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Experimental Methods of Assessment of PVD Coatings Properties

Mária HAGAROVÁ

Department of Materials Science, Faculty of Metallurgy,
Technical University of Košice, Slovakia

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Abstract

The paper investigates the properties of thin coatings deposited by Physical Vapour Deposition (PVD) technology. This is a modern technology of depositing thin coatings onto the surface of machine parts. Technological procedures of deposition of thin coatings undergo constant improvement. At the same time, new types of coatings are developed. These developments necessitate evaluation of structure, composition and properties of the coatings. The basic methods of determination of properties of thin coatings include determination of their thickness and hardness, structure and chemical analysis, adhesion-cohesion and tribological properties.

Key words : PVD, coating, experimental methods, properties

Introduction

Thin PVD coatings deposited onto surface of machine parts and tools are widely used in a range of sectors, for example in machine and electrical engineering and medicine. Hard thin layers are deposited particularly onto cutting tools, forms and other mechanically stressed parts. These materials are used in operations which need high demands on their hardness, wear resistance and also thermal and chemical stability of the surface. Coatings produced by physical vapour deposition from the metal vapours or alloys applied to the surface of the stressed materials satisfy conditions related to maintaining sufficient quality of the coated parts during their application in practice. The coating itself must be of high quality and fulfil all predetermined requirements.

Various methods have been used to determine the properties of the coated materials. The following paragraphs describe some basic methods of determination of physical and mechanical properties of thin PVD coatings.

Methods of Evaluation of Thin Coatings Properties

Hardness

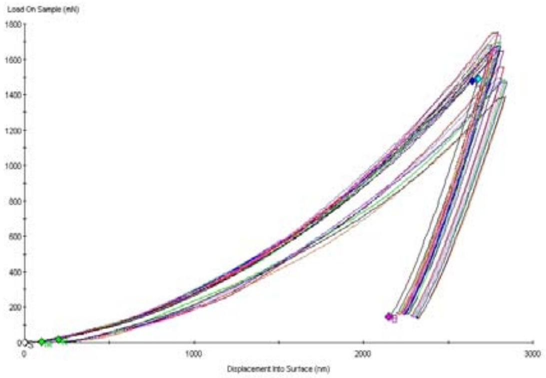
Hardness of materials is in principle their resistance to local plastic deformation induced by applying a load on an indenter.[(1)] A cause of tool wear is abrasive wear. Therefore, high hardness is one of the basic parameters of quality of abrasion-resistant coatings.[(2)]

The thickness of PVD coatings most frequently reaches several micrometers. When measuring the hardness one must make sure that the measuring tip penetrates no deeper than 1/10 of the coating thickness.[(3)] This will ensure that the measurement of hardness of the respective layer will not be affected by properties of the substrate. Nanoindentation methods allow for measuring hardness at very low loads (in mN) while the instrument records and analyses reaction of the material to the respective way of loading. 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 the thin coatings. Figure 1 shows an apparatus NanoIndenter XP, designed for measurement of hardness and modulus of elasticity of thin coatings.

Figure 1. NanoIndenter XP Apparatus

Figure 2 depicts the relationship between the loading force and the depth of indentation recorded during the measurements.

Figure 2. Relationship between the applied load and the depth of indentations for TiAlN coating [4]



Indentation curves correspond to the course of hardness and elasticity modulus in dependence on the depth of indentation, Figsure 3 and 4.

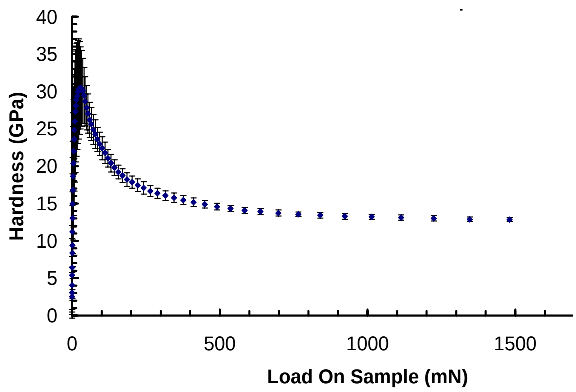


Figure 3. Relationship between hardness and the depth of indentation [4]

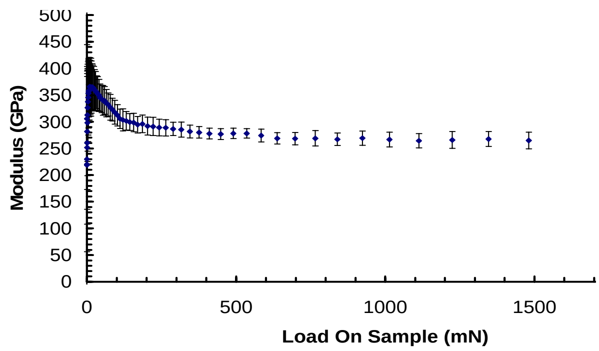


Figure 4. Relationship between elasticity modulus and the load of indentation [4]

Thickness

The thickness of coatings deposited on cutting tools is one of the most important characteristic for their practical application as it affects the cutting life of these tools. Rapid and simple determination of thin coating thickness is carried out by a Calotest, Figure 5 The principle of this method is that a depression in the shape of a spherical cap is abraded in the specimen by means of a rotating steel sphere, Figure 5 By determining dimensions of the depression under a microscope, needed for calculation, Figure 6 one can determine the coating thickness according to the formula:

$$h = \sqrt{R^2 - r_2^2} - \sqrt{R^2 - r_1^2}$$

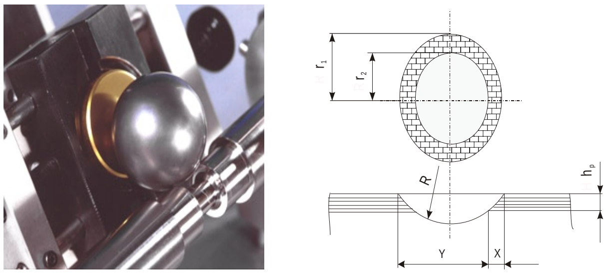
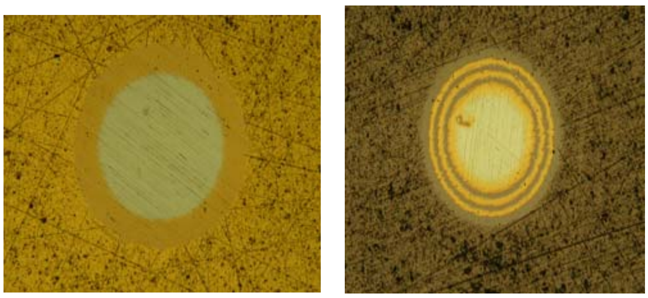


Figure 5. "Calotest" Apparatus [5] and determination of coating thickness by a Calotest [6]



a)

Figure 6. The trace in the coating after Calotest testing: a) single-layer, b) multilayer coating [7]

Another method for determination of coatings thickness is the fracture surface obtained by brittle failure of the specimen at the temperature of liquid nitrogen.

Observation under an electron microscope allows determining not only thickness of the coating but also its structure, Figure 7

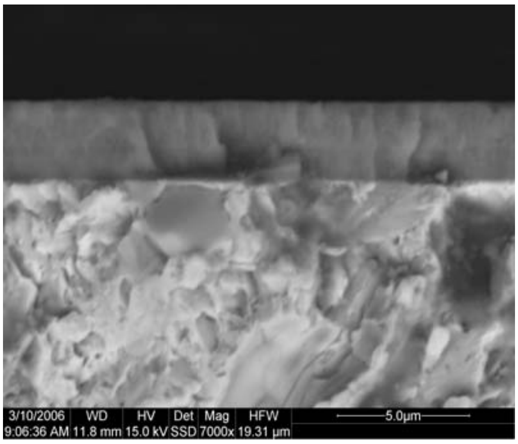


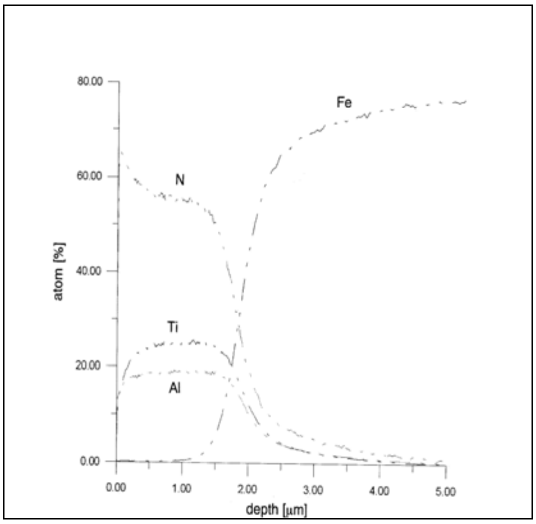
Figure 7. Determination of thickness and structure of the TiAlN coating by SEM [8]

Evaluation of Chemical Composition

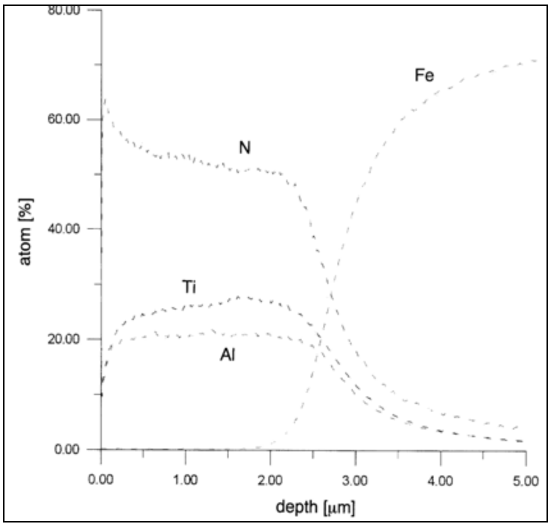
Another important characteristic affecting the properties of coated systems is the concentration profile of individual elements in dependence on depth from the surface. GDOES analysis (Glow Discharge Optical Emission Spectroscopy) allows to document the depth concentration profiles of layers in wt. % proportion of atom. An optic spectrometer, Figure 8 uses a discharge arc as an excitation source (the so-called Grimm lamp) to excite atoms in the specimen. In this process the radiation of wavelength typical of the respective element is obtained.[(5)]

Figure 8. Spectrometer [5]

The procedure described provides a concentration profile dependent on depth of deducting, Figure 9. The graph presented allows for reading the coating thickness with relatively good accuracy.



a)



b)

Figure 9. GDOES depth profile of: a) 2 µm TiAlN, b) 4 µm TiAlN, deposited on steel substrate [8]

Evaluation of Morphology of Coating Surface

The macro-particles which develop during the deposition process and remain incorporated in the coating are a source of increased roughness of the coating surface, Figure 10.[(6)] The surface roughness increases the cutting forces which results in a considerable thermal and mechanical stress, for example of cutting edges of tools. This negative influence can be eliminated particularly by modification of the technological procedure of coating or by adjustment of the surface of coatings.[(2)]

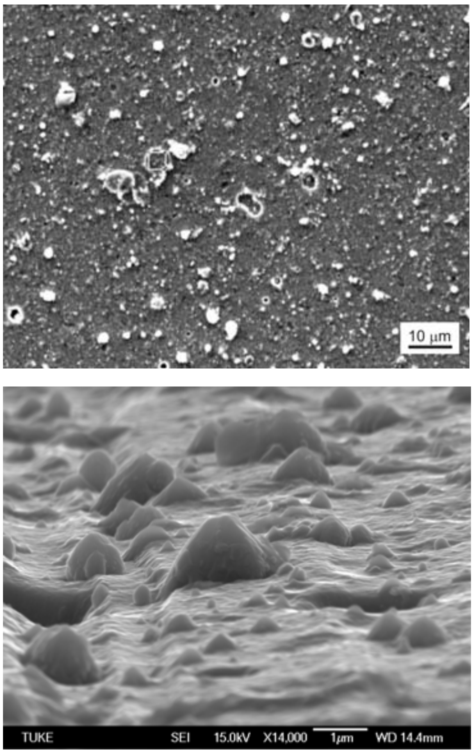


Figure 10. Surface of the TiAlN coating with incorporated macro-particles [8]

Morphology of the surface is most frequently measured with a profilemeter, Figure 11 After the measurement, the micro-geometrical characteristics of the surface are defined and evaluated according to the respective standards.(9, 10) Another method is based on evaluation by Atomic Force Microscopy.(2)

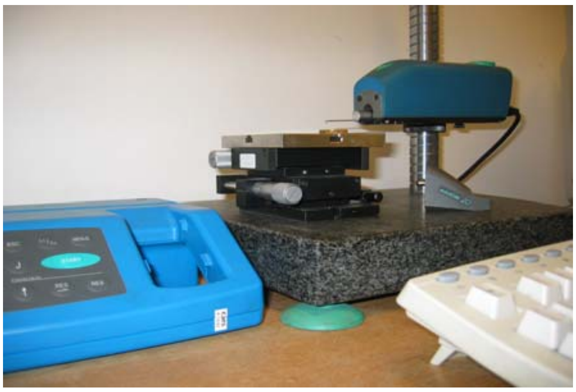
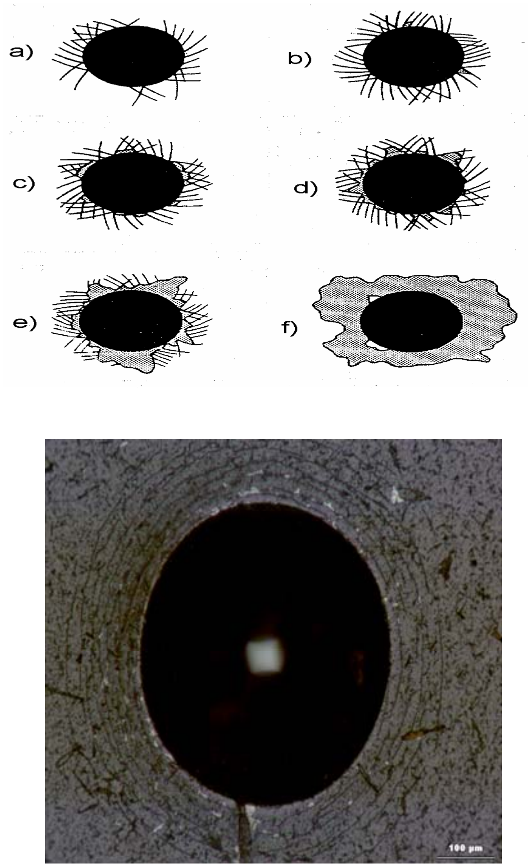


Figure 11. Profilemeter Hommel Tester T 1000

Adhesive-Cohesive Behaviour of Coatings

Maximum utilization of deposited abrasion-resistant coatings is possible particularly in those cases in which excellent adhesion of coating and the substrate is ensured. In order to achieve high quality coating-substrate adhesion, superior preparation of the substrate (its thermal processing and adjustment of the surface by polishing and cleaning) and correct selection of technological parameters of the deposition process must be ensured.(2) The methods most frequently used in practice to determine adhesion are indentation and scratch tests.

The indentation test is carried out by means of a hardness tester. It is based on forcing the Rockwell indenter into a surface of the specimen using a constant load of 1,500 N. The indentation test results in formation and propagation of cracks. Resistance to propagation of these cracks along the interface is a criterion of adhesion of coating to the substrate. Figure 12 shows the HF scale used for evaluation of coatings quality(5) and results of the indentation test.



g)

Figure 12. Evaluation of adhesion by the indentation Test : a) good adhesion, b) acceptable adhesion, c) d) decreased adhesion, e) f) insufficient adhesion[5] g) acceptable adhesion of the TiAlN coating deposited on PM steel [4]

Adhesion-Cohesion Properties of Thin Coatings

The scratch test is one of the basic and most frequently used tests for determination of adhesion of thin coatings to the surface of substrates. The principal mechanism of the "scratch test", Figure 13 is the movement of the specimen and gradual penetration of the indenter into the specimen surface. Either constant or continuously increasing force is applied to the indenter which penetrates into the specimen surface and produces a scratch. The load that results in damage to the coating is called critical load and is a criterion of adhesion of the coating to the substrate.(1)

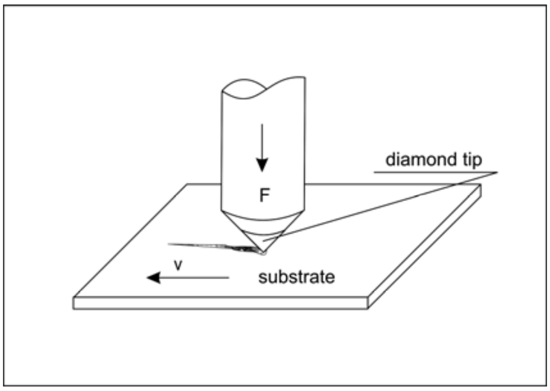


Figure 13. Principle of "scratch test"[9]

This test allows to scan the signal of acoustic emission as well as observe the small subsurface cracks which develop due to the external stress. The apparatus records the course of the force applied to the indenter, magnitude of friction coefficient and signal of acoustic emission, Figure 14

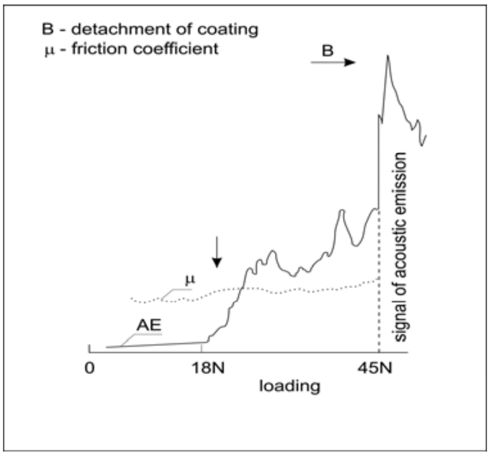
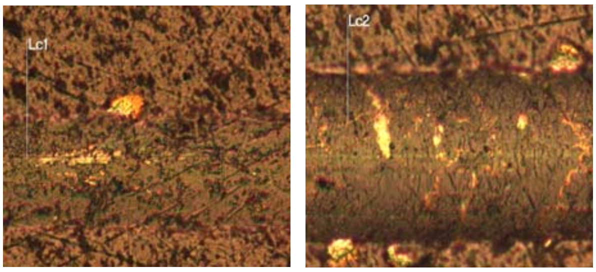


Figure 14. The graphical record of scratch test [6]

Another way of evaluation of the scratch test is the microscopic observation. By using light or electron microscope it is possible to identify the sites of the first failure of the coating Lc_1, its first adhesion failure Lc_2, first large scale adhesion failure Lc_3 and complete exposure of the substrate Ls.(5) Figure 15a shows the site of the first failure of the coating Lc_1, and Figure 15b the site of the first adhesion failure of the coating Lc_2.

The accuracy of the values measured after the "scratch test" is affected particularly by the state of the surface, feed speed of the specimen, rate of loading and wear of the indenter tip.(1)



a) b)

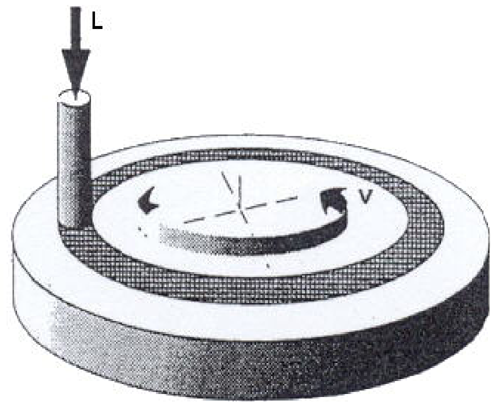
Figure 15. Microscopic analysis of the scratch test: a) site of the first failure of the coating, b) site of the first adhesion failure of the coating [7]

Tribological Properties

Friction between two surfaces is accompanied by processes of wear, either abrasive or adhesive. Tribological tests by means of a tribometer, Figure 16, are intended to determine first of all the friction coefficient and its changes during the test which also provide information about adhesion-cohesion behaviour of the material.(5)

Figure 16. CSM High temperature tribometer

The principle of the "pin-on-disc" test, Figure 17 a, is that a well-fixed stationary testing body in the shape of a sphere or pin is forced into the surface of a specimen disc rotating at a speed v by a defined force L. This is done with a disc of a predetermined radius r and defined number of cycles N. The direct output of the measurements is the course of friction coefficient in dependence on the number of cycles, Figure 17 b.



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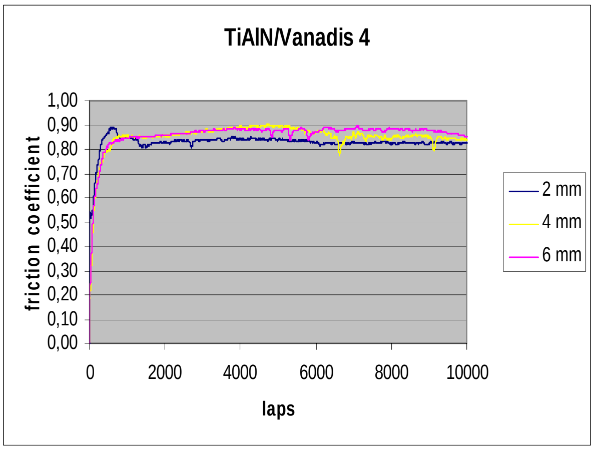
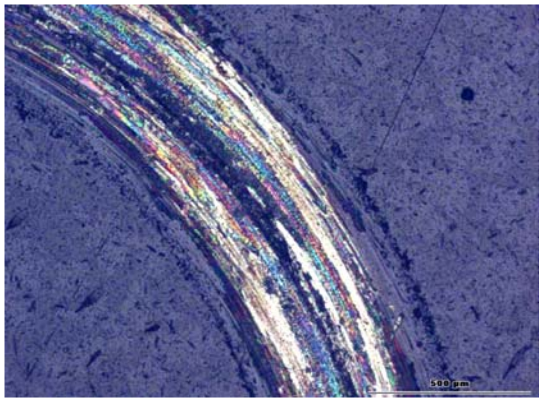


b)

Figure 17. Tribological test "pin-on-disc" : a) principle of the method [11], b) course of the friction coefficient in dependence on the number of cycles for the defined radius *r* [4]

The wear track appearance after the "pin-on-disc" test is evaluated by light and electron microscopy. Figure 18 shows the tribological wear track observed under a light microscope. Evaluations include the character and dimensions of the tribological track observed on the specimen, intensity of wear of the "pin" and proportion of particles adhered to the wear track after the test.(5)



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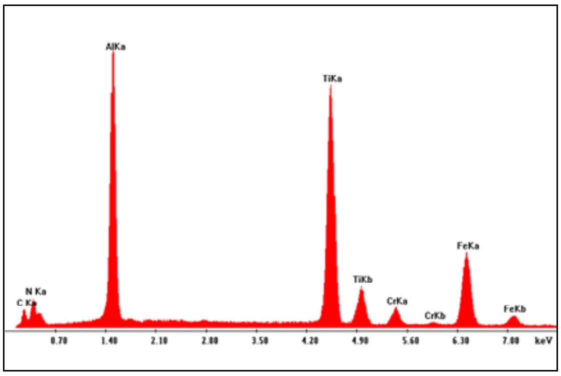

b)

Figure 18. "Pin -on - disc" test: a) tribological track on the surface of a specimen with deposited TiAlN coating, b) EDX analysis of tribological track [4]

Conclusion

Thin coatings applied to the surface of machine parts and tools must comply with the relevant requirements, such as high hardness, resistance to wear and thermal and chemical resistance. Determination of properties of thin PVD coatings by correctly selected methods contributes to their successful application in the practice.

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