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IMPROVEMENT OF OPERATION LIFETIME OF MACHINE PARTS WITH COMPOSITE COATINGS IN WEAR CONDITIONS

L.G. Petrova, V.A. Aleksandrov, A.Yu. Malakhov, V.M. Zinchenko, V.I. Karagodin

Moscow Automobile and Road Construction State Technical University (MADI),
Leningradskiy Prospekt 64, Moscow, 125319 Russia

ABSTRACT

This work is aimed at improvement of operation lifetime of parts made of carbon steels due to increase in their wear resistance upon formation of composite multilayer coating by thermo-chemical treatment. A new method is proposed, which provides surface strengthening of parts made of structural steels including nitriding of items with preliminary formation of thin copper oxide film characterized by catalytic properties. It is demonstrated that adjusting thickness of deposited copper precursor it is possible to achieve required structure of nitrided layer, favorable for improvement of wear resistance of parts operating under the conditions of friction. Weight loss of medium carbon steel during wear testing decreases by 1.5…2.3 times, linear wear – by 1.2…2 times, in comparison with conventional nitriding. With optimum thickness of CuO film the operation lifetime of gears increases by nearly two times.

Keywords: operation lifetime, machine parts, structural steels, wear resistance, composite coating, nitriding, catalytic film.

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1. INTRODUCTION

Current development of mechanic engineering leads to more complicated machine designs and their operational conditions. Machine parts should comply with specialized requirements to provide fail-free operation during overall lifetime, including extreme conditions.

In the case of parts' operation under conditions of friction, 80-90% of failures are caused by their surface wear [1]. Surface wear damages serve as stress concentrators and can cause fatigue breakage even at minor loads. Contact of worn parts violates their normal interaction in assemblies, this can result in impacts and vibration in joints and, hence, be the reason of unexpected breakage [2].

For instance, gears used in nut wrenches are exposed to intensive wear during operation. The wrenches applied for fitting operations cannot be repaired in case of gear failures. According to statistic data average amount of wrenches failed due to gears wear at large engineering company is 10 pieces per year, this requires an approach to increase their operation lifetime.

It is known that wear resistance of steel parts can be improved by various methods of formation of modified strengthened layers [3-5]. High wear resistance is characteristic for coatings and materials with composite structure [6, 7]; such coatings can be obtained by thermo-chemical treatment (TCT). The most popular types of TCT are low temperature methods, such as nitriding. This method is comprised of diffusion saturation of metals with nitrogen; it has certain advantages in comparison with other types of thermo-chemical treatment. Comparatively low process temperatures (500…600°C) stipulate lower power consumptions in comparison with quenching and carburizing. Nitriding does not lead to modification of geometrical sizes, since this process is not accompanied with phase transformations in steel. Therefore, no mechanical finishing is required after nitriding. The core structure is retained, providing resistance against fatigue and dynamic loads of overall part.

The greatest advantage of nitriding is the possibility of targeted formation of the required structure of modified layer, including composite one. This is diffusion coating, formed in metal and not on its surface, which is especially important for items with precious geometry, such as gears.

Nitriding provides vast complex of operational properties of parts: high hardness, wear resistance, scuff resistance, thermal stability, corrosion resistance and fatigue strength. The requirements to the structure of nitrided layer providing efficient resistance against various types of wear are known [8]. It is assumed that sufficiently good resistance against wear is provided by high-nitrogen ε-phase which increases surface wear-in of nitrided item. For parts which operate under conditions of friction at high and medium pressures it is required to obtain well developed nitride zone with moderate nitrogen content (up to 8%). Formation of carbon-nitride ε+γ' zone decreases friction coefficient and promotes wear-in of contacting parts during operation [9]. In all cases it is required to obtain an internal nitriding zone under a compound layer, which supports external layer with high hardness. It acts as transition layer and decreases possible stresses at the interface between coating and core. The main property of transition layer existence is a smooth profile of microhardness distribution across overall layer from surface to core.

One of the approaches to improve nitriding is searching for the process intensification, since low temperature saturation of steel with nitrogen requires long time for obtaining modified layers of necessary thickness. Nitriding can be significantly intensified by increase of nitrogen concentration in saturating atmosphere which contacts with item surface. The nitrogen concentration in saturating atmosphere can be increased upon activation of ammonia dissociation. Possibility to accelerate ammonia dissociation upon formation of oxide layer on steel surface was demonstrated [3]. It was found that copper oxide film is an efficient reducing agent for ammonia dissociation [10]; under certain conditions of the film application onto surface it acts as catalyst.

This work is aimed at investigation of possibility to improve operation lifetime of items made of carbon steels due to increase in their wear resistance after nitriding of surface with preliminary applied catalytic film of copper oxide.

2. METHODS

The tests were performed on samples made of structural medium carbon steels, grades 45 and 40Kh. The samples were processed as follows:

1. Formation of precursor - nanosize film of pure copper on sample surface. Cu film was deposited from copper sulfate solution containing 3 g of powdered $CuSO_4$ in 50 ml of distilled water. The thickness of applied copper film was adjusted by variation of holding time in the solution.
2. Formation of CuO film. Copper film was oxidized in furnace in air environment at 585°C in the time intervals required for complete oxidation of copper layer (1…3 min).
3. Nitriding of sample with applied CuO film. Nitriding was carried out in ammonia environment (100% NH_3) at 585°C in 1…3 h. Samples were cooled in furnace in ammonia.

Metallographic study of microstructure of modified layer was carried out using an AXIOVERT 25CA optical microscope with magnification from 100 to 1500 in reflected light in the modes of bright or dark field image as well as in polarized light. The layer microhardness was determined using a PMT-3 hardness tester with the load of 0.02 N and 0.05 N. Hardness was measured on polished sections in the direction perpendicular to the surface. The profiles of microhardness distribution across the depth were obtained by averaged data of eight measurements. The effective thickness of diffusion layer was estimated by microhardness equaling to 1.25HV.

Chemical composition of the layers was determined using a FAUDRY MASTER vacuum spectrometer and a JEOL JSM-6480LV scanning electron microscope equipped with analytical unit: spectrometer with energy and wavelength dispersion. Element concentration profiles in the layer thickness were obtained. Variations of phase compositions across the depth of nitrated layer were obtained by comparison of nitrogen concentrations in various layer segments with equilibrium areas of phases in Fe–N system.

Wear tests were carried out using a 2168UMT tribo-technical facility according to “ring-to-ring” pattern in 60 min with nominal pressure of 2 N. Weight loss of samples and variation of their sizes before and after tests were considered as wear resistance characteristics. Weight loss as a consequence of wear was determined by weighing of samples on analytical balance with measurement error not higher than 0.1 mg. Linear sizes of samples were measured with the accuracy of 0.005 mm.

3. RESULTS

Study of structure of composite coating. It was experimentally demonstrated that during nitriding of medium carbon steel samples with catalytic copper oxide film the multicomponent nitrided layer is formed with the structure of composite coating (Fig. 1a). External surface layer is comprised of ε-phase (Fe_3N) with the thickness of 8…12 μm and nitrogen concentration up to 8.9 wt %. The second sublayer of nitride zone is located under it comprised of the mixture of ε+γ'-Fe_4N phases with the thickness of 10…15 μm. Below it there is the internal nitriding zone with the thickness more than 60 μm, consisting of nitrogen solid solution in α–Fe with precipitations of γ'-phase (Figure 1b).

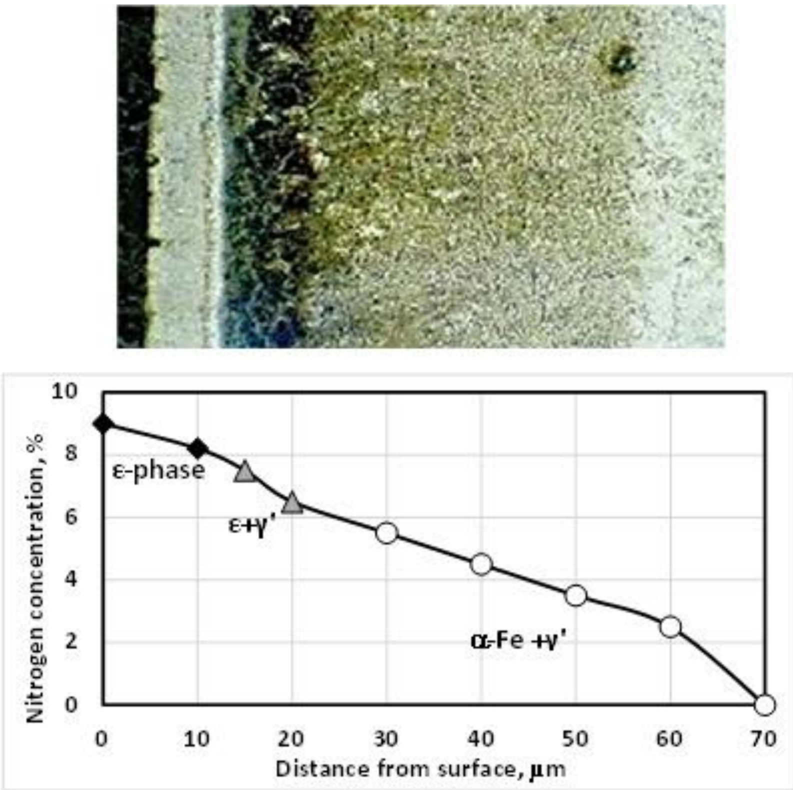

Figure 1 Microstructure of steel, grade 40Kh after nitriding at 585°C in 3 h with preliminary applied copper oxide film (a) and nitrogen concentration profile over the thickness of nitrided layer (b).

Mechanical properties of steels with composite coating. It is experimentally established that the composite coating with catalytic film obtained during nitriding is characterized by increased microhardness. Microhardness of the surface nitride zone corresponds to that of ε-nitride. Microhardness of the internal nitriding zone under catalytic film is higher than that of the zone after conventional nitriding due to increased nitrogen concentration. Due to the same reason, the length of the layer of higher microhardness increases. The highest increase in microhardness is observed upon formation of catalytic film with the thickness of 2.5 µm.

Multilayer structure of composite coating increases wear resistance of steel surface. Hard nitride zone on the surface improves wear-in of contacting items in initial wear period. Multicomponent nitrided layer consisting of the mixture of ε- and γ'-phases is resistant against wear during long term operation under the conditions of friction. Transition internal nitriding zone with smooth hardness gradient decreases the risk of embrittlement and delamination of composite coating.

Tests of steel samples, grade 45 revealed decrease in weight loss and linear wear of samples nitrided with catalytic film in comparison with those nitrided without application of copper oxide (Table 1). Weight loss during wear tests decreases with increase in the thickness of catalytic oxide film. The highest wear resistance is achieved for the sample nitrided with deposited copper film with the thickness of 2.5 µm as a consequence of significant thickness of nitride zone, high total distance of nitrided layer and smooth microhardness gradient across its thickness. Wear resistance of steel, grade 45, nitrided with application of catalytic copper film with the thickness of 2.5 µm decreases more than by two times in comparison with the samples after conventional nitriding.

Table 1 Results of comparative wear tests of steel, grade 45

no	Treatment	Weight loss, g	Linear wear, mm
1	Conventional nitriding	0.16	0.06
2	Nitriding with applied CuO film with the thickness of 0.8 μm	0.11	0.05
3	Nitriding with applied CuO film with the thickness of 2.0 μm	0.08	0.04
4	Nitriding with applied CuO film with the thickness of 2.5 μm	0.07	0.03

Nitrided samples with preliminary deposited catalytic film are characterized by decreased friction coefficient (Fig. 2), which increases operation lifetime of a part in contacting couple. Increase in thickness of copper oxide film increases the thickness of nitride zone of ε-phase responsible for increase in wear resistance and decrease in friction coefficient. With the thickness of CuO film equaling to 2…2.5 μm the average friction coefficient decreases by about two times in comparison with that after conventional nitriding.

Commercial trials of nitrided gears of wrenches with preliminary deposition of CuO film with the thickness of 2.5 μm demonstrated increase in their operation lifetime by 1.9 times as a consequence of increased wear resistance.

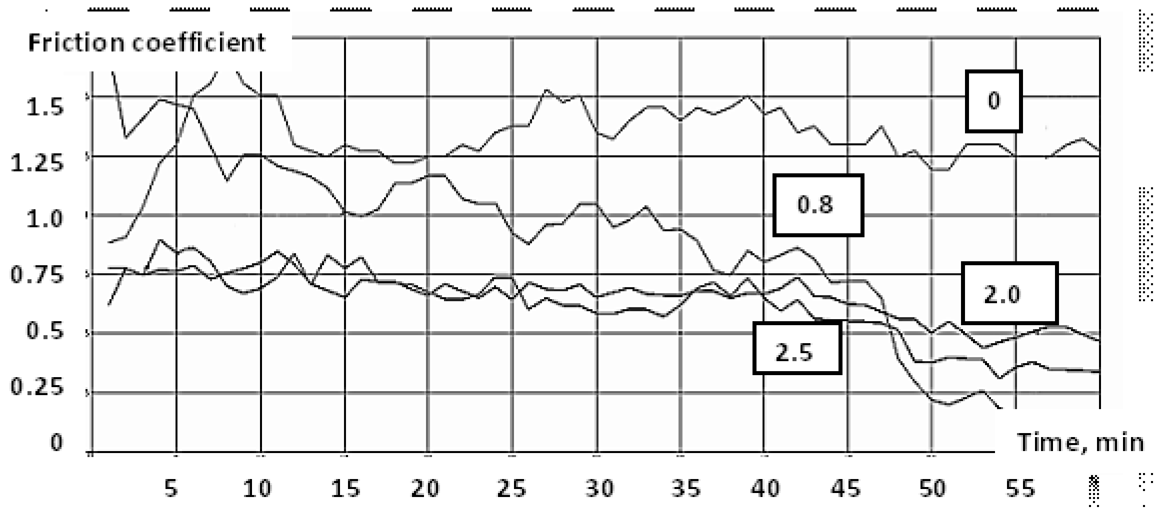

Figure 2 Friction coefficients during wear testing of steel, grade 45 after nitriding in ammonia at 585°C in 3 h: curve 1 – conventional nitriding, curves 2-4 – nitriding with application of CuO film (numbers near curves – film thickness).

4. DISCUSSION

Mechanism of catalytic effect of copper oxide upon nitriding of iron. Depending on their thickness and features of structure, oxide films can be characterized by catalytic of inhibiting properties as well as by barrier pattern. Copper oxide CuO satisfies the required conditions of formation of film with catalytic properties. Copper is oxidized well in air environment upon heating to moderate temperatures. Copper oxide is characterized by lower thermodynamic stability in comparison with iron oxide. Moreover, there is a simple and available method of copper application on iron surface: its spontaneous reduction on iron from solution of copper sulfate $CuSO_4$. Herewith, iron from steel is transferred to solution in the form of ions, and copper is deposited on surface of steel sample.

Copper oxide CuO is formed upon copper oxidation in oxygen (air) flow at the temperatures above 500°C. Exactly this oxide can manifest catalytic properties, since it promotes ammonia dissociation with formation of nitrogen as follows:

$$3CuO + 2NH_3 \rightarrow N_2 + 3Cu + 3H_2O \quad (1)$$

As a consequence of this process the concentration of nitrogen ions adsorbed on the film surface significantly increases. With low thickness of the copper oxide film a portion of nitrogen ions penetrates through it into iron core. Therefore, the thickness of catalytic film determines the concentration of active nitrogen ions on steel surface.

During saturation in ammonia environment two parallel processes run: diffusion of nitrogen ions into the depth of metal with formation of diffusion layer and reduction of pure copper from oxide which leads to thickening of catalytic layer and possible diffusion of metallic copper into nitrided layer.

Taking into consideration the aforementioned it is possible to assume that the thickness of initial copper film (with its complete oxidation) can be considered the adjusting parameter of formation of diffusion layer upon subsequent nitriding.

The thickness of CuO catalytic film determines phase composition of layer segments and their length. With increase in the thickness of copper oxide film the thickness of nitride layer of ε-phase increases. It is demonstrated that catalytic properties of the film and, as a consequence, formation of composite coating are manifested at its thickness above certain critical value. When the thickness of copper film exceeds 10 µm, there occurs nitriding without formation of surface layer of ε-phase, that is, the film behavior is of inhibiting pattern. This can be attributed to incomplete oxidation of thick copper layer at the stage of heating in air environment. We assume that the existence of non-oxidized metallic copper in film suppresses nitrogen diffusion upon nitriding.

Therefore, the method of nitriding with applied catalytic film forming the surface composite of two-layer nitride zone supported by sublayer of nitrogen solid solution in α-Fe with dispersed nitrides can be used for improvement of operation lifetime of parts working under conditions of intensive wear (gears, shafts, splines, rollers, piston pins, etc.).

5. CONCLUSION

The developed method of surface strengthening of parts made of structural steels including nitriding with preliminary applied catalytic copper oxide film makes it possible to form composite multilayer coating. It is comprised of surface layer of nitride zone of ε-phase, intermediate sublayer of nitride zone of ε+γ' phases and internal nitriding zone on the basis of nitrogen solid solution in α iron with particles of chromium nitrides and/or γ' phase. The layers are characterized by higher thickness and microhardness in comparison with samples nitrided by conventional procedures without application of copper film.

The thickness of precursor, initial copper layer deposited onto steel surface by chemical method from copper sulfate solution, is selected as the main parameter of nitriding with catalytic film. The thickness of copper precursor makes it possible to adjust the structure and phase composition of composite multilayer coating.

The composite coating possesses higher wear resistance in comparison with that of conventionally nitrided steel. During wear testing of medium carbon steel the weight loss is by 1.5-2.3 times lower and linear wear is by 1.2-2 times lower than the respective indices for conventional nitriding. With optimum thickness of CuO film equaling to 2.5 µm operation lifetime of gears increases by nearly two times.

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