Change of a Structural – Phase State and Tribological Behavior of Surface Layers at Ultrasonic Modification and Ion Nitration

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Abstract – The ultrasonic modification of steel surface provides formation of microcrystal structures on the surface and gradient change of structure and properties in depth. The properties of the steel surface influence on development of nitration processes. The influence of ultrasonic modification on the structure and properties of nitrated layers received by nitration at low-temperature to plasma of the glow discharge, in plasma of the arc discharge of low pressure and high-intensive implantation with nitrogen ions is investigated. Metallographic researches, electron-microscope analysis, X-ray SA and the analysis of mechanical and tribotechnical properties have shown, that the ultrasonic modification of the steel 40X13 increases depth, changes of phase structure, increases microhardness and wear resistance of nitrated layer. The ultrasonic finishing processing is offered as a method of treatment of a surface before ion nitration.

1. Introduction

Among the methods of dressing-reinforcing treatment by surface plastic deformation ultrasonic finishing treatment has recently found a wide application [1]. Earlier the authors researched influence of ultrasonic treatment on structure and properties of surface layers of carbon and alloy steels [2]. Repeated plastic deformation of the surface by ultrasonic instrument results in changing surface geometry, physical state of a surface layer and its structure. Relatively high voltage influence and multiple loading cause on a product surface formation of a layer with gradient structure, stipulated by changes according to depth: physical-mechanical properties (microhardness); dimensions of structure elements (grain size, dislocation substructure, particles of s cond phases); structure characteristics (scalar density of dislocations, elasto-plastic stress fields). All above said made possible to discuss modification influence of ultrasonic treatment on steel surface layers' structure.

It is known that enlarging crystal structure of metal alloys intensifies diffusive processes taking place by chemical-thermal treatment. Authors of a number of articles suggest applying various methods of cold plastic deformation to raise efficiency of cementation, borating and nitration of different steel types [3, 4].

By ultrasonic treatment a modification layer, bigger than the depth of diffusive penetration of atoms by ion implantation, is formed on the steel surface, which allows us to suggest this method of surface plastic deformation as a method of preliminary preparation of the surface before high-intensity low-energy implantation with nitrogen ions [5].

2. Material and methods of experiment

For analysis we used alloy steel of martensite class 40X13 (oil quenching and tempering from 1050° with the following post-tempering by 600 °C within 2 hours with air-cooling). Samples were grinned at the end surface, then the half of them were processed ultrasonic finishing treatment. Then all samples were exposed by nitration in low-temperature to plasma of the subnormal discharge, in plasma of the arc discharge of low pressure and high-intensive implantation with nitrogen ions [5, 6]. In all used methods nitration varied time and temperature nitration.

3. Experiment and results

Preliminary researches confirmed that after ultrasonic finishing treatment on the sample surface of steel 40X13 a modification layer of thickness 10 micrometers, revealed by metallographic analysis, is formed. Initial structure – tempered martensite of acicular structure is crushed, moreover, a "white" layer of a few micrometers can be observed on the surface (Fig. 1).

Results of X-ray SA confirmed that at ultrasonic finishing treatment of steel 40X13 changes of phase composition in the surface layer do not take place. Steel contains matrix α-phase and carbide particles $Cr_{23}C_6$. In the surface layer crystal lattice spacing of α-phase changes and surface layer texturing is registered (Fig. 2).

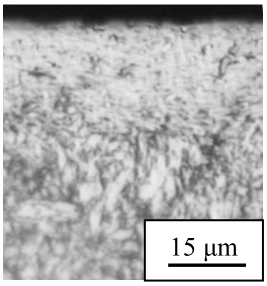

Fig. 1. Metallographic picture of the surface layer of steel 40X13 after ultrasonic modification

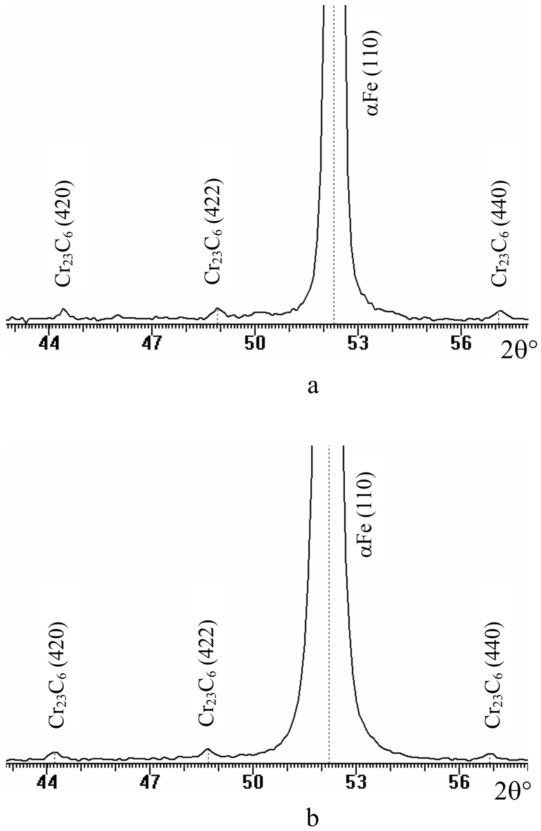

Fig. 2. Fragments of X-ray difractograms (CoK_α) from the surface layers of steel 40X13: a – after grinding, b – after ultrasonic modification

Previous electron-microscope of the analysis demonstrated that ultrasonic deformation causes in the steel surface layer partial destruction of martensite structure with fragmentation dislocation substructure takes place along with decomposition of carbide particles (Fig. 3).

Increase in defectiveness of surface layer structure leads to its reinforcing. Micro-hardness value measured under the loading 30 g changes from 4250 MPa at the sample depth to 5980 MPa on the surface.

Ion implantation of the samples, grinned and processed by ultrasonic treatment, causes formation of a nitrated layer, thickness and phase structure of which depends on implantation temperature and method of surface pretreatment.

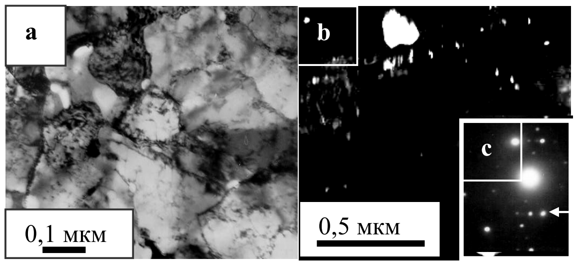

Fig. 3. Electron-microscopic images of structure of surface layers of steel 40X13 after ultrasonic modification: a – light field, b – dark field in the reflection [115] $Cr_{23}C_6$, c – microelectron diffraction

With the increase in treatment temperature the depth of a nitrated layer rises for all samples. At the same time thickness of the nitrated layer, obtained within one temperature condition, of the samples after ultrasonic modification is bigger than of grinned ones. So, under temperature implantation of 450 °C nitrated layer thickness is 26 micrometers and 20 micrometers accordingly (Fig. 4).

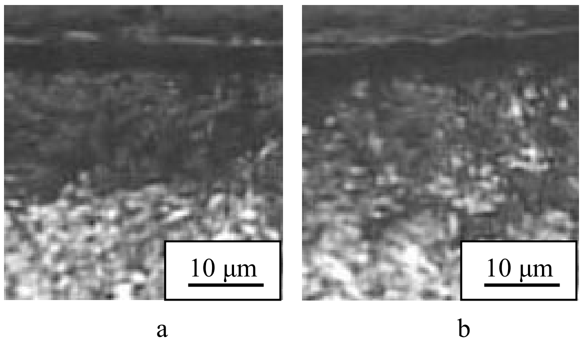

Fig. 4. Nitrated layer on steel 40X13 by T=450 °C: a – implantation N^+ after grinding, b – implantation N^+ after ultrasonic modification

With the temperature increase of ion implantation influence of ultrasonic modification of the surface on the ion nitrogen diffusion is getting bigger. When by the ion implantation temperature of 400 °C difference between layers' thickness was 3 micrometers, by the temperature of 500 °C it was 10 micrometers. This effect is most likely caused by the fact that temperature of 450–500 °C initiates processes of re-crystallization in ultrasonic deformed layer of steel 40X13. Return and polygonization of dislocation structures speed up nitrogen progress deep into the sample.

X-ray SA of the compared samples illustrates the following. Phase composition of implanted layer, irrespective of the pretreatment method, includes nitrides ε-$(Fe,Cr)_3N$, γ'-$(Fe,Cr)_4N$ and α''-$(Fe,Cr)_8N$ as well as nitrogen martensite. In a thin superficial layer by methods electron-microscope of the analysis are not identified a α''-phase and nitrogen martensite. Hence, they settle down on the greater depth, forming a zone internal nitration.

In the ultrasonic modification steel layer, modified by nitrogen ions, processes of nitride formation are more intensive at implantation. Content of high-nitrogenous phase ε-$(Fe,Cr)_3N$, decreases and formation of lot of low-nitrogenous phase $\gamma'(Fe,Cr)_4N$. Besides in the layer release of nitride particles CrN and Cr_2N takes place.

Raise of inclusion volume fraction of cubic γ'-phase instead of porous and frail ε-phase and release of particles CrN and Cr_2N result in microhardness increase in all nitrated layer of the sample, obtained by implantation N^+ ultrasonic treatment (Fig. 5).

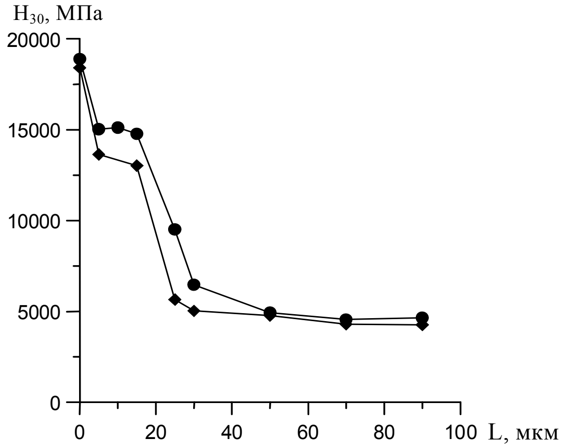

Fig. 5. Changes in microhardness values to the depth of the implanted layer at T=450 °C: ♦ — implantation N^+ after grinding, • — implantation N^+ after ultrasonic treatment

By consideration of change of microhardness on depth nitrated of a layer formed in low-temperature to plasma of the subnormal discharge the same dependence is observed.

Research of influence of preliminary ultrasonic treatment on wear resistance of nitrated layers in plasma of the arc discharge of low pressure received has shown the following.

At nitration at temperature 500 °C during 40 minutes on a surface of samples is formed continuous nitrated a layer having high value of microhardness. The character of wear process shows, that on samples with ultrasonic modification the period wear-in of a surface is increased twice (fig. 6).

Thus on the data of loss of weight of samples wear resistant grows by 30 %.

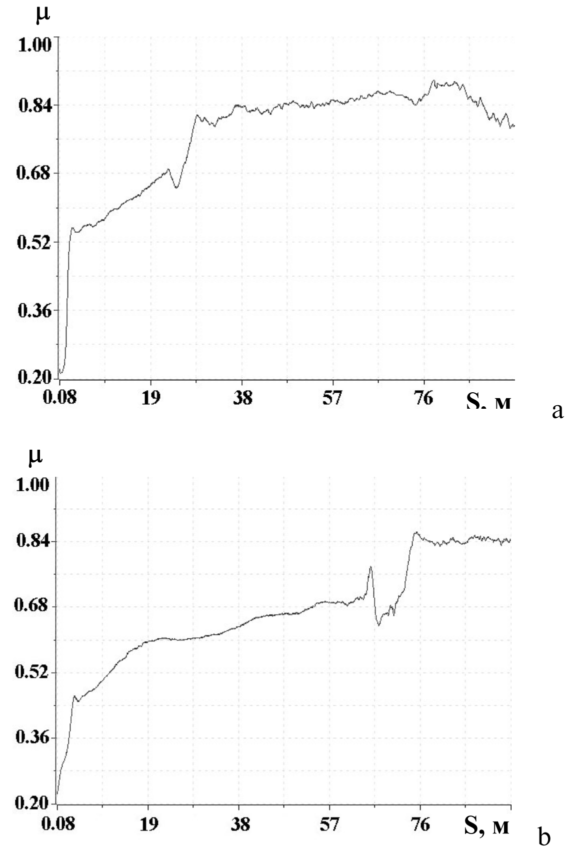

Fig. 6. Dependence of size of factor of friction on length of a way indentor on steel 40X13 with nitrated by a layer: a — after grinding, b — after ultrasonic modification

4. Conclusion

Thus, on the basis of the shown researches is established, that the ultrasonic modification of the steel 40X13 increases depth, changes phase structure, increases microhardness and wear resistance of nitrated layer. The ultrasonic finishing processing is offered as a method of treatment of a surface before ion nitration.

References

[1] Hkolopov Y.V., Metal-working 4, 5 (2001).

[2] Klimenov V.A., Kovalevskaya Zh.G., Uvarkin P.V., Tolstov V.P., Stepanov I.B., *in Proc. the 7th Korea–Russia International Symposium on Science and Technology*, 2003. pp. 279–284.

[3] Vlasov V.M., Zhigunov K.V., Ivankin I.S., M&HT 9, 39 (2002).

[4] Baraz V.P., Shremt A.M., Kirianova N.S., Izvestia VUZov. Ferrous metallurgy 8, 101 (1989).

[5] Kukareko V.A., Lobadaeva O.V., Shihk S.K. Influence of high-intensive implantation of nitrogen ions on structure and characteristics of steel 40X under the conditions of friction and wear

[6] Goncharenko I.M., Ivanov Y.F., Grigorev S.V., Koval N.N., Shanin P.M., Yang Si-Ze, *Physical mesamechanics*, Special release, 201 (2004).