Influence of hydrogen content in working gas on growth kinetics of hardened layer at ion nitriding of 16MnCr5 and A290C1M steels

Y G Khusainov, R S Esipov, K N Ramazanov, E L Vardanyan, P V Tarasov and S R Shekhtman
Ufa State Aviation Technical University, 450008, Ufa, Russia
E-mail: ramazanovkn@gmail.com

Abstract. The impact of hydrogen content from 10 to 30% in the mixture of operating gas environment on the growth kinetics and structure of a hardened layer during ion nitriding of martensite and pearlite steels has been studied. It is stated that hydrogen in the vacuum chamber in the range from 10 to 30% contributed to a 1.2-time increase of nitrogen diffusion rate in 16MnCr5 steel and 1.3-time increase in A290C1M steel. The dependence of the hardened layer thickness and microhardness distribution over the depth on the gas environment composition is stated. The structure of the nitrided layer of steels at different hydrogen contents during ion nitriding is studied.

1. Introduction
At present most critical parts of machines are manufactured from high-strength alloyed structural steels. An example of such a part is a gear wheel, the working surface of which is a tooth bearing. This tooth bearing of the part is subjected to alternating and contact stresses, which determines the application of surface hardening techniques in production. One of the widely used hardening techniques applied to the surface to structural steels is glow discharge nitriding (ion nitriding) [1]. However, the significant disadvantage of ion nitriding is the long duration of processing (25–30 h) [2].

It is stated in [3–5] that during glow discharge nitriding the composition of the gas environment significantly effects the structure, properties and growth rate of the hardened layer. Use of operating environments, which contain nitrogen and hydrogen (up to 5 %), allows increasing the rate of diffusion saturation in the surface due to chemical etching of oxides on the processed surface [3]. Use of argon (up to 80 %) in the operating environment also enables considerably increasing the growth rate of the diffusion layer, as under the effect of electrical field argon ions diffuse the nitride layer on the surface of a cathode-part, which results in the reduction of the barrier-layer effects [4, 5]. However, at present there are no published data on the impact of multi-component saturating environments on the rate of diffusion growth and mechanical properties of the strengthened layer.

Thus, the study on the impact of the operating gas environment composition on the growth kinetics of the nitride layer and regulation of the ratio between the gas mixture components with the aim to enhance the efficiency of ion nitriding of structural steels is an urgent problem.

2. Experimental procedures
Heat-treated structural steels 16MnCr5 and A290C1M were subjected to ion nitriding. Low-carbon steel
16MnCr5 of the martensite type is widely used to produce critical parts of machines, for example, high-loaded gear wheels. Nitride forming elements allow using it as a nitride one to increase the wear resistance of machine parts. Steel A290C1M is the pearlite one and is used to produce gear wheels, cylinder barrels, worms, spindles, barrels and other parts operating at temperatures up to 450 °C.

Ion nitriding was performed on the modernized set ELU-5M designed to conduct thermal and chemical and thermal treatments in the vacuum. Before nitriding the samples were subjected to ionic clearing for 10 minutes at P=10 Pa in the argon environment, the surface temperature did not exceed T=250 °C. The diffusion saturation was performed in the operating mixture of nitrogen N₂, argon Ar and hydrogen H₂. The operating gas environment composition and nitriding regimes are listed in table 1.

### Table 1. Operating gas environment composition and ion nitriding parameters.

| № | Composition of gas, % | Temperature, °C | Pressure, Pa | Duration, h |
|---|------------------------|------------------|--------------|------------|
| 1 | Ar 60 N₂ 30 H₂ 10      |                  |              |            |
| 2 | Ar 55 N₂ 30 H₂ 15      |                  |              |            |
| 3 | Ar 50 N₂ 30 H₂ 20      | 550              | 150          | 6          |
| 4 | Ar 45 N₂ 30 H₂ 25      |                  |              |            |
| 5 | Ar 40 N₂ 30 H₂ 30      |                  |              |            |

The microhardness measurements of the surface were carried out on the microhardness tester Struers Duramin-1. The statical load value applied to the diamond indenter for 10 seconds was 980.7 mN (100 g). The microstructure studies of the samples were performed with the help of an optical microscope Olympus GX-51. Prior to metallographic studies the cross section samples of steels were etched in 3 % solution of HNO₃.

### 3. Results and discussion

Figure 1 displays the optical images of the surface layer microstructure of steel 16MnCr5 samples after ion nitriding with the hydrogen content in the operating gas environment in the range from 10 to 30 %.

The microstructure analysis of the 16MnCr5 steel samples after nitriding shows that the microstructure of all the samples consists of three zones: I – zone of nitrides, II – diffusion zone, III – matrix. The transition between the diffusion zone (II) and matrix (III) is smooth. The zone of nitrides (I) presumably consists of iron nitrides and alloying elements, and the diffusion layer consists of solid solution of nitrogen in α-Fe [7, 8]. It is stated that during nitriding of 16MnCr5 steel samples (when the hydrogen content changes in the operating gas environment in the range from 10 to 25 %) (figure 1(a)–(c)) a modified layer forms with a thickness (h) of 185 μm. This is explained by the fact that the nitrogen content in the gas mixture is constant of 30 % (table 1). However, after nitriding of the 16MnCr5 steel sample with the hydrogen content in the operating gas environment of 30 % (figure 1(e)), there is no nitride layer (I), the thickness of the diffusion zone is h ~180 μm. The absence of the nitride layer is possibly connected with its spalling during specimen preparation. Spalling of a nitride layer is conditioned by its hydrogen embrittlement, as the hydrogen content in the saturating atmosphere is about 30 %. The reduction of the diffusion zone thickness is caused by a low argon content in the operating gas environment (40 %), which results in the efficiency loss of the barrier nitride layer diffusion.

Figure 2 displays the optical images of the microstructure of the surface layer in steel A290C1M samples after ion nitriding at different contents of the operating gas environment. The microstructure analysis shows that in all the samples the transition between the diffusion zone (II) and the matrix (III) is smooth. The nitride zone (I) consists of iron nitrides, the diffusion zone (II) is the solid solution [7,
There were no microcracks and cleavages on the surface. It is stated that during nitriding of A290C1M steel with the hydrogen content in the operating gas environment of 10, 15, 20, 25, 30 % modified layer with a thickness of $h \approx 195, 230, 240, 250$ and 230 $\mu$m, forms, respectively. The decrease of the hardened layer thickness in the sample after nitriding with a hydrogen content of 30 % in the operating gas environment is explained by a low volume of argon in the saturating atmosphere.

**Figure 1.** Microstructure of $16$MnCr5 steel samples after ion nitriding with different content of hydrogen in the operating gas environment: (a) – 10 % $H_2$; (b) – 15 % $H_2$; (c) – 20 % $H_2$; (d) – 25 % $H_2$; (e) – 30 % $H_2$.

**Figure 2.** Microstructure of A290C1M steel samples after ion nitriding at different contents of hydrogen in the operating gas environment: (a) – 10 % $H_2$; (b) – 15 % $H_2$; (c) – 20 % $H_2$; (d) – 25 % $H_2$; (e) – 30 % $H_2$. 
Figure 3 displays the dependence of the microhardness distribution over the thickness of the surface layer of 16MnCr5 and A290C1M steel samples after ion nitriding with different contents of hydrogen in the gas environment.

![Figure 3](image)

**Figure 3.** Dependences of microhardness distribution over the thickness of the surface layer after ion nitriding with different contents of hydrogen in the gas environment: 16MnCr5 (a), A290C1M (b).

The microhardness distribution graphs over the depth of steel 16MnCr5 after ion nitriding (figure 3(a)) show a smooth change of the microhardness over the thickness of all the studied compositions of the operating gas environment. When the hydrogen content in the vacuum chamber changes from 10 to 30% some the hardened layer thickness increases from 170 to 190 µm and the surface microhardness enhances from 1025 to 1150 HV0.1. The microhardness increment is conditioned by the increase of microstresses in the modified layer under the impact of enhanced concentration of dissolved nitrogen in α-Fe [9, 10]. Thus, the hydrogen content increase in the operating gas environment from 10 to 30 % contributes to hardness enhancement of the hardened layer of 16MnCr5 steel by 2–2.5 times.

The dependence graphs of microhardness distribution over the depth in steel A290C1M after ion nitriding (figure 3(b)) demonstrate a smooth change in the microhardness over the thickness in all the studied compositions of the operating gas environment. When the hydrogen content increases in the vacuum chamber from 10 to 25 %, the hardened layer thickness enhances from 195 to 240 µm, and the surface microhardness grows from 1000 to 1175 HV0.1. When the hydrogen content in the gas environment is about 30 %, the modified layer thickness decreases to 240 µm, and the hardened layer microhardness is 1200 HV0.1. Thus, the hydrogen content increase in the gas environment from 10 to 30 % contributes to enhancement of the hardened layer hardness in steel A290C1M by 2.5–3.5 times.

4. Conclusions

As a result of the studies on the impact of the hydrogen content in the operating gas environment on the growth kinetics of the hardened layer during ion nitriding of steels 16MnCr5 and A290C1M, it was states that:

- the growth kinetics of the hardened layer during the hydrogen content increase in the gas environment from 10 to 25 % for 16MnCr5 steel practically does not change, and that for A290C1M steel increases by 1.3 times. The use of the operating gas atmosphere with the hydrogen content of about 30 % and argon of 40 % for the studied steels results in the reduction of the growth kinetics of the hardened layer, as in the mixture of gases the argon content reduces, and the barrier effect takes place due to formation of a nitride layer. The nitride layer prevents nitrogen atoms to penetrate the matrix.
the hydrogen content increase in the gas environment from 10 to 30 % results in the enhancement of the efficiency of oxides etching on the steel surface and increase of the dissolved nitrogen concentration in α-Fe. As a result the microhardness increment occurs in the hardened layer for 16MnCr5 steel by 2–2.5 times and that for A290C1M steel increases by 2.5–3.5 times.

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