Chapter 15
Wear Resistant Nanostructured Multi-component Coatings

A. Urbahs, M. Urbaha, K. Savkovs, and S. Bogdanova

Abstract The authors offer the restoration technology of precision pair parts – valves, plungers and injectors of vehicle fuel pumps. The technology stipulates the creation of a special restoring wear-resistant coating on the basis of Ti–Al–N (titanium-aluminum-nitrogen) deposited by ion-plasma sputtering. The possibility of combining the methods of electric arc and magnetron sputtering, which arose as a result of the modification of the installation, makes it possible to partially reduce the drawbacks of both methods by simultaneously using their basic advantages. This technique, in particular, gave the opportunity to reduce the drop phase without reducing the efficiency of ion bombardment. In addition, the spectrum of materials being sputtered has been expanded and their quality has been improved.

Keywords Nanostructured wear-resistant coatings • Restoring coatings machine parts surface

15.1 Introduction

Most failures of vehicle power units including failures of hydraulic units occur as a result of damage of regulating and distributing devices as well as plunger and piston pairs of pumps and hydraulic motors. All types of failure and destruction of precision pair parts occur according to one of the performance criteria (wear, corrosion, etc.) and usually start from the surface of a part. All service properties of parts and their parameters are closely connected with geometrical and physico-mechanical properties of surface layers.

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The analysis of different methods of increasing wear resistance (constructive, technological or maintenance) shows that the most perspective method is related to the improvement of part surface properties by creating special protective coatings. Innovative nanostructured coatings, which are created on the basis of ion-plasma sputtering technologies, are of special interest. Coatings of this type differ by technological effectiveness and a wide range of physico-mechanical and service properties [1–3].

15.2 Experiment Techniques

A process installation, which makes it possible to apply the method of ion-plasma sputtering of coatings, represents a vacuum installation that includes a working chamber with a built-in plasma evaporator and an arc initiation system, a power supply source, a gas station for feeding working gas into the chamber as well as measuring and control equipment [1].

Deposition of evaporator material on the surface of parts occurs under the effect of constant electric and magnetic fields, which intensify the process of sputtering and enhance the density of the coating and its adhesion to the base.

The vacuum installation has been modified in order to improve its performance. A planar magnetron sputtering device “Magnetron 2” has been installed instead of one of the arc sources (Fig. 15.1).

The possibility of combining the methods of electric arc and magnetron sputtering, which has appeared as a result of modification of the installation, makes it possible

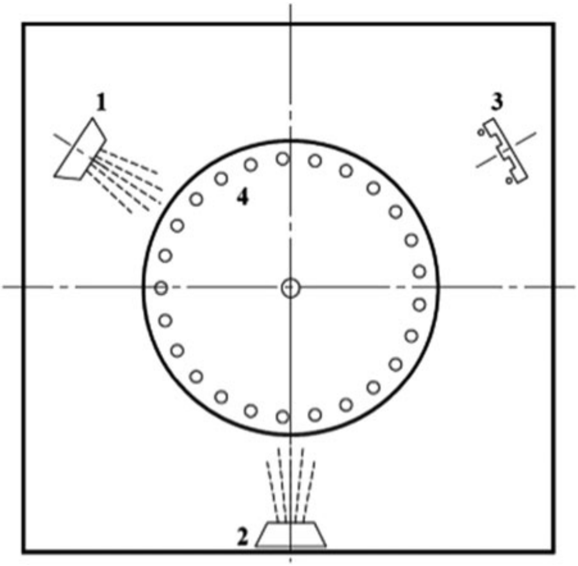

Fig. 15.1 Location of evaporators on the modified vacuum installation: *1*, *2* electric arc evaporators; *3* magnetron; *4* turntable

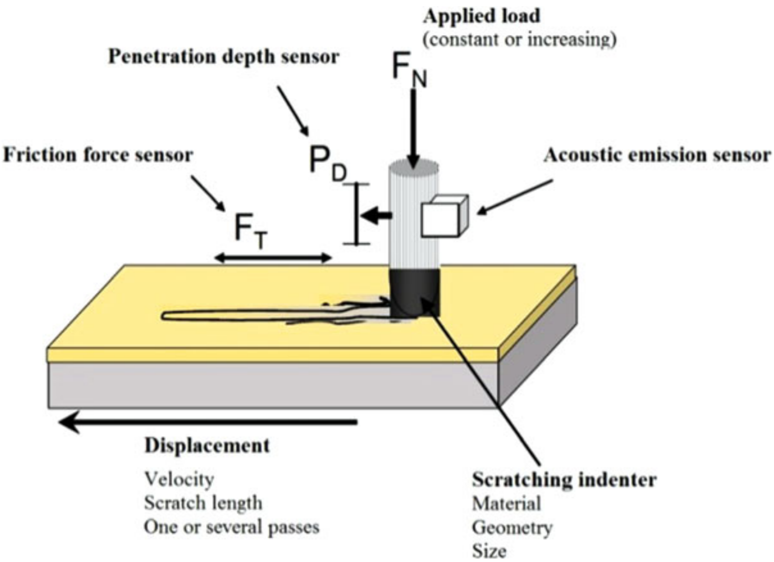

Fig. 15.2 Schematic diagram of operation of CSM Revetest Xpress installation intended for testing adhesion strength of coatings [4]

to partially reduce the drawbacks of both methods by simultaneously using their basic advantages. This technique, in particular, gives the opportunity to reduce the drop phase without reducing the efficiency of ion bombardment. In addition, the spectrum of materials being sputtered has been expanded and their quality has been improved.

In order to carry out the investigation of microstructure of the created coatings on a nano-level, a scanning electron microscope SEM HITACHI–S3000N was used.

The Chemical analysis of composition of the created nanostructured coatings was carried out by the method of micro X-ray spectrum analysis, which is one of the most sensitive analytical methods. For this purpose, a micro X-ray energy dispersive analysis system (EDS) BRUKER-QUANTAX 200 was used.

The measurement of geometric parameters and roughness of coated and non coated sample surface was carried out with the help of a contact profilograph – profilometer "Form Talysurf Intra 50" manufactured by Taylor Hobson company.

The device gives the opportunity to simultaneously measure the size, form and texture of a sample surface.

The investigation of coating adhesion properties on a micro-level and nano-level was carried out with the help of a scratch tester CSM Revetest (CSM Instruments) (normal force range – 0.5...200 N; maximum friction force – 200 N; maximum scratch length – 70 mm; resolution by depth – 1.5 nm) through the testing of samples and products by scratching. The installation was intended for determining adhesion strength, scratch resistance and coating destruction mechanism.

The indenter (diamond or tungsten carbide) (see Fig. 15.2) in automatic mode is moved along the surface of a sample by applying constant, stepwise increasing or

Fig. 15.3 Details of power-plants and hydro-fuel equipment of vehicles

progressive normal load FN. In order to determine the adhesion strength of a coating, the load is increased linearly, in a specified range.

Experimental samples, i.e. steel plates (100 × 30 × 1.5 mm), precision pair (PP) of power-plants for sea and railway vehicles (gas-turbine installations, diesel engines, combustion engines) and details of the hydro-fuel equipment (valve pairs and plungers of hydro pumps, hydro motors, etc.) have been used as the object of the research (see Fig. 15.3).

The test mode for coated plungers complied with the following conditions: the load on indenter FN was increased linearly in the range from 0.3 to 80 N, the loading rate was 3.0 N/s, the rate of indenter moving was 6 mm/min, the scratch length was 3 mm.

During the tests, the recording of different physical parameters depending on the applied load and scratch length was carried out. The moment of coating adhesion or cohesion destruction was fixed after the tests, either visually with the help of an optical microscope equipped with a digital camera or on the basis of a change of one of the five parameters:

- acoustic emission,
- friction force Ft,
- friction coefficient,
- indenter penetration depth.

A coating starts to destroy under a certain critical load. The moment when the critical load is reached is very precisely fixed based on the results of the recording acoustic emission signals (AE). AE signals have been recorded with the help of a portable acoustic emission device (Vallen Systeme GmbH) built in the scratch tester, which makes it possible to record acoustic emission parameters. The range of AE channel frequencies is from 100 kHz to 1 MHz. For multiplication of the initial signal, a preamplifier with constant amplification of 26 dB in frequency range from 100 kHz to 1 MHz has been used. AE sensor SE 150-M (Dunegan Engineering Company) is structurally built in the loading device. During the experiments, the amplitude of AE signals was recorded.

As a result of the tests, the minimum (critical) load (FNc) leading to the destruction of a coating can be determined. The given method complies with the international standard ISO 20502.

15.3 Results and Discussion

One of the multicomponent nanostructured two-layer coatings obtained by the combined method (KJONBOMU – condensation and ion bombardment + magnetron sputtering) is presented in Fig. 15.4.

It is seen from the figures that the influence of the drop phase on the process of coating formation is substantially reduced (sporadic drops of much smaller size are observed), and the coating structure is homogeneous.

The total thickness of the coating obtained by the KJONMOBU method changes within the range of 5...15 μm. The application of the combined method results in a substantial (four times) decrease of the coating roughness.

It has been stated that in coatings obtained by the combined method, the height of separate "drop phase" peaks reaches only 2.25 μm with the average value of roughness parameters Sa = 0.154 μm and Ra = 0.062 μm (see Figs. 15.5 and 15.6).

Alloying of a coating with aluminium (up to 4%...5% of the composition) leads to the formation of nanocomposite structure with crystalline phase TiN base grain sizes 15...20 nm.

The obtained results (see Figs. 15.7 and 15.8) gave the opportunity to analyze the failure mechanism and evaluate the adhesion strength of coatings created by the KJONBOMU method.

The comparative analysis of the experimental investigation data shows that the most informative parameter for evaluation of the failure mechanism and adhesion strength of coatings is the parameter of AE signals amplitude A_{AE} (see Fig. 15.6). At the same time, the behavior of the friction coefficient μ_v and the friction force F_t is comparable with the changes of parameter A_{AE}.

The values of loads that correspond to certain stages of coatings damage have been determined on the basis of the results of AE measurements. For the considered example, the minimum (critical) load F_{Nc1} equal to 25.02 N corresponds to the first peak of AE amplitude and is the evidence of first damages on the microlevel in the system "coating-plunger" (point A Figs. 15.6 and 15.7). Alongside with the substantial increase of parameter A_{AE} when reaching F_{Nc1} the increase of parameters μ_v and F_t is also observed (see Fig. 15.6), which increases the reliability of the conclusions about the appearance of first damages in the coating material. The comparative analysis of structural peculiarities of the scratch gives the

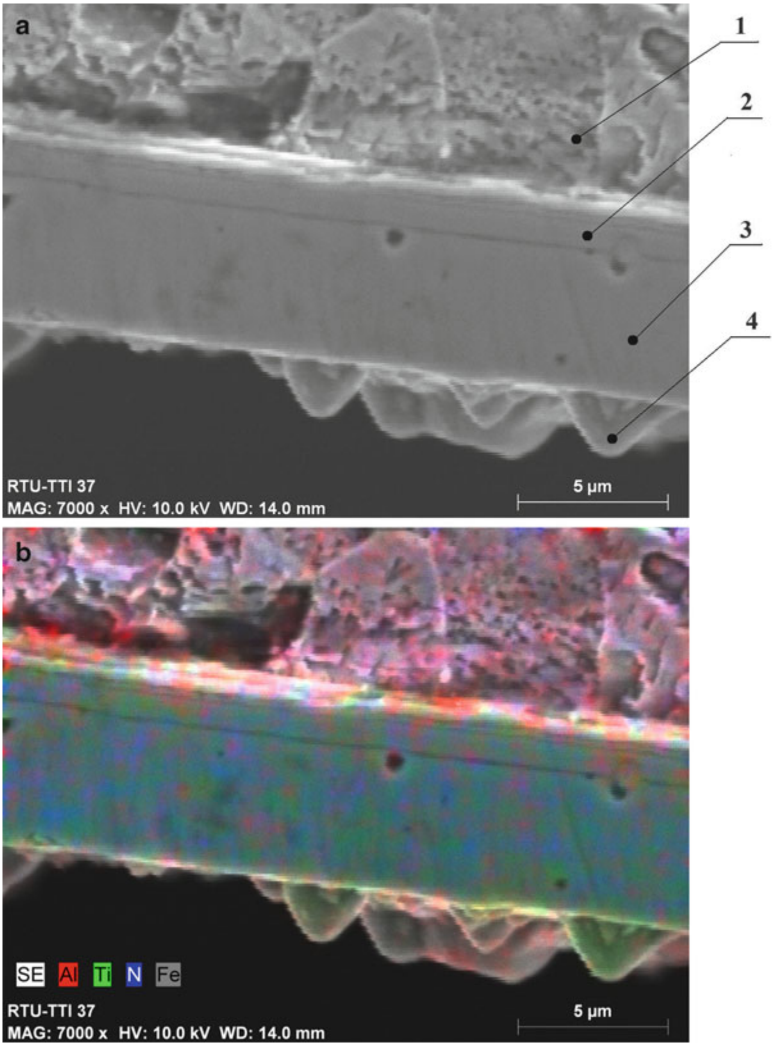

Fig. 15.4 Structure of two-layer coating material of the product (**a**) and results of microrentgen-spectral analysis of the chemical composition of material (**b**): *1* basic material; *2* internal layer of the coating; *3* external layer of the coating; *4* "drop phase"

opportunity to conclude that on the initial stage of loading (load less than 25.02 N) the indenter practically does not leave any marks on the coating (see Fig. 15.7a). The diamond indenter slides across the coating with a very low coefficient of friction (less than 0.1).

A further increase of the load leads to the formation of multiple chips on the coating, which is reflected in AE amplitude curve (with the load of $F_{Nc2} = 30.05$ N) (point B Figs. 15.6 and 15.7b). Load $F_{Nc3} = 42.63$ N corresponds to the point when

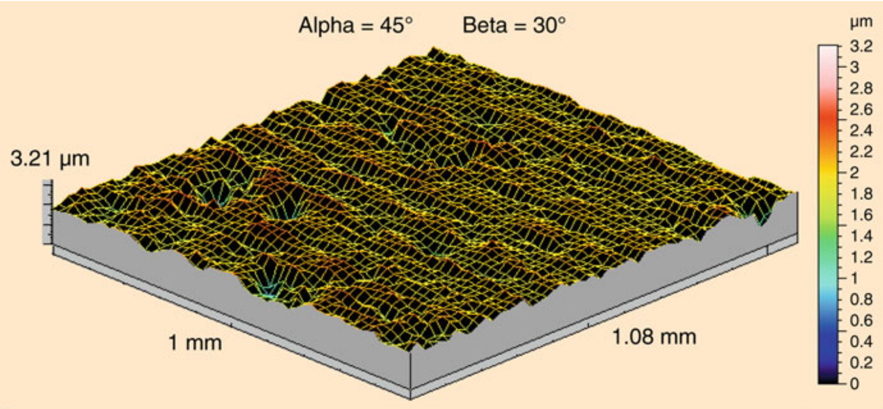

Fig. 15.5 Coating surface roughness (KJONBOMU method)

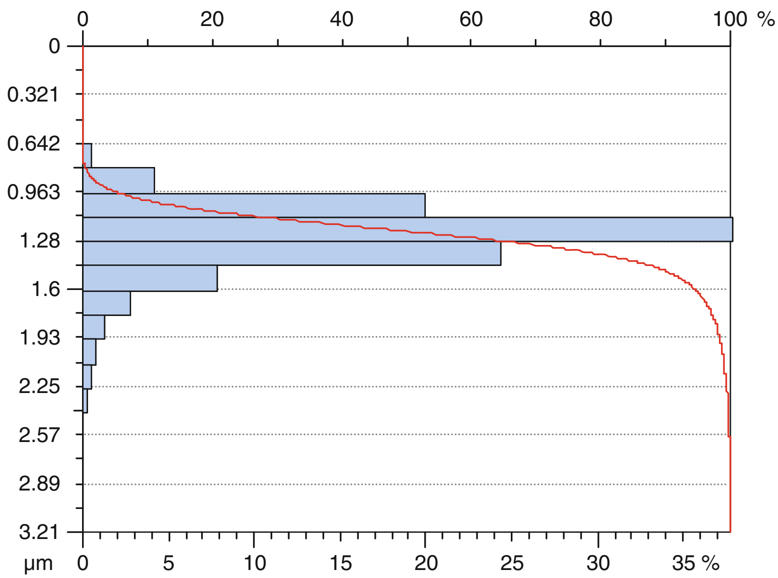

Fig. 15.6 Coating surface support curve and a histogram of peaks distribution

multiple flaking of some areas of the coating begins (point C Figs. 15.6 and 15.7c), which then is followed by the process of mass flaking with the load of $F_{Nc4} = 47.32$ N (point D Figs. 15.6 and 15.7d). The formation of this type of damage entails the growth of AE signal amplitude as well as the monotonous increase of parameters μ_v (up to 0.2) and F_t. The appearance of multiple chips on the coating is followed by sharp bursts of curve μ_v and F_t upwards, while curve h goes downwards (see Fig. 15.6).

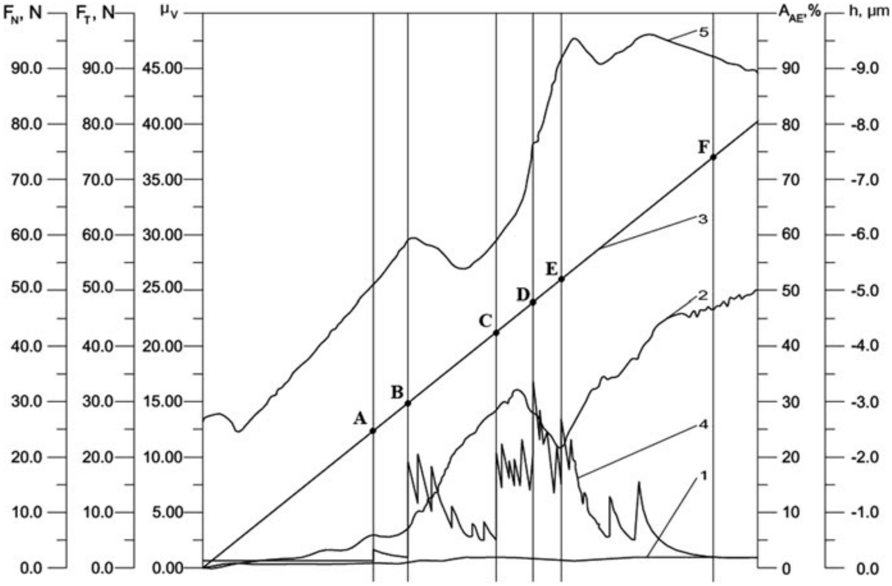

Fig. 15.7 Results of adhesion tests of the system « coating (TiAlN) – plunger (stainless steel)»: *1* friction coefficient μv; *2* friction forces Ft; *3* normal load FN; *4* amplitude of acoustic emission signals A A_{AE}; *5* indenter penetration depth *h*

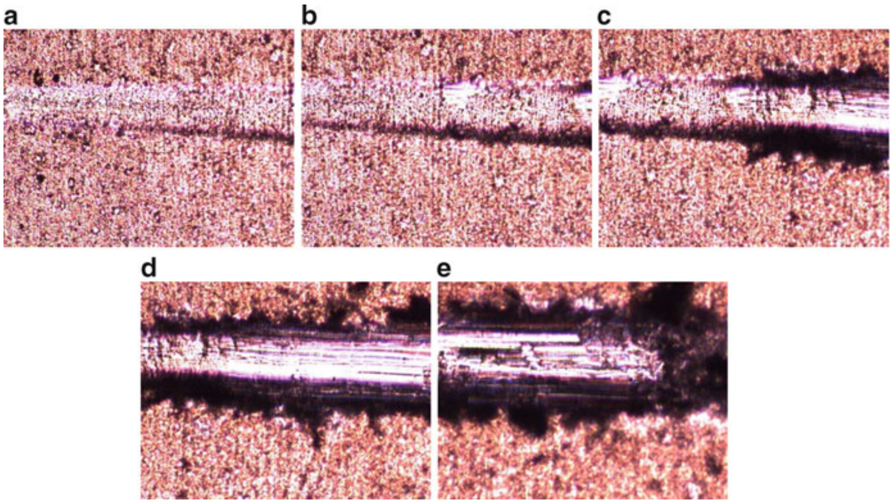

Fig. 15.8 Photos of plunger coating scratches during observation through the optical microscope for the following indenter loads: (**a**) 25.02 N; (**b**) 30.05 N; (**c**) 42.63 N; (**d**) 47.32 N; (**e**) 73.68 N

The appearance of substrate material on the bottom of the scratch under high loads $F_N = 40\ldots50$ N indicates, in its turn, at the high adhesion strength of the coating.

The following stage (point E Fig. 15.6) with the load of $F_{Nc4} = 52.51$ N corresponds to the transition of the coating destruction process to the subsequent plastic deformation (wear) of the substrate material (plunger). On the last stage of indenter travel across the surface of the object being tested with the load of $F_{Nc5} = 73.68$ N a mass concentration of destruction fragments that represent a conglomerate of coating and substrate materials is observed (point F Figs. 15.6 and 15.7f). This circumstance is indicative of the fact that the scratching coatings wear off and separate together with the basic material of a product, i.e. the destruction occurs by a cohesive mechanism, which is related to plastic deformation and formation of cracks in the coating material.

Thus, the coating adhesion strength for the considered example is $F_{Nc1} = 25.02$ N.

Based on the obtained experimental data for a rather large sampling (59 measurements for seven samples and 40 measurements for five products), the mean value of critical load F_{Nc1} is 25.15 N. The received value of the adhesion strength of the created nanostructured wear-resistant coatings considerably (4...5 times) exceeds the known data for coatings of such type.

15.4 Conclusion

A new high-performance ion-plasma technology for obtaining a nanostructured wear-resistant composite coating has been developed by the combined method (KJONBOMU). The combined technology makes it possible to create nanostructured coatings, to ensure high microhardness and wear-resistance of a coating, its adhesion and thickness uniformity on a large area. It gives the possibility to diversify the composition of a coating in a wide range within a single technological cycle, to obtain high smoothness of a coating surface (the influence of a “drop phase” and the parameters of the coating roughness decrease considerably) and more uniform coatings from the point of view of composition, as well as, to ensure ecological cleanliness of the production cycle.

A prototype of nanostructured composite coating (two-layer, three-component) on Ti–Al–N basis with the increased wear-resistance has been developed. The coating is intended for the protection and restoration of tribo-element surface, i.e. for the parts of valve and plunger precision pairs in the process of their manufacturing and repair. The developed multicomponent coating provides the opportunity to reduce the friction coefficient and the wear of contact surfaces by two times and, as a result, to increase the life of friction parts.

The experimental investigation of the failure mechanism of the created wear-resistant nanostructured coating has been carried out and the assessment of its adhesion strength by the system “coating-sample” and “coating-part” has been performed using scratch tester CSM Revetest Xpress. Based on the experimental measurements of different physical parameters (acoustic emission, friction coefficient, indenter penetration depth, normal load, friction force), the failure mechanisms and threshold values of certain critical loads leading to coating destruction have been described. The value of coating adhesion strength exceeds the strength of the coatings obtained by traditional methods of ion-plasma sputtering by 4. . .5 times. On the basis of the offered probabilistic approach and the carried out experimental investigation, a quality assessment methodology for the coatings of tribo-elements depending on the threshold values of critical load has been developed, which leads to the deterioration of coatings adhesion and can be determined with high sensitivity by the acoustic emission method.

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