

Influence of hydrogen content in working gas on diffusion processes at ion nitriding of martensitic and austenitic steels

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Abstract. The paper describes the influence of hydrogen content in a working gas on diffusion processes at ion nitriding of martensitic and austenitic steels. It is found that the presence of hydrogen in a working space of a vacuum chamber contributes to the increase in nitrogen diffusion depthward the material. Dependence of the hardened layer thickness and depth distribution of microhardness on gaseous composition was obtained. The structure of steel nitrated layer with various content of hydrogen in a vacuum chamber was studied.

1. Introduction

At present the emphasis is placed on the increase in wear resistance of workpiece surface operating under friction. One of the efficient surface hardening methods applied for machine components and mechanisms is glow-discharge nitriding (ion nitriding) [1, 2]. This method gained widespread use due to some advantages: high growth rate of the nitrated layer; diffusive layers with set phase composition and structure; ecological compatibility of the process [3]. Despite all mentioned advantages ion nitriding is time consuming (25–30 h).

It is known [4, 5] that in case with glow-discharge nitriding the composition of gaseous medium has significant impact on structure, properties and growth rate of a hardened layer. Hence, the use of working fluids containing argon with small amount of hydrogen allows to increase the speed of nitriding (up to 5 %). Being good reducing agent, hydrogen prevents the formation of iron oxides on a workpiece surface, which in their turn make nitriding more complicated [4]. When subject to bombardment, argon with its high weight forms numerous defects contributing to acceleration of nitrogen diffusion in metals [5, 6]. Thus, in order to enhance the efficiency of ion nitriding it is critical to control the composition of a working gas. However, at present there is no data on the influence of multicomponent saturating fluids on the diffusion speed of a hardened layer.

2. Experiment

Ion nitriding was carried out on installation ELU-5M. Prior to ion nitriding, samples of martensitic and austenitic steels were treated by heat to obtain a homogeneously grained microstructure. The chemical composition of steels and the parameters of heat treatment are given in table 1. Prior to nitriding samples were subject to sputter cleaning at temperature of 250 °C under pressure of 10 Pa at argon atmosphere. Ion nitriding was performed in flowing N₂, Ar, H₂ at 550 °C for 6 h under 150 Pa pressure. Microindentation was conducted on the Struers Duramin 1/-2 microhardness testing machine with a



load of 100 g (980.7 mN) for 10 s. At least four measurements were taken at each given depth. The microstructure of the specimen was observed under optical microscope Olympus GX51.

Table 1. Chemical composition of steels and parameters of heat treatment.

Type of steel	Chemical composition, %	Heat treatment
Martensitic	0.10–0.16 C; 10.5–12.0 Cr; 1.5–1.8 Ni; 0.18–0.30 V; 0.35–0.50 Mo; 1.60–2.0 W	Quench 1050 °C + annealing 800 °C
Austenitic	≤0.12 C; 17–19 Cr; 9–11.0 Ni; ≤0.8 Ti	Quench 1050 °C

3. Results and discussion

It is known that the surface temperature at glow discharge nitriding without additional heating of a vacuum chamber working space (cold wall nitriding) is determined by energy and density of charged particle flux. The density of ion flux regarding any component of a gaseous mixture depends on its percentage in a vacuum chamber and pressure of the environment, while the energy depends on the mass of particles and voltage between electrodes. Thus, at a different equivalent ratio of working gas components there is a change in surface temperature and workcycle parameters. In order to determine technological parameters of ion nitriding the dependence of voltage between electrodes on working gas composition in a vacuum chamber at a temperature $T = 550$ °C and pressure $P = 150$ Pa (figure 1) was obtained.

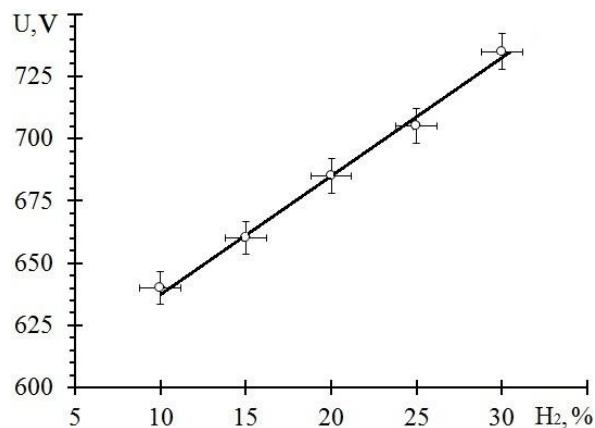


Figure 1. Dependence of the potential difference between the electrodes on the hydrogen content in the vacuum chamber, at which the temperature of the sample is $T = 550$ °C.

The analysis of the given dependence (figure 1) showed that when hydrogen content in a working gas varies from 10 to 30 % there is a need to increase the energy of particle flux due to the increase in charge voltage from 640 to 730 V to ensure the desired treatment temperature (550 °C). This is caused by the fact that at the decrease in argon concentration in gaseous mixture there is a reduction of density of argon ion flux.

Heat treatment of martensitic and austenitic steels is carried out to ensure stabilization of structure and to obtain homogeneity of material bulk properties. The structure presented as diversely focused martensitic grains is formed as a result of heat treatment of martensitic steel. Upon quenching and annealing the hardness of a sample made 365 ± 10 HV₁₀₀. The microstructure of austenitic steel after quenching is characterized by uniform structure of austenite grains with twins and a small amount of TiC carbides on crystallite boundaries. Sample hardness after heat treatment made 190 ± 10 HV₁₀₀.

In order to study diffusion processes the following distributions of microhardness on depth were obtained (figure 2).

The analysis of the measured data showed that the distribution of microhardness on depth is characterized by area (I) where the hardness steadily decreases with the increase of distance from the

surface, which is followed by its sharp decrease (transition zone II) with subsequent smooth alignment to the hardness of the basic material (area III). When comparing dependences of microhardness distribution on depth it was identified that the diffusion zone of martensitic steel has smoother decrease of hardness without sharp transition area (II). Sufficient hardening of a steel surface happens when the content of hydrogen in a vacuum chamber varies from 15 %. With further increase in hydrogen content up to 30 % the hardness of martensitic steel surface increases slightly and falls within the limits of $1650 \pm 30 \text{ HV}_{100}$. The hardness of austenitic steel surface increases from 820 to $1100 \pm 10 \text{ HV}_{100}$ in case hydrogen content ranges from 15 to 20 % and slightly changes ($1100 \pm 30 \text{ HV}_{100}$) with its further increase.

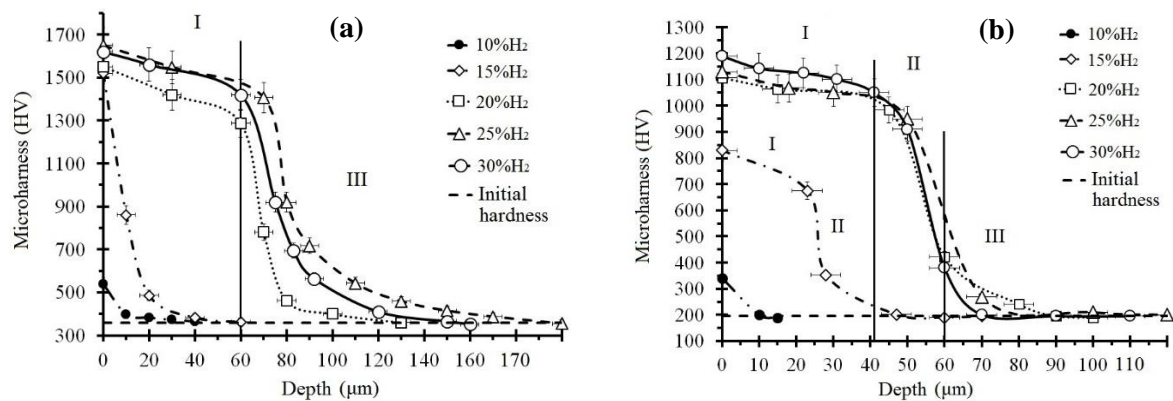


Figure 2. Distribution of microhardness along depth of a hardened layer of martensitic (a) and austenitic (b) steels depending on hydrogen content in a vacuum chamber.

When using gaseous media one shall consider the phenomenon of hydrogen embrittlement of a hardened layer. Bitmap images of sample sections were obtained to study the structure of a nitrated layer (figure 3).

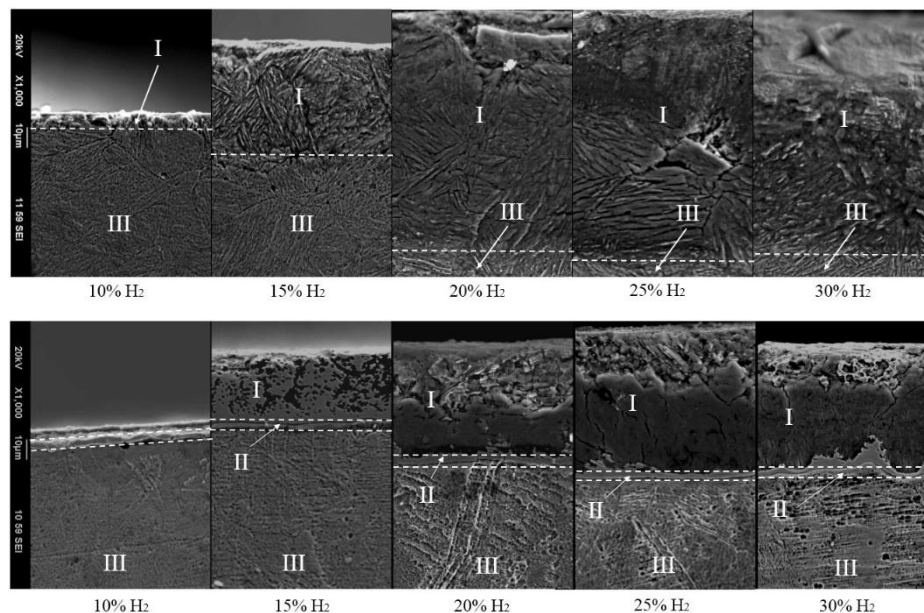


Figure 3. Bitmap images of a surface layer of martensitic (a) and austenitic (b) steels after nitriding with various content of hydrogen in a working gas.

The study of a hardened layer structure showed correlation with distribution of microhardness on depth of all samples. The structure of steels after ion nitriding is characterized by the presence of three

zones: I – nitriding layer; II – diffusion zone; III – basis. It was found that depending on hydrogen content in a working gas and a category of steel the morphology of a hardened layer differs essentially. Intense crack formation is observed on martensitic steel (figure 3(a)) when hydrogen content in gas medium is from 25 %, whereas chips of a near-surface area (I) are local and there are some cracks along the boundaries of martensitic grains. In case of austenitic steel (figure 3(b)) when hydrogen content in a vacuum chamber ranges from 20 to 30 % in near-surface sections of a zone of nitriding layer (I) there are cracks and chips along the whole cross-section, which indicates brittle nature of a hardened layer. The above data confirm diffusion saturation of steel samples with hydrogen when its content in a vacuum chamber falls within the range from 20 to 30 %. The absence of cracks and chips in a surface layer of samples treated with hydrogen content of 10 and 15 % indicates low hydrogen diffusion on a surface due to low concentration.

4. Conclusion

As a result study of the influence of hydrogen content in a working gas on diffusion processes at ion nitriding of martensitic and austenitic steels the following was identified:

1. Efficient hardening of a surface of martensitic and austenitic steels happens when hydrogen content in a vacuum chamber is from 15 %. With further increase the hardness of a steel surface does not considerably change.
2. Change of hydrogen content from 10 to 25 % leads to the increase in nitrogen diffusion depthward the material. As a result, the growth rate of the nitrated layer increases. When hydrogen content exceeds 25 % due to predominance of surface diffusion coating with hydrogen there is the reduction of nitrogen diffusion and the thickness of a hardened layer decreases.
3. Presence of hydrogen in a working chamber in the amount of over 20 % leads to steel saturation with hydrogen which causes hydrogen embrittlement of a hardened layer. Therefore, there is near-surface damage of a nitrated layer.

Acknowledgments

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References

- [1] Park M J, Leyland A and Matthews A 1990 *Journal of Surf. and Coat. Technol.* **43** 481
- [2] Michel H, Czerwicz T, Gantois M, Ablitzer D and Ricard A 1995 *Journal of Surf. and Coat. Technol.* **72** 103–11
- [3] Gary G T 1974 *Journal of Applied Physics* **45** 5072–3
- [4] Mehmet K, Akgün A and Ayhan Ç 2002 *Journal Materials Characterization* **49** 241–6
- [5] Budilov V V, Ramazanov K N, Khusainov Yu G and Zolotov I V 2014 *Reinforcing technol. and coatings.* **120** 27–30
- [6] Shinichiro A and Nobuhiro U 2012 *Journal of Thin Solid Films* **523** 11–4