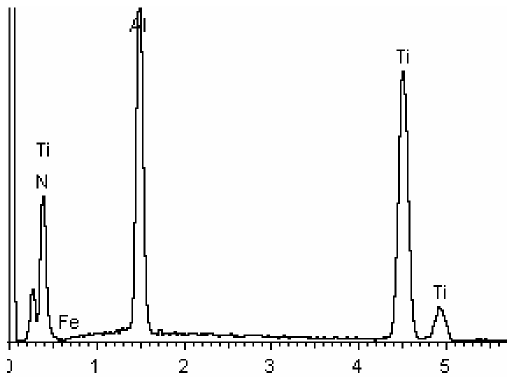

Fig.3 EDX spectrum of TiAlN coating, SEM

The structure of TiAlN coating was columnar. The structure of nanocomposite is compact and we can dispose it, according to Thornton model, to T-zone [1]. The character of structure depends on technological parameters: substrate temperature, gas pressure and acceleration voltage on the substrate. Existence of microparticles, on the surface coated samples of both coatings, (Figs. 2 and 4) were formed during PVD process resulted in the increases of coated surface roughness after deposition [4].

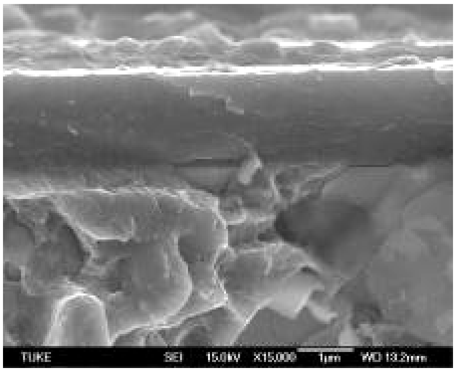

Fig.4 The specimen surface with nACo coating, SEM

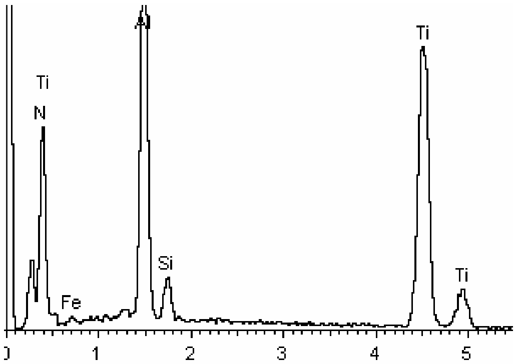

Fig.5 EDX spectrum of nACo coating, SEM

Nanoindentation method carried out by NanoIndentor XP apparatus, is allow for measuring hardness at very low loads (in mN). This is due to the fact that it is possible to define depth changes in hardness and indentation modulus of elasticity in the course of penetration of the indenter into the material. The values measured are used to calculate the hardness and module of elasticity of thin coating. In Fig. 6 and 7 are documented the results of hardness test. Result of nanoindention test are summarized in Table 3.

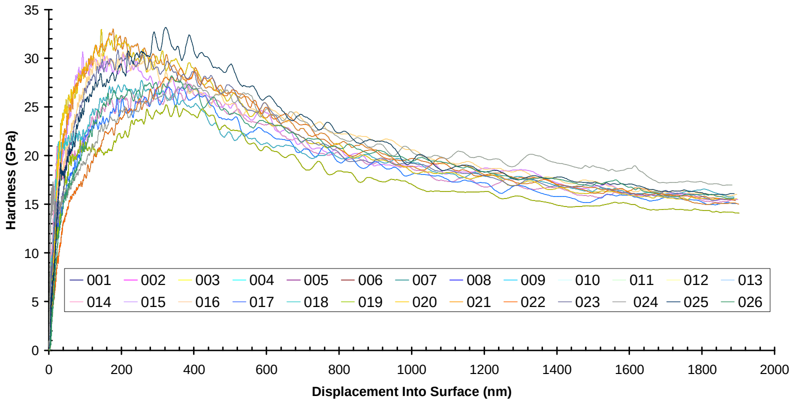

Fig.6 Relationship between hardness and the depth of indentation, TiAlN coating

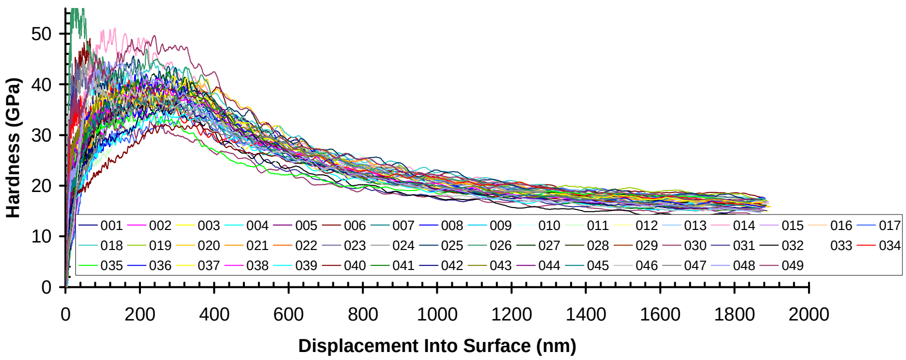

Fig.7 Relationship between hardness and the depth of indentation, nanocomposite

Table 3 Nanoindentation test

Coating	TiAlN	nanocomposite
Module of elasticity E [GPa]	375±42	400±82
Hardness [GPa]	28±2	38±4

Calotest is method for assessment of thin coating thickness. The results of Calotest are documented in Fig. 8 and 9.

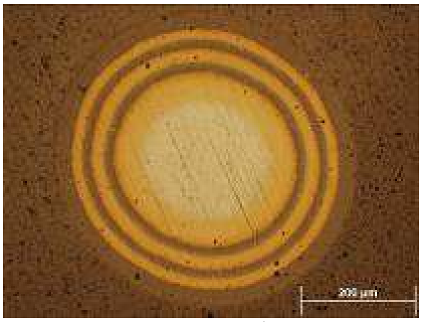

Fig.8 Calotest. TiAlN coating of 2.6 µm thickness

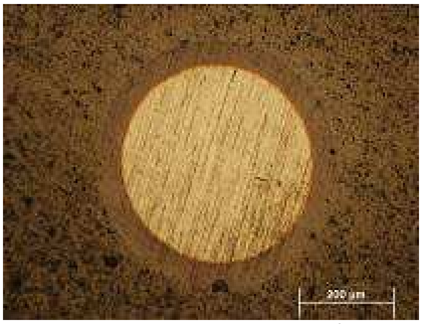

Fig.9 Calotest.nc-(Ti1-xAlx)N/a-Si3N4 of 1.8µm thickness

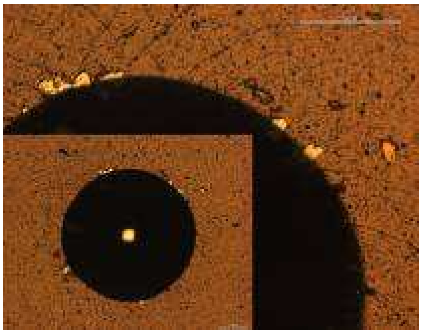

Fig.10 Rockwell test. TiAlN coating

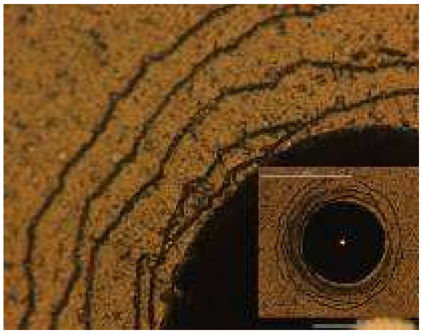

Fig.11 Rockwell test; nACo coating

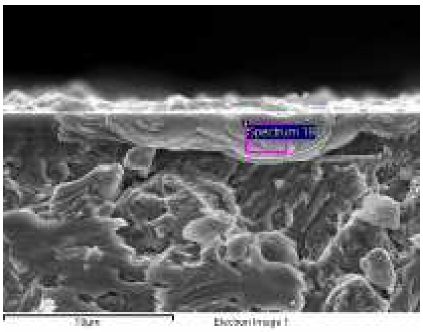

Fig.13 Spectrum of locality (see Fig. 10), SEM

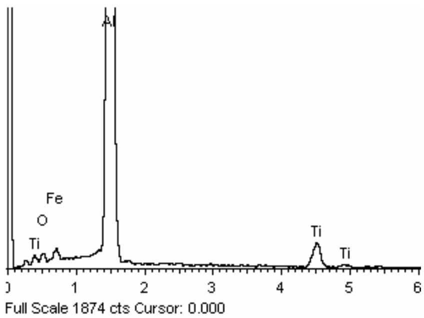

Fig.12 The locality with high average of aluminium, SEM

Adhesive properties were realised by Rockwell indentor. The results of measurement were evaluated according to HF scale (see Fig. 1) and they documented in Figs. 10 and 11. The

TiAlN coating has acceptable adhesion HF2. The coating was flaking along cracks. The places with high aluminium average, Fig. 12 and 13 and Table 4 were identified by scan microscopy in these localities. Coating nc-$(Ti_{1-x}Al_x)N/a-Si_3N_4$ has a good adhesion, HF1 with a few cracks. A good adhesive behaviour is dependent on the qualities of substrate surface preparation and accurate selection of technological parameters of deposition process.

Table 4 Chemical composition of locality into TiAlN coating

Element	Weight (%)	Atomic (%)
Al	86.99	89.67
Ti	6.50	3.78
Fe	3.83	1.91

4. Conclusion

The set of observations performed together with the measurement of properties of the system thin coat - steel substrate allowed us to conclude the following:

1. The character of structure depends on technological parameters: substrate temperature, gas pressure and acceleration voltage on the substrate. The structure of TiAlN coating was columnar. The structure nc-$(Ti_{1-x}Al_x)N/a-Si_3N_4$ was compact.
2. Hardness of the deposited thin coatings is higher than the hardness of the steel substrate.
3. Calotest is method for assessment of thin coating thickness. The thickness of TiAlN coating was 2.6 μm and nc-$(Ti_{1-x}Al_x)N/a-Si_3N_4$ was of 1.8 μm.
4. The adhesion properties of the coated system were evaluated by Rockwell test. The samples coated by TiAlN had a acceptable adhesion according to HF scale. The microlocalities with high average of aluminium caused the lower adhesion of TiAlN coating. Nanocomposite coating had a good adhesion, HF 1.

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