Multicomponent Tribotechnical Coatings Based on Ti–C Combinations with Dopes[1]

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Abstract – Research results of multicomponent tribotechnical coatings of Ti–Si–Mo–S and Ti–Si–B–N composition produced either by magnetron or electroarc coating are given in the scientific paper. Cathodes made by means of self-propagation synthesis with simaltaneous pressing were used in the research. It has been found out that magnetron and electroarc coatings of Ti–Si–Mo–S composition, having the same initial cathodes composition, are significantly different in values of microhardness, friction coefficient in the couple with the mating surface made of ShH-15 hardened steel, wear resistance. Friction coefficient and microhardness of Ti–Si–Mo–S coatings, deposited by the electroarc method, are, in average, 2 times less than the ones of the coatings produced by magnetron sputtering, but their durability is higher. Differences in quantity content of these coatings componens, their phase composition and structure have been discovered by the methods of X-ray spectrum, X-ray structure analysis and transmission electron microscopy. Comparative tribotechnical tests of magnetron and electroarc coatings of Ti–Si–B–N composition have been carried out. It has been detected that both magnetron and electroarc coatings of this composition have a high value of hardness (about 30 and 40 GPa, respectively), and a noticeable decrease of friction coefficient in the couple with structural steel is revealed only in case of additional polishing of these coatings.

1. Introduction

The main requirement to tribotechnical coatings on details of friction couple is the combination of low friction coefficient and high wear resistance due to hardness, good coupling (adhesion) with base layer, chemical inertness in relation to mating surface material and components of ambience.

Traditional antifriction materials, such as molybdenum disulfide (MoS_2), sulfides of other metals, graphite or plastic metals do not correspond to this requirement, primarily, because of low hardness and wear resistance. But solid coatings like carbides, nitrides, oxides (TiC, TiN, Al_2O_3, TiAlN, etc.) are usually characterized by high friction coefficient in relation to metals and alloys.

Thus, research work to develop tribotechnical coatings of compositional type which are based on multicomponent composition and possess all properties mentioned above, is being carried out nowadays.

The research work [1] shows that magnetron multicomponent coating of TiN + MoS_x composition, when Mo + S is about 7% of weight, is characterized by intermediate values of friction and hardness coefficients in comparison with TiN and MoS_x coatings, but it significantly (2.7–3 imes), exceeds them in wear resistance. Similar positive results due to hardness as well as antifriction have been received in magnetron coatings $TiB_2 + MoS_2$ [2], Ti–Si–N: MoS_x [3], Mo:S:C:Ti:B [4].

Not only properties of molibdenum disulphide but of other combination of elements as well, have been studied in tribotechnical multicomponent coatings. For instance, in work [5], it has been revealed that the coating of Ti–C–B(9%)–N composition, which is characterized by nanocrystal structure and superhigh hardness value (42 GPa), has the lowest value of friction coefficient in tests with hardened steel in comparison with less hard TiC and TiN coatings. This coating is produced by a plasma-enhanced chemical vapor deposition.

Coating properties of the same Ti–C–B–N component composition, but made by magnetron sputtering of composite targets TB_2–TiC, synthesized by SHS-consolidation technique, are studied in works [6, 7].

As there is not much information concerning the use of well-known electroarc method in national industry in scientific works about the synthesis of coatings mentioned above, it is necessary to investigate and compare tribotechnical characteristics of magnetron and electroarc coatings with sputtering of similar component composition cathodes. Cathodes of TiC + MoS_x composition come first, then TiC + TiB_x composition is studied.

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2. Equipment and experimental methods

To solve research objectives by means of self-propagation high temperature synthesis with simultaneous pressing (SHS-consolidation technique) sputtering targets and cathodes for magnetrone and electroarc deposition of coatings with TiC + MoS_x and TiC + TiB_x composition were made. Magnetron deposition of coatings was carried out by using a planar duomagnetron of MIR-2. Electro arc deposition of coatings was made by using two vaporizers of NNV 6.6-I1 installation. Magnetron and electroarc coatings TiC:MoS_x were deposited in argon atmosphere; magnetron coatings (TiC:TiB_x)N were deposited in gas mixture Ar + 25%N and electroarc coatings (TiC:TiB_x)N – in nitrogen. Working pressure in the chamber was $2.66 \cdot 10^{-1}$ Pa in all cases. Accelerating voltage was 100 V. Base layer temperature during the process of magnetron deposition was about 250 ºC and of electroarc one was about 450 ºC. ShH-15 and 40 H steel types (hardened, ground and polished) were used as base layers for coatings deposition and subsequent friction tests. These types of steel were used as they are widely applied in national industry to produce kinematics transmission when sliding friction is of great importance. Steel types for friction tests were made in the shape of discs (diameter – 50 mm, thickness – 5 mm). After thermal treatment, i.e., hardening with subsequent tempering, hardness was 52–53 Horace for ShH-15 steel, 37–38 Horace for 40 H steel. These values correspond to real hardness values, which are common for some kinematics transmissions: screw gears and intermediate rollers.

Magnetron coating thickness was 1.3–1.5 µm and electroarc coating thickness was 2.5–3.0 µm. Microhardness was measured by the device PMT-3 with diamond pyramid load of 0.2 and 0.1 N. Coating roughness was determined with the profilometer (model 296). Friction testing was carried out with the help of the device “Cyclometr” according to the scheme – fixed indenter (a ball with 3.4 mm diameter made of hardened steel ShH-15) was a rotated disk.

Disk rotation velocity was 250 turn/min. The radius of a circular trace of indentor friction interaction with a disk surface was 15 mm. Thus, the velocity in relation to the recycle of the mating surface (an indenter and a disk) was 0.4 m/s. Indenter load was 0.15 N. Testing time was 10 min (for all samples), the change of friction coefficient was being detected during this time. X-ray spectrum analysis of the coating component composition was carried out at the device “Qunta-200-3d”, X-ray structure analysis of phase composition was made with the device “Shimadzu”, electron-microscopic investigation was carried out with the microscope EM-125.

3. Results and discussion

This research has shown that magnetron and electroarc TiC:MoS_x coatings have quite different hardness. Magnetron coating microhardness is H_{20} = 9–9.5 GPa but electroarc coating microhardness is H_{10} = 4.2–4.5 GPa. The value of magnetron coating roughness Ra in different base layers ranged from 0.15 to 0.33 µm, i.e., there was not much difference in values R_a of magnetron coatings deposited on base layers subjected to previous mechanical working of various kinds.

The same conclusion has been made concerning electroarc TiC:MoS_x coatings, deposited on ground and polished base layers. But only electroarc coatings R_a increased to 0.3–0.5 µm. These coatings were characterized by low adhesion with the base layer. Further experiments showed that they deposited on a special intermediate sub layer. Comparative friction tests have shown significant difference of magnetron and electroarc TiC:MoS_x coatings. Magnetron coatings are characterized by rather long period of running – in at a relatively high (0.4–0.5) friction coefficient (Fig. 1).

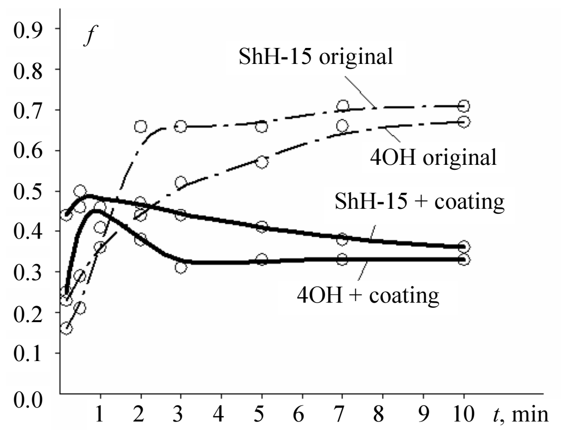

Fig. 1. Friction coefficient change, depending on time, for TiC:MoS_x about magnetron coatings

Friction coefficient stabilization of the coatings occurred at values of about 0.3.

The time of electroarc coatings running-in is much less (about 1 min of testing), and the value of friction coefficient is stabilized at 0.2 (Fig. 2).

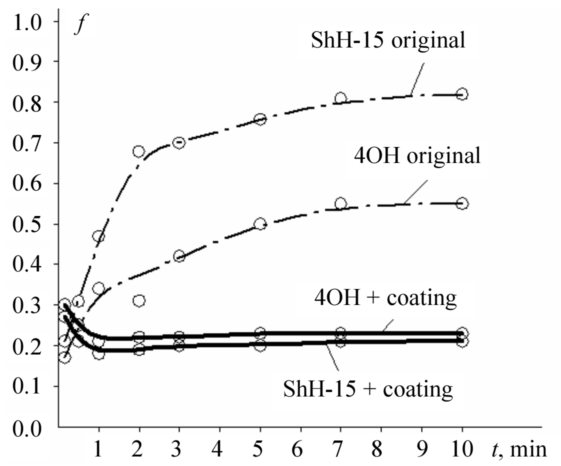

Fig. 2. Friction coefficient change, depending on time, for TiC:MoS_x electroarc coatings

Such difference in the values of hardness and friction coefficients required further investigations of composition and structure of these two coatings. By X-ray spectrum method it was found out that the coating deposited by means of magnetron sputtering had the following composition: S – 10.60%, Ti – 37.91%, Mo – 11.32%, C – 33.71%, O – 6.46% (at %), while the coating deposited by the electroarc method contained S – 33.62%, Ti – 27.60%, Mo – 5.70%, C – 19.23%, O – 13.85% (at %). Thus, it is obvious that sulphur concentration increased in the coatings produced by the electroarc method. Probably, it is the result of the fact that the electroarc method is characterized by higher energy surface activation by ions and high deposition temperature which direct to higher diffusion mobility of sulphur atoms, which, in turn go beyond grain boundaries, limiting their growth, and take a low energy position there. This, in its turn, results in the decrease of sulphur sputtering efficiency and the increase of sulphur content in the coating. It is evident that such effect cannot be observed in case with molybdenum, as molybdenum and sulphur have different thermodynamics characteristics.

The change of the composition and the deposition method directs, no doubt, to the modification of the structure – phase state of coatings. This investigation was carried out by X-ray structure analysis in the application to the X-ray diffraction spectra. As a result of machine processing of X-ray spectra, the following characteristics of magnetron coating phase composition were received: TiC SG196 cubic – 39 ± 10%, TiC SG225 cubic – 10%, MoS_2 SG160 trigonal/rhombohedral – 5%, MoS_2 SG194 hexagonal – 1%, X-ray amorphous phase – 45 ± 10% (vol.)

Electroarc coating phase composition was: TiC SG196 cubic – 0, TiC SG225 cubic – 44 ± 10%, MoS_2 SG160 trigonal/rhombohedral – 1%, MoS_2 SG194 hexagonal – 5%, X-ray amorphous phase – 50 ± 10% (vol.).

Taking these data into consideration, it is necessary to point out 1) their quality correlation with the change of element composition of coatings when a deposition method is changed, i.e., the correlation of sulphur content increase and volume part of X-ray amorphous phase in electroarc coatings; 2) the change of crystal lattice type of the main volume composite of MoS_2 phase from trigonal to hexagonal in the transfer from magnetron to electroarc deposition, that points out to conditions change (kinetic and thermodynamics ones) of its lattice formation, which can be connected with deposition regime and temperature.

As electron microscopic investigations have confirmed, phase composition and structure of deposited coatings really depend on the method they are received. Dark field analysis of disorientation peculiarities of crystal lattice coatings, received by the magnetron deposition (Figs. 3, *a* and *b*) has shown that they have two-level grain structure in which grains with size up to 1 μm are fragmented to sub grains with size of 20–30 nm (shown by the arrow, Fig. 3, *b*) by small angle boundaries.

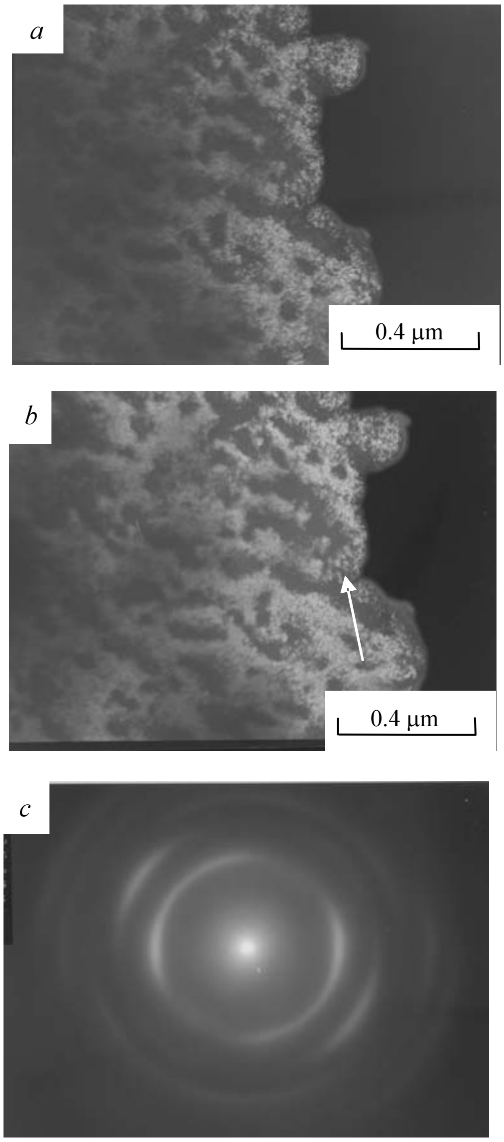

Fig. 3. Dark field pictures (*a*, *b*) and diffraction picture of TiC:MoS_2 coatings structure (*c*) produced by magnetron sputtering: *a* – slope angle of goniometry 0°; *b* – slope angle of goniometry 3°

Micro diffraction (Fig. 3, *c*) revealed that the coating is one-phase titanium carbide (TiC) with lattice parameter ~ 4.35 A°. But an elevated level of diffusion background in micro diffraction (Fig. 3, *c*) allows a suggestion that there is an amorphous component in the structure that qualitatively coincides with X-ray diffraction data. Besides, diffraction picture shows that the coating has azimuth – indefinite/unclear plane texture of increase (100) that is peculiar for column growth mechanism of coatings at high level of internal tension.

Quite different structure has been found out during electron – microscopic investigation of electroarc coatings (Fig. 4). This structure is characterized by much widened quasi-ring reflections that speaks of tiny grain size, which is about 2–5 nm as the analysis of dark field image shows (Fig. 4, *a*). As reflexes in the picture of diffraction of the surface layer structure of the coating are unclear, it is impossible to determine the phase composition. But it is necessary to point out that detected reflexes can be described, explained by the superposition of diffusion unclear reflections of nanosize particles of phases detected by the X-ray structure analysis. Meanwhile, the level of diffusion background in micro diffraction (Fig. 4, *b*) is higher for this deposition method.

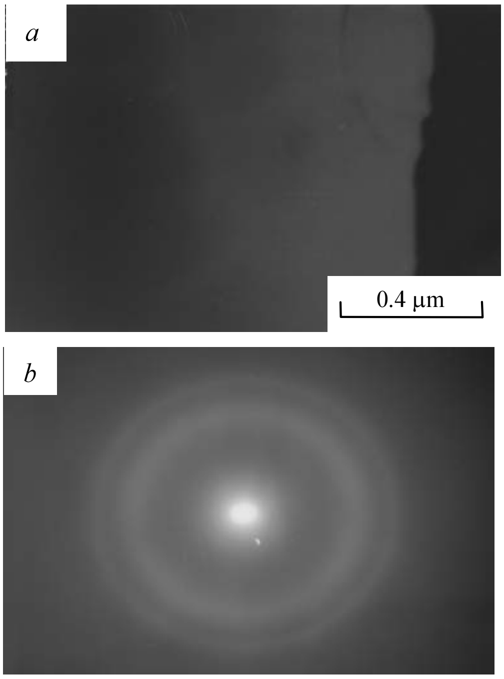

Fig. 4. Dark field pictures (*a*) and diffraction picture of TiC:MoS_2 coatings structure (*b*), deposited by electroarc evaporation

Investigations of hardness and friction coefficient were carried out in magnetron and electroarc coatings of (TiC:TiB_x) N composition. Microhardness values of magnetron coatings of this composition exceed 30 GPa, and in electroarc ones they exceed 40 GPa. As a result of such high hardness, friction coefficient of (TiC:TB_x)N coatings with initial roughness R_a = 0.3–0.5 μm in the couple with ShH-15 steel is characterized by a high value (about 0.4 or higher). But after additional polishing of such coatings to values R_a = 0.1–0.15 μm friction coefficient became lower (about 0.1–0.2).

4. Conclusion

1. It has been confirmed experimentally, that when molybdenum disulphide MoS_2 or bore are added to the content of magnetron and electroarc coatings TiC, their tribological characteristics are improved in certain conditions of frictions testing.

2. Differences in hardness and tribological properties of magnetron and electroarc TiC:MoS_x coatings are connected, first of all, with higher sulphur content in electroarc coatings (about 30% at), that results in differences both in the phase composition and in the structure of these coatings.

Electroarc TiC:MoS_x coatings, produced as a result of experimental work, are characterized by nanocrystal structure with grain-size of 2–5 nm, a short period of running-in, low friction coefficient and higher wear resistance.

3. As electroarc TiC:MoS_x coatings, even with higher value of surface roughness, do not require additional polishing operation, they are considered to be more perspective for practical application in comparison with (TiC:TiB_x)N coatings.

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