Investigated of the Modified Surface Layers of High-Chromium Steel[1]

Zh.G. Kovalevskaya, I.M. Goncharenko*, K.A. Koshkin*,
P.V. Uvarkin, V.A. Klimenov**, and V.A. Dolomanova**

Institute of Strength Physics and Materials Science of SB RAS, 2/1, Akademichesky ave., Tomsk, 634021, Russia
Phone: +8(382) 28-68-87; E-mail: uvarkin@ispms.tsc.ru
**Institute of High Current Electronics, Tomsk, Russia*
***Tomsk Polytechnic University, Tomsk, Russia*

Abstract – Investigated structure and properties of the diffusion layers obtained at a low temperature nitration of steel 20X13 surface, modified with ultrasound processing. It is determined that when steel is nitrated in arc-discharged plasma of low pressure at 200 ºC it becomes saturated with nitrogen and its surface layer strengthens. Preliminary modification of surface allows reducing nitration time considerably due to the intensification of the process.

1. Introduction

Relatively low diffusion mobility of nitrogen atoms in subcritical temperature area always restricted the temperature regime of ion nitration to 500–550 °C. Thus, nitration for the surface hardening of structurally nonequilibrium materials was not considered [1].

It was shown earlier that there is the possibility in principle to conduct the nitrogen diffusion saturation of structural steels at significantly lower temperatures by the use of plasma of the arc discharge of low pressure, intensifying the process due to the radiation-enhanced diffusion [2].

The present work is devoted to the investigation of the low temperature nitration process of high-chromium steel 20X13 with the structural nonequilibrium surface layer, obtained by the ultrasonic modification.

Annealed steel 20X13 was chosen as an investigation material, since its equilibrium structure is significantly modified under the surface ultrasonic treatment. Chromium, entering into the steel composition is the nitrogen-forming element and increases the solubility of nitrogen in ferrite, which gives an opportunity to obtain the nitrated layer at low temperatures of nitration.

2. Material and methods of experiment

Samples from steel 20X13 were prepared for the fulfillment of the present work. Samples were grinned at the end surface, then the half of them were processed ultrasonic finishing treatment [3].

Ultrasonic treatment was carried out by the ultrasonic finishing treatment device with power 200 W; indentation load 75 N; indenter vibration frequency 24 kHz, and indenter amplitude 10 μm.

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 [1, 2]. In all used methods nitration varied time and temperature nitration. Nitration temperature made up 200 and 500 °C and nitration took from 40 min to 8 h.

For analysis, we used мetallographic researches, X-ray SA and the researches of microhardness.

3. Experiment and results

The structure of the steel surface layer in the initial state and after the ultrasonic treatment was investigated with the help of X-ray SA.

Results of X-ray SA confirmed that steel contains α-phase matrix and carbide particles $Cr_{23}C_6$.

Under ultrasonic finishing treatment of steel 20X13 changes of phase composition in the surface layer do not take place. However the α-phase lattice parameter increases from $a = 0.2871$ to 0.2874 nm and the intensity of $Cr_{23}C_6$ carbide reflection decreases, which confirms the partial dissolution of $Cr_{23}C_6$ carbide and transition of carbide and chromium atoms to the α-phase matrix and to the defects of the matrix structure [4]. Along with the abovementioned changes in the matrix structure, the decrease in the size of coherent scattering regions is observed after the ultrasonic treatment of the surface layer, which signifies the substructure refinement. The analysis of internal elastic stresses showed that compression stresses exist on the steel surface. Ultrasonic treatment stipulates the increase of internal stresses in the surface layer. Such structural transformations take place under the ultrasonic treatment of other structural carbon and alloyed steels, which is described in the papers of other researchers [5, 6].

The modification of the structure results in the hardening of the surface layer. After the ultrasonic treatment the microhardness of steel increases from 2520 to 3570 MPa.

As it is known the state of the work-hardened layer is thermodynamically unstable at all temperature regimes. Free energy of the surface layer increases due to the distortion energy, created by dislocations and

[1] The work has been supported by Program of fundamental research SB RAS (Project No. 3.6.2.1) and RFBR (Grant No. 06-08-01220).

point defects, introduced during the deformation. Thus at nitration the transition of the deformed metal to a more stable state with less free energy takes place along with the diffusion processes of nitride propagation in the steel surface layers. This process is not strictly bounded by the certain temperature. The process of polygonization is possible in the investigated temperature interval from 200 to 500 °C.

To estimate the contribution of structural transformation data to the process of low temperature nitration we conducted the annealing of all samples that underwent ultrasonic treatment at temperatures: 200 °C during 2 and 8 h and at 500 °C during 40 min and 2 h.

The change in structure of the deformed material under heating and its approximation to the structure of the undeformed metal is accompanied by the recovery of properties to the initial values before plastic deformation. However obtaining of the initial material structure and properties is optional.

Figure 1 demonstrates plots of microhardness values on the surface of samples with different temperature and time of annealing.

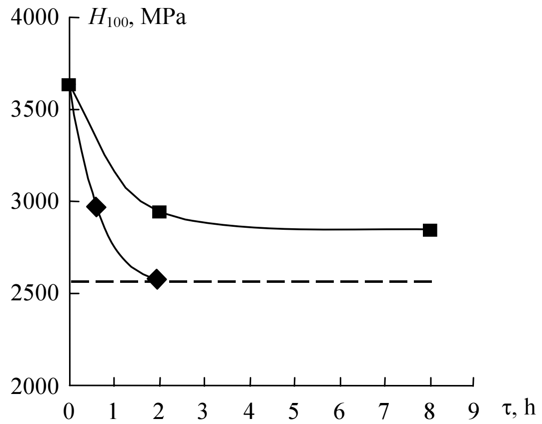

Fig. 1. Dependence of microhardness on the 20X13 steel surface from annealing time: *1* – at 200 °C; *2* – at 500 °C. The dashed line shows the microhardness of steel 20X13 with the equilibrium structure

After the annealing of face-hardened samples at 500 °C during 2 h the decrease of microhardness from 3570 to 2520 MPa takes place. After sample annealing at 200 °C during 8 h the microhardness decreases to 2760 MPa.

Comparing microhardness values on the surface of samples after annealing at 500 °C and 200 °C it is evident that under annealing at 500 °C the microhardness values become equal to the values of surface microhardness in the equilibrium. Under a long-term heating at 200 °C, microhardness on the surface stabilizes not reaching equilibrium values. The process of polygonization is assumed to take place in 20X13 steel in both annealing regimes. However, the heating temperature (200 or 500 °C) can change the ways of dislocation redistribution and thus the character of the polygonal structure. At lower temperatures (200 °C), dislocations predominantly slip and the stable boundaries of a small curvature are formed. With temperature increase (500 °C), the diffusion climb plays a greater role and polygons with boundaries of greater curvature and mobility appear. Besides, the processes of recrystallization might appear at 500 °C, which results in the decrease of the microhardness value [7].

On the basis of the conducted experiment, it is shown that the modified layer formed under the ultrasonic treatment is structurally unstable. The heating in conditions, corresponding to the nitration process at 500 °C during 2 h removes the hardening that appeared under the ultrasonic treatment. Heating to 200 °C leads to the removal of the strain hardening with further stabilization of the modified structure and properties.

Nitration was carried out at the annealing temperature and time: at 200 °C in the time interval from 2 to 8 h and at 500 °C during 40 min and 2 h. The X-ray SA showed that after the nitration in the whole temperature and time interval the nitration layer is formed on the surface. It has the following content: the main phase is α-phase and CrN is formed with a volume factor from 10 to 23% depending on nitration temperature and time. Reflections of the carbide phase are not observed on difractograms.

Decarbonization of the nitrated layer formed at the ion bombardment is connected with the increased sputtering of carbon that is in the surface layers of steel and partial dissolution of carbon in the nitration phase. When the nitration layer is formed on the modified surface, the part of the nitration phase is increased in the surface layer.

The analysis of mircohardness values showed that at nitration the hardened layer is formed on the surface of samples in all presented regimes. At nitration temperature 200 °C the degree of hardening is not high. Nitration at 500 °C results in the significant increase of the microhardness value on the surface. In both nitration temperature regimes the microhardness values, obtained on the modified surface are high. Microhardness grows with the nitration time increase (Fig. 2).

The works of the previous researchers show that the superimposition of the diffusion saturation of the nitrogen surface on the polygonization of the deformed structure intensifies the growth kinetics of the nitride layer and increases the phase content [8]. This mechanism is observed in the present investigation.

However according to the research the process of polygonization finishes much earlier than nitration at long time intervals. Despite this fact the growth of microhardness on the surface of samples, modified by the ultrasonic treatment is preserved and even increases.

Under nitration at 200 °C, it is explained by the stabilized modified structure in the surface layer. Under nitration at 500 °C, the stabilizing of the modified structure does not take place. This conclusion has been made from the analysis of microhardness values on the surface of sample, hardened by the ultrasonic treatment and annealed at 500 °C during 2 h (Fig. 1).

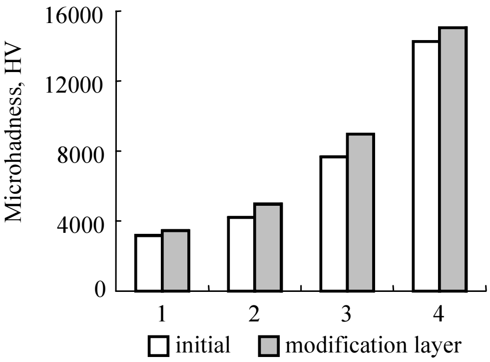

Fig. 2. Microhardness value on the surface of 20X13 steel samples, annealed in the arc discharge of low pressure: *1* – 200 °C, 2 h; *2* – 200 °C, 8 h; *3* – 500 °C, 40 min; *4* – 500 °C, 2 h

It is shown that hardening on the surface is totally removed. However, it is possible that the polygonized structure is preserved and thus the process of nitration is intensified. To confirm this conclusion the additional investigations are being carried out.

4. Conclusion

It is shown that at the nitration of steel 20X13 in low-temperature in plasma of the arc discharge of low pressure the steel is saturated with nitrogen and its surface is hardened. Preliminary ultrasonic modification of the surface allows intensifying the nitration process along the whole investigated temperature interval. The process of diffusion saturation of the steel modified surface by nitrogen takes place simultaneously with the polygonization of the deformed structure and probably together with the stabilization of the polygonized structure.

References

[1] P.M. Schanin, N.N. Koval, I.M. Goncharenko, and S.V. Grigoriev, Fizika i Khimia Obrabotki Materialov **16**/3 (2001).
[2] I.M. Goncharenko, Y.F. Ivanov, S.V. Grigorev, N.N. Koval, P.M. Shanin, and Si-Ze Yng, Phys. Mesomechanics. Special release **201** (2004).
[3] V.A. Klimenov, Zh.G. Kovalevskaya, P.V. Uvarkin, V.P. Tolstov, and I.B. Stepanov, *in Proc. the 7th Korea-Russia Int. Symp. on Science and Technology* **1**, 2003, p. 279.
[4] E.V. Kozlov, N.A. Popova, Yu.F. Ivanov et al., *Phase transformations in carbide subsystem of structural steel at thermal and deformation influence, in Problems of Steel and Alloy Physics*, Yekaterinburg, 2008, p. 46.
[5] E.V. Kozlov, N.A. Popova, L.N. Ignatenko et al., Fizika i Khimija Obrabotki Materialov **6**, 77 (1993).
[6] V.A. Klimenov, O.N. Nekhoroshkov, P.V. Uvarkin, Zh.G. Kovalevskaya, and Yu.F. Ivanov, Physical Mesomechanics **173**/ 9 (2006).
[7] S.S. Gorelik, S.V. Dobatkin, and L.M. Kaputkina, *Recrystallization of metals and alloys*, Moscow, MISIS, 2005.
[8] V.A. Klimenov, N.N. Koval, Zh.G. Kovalevskaya, I.M. Goncharenko, Y.F. Ivanov, V.A. Kukareko, O.B. Perevalova, B.B. Ovechkin, P.V. Uvarkin, and A.I. Tolmachov, Izv. Vyssh. Uchebn. Zaved. Fizika. Prilozh. **8**, 358 (2006).