Influence of ion-plasma nitriding on wear-resistance of Cr6VW die steel

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Abstract. The paper presents research data on the surface structure, phase state, microhardness, and tribological properties (wear resistance, friction coefficient) of Cr6VW steel nitrided in the plasma of a non-self-sustained low-pressure hollow-cathode glow discharge at a bias of -200 V and temperature of 460 and 520°C for 3 and 8 h.

1. Introduction
As has been shown [1, 2], coating deposition and plasma nitriding allow an up to several-fold increase in the service life of steel tools. Most of the related studies concern gas nitriding and plasma nitriding in anomalous glow discharges in which the pressure is two to three orders of magnitude higher and the nitriding rate is two to three orders of magnitude lower than the values typical of low-pressure discharges. Nitriding in low-pressure arc plasmas requires a rather high negative bias [3] or indirect heating [4], i.e., it provides a rather low (about 1–2 mA/cm²) ion current density which correlates with the amount of nitrogen to the surface. The non-self-sustained glow discharge provides a plasma density of about 10¹⁸ m⁻³ and ion current density of more than 10 mA/cm² in relatively large (>0.1 m³) vacuum volumes [5], making possible nitriding at a bias of 100–200 V without indirect heating. By now, insufficient data are available on the structure and properties of die steels after such treatment.

Here we analyze the surface structure, phase state, and physicomechanical properties of Cr6VW die steel nitrided in the plasma of a non-self-sustained low-pressure glow discharge and determines the modes which are most efficient for increasing the wear resistance of die steels operated under severe conditions.

2. Material and research techniques
The test material was hardened Cr6VW die steel (1.05C, 5.5Cr, 0.5V, 1.1W wt%) shaped as cylinders of diameter of 30 mm and height of 5 mm. The steel surface was nitrided in the plasma of a non-self-sustained low-pressure hollow-cathode glow discharge (high purity nitrogen (99.999%), 2 Pa) on a setup described in detail elsewhere [5].

The specimens were fixed on a holder to which a negative bias of 200 V was applied from a separate power supply. The specimen temperature during nitriding was 460 and 520°C, and the nitriding time was 3 and 8 h. The specimen surface was heated and cleaned via nitrogen ion bombardment.

The structure and the phase composition of the steel before and after modification were examined on a µVizio- MET-221 optical microscope, Philips SEM-515 scanning electron microscope, and Shimadzu XRD diffractometer in CuKα radiation. For analyzing the phase composition, we used PDF 4+ data bases and POWDER CELL 2.4 software. The Vickers microhardness was measured on a PMT-3 device at an indenter load of 0.5 N.
The tribological tests were carried out on a Tribotechnic tester using pin-on-disk configuration under conditions of friction with lubrication during the reciprocal motion of the sample relative to the counterbody. As a counterbody a ruby ball with a diameter of 6 mm was used. The load on the ball was 12 N, the amplitude of the reciprocal motion was 5 mm, the friction path was 1200 m. A soap solution based on water (1:125) was used as the lubricant. Comparative tests of the wear resistance were made for the steel samples in the initial state and after nitriding in plasma of glow discharge. The coefficient of wear was estimated by the formula 

\[ V = \frac{(S \cdot E)}{F_n \cdot S_l} \]

where \( S \) is the track cross-section (mm\(^2\)), \( E \) is the eccentric (mm), \( F_n \) is the normal load on the friction surface, and \( S_l \) is the sliding length (m). The friction coefficient was estimated through recording its current values. Before tribological tests, the steel surface was polished with a diamond paste to a roughness \( r_a = 0.04 \mu m \). The thickness of the removed layer was several hundred nanometers.

### Results and discussion

The surface characteristics of Cr6VW steel before and after nitriding in different modes are listed in table 1.

| Mode | Nitriding temperature, °C | Nitriding time, h | Friction coefficient | Specific wear rate, mm\(^3\)/N\(\cdot\)m | Nitriding depth, µm | Nitride layer thickness, µm |
|------|--------------------------|------------------|---------------------|----------------------------------------|-------------------|-----------------------------|
| 1    | –                        | –                | 0.08                | 6.9*10\(^{-8}\)                        | –                 | –                           |
| 2    | 460                      | 3                | 0.09                | 2.4*10\(^{-8}\)                        | 50                | 3–4                         |
| 3    | 460                      | 8                | 0.08                | 2.1*10\(^{-8}\)                        | 80                | 4–5                         |
| 4    | 520                      | 3                | 0.07                | 2.8*10\(^{-8}\)                        | 80                | 5–6                         |
| 5    | 520                      | 8                | 0.08                | 2.5*10\(^{-8}\)                        | 170               | 9–10                        |

Figure 1 shows the microhardness distribution in depth of Cr6VW steel nitrided in plasma of a hollow-cathode glow discharge at 460 and 520°C. It is seen that the modification greatly increases the surface microhardness of the steel. The highest surface microhardness is attained after nitriding at 460°C. Its measured value was about 12 GPa, which is almost 20% higher than the microhardness after nitriding at 520°C. This can be explained, among other things, by the higher nitrogen content in the surface layer. The
nitride layer thickness after nitriding at 460°C for 3 and 8 h is 50 and 80 µm, respectively. At 520°C, it increases to 80 and 170 µm, respectively (table 1). It should be noted that the main bulk of hardened Cr6VW steel heated to 460°C and nitried for 3 h does not show any softening (figure 1a). Increasing the nitriding time only slightly decreases its microhardness. When nitried at 520°C, the steel is tempered more intensively, and this decreases the microhardness in its bulk by 25–35% (figure 1b).

The analysis of the Cr6VW microstructure shows that a thin nitride layer with a diffusion saturation zone beneath it is formed in all nitriding modes (table 1). The nitride layer consists of ε-Fe2–3N and γ′-Fe4N phases with ε-Fe2–3N being the main phase (figure 2). As the nitriding temperature is increased, the volume fraction of the γ'-phase in the surface layer increases, and that of the ε-phase decreases. After nitriding at 460°C, chromium nitrides in the surface layer are not found, whereas their small amount is detected after nitriding at 520°C.

After nitriding in all modes, the specific wear rate of Cr6VW steel decreases to (2.5-3) times compared to its initial value (table 1), and its friction coefficient is the same as the initial one.

Figure 2. X-ray diffraction patterns of Cr6VW steel surface layer after nitriding for 8 h at 460 (1) and 520 °C (2).

4. Conclusion

Thus, our analysis of the surface structure, phase state, and tribological and mechanical properties of Cr6VW steel nitried in the plasma of a non-self-sustained glow discharge shows the following. The surface microhardness of the steel nitried at 460°C increases to 12 GPa with no change in the initial microhardness of the steel bulk. Increasing the nitriding temperature to 520°C increases the nitride layer thickness but decreases the surface microhardness of the steel to 10.5 GPa and the microhardness in its bulk by 25–35% compared to their initial values. The nitride layer formed on the steel surface consists of ε-Fe2–3N and γ′-Fe4N phases. After nitriding at 520°C, chromium nitride appears in the steel surface layer. The research results demonstrate the efficiency of nitriding in non-self-sustained glow discharge plasmas for increasing the wear resistance of Cr6VW steel.

Acknowledgments

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References

[1] Bjork Th, Westergard R and Hogmark S 2001 Wear 249 316
[2] Wang B, Zhao X, Li W, Qin M and Gu J 2018 Appl Surf Sci 431 39
[3] Schanin P M, Koval N N, Goncharenko I M and Grigoryev S V 2001 Fiz Khim Obr Mater 3 16
[4] Andreev A A, Shulaev V M, Sablev L P 2006 Fiz Inzh Poverkhn 4 191
[5] Akhmadeev Y H, Denisov V V, Koval N N, Kovalsky SS, Lopatin IV, Schanin P M and Yakovlev V V 2017 Plasma Phys Rep 43 67