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Adhesive-cohesive behaviour of the system with thin layers*

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Process of thin coating deposition strongly influences the behaviour of the whole thin coating-substrate system in operation. Thin layer is not selfportable and therefore the deterioration of the whole system should be investigated in details. In paper adhesive-cohesive behaviour of steel substrate (carbon and highspeed steels) with TiN thin layer by help of Vickers hardness test is investigated.

1. INTRODUCTION

Contemporary wide sort of materials is a product of allworld many years passing intensive research and development.

Permanently higher requirements are puct on these materials with respect to their applied properties. Surface treatment by help of thin and thick coatings modification of material surface, etc. bring improvement of surface properties. Thin coatings find wide application in microelectronics, optics, machinery, automobile industry, etc.

Many applicable properties are determined by surface state (surface roughness different kinds of abrasion, different kinds of damage, oxidation, corrosion, etc.). Mechanical loading of body surface changes surface properties and nucleation and extension of first defects are then stimulated. Service life and corrosion resistance of material are dependant on extension velocity and abrasion intensity and on working conditions of material.

Special thin coatings manytimes prolong service life and improve corrosion resistance.
Basic material being superfacially treated is an important parameter and strongly influences the process of thin coatings deposition and behaviour of the whole-system „thin coating-substrate” in operation conditions. Thin layer is not selfportable and therefore the deterioration of the whole system „thin layer-basic material” and modification of properties of the whole system after certainly defined load modes should be investigated.

Evaluation of adhesive-cohesive behaviour of „thin layer-substrate” system in comparison with cohesive behaviour of basic material is the important area.

In the paper adhesive-cohesive behaviour of steel substrate (carbon and highspeed steels) with TiN thin layer on the base of macroindentations developed by Vickers hardness tests is investigated.

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2. ADHESIVE BEHAVIOUR OF „THIN LAYER-SUBSTRATE“ SYSTEM (L-S)

Adhesion of the layer to the substrate is one of the most important parameters deciding succesfull application of thin layers for increase of tools and components service life.

Adhesion is defined as a state when two surfaces are mutually kept by bond forces influencing the interface of these surfaces [4].

Adhesion of the layer to the substrate is significantly influenced by the intensity of atoms and ions kinetic energy incidencing on the surface during deposition; it can be influenced by deposition parameters [3].

Adhesion of sputtered layers is dependent on the bond of layer to substrate, on the development of sliding stresses on the interface „layer-substrate“ and on the layer microstructure.

On the interface following bonds can be found [3]:

- chemical-atoms existing in positions suitable for mutual sharing of electrons
- van der Waals – result of polarizations between atoms
- electrostatic – result of creation of electrical doublelayer between layer and substrate; at some metal – dielectricum interfaces is comparable with van der Waals bond
- mechanical – connection based on impinging of layer into unequities of substrate in equidistant direction with interface

For the existence of perfect bond is necessary to remove from the surface of substrate weakly bonded atoms and molecules of impurities adsorped on surface.

If the crack appears on the L-S interface – adhesive deterioration takes place. If the crack appears in L or S – cohesive deterioration takes place [6].

3. METHODS OF MEASUREMENT AND EVALUATION OF ADHESIVE-COHESIVE BEHAVIOUR OF SYSTEMS

Energy necessary for deterioration of bonds on interface L-S is decisive. Methods of layers adhesion determination are based on creation of defined stress on interface and determination of critical value, at which interface is deteriorated and a part of layer is spalled.

Most of methods are destructive or nonusable for checking of different parts with layer and it is very difficult to compare them. Optimum method is dependent on the layer type, its thickness and properties.

Adhesion measuring methods are usually divided into three groups: nucleation methods, mechanical methods, combined methods.

In practice mechanical and some combined methods are used. At the evaluation of adhesion by mechanical methods the energy necessary for deterioration of bonds on the L-S interface is evaluated. Adhesion is evaluated by the value of force necessary for spalling of layer related to the unit of plane or by magnitude of work connected with it.

Mechanical methods for adhesion determination [6]:

Qualitative:

- Strip test. Spalling test
- Abrasive test
- Band slide and tensile test

Quantitative:

- direct tensile method
- laser spalt test
- indentation test
- ultracentric test
- scratch test

3.1 Indentation (penetration) tests methods for evaluation of systems

Indentation tests (hardness tests) can be divided according more points of view. According to test principle tests can be divided in scratches indentations, impacts and reflections. According to the rate of loaded force the tests are either static or dynamic. According to measurement reason tests of macro- or microhardness are conducted.
Exact survey of tests is given in table 1.

Table 1: Classification of indentation tests

Macrohardness tests	Static	Scratch	Martens method
		Indentation	Brinell method
			Vickers method
			Rockwell method
	Dynamic	Plastic impact	Free drop method
			Compresed spring method
			Comparable method
		Elastic Reflects	Free drop method (Shore)
			Pendulum method (Duroskop)
Microhardness tests	Static	Indentation	Vickers method at loading > 4,9 N

4. EXPERIMENTAL PROGRAMME

Hard abrasive resistant thin layers were deposited on structural steels 12050, 11500 and high speed steels 19830, 19852. Chemical composition of steels is given in table 2.

Table 2: Chemical composition of steels [% wt.]

Carbon steel 12050

	C	Mn	Si	P	S
Min.	0,42	0,5	0,17		
Max.	0,5	0,8	0,37	0,55	0,05

Carbon steel 11500

	C	P	S
Max.	0,38	0,55	0,05

High-speed steel 19830

	C	Mn	Si	Cr	W	V	Mo	P	S
Min.	0,8			3,8	5,5	1,5	4,5		
Max.	0,9	0,45	0,45	4,6	7,0	2,2	5,5	0,035	0,035

High-speed steel 19852

	C	Co	Mn	Si	Cr	W	V	Mo	P	S
Min.	0,8	4,3			3,8	5,5	1,5	4,5		
Max.	0,9	5,2	0,45	0,45	4,6	7,0	2,2	5,5	0,035	0,035

TiN layer deposition was conducted in deposition equipment NNO 150 working on the principle of low voltage are evaporation in vacuum (PVD method).

Thickness of thin layer was measured by help of „calotest“ method [1]. Results of thickness measurements are following: 12050 – 2,59 μm; 11500 – 2,83 μm; 19830 – 3,62 μm; 19852 – 3,43 μm.

Hardness measurements were conducted by help of macrohardnesstester Ernst AT 180 D, Vickers method. Three loading values were elected: 60 kg, 125 kg, 187,5 kg.

4.1 Macroindentation evaluation

Optical microscope NIKON Optiphot 100S and corresponding software equipments (program Image - Pro Plus and Photo Deluxe) were used for metalographical evaluation.

Material 12050
In L-S system cracks appear also in the neighbourhood of edges and their direction is almost equidistant with indentation edges.

Material 11500
Cohesive deterioration of basic material is dependent on deterioration of individual structure components (fig. 1). In L-S system cracks appear in corners and around edges.

Material 19830
In comparison with previous cases indentation is of regular shape the whole deformation is lower. In L-S system smaller cracks corresponding to direction of diagonal appear in corners.

Material 19852
Structure characteristics is similar to previous one.

Substrate L-S system

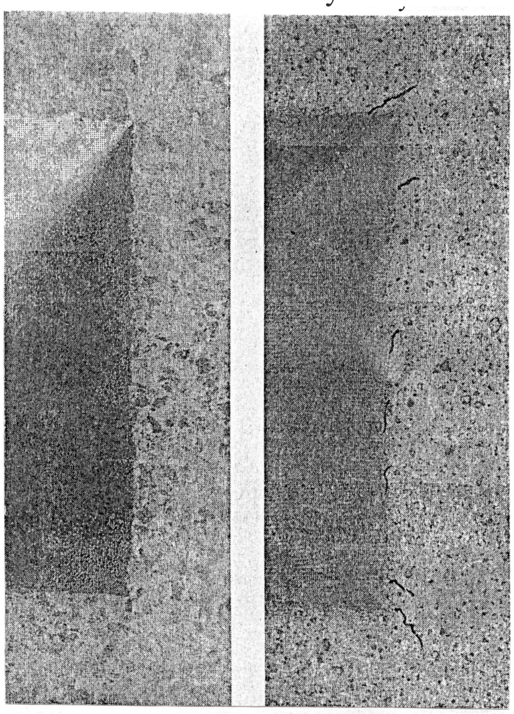

Fig. 1 Macroindentation at indentor loading / 1875,5 kg, 500x

Cross section view of identation in steel 11500 is given in fig. 2. Inner deformation of identations as well as depth deformation were evaluated.

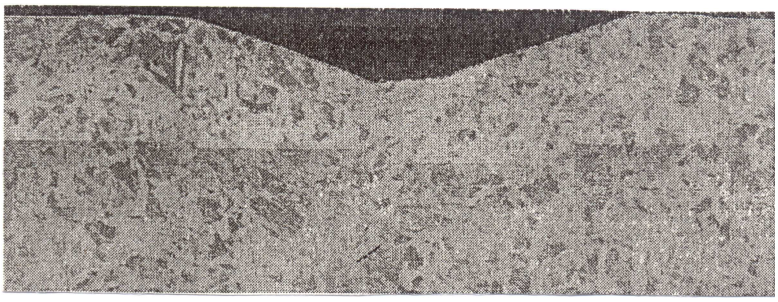

Fig. 2 Cross section of L-S system. Indentor loading 187,5 kg, 500x

5. DISCUSSION OF RESULTS

Macroindentation evaluation was conducted at three different loads of macroindentors and measurements were conducted on four different basic materials and on four L-S systems. Concentrated attention is paid to cohesive deterioration at indentation edges, in corners and closed neighbourhood of indentation.

In substrates of carbon steels indentations are irregular and with increasing load strenghtening of material appears and indentations are more regular than at lower loads. In some corners of indentations fine cracks in direction of diagonal are found. Cohesive deterioration is clearly coherent with structure.

In substrate of highspeed steels indentations are of regular shape in comparison with previous carbon steels. Whole deformation is also much lower. Cohesive deterioration is not initiated by the structure of material.

In L-S systems of carbon steels irregularity of indentations is less caused by strenghtening of surface and edge of indentation. Cracks are extended from corners mostly in directions equidistant with diagonals. Cracks in neighbourhood of edges almost equidistant with edges are appeared. Cohesive deterioration in edges is less extended in comparison with basic substrate.

In L-S systems of highspeed steels indentations are regular, without strong deformation on edges, small cohesive deterioration corresponding to the influence of basic material structure are appeared on edges.

From cross section cut of carbon steel substrate with thin layer deformation of indentation by rounding of edges is evident. Indentation bottom keeps the accuracy of cut till to the center of indentation. No adhesive abruption of layer was found.

In L-S highcarbon steels deformation is smaller and no adhesive deterioration was also found.

From described results is evident, that the extent of deformation of individual materials connected with macroindentation process, cohesive deterioration of substrate and its clear connection with structure composition and hardness, deterioration of thin layer (that is not selfportable) are dependent not also on substrate; it is frequently neglected.

Thin layers deposited on tested materials prove very good adhesive properties and the behaviour of all tested materials is significantly improved.

6. CONCLUSIONS

Realized indentation measurements proved kinds of deformation of material in inner indentation and in its near surrounding and how the individual basic materials are deteriorated. Adhesive-cohesive behaviour of the whole system L-S was studied.

Hard substrate is deteriorated by cohesive cracking, non selfportable layer follows brittle deterioration of substrate, softer substrate is deteriorated by plastic deformation, increased loading strengthens the edges, cohesive deterioration follows individual structure components and is of higher extension than in hard substrate.

Thin layer evidently improves surface properties of thin layer system. TiN layer proves very good adhesion to substrate. Its deterioration is much dependent on cohesive deterioration of substrate. Deterioration of layer is developed from hardness and substrate structure; cohesive deterioration copies cohesive deterioration of substrate.

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