Friction Surface Modification by Nanodiamonds of Denotational Synthesis

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Abstract. The paper deals with the technology and equipment for making a hardened antifriction surface layer of machine elements on the basis of using carbon nanomaterials in order to improve wear resistance. The paper presents the results of tribotechnical tests of pins according to standard techniques using an automated system for research.

1. Introduction
The perspective directions of solving the problem of increasing wear resistance of friction surfaces of machine parts include the use of nanodiamonds as a reinforcement (reinforcing) of the surface layer of particles of a small size.

The diversity of principally new properties of nonmaterials allows using them for new applications in various industries including friction pairs of machine elements. In technologies of obtaining and applying nonmaterials of 1…500 nm carbon clusters are becoming more and more popular, the most promising of them are carbon nonmaterials, super disperse nanodiamonds of denotation synthesis in particular.

Synthesized in highly non-equilibrium conditions, nanodiamonds do not have a clear cut crystal. The rounded shape of nanodiamonds along with modification of their surface during detonation synthesis provides an effective application to improve mechanical characteristics of materials, and, in some cases, it provides unique tribological properties due to the formation of spatial grid of physical connections at the interface of the material structure with nanoparticles with high adsorption properties.

Making surface layers with implanted nanodiamonds as reinforcing nanoparticles of the layer increases its density, strength and provides a nanostate, which gives improved wear resistance during friction.

The micro- and nanostate of friction surface is formed by electromechanical working (EMW) for making surface layers with implanted highly consistent disperse particles consisting of coagulated nanodiamonds. The distribution pattern and morphology of the disperse strengthening phase (a strengthening composition effect as a result of decay of supersaturated solid solutions of the material structure) have the main influence on wear resistance of the worked surface. Electromechanical working is characterized by wide technological possibilities to control micro- and nanostate and tribotechnical parameters of friction surfaces of machine elements and also by reduction of cost, labor and energy intensity by 3…5 times in comparison with other more widespread technological methods, in particular laser and plasma working. At the same time plant-performance Figures of components rise by 1.5…3 times.
The technology of electromechanical working is based on the combination of thermal and power influence on the surface of the work piece, that leads to changing physical-mechanical microgeometric parameters of the surface layer (strength increase, reduction of roughness parameter, etcetera) and, as consequence, improvement of plant-performance Figures of components, in particular wear resistance, contact stiffness and strength, endurance limit, heat resistance, fretting resistance.

A combined electromechanical working (CEMW) technology was developed to get wear-resistant surface layers on friction surfaces of machine elements with implanted nanodiamonds of denotational synthesis. This technology includes implanting nanodiamonds of denotational synthesis into a friction surface and subsequent reinforcing the worked surface. This technology is referred to additive technologies [1].

If we consider any tribosystem functioning under the conditions of adhesive, fatigue, oxidative and abrasive wear, than nonmetallic hard materials – carbides, nitrides, borides, composite and nanocomposite materials, cermets and diamond are the most perspective [2].

2. Research technique
The technology additionally includes a special set representing the technological complex: a universal machine (used for mechanical treatment of billets) with appropriate tools and devices for fixing workpieces and supplying an electric current of great strength and low voltage; a power conversion unit for electric current; the control unit processing modes; and switching means for supplying lubricating and cooling technological environment; the interface unit with the PC. Implanting nanodiamonds into the surface layer during EMW is done under certain conditions in a coagulated state (powder fractions of (200…250) nm).

They are coated to the surface before working, having been mixed with consistent graphite grease in a certain proportion. Most of coagulants penetrate into the formed surface layer reinforcing it. Then EMW is performed on the same surface to harden it.

Complex comparative tests of samples were performed at an automatic plant based on the friction machine MI-1M and intended for tribotechnical tests of pins made of metals and alloys. The tests give the opportunity to determine surface tribotechnical characteristics in the conditions of sliding friction and with boundary lubrication.

The tests were carried out under the following conditions: sliding velocity $v = 1$ m/s; normal loading force $N = 100 \pm 0.5 \%$, $H$ (corresponds to pressure in Hz, about 150 MPa) ; initial contact – concentrated plastic contact; boundary lubrication; lubrication type – dipping; key wear type - fatigue wear; lubricant – industrial oil I – 20A ГОСТ 20799 – 88; indenter material - hard alloy VK8.

The complex comparative tests of wear resistance were performed on outside cylinder surfaces of samples made of medium-carbon steel 45 and in accordance with the following technology:

- combined electromechanical working (CEMW) – forming a surface layer with implanted nanodiamonds and subsequent reinforcing the worked surface, microhardness is 950 HV.
- bulk hardening, backing, hardness is 55 HRC;
- electromechanical reinforcing of the worked surface, microhardness is 750 HV.
- nanocoating of system Si-O-C-N, performed by using the technology of plasma finishing in Ltd “NPF “Plazmatsentr”, St. Petersburg, coating nanohardness is 23GPa.

According to the results of parameters analysis we determined the following values of tribotechnical properties:

- aging time $t_0$, in hours, defined as the time from the test start till the moment when wear curve comes to the piece of normal wear;
- aging wear $h_0$, in microns, an approach value, defined at the moment of aging finishing $t_0$;
- average friction coefficient during normal wear $f$;
- $f_0 / f$ – ratio of a maximum friction coefficient during aging $f_0$ to its average value during normal wear $f$;
- average value of wear intensity in the period of normal wear:
\[ I_h = (h - h_0) / (L - L_0), \]
where \( h, \) micron, - total value of sample wear at the test time; \( L, \)
micron, - friction distance of the sample surface at the test time; \( L_0 = 3.6 \times 10^9 \cdot t_0 \cdot \nu, \)
micron, - friction distance of the sample surface at the aging time.

3. Research results
There is austenization of the steel surface layer in the contact zone in the process of high temperature plastic deformation and under the influence of high temperature and pressure. Carbon of the coating consisting of graphite and nanodiamonds is diffused during a solid phase saturation process into the surface layers increasing the carbon content in the austenite.

With the increase of the carbon content and a higher cooling rate the steel matrix structure acquires a higher share of pearlite with intensive decrease of ferrite fringe thickness around pearlite grains. Intensive cooling forms a steel structure, which consists of alternate layers of various structure arrangement – normalized ferrite-pearlite and sorbate-troostite (hardening structure) zones (Figure 1).

Thus, in the hardening process by CEMW steel is simultaneously subjected to normalizing with faster cooling and formation of the ferrite-pearlite structure and hardening with formation of the ferrite-sorbite-troostite structure. As a result of high temperature plastic deformation there is a formed sintered graphite layer with implanted nanodiamonds on the steel surface (Figure 1, e).

This layer consists mainly of sintered mass of graphite and nanodiamonds (Figure 2, a). A thin layer of over strained metal is formed on the outer surface of this layer, which is removed by diamond smoothing (Figure 2, b). The structure of the nanodiamond graphite layer (Figure 2, a) consists of the base, sintered graphite, nanodiamonds of various size groups and different carbide inclusions, cementite for example. This layer has good tribotechnical characteristics as its base (sintered graphite) is a high-performance antifriction material, reinforced by implanted nanodiamonds and disperse carbide particles of various types, for example, cementite. Figure 3 shows the structure of a sintered layer.

4. Discussion of results
The underlying hardened ferrite-sorbite-troostite layer (Figure 1) has a wear-resistant fine-grained textured structure. A significant component of this structure is milled grain ferrite obtained by recrystallization of heavily deformed austenite during Electromechanical hardening. The grain size of the ferrite compared to the base material is 15-fold reduced (Table 1).

| № p/p | Layer                                | Size, mc mum | Positioning in Figure |
|-------|--------------------------------------|--------------|-----------------------|
| 1     | Ferrito-pearlitic                     | 350...500    | 1,б                   |
| 2     | Ferrite-sorbite-troostite             | 300...450    | 1,в                   |
| 3     | Transition                            | 30...70      | 1,г                   |
| 4     | Graphite with nanodiamonds           | 200...250    | 2,а                   |
| 5     | Fragments overstrained steel          | 100...120    | 2,б                   |
|       | The total thickness of the hardened layer | 850...1200  |                       |

A ferritic grain size of steel (medium)

| № p/p | Layer                                | Size, mc mum | Positioning in Figure |
|-------|--------------------------------------|--------------|-----------------------|
| 6     | Matrix steel main                    | 30           | 1,а                   |
| 7     | Ferrito-pearlitic layer              | 20           | 1,б                   |
| 8     | Ferrite-sorbite-troostite layer      | 2            | 1,в                   |

The reinforced lower ferrite-sorbate-troostite layer (Figure 1, c) has a fine grain-oriented wear resistant structure. The significant component of this structure is a ground ferrite grain, obtained as a
result of recrystallization of greatly strained austenite by electromechanical reinforcement. The ferrite grain size in comparison with the base material decreases by 15 times.

**Figure 1.** Multilayer structure of the surface content of steel 45 after forming the layer with implanted nanodiamonds and subsequent electromechanical reinforcing (×100): a - lower layer – material base, standard initial steel structure; b - 1st following layer – normalized structure formed by high cooling velocity when heating; c - 2nd following layer – a zone with the higher carbon content consisting of hardened sorbate-troostite structures; d - transition layer or ‘white’ layer; e - upper layer – sintered graphite layer implanted with nanodiamonds

Table 2. Results of tribotechnical tests of samples made of steel 45 after different working techniques

| Tribotechnical property | Index               | Values for different working techniques |
|------------------------|---------------------|----------------------------------------|
|                        |                     | Backing | EMW | Plasma finishing | CEMW |
| Running ability        | $t_0$, h            | 2.75    | 2.85| 1.94   | 2.13 |
|                        | $h_0$, mcmm         | 11.5    | 6.0 | 3.5    | 2.5  |
|                        | $f_0 / f$           | 1.58    | 2.94| 3.28   | 2.60 |
| Antifrictionality      | f                   | 0.33    | 0.17| 0.035  | 0.098|
| Wear resistance        | $I_k \cdot 10^{-10}$| 2.41    | 0.8 | 0.96   | 0.75 |

Microhardness of the surface layer (by medium-carbon steel working) reaches 1000 HV on the surface. The depth of the general reinforced layer is up to 1.2 mm. Besides, there is blending of reinforced layer hardness from the surface with the non-reinforced core of the element.

So, it does not cause delaminating at dynamic loads because of the integrated phase condition of the layer metal matrix, different in its structure. The obtained microgeometrical parameters of the worked elements are: $Ra = 0.2 ... 3.2$ micron; $Sm = 0.025 ... 0.36$ mm; $tm = 50 ... 70$ %; $Wz = 0.4 ... 8.0$ micron, $max = 6 ... 20$ micron.

The results of tribotechnical tests of samples made of steel 45 after different working techniques are given in Table 2. The results of tribotechnical tests showed that the wear resistance of samples with implanted nanodiamonds with subsequent electromechanical reinforcing in comparison with
heat-treated samples rises by 3.2 times, in comparison with reinforced by EMW – by 1.1 times, in comparison with plasma finishing – by 1.3 times.

Figure 2. Sintered graphite layer with implanted nanodiamonds on the friction surface of steel 45(a), covered with layer fragments of overstrained steel (b) during CEMW, (x500)

5. Findings
1. Modification of the steel friction surface by means of the forming sintered graphite layer, implanted and reinforced by nanodiamonds and carbides of various nature along with the formation of the reinforced ferrite-sorbate-troostite and ferrite-pearlite fine-grained structure of lower layers by CEMW allows improving wear resistance of friction surfaces, which is proved by tribotechnical tests.
2. A multilayer structure with the general metal matrix of the base material of a homogeneous phase provides monolithic cohesiveness of reinforced layers without continuity violation during wear at heavy loads, which is also proved by tribotechnical tests.
3. It is economically sound to use relatively cheap medium-carbon steels for critical friction pair instead of expensive high-carbon and high-alloy steels. It is possible to get the same microhardness and higher wear resistance of medium-carbon steels, which is not possible by using other heat processing and surface reinforcing methods. This is the main advantage of CEMW. The coefficient of reinforcement reaches $k = 3.5...4$.

6. Conclusion
This research urgency and scientific significance consist in a system approach to the study of the formation of the surface layer quality, its phase, micro- and nanostructural states, tribotechnical characteristics of machine element surfaces. This approach is a methodological foundation of the research. It allows applying scientific ways in order to choose, standardize and optimize the modes of combined electromechanical working taking into account functionality of the worked elements.

References
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[2] Stanski D et al 2015 Met Sci Heat Treat. 7(721) 77-83