Influence of martensitic steel ultrafine-grained structure on diffusion processes at low-temperature ion nitriding

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Abstract. The paper presents research results on the influence of martensitic steel ultrafine-grained (UFG) structure on diffusion processes at low-temperature ion nitriding. UFG structure was obtained through severe plastic deformation by torsion (SPDT) of coarse-grained (CG) steel samples. Ion nitriding was performed under the constant voltage mode during 4 hours at a temperature of 420 °C. Optical microscope was used to study the microstructure of a sample. Thickness of a modified layer was defined according to measurements of microhardness by depth. The analysis of research results showed that UFG steel structure enhances effective intensification of diffusion processes by 1.7 times, which in its turn contributes to low-temperature modification of a surface. At the same time, surface microhardness of UFG steel after treatment amounted to 1,200 HV and 525 HV of CG steel correspondingly.

1. Introduction
At present various methods of surface structural and phase modification based on diffusion processes, such as ion nitriding, are used to improve surface tribological properties of construction steel. However, such methods can only be applied at high temperatures (550-680 °C) and prolonged exposure time (up to 24 hours) [1,2]. As a result of such treatment, parts with complex geometry are subject to buckling, and the surface is scattered by saturating ions [3]. Besides, at temperatures over 600 °C the diffusive zone depth may decrease due to the reduction of ion current rate under the influence of thermionic emission from the cathode surface [1]. The aforesaid disadvantages may be cleared out by reducing the treatment temperature, which, however may lead to the essential delay of diffusion processes, increase of treatment cycle duration and rise of energy costs.

It is known [4,5] that ultrafine-grained (UFG) steel structure obtained via severe plastic deformation (SPD) is characterized by the grain size of 100-300 nm and non-equilibrium grain boundaries with enhanced concentration of vacancies and dislocations. Such crystalline defects have the stimulating impact on the progress of diffusing element atoms into the material [6-8]. Thus, UFG structure of a material allows to intensify the process of diffusion saturation at low-temperature structural and phase modification.

The purpose of this paper is to study the influence of martensitic steel structure on diffusion saturation at low-temperature ion nitriding.
2. Experimental approach

Table 1 shows chemical composition of martensitic steel 13X11N2V2MF-SH, which was used as a basic material in the research. Steel was subject to thermal treatment at 1,050 °C during 1 hour with subsequent air cooling and tempering at 800 °C within 1 hour. As a result of such treatment the microhardness of a material made 340 HV.

| C   | Cr  | Ni  | W   | Mo | Mn | Si  | V   | Cu  | Fe  |
|-----|-----|-----|-----|----|----|-----|-----|-----|-----|
| 0.134 | 11.4 | 1.66 | 1.77 | 0.46 | 0.44 | 0.32 | 0.21 | 0.12 | residual |

Discoidal samples with UFG and coarse-grained (CG) structure of 20 mm in diameter and 1 mm thickness were prepared for further ion nitriding. UFG structure was obtained via severe plastic deformation by torsion (SPDT) (Fig. 1) at the temperature of 300 °C and pressure of P=6 GPa with 10 revolutions per minute. Microhardness of steel surface after SPDT made 750 HV.

![SPDT for martensitic steel sample.](image)

In order to assess thickness of a nitried layer, microhardness by depth was measured using microhardness tester Duramin-1/-2 under the load of 10 g during 10 seconds. Net thickness of a diffusion zone was determined according to hardness distribution curve up to the hardness value of non-nitried material.

The structure of a nitried layer was studied against angled laps using an optical microscope Olympus GX51.

3. Results and discussion

Figures 3a and 3b show microhardness distribution curves in cross section of samples after ion nitriding during 4 hours at 420 °C and at the pressure of 150 Pa. The results of surface microhardness measurements are presented in Table 2.
The analysis of microhardness distribution curves in cross section after ion nitriding (Fig. 3) shows smooth decrease in hardness from the surface to the basic material for all samples under study. At the same time the net thickness of the modified layer of UFG steel is approximately 1.7 times higher than the same value of the CG steel. Surface microhardness of UFG steel was about 1,250 HV that justifies high concentration of nitrides on a surface [6,7]. Surface microhardness of CG steel made about 525 HV.

| Table 2. Surface microhardness |
|--------------------------------|
| Initial state, HV<sub>0.1</sub> | Surface microhardness | Surface microhardness | Surface microhardness |
|                                 | of UFG samples, HV<sub>0.1</sub> | of CG samples after nitriding, HV<sub>0.1</sub> | of UFG samples after nitriding, HV<sub>0.1</sub> |
| 340±20                          | 750±20                          | 525±20                          | 1250±20                        |

Higher thickness of UFG steel nitrided layer is caused by intense diffusion processes due to multiple grain boundaries and crystalline defects that contribute to the progression of nitrogen atoms into the material at nitriding temperature of 420 °C [6,7]. High increase of UFG steel surface microhardness leads to the formation of γ’, ε and α’-phases in the surface layer of a material [10]. At the same time, due to multiple defects of UFG steel the formation of phases is heterogeneous [7].

**Figure 2.** ELU-5M unit for ion nitriding.

**Figure 3.** Microhardness distribution curves in cross section of samples after ion nitriding at 420 °C during 4 hours: (a) CG steel, (b) UFG steel.
Figures 4a and 4b show optical images of samples microstructure after ion nitriding at 420 °C during 4 hours.

![Microstructure of samples at angle laps after ion nitriding at 420 °C during 4 hours: (a) UFG steel, (b) CG steel.](image)

**Figure 4.**

Diffusion zone – I and the zone of a basic material – II were identified on optical images of sample microstructures. The total thickness of UFG steel modified layer was about 220 microns. Thus the transition time between diffusion zone and the zone of a basic material is smooth, which is justified by measurements of microhardness. Due to its fine structure it was impossible to identify the grain boundaries of a UFG sample (Fig. 4a). During metallographic study CG steels were characterized by enhanced etchability along the grain boundaries with about 130 microns thickness, which correlates with microhardness measurement.

The analysis of optical images of sample microstructures showed that UFG structure contributes to the effective increase in speed of diffusive processes at low-temperature ion nitriding during 4 hours at a temperature of 420 °C, thus allowing to reduce the duration of treatment by 1.7 times.

4. Conclusion

The study of the influence of the UFG structure of martensitic steel on diffusion processes at low-temperature ion nitriding proved that preliminary treatment by pressure that forms UFG structure via SPD contributes to the effective increase in the modified layer speed by 1.7 times at 420 °C and within 4 hours.

It was also defined that UFG structure with high volume ratio of non-equilibrium grain boundaries initiates nitrogen grain boundary diffusion mechanisms. At the same time the surface layer is characterized by heterogeneous formations of γ', ε and α'-phases that define high values of surface microhardness after treatment.

References

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