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Physical and Technological Parameters of Cr28 Steel Nitriding in an Ammonia Environment

T.V. Loskutova[1], I.S. Pohrebova[1], S.M. Kotlyar[1], M.M. Bobina[1], D.A. Kaplii[1], N.A. Kharchenko[2], T.P. Hovorun[2,*]

[1] *National Technical University of Ukraine "I. Sikorsky Kyiv Polytechnic Institute", 37, Prosp. Peremohy, 03056 Kyiv, Ukraine*
[2] *Sumy State University, 2, Rimsky Korsakov Str., 40007 Sumy, Ukraine*

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The paper examines the effect of technological parameters of gas nitriding (temperature and time) on the phase composition, structure, microhardness, and wear resistance of the Cr28 ferritic class corrosion-resistant steel. Nitriding was carried out in an environment of dissociated ammonia in the temperature range of 550-950 °C. The dependence of the phase composition of the formed coatings on the nitriding temperature was established. X-ray structural, metallographic, and durometric analyses determined that the following phases are formed due to nitriding: Fe_2N, Fe_4N, Fe_α, Fe_γ, and CrN. The maximum microhardness of 15.5-16.0 GPa was recorded for coatings on Cr28 steel after nitriding at a temperature of 550 °C. The influence of the annealing temperature on the microhardness of the nitrided coating was analyzed. A decrease in the microhardness of the nitrided coating on Cr28 steel starting from the annealing temperature of 600 °C was recorded. It was established that the minimum reduction in microhardness at an annealing temperature of 750 °C is characteristic of Cr28 steel nitrided at a temperature of 550 °C. The optimal mode of nitriding (temperature 550 °C, time 6 hours) was determined, which allows for obtaining the maximum abrasive resistance of Cr28 steel after nitriding. At the same time, a 2.8-fold increase in the wear resistance of nitrided Cr28 steel compared to the original structure is recorded.

Keywords: Nitrided coatings, Microhardness, Wear resistance.

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1. INTRODUCTION

Various types of alloys are used in modern production and electronic engineering [1, 2]: metallic, multi-component, granular, high-entropy, arranged in bulk and film states.

Nitriding of metals and alloys is currently one of the widespread methods of increasing the surface hardness, strength, and corrosion resistance of products [3-6]. Well-known literary sources on nitriding of stainless steels mainly describe the study of the structure and properties of coatings obtained under generally accepted technological regimes [3, 4, 6].

The significant length of the process can be attributed to the disadvantages of nitriding. For many years, work was carried out to intensify the process of steel saturation with nitrogen. In the scientific and technical literature, results are given regarding the acceleration of nitriding processes under the action of ultrasound, radioactive radiation, glow discharge, high temperatures, etc., of which nitriding at high temperatures is probably the most effective. In this case, it is possible to hardening nitrided steels with subsequent tempering or cold treatment.

The task of the work is to carry out a critical analysis of the influence of temperature and time of the nitriding process on the formation of the phase composition and structure of corrosion-resistant steel Cr28. In addition, the goal is to determine and provide recommendations on the optimal mode of nitriding and subsequent annealing, which would ensure the formation of the Cr28 steel structure with high microhardness and abrasion resistance.

2. MATERIALS AND METHODS

Corrosion-resistant steel of ferrite class Cr28 were proposed as the object of research in the work. It should be noted that scientific and technical information on the chemical-thermal treatment of Cr28 steel and even steels of this class is practically absent or severely limited. The main alloying element of Cr28 steel is chromium, the concentration of which is 28.1 % by weight. In addition, the steel composition includes carbon, with a concentration of 0.1 % by mass, and silicon and manganese, respectively, with a concentration of 1.0 and 0.8 % by mass. In the first stage of the research, we carry out the process of nitriding in different modes, in the temperature range of 550-950 °C. In the second stage of work, we implement annealing to varying temperatures of 400-750 °C. The research results were obtained by a complex method based on: X-ray phase, microstructural and durometric methods, which corresponded to modern methods of physical materials science.

3. RESULTS AND DISCUSSION

The results of studies of growth kinetics, microstructure, microhardness, and phase composition of nitrided layers on Cr28 steel are shown in Fig. 1-Fig. 3 and Table 1.

The growth kinetics of nitrided layers on Cr28 steel is shown in Fig. 1. The analysis of the obtained data showed the nature of the relation between the thickness

* hovorun@pmtkm.sumdu.edu.ua

of the nitrided coatings and the saturation time. The established relation is close to parabolic, which indicates diffusion processes during nitriding of Cr28 steel. First of all, it is talking about the diffusion of nitrogen, which is accompanied by the formation of α-iron, nitrides of iron and chromium in the nitrided coatings.

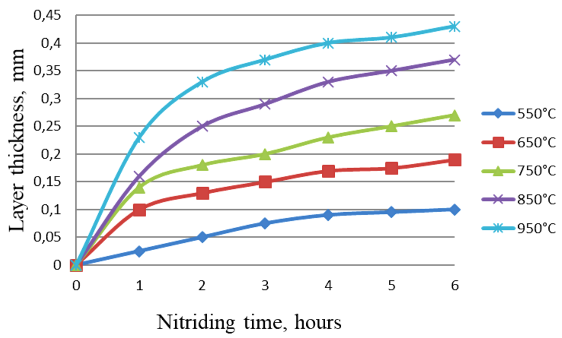

Fig. 1 – Growth kinetics of nitrided layers on Cr28 steel

Layer-by-layer studies of the phase composition of nitrided coatings showed that the phase composition, the arrangement of phases in the coating, and their number depend on the nitriding temperature. The diffraction lines of compounds Fe_2N, Fe_4N, CrN are present on the X-ray pattern of the surface of Cr28 steel nitrided at the temperature of 550 °C. At a certain thickness from the surface, α-phase lines appear, and at the distance of 40-50 μm from the surface, lines of α-phase and CrN nitride appear. At the nitriding temperature of 650-850 °C, the γ-phase appears on the nitrided surfaces, which is present in coatings up to 40-50 μm thick. The following compounds are present on the surface of the nitrided layers obtained at a nitriding temperature of 850 °C: α-, γ-iron, CrN.

In works [7, 8], the mechanisms of phase formation on the surface of corrosion-resistant steels during nitriding were studied. At the initial stages of chemical-thermal treatment (CTT), α-iron is saturated with nitrogen, which is typical for the entire selected range of saturation temperatures. After reaching the limit of nitrogen content in α-iron, Fe_4N nitride (γ'-iron) is formed, then Fe_2N nitride (ε-iron). Due to the large concentration of nitrogen to chromium, the formation of CrN compounds in coatings occurs immediately after reaching the limit of nitrogen content in the α-phase [5, 6]. The interference pattern from CrN nitride is detected simultaneously with the interference pattern from Fe_4N compounds. It should be noted that the chromium concentration in Cr28 steel is significantly higher compared to the concentration of chromium in the steels of works [5, 6] (20Cr13, 40Cr13, Cr18N9). It can be assumed that the probability of CrN nitride formation in the nitrided coating of Cr28 steel is significantly higher than that of the steels of works [7, 8]. According to the results of X-ray structural analysis, CrN compounds are present in nitrided coatings on Cr28 steel throughout the thickness obtained in the temperature range of 550-850 °C.

In the structure of coatings on nitrided Cr28 steel, the following zones can be distinguished: the zone of compounds (Fe_4N, FeN, CrN, Fe_α) and the zone of internal nitriding (Fig. 2). Nitriding at the temperature of 650 °C is accompanied by the formation of the zone of compounds, which consists of equiaxed crystals with dimensions of 3.8-8.0 μm. At the border between the internal nitriding zone and the compound zone, microstructural analysis revealed the light compound several microns thick. As the nitriding temperature increases, the boundary strip increases in thickness, the border between the zone of compounds and the zone of internal nitriding becomes blurred, the etchability of the zone of compounds decreases. On the gray background of the etched zone of compounds, individual inclusions of almost white color with clearly defined habits can be detected. These inclusions correspond, most probably, to chromium nitride or carbonitride.

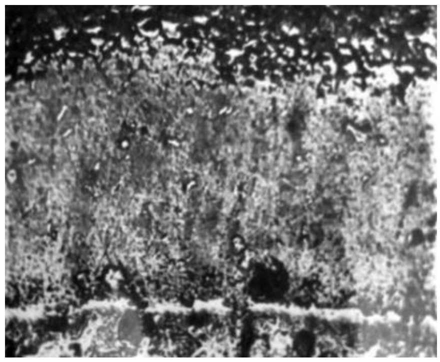

Fig. 2 – Microstructures of corrosion-resistant steel Cr28 after nitriding (650 °C, time is 2 hours), ×500

The structure of the outer side of the obtained coatings is typical of nitrided steels [3]: it is the presence of pores, cracks, and layering in the coating. The thickness of this zone is 15.0-25.0 μm.

The results of studies of the coatings microhardness are shown in (Fig. 3). Analysis of changes in the microhardness of nitrided layers by thickness showed that there are maxima at a distance of 20.0-40 microns from the surface. The maximum microhardness is 15.0-15.5 GPa, found on coatings obtained at the temperature of 550 °C. As the nitriding temperature increases, the microhardness of the coatings decreases and is 7.1-7.6 GPa after nitriding at temperatures of 850-950 °C. The high microhardness of the coatings obtained at the temperature of 550 °C is caused by the nitride dispersion, the decrease in microhardness with increasing nitriding temperature is caused by the agglomeration of nitrides.

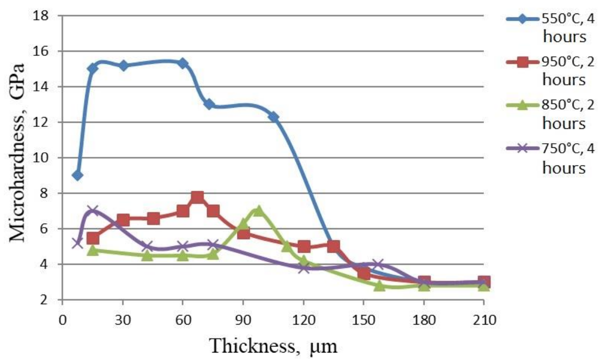

Fig. 3 – Microhardness distribution of coatings obtained during nitriding of corrosion-resistant steel Cr28

Table 1 – Effect of the saturation temperature and time on the total thickness and phase composition of nitrided coatings obtained on the surface of corrosion-resistant steel

Distance from the surface, μm	Nitriding temperature and time				
	550 °C, 10 hours	650 °C, 1 hour	650 °C, 10 hours	750 °C, 1 hour	850 °C, 0.5 hour
	Phase composition				
surface*	Fe_2N_3, Fe_4N, CrN	Fe_2N, Fe_4N, CrN, γ	Fe_2N, Fe_4N, CrN, γ	CrN, Fe_2N, Fe_4N, γ	α, CrN, γ
10	Fe_4N, Fe_2N, CrN, α	CrN, Fe_4N, γ, α	CrN, Fe_2N, Fe_4N, γ	CrN, γ, Fe_4N, α	α, CrN, γ
20	Fe_4N, Fe_2N, CrN, α	γ, α, CrN	CrN, γ, α	γ, CrN, α	α, CrN, γ
30	α, CrN, Fe_4N	α, γ, CrN	γ, α, CrN	γ, CrN, α	α, CrN, γ
40	α, CrN	α, CrN	γ, α, CrN	α, CrN, γ	α, γ, CrN
50	α, CrN	α, CrN	γ, α, CrN	α, CrN	α, **
60	α, CrN	α, CrN	α, γ, CrN	α, CrN	α, **
70	α, CrN	α, CrN	α, CrN	α, CrN	α, **
80	α, CrN	α, $Cr_{23}C_6$	α, CrN	α, CrN	α, **
100	α	–	α, CrN	α, $Cr_{23}C_6$	α, $Cr_{23}C_6$
120-200	–	–	α, CrN	–	–
250	–	–	α, $Cr_{23}C_6$	–	–

* are phases Cr_2O_3, Fe_3O_4 located on the surface
** are undeciphered compounds

Annealing of nitrided steel Cr28 samples was carried out in the temperature range of 400-750 °C. At annealing temperatures of 400 and 500 °C, there were no changes in the microhardness of the nitrided layer. In coatings obtained at the temperature of 550 °C, annealing at the temperature of 750 °C leads to the sharp decrease in microhardness to 8.5 GPa in the zone at the distance of 75.0-80.0 μm from the surface. The increase in the annealing temperature to 650-750 °C leads to the decrease in the microhardness of all the studied coatings. At the same time, the maximum microhardness after annealing at the temperature of 750 °C (Fig. 4) is established for coatings obtained at the temperature of 550 °C.

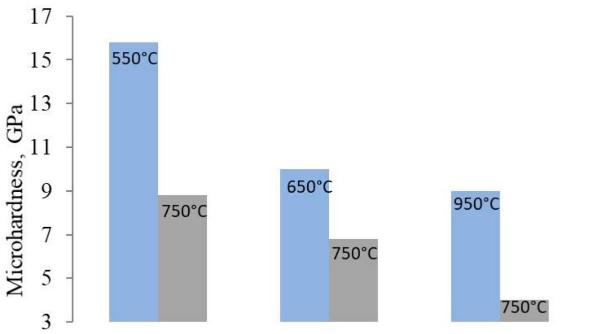

Fig. 4 – Microhardness histogram of nitrided layers on Cr28 steel a, b, c – microhardness of coating on Cr28 steel after nitriding at temperature 550 °C(a), 650 °C(b), 950 °C(b); g, d, e – microhardness of nitrided coating after annealing at the temperature 750 °C

The results of testing the abrasive wear resistance of Cr28 steel in its initial state and nitrided at temperatures of 550 °C are shown in Fig. 5. The analysis of the obtained data showed that the wear resistance of the initial steel was higher than the wear resistance of the nitrided steel for the first 0.5 hour of testing. The non-obvious dependence is related to the presence of the defective zone with cracks, pores and chips on the outside of the nitrided layer. The defective layer is removed from the surface within 0.5 hours. During the next period of the test, the wear resistance of nitrided Cr28 steel turned out to be higher than the wear resistance of the initial steel.

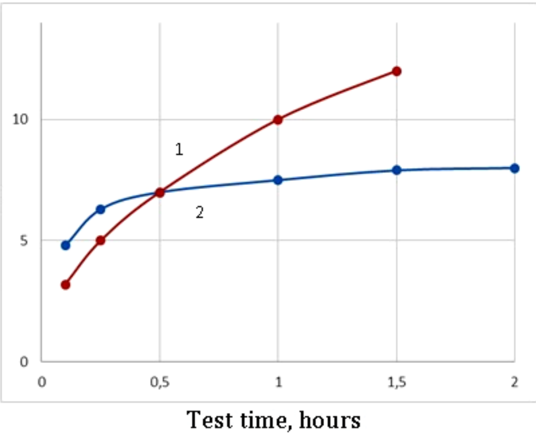

Fig. 5 – Dependence of abrasive wear on test time: 1 – Cr28 steel (initial state), 2 – nitrided Cr28 steel (550 °C, 8 hours)

The analysis of known results [7-9] showed that destruction in the process of abrasive wear occurs in two stages, which are controlled by different mechanical properties. The first one, it is penetration of abrasive particles into the coating, which corresponds to the penetration of a diamond indenter into the material when measuring microhardness. It can be assumed that the processes of the first stage are determined by microhardness. During the movement of abrasive particles on the surface, which is the second stage of wear, the nitrided layer is destroyed and carried away by abrasive flows.

The tendency to destruction of high-hardness materials will be determined by their fragility. As mentioned above, nitriding of Cr28 steel at the temperature of 550 °C leads to the formation of diffusion layers with the microhardness of 15.5-16.0 GPa. At the same time, no cracks were found near the indenter impressions. Thus, high hardness and high crack resistance of nitrided layers on Cr28 steel contribute to high abrasive wear resistance of coatings.

The results of studies of phase composition, structure, microhardness and wear resistance of nitrided Cr28 steel obtained in the work make it possible to recommend this type of coating for protection of Cr28 steel from the abrasive wear.

4. CONCLUSIONS

1. The effect of the nitriding temperature in the dissociated ammonia environment on the phase composition, structure, microhardness and wear resistance of Cr28 steel with the coating was determined.

2. Coatings nitrided at the temperature 550 °C consist of compounds Fe_2N, Fe_4N, CrN, Fe_α; nitrided in the temperature range of 650-750 °C contain Fe_2N, Fe_4N, Fe_α Fe_γ, CrN; and nitrided in the temperature range of 850-950 °C contain Fe_α Fe_γ, CrN are shown.

3. The maximum microhardness of 15.5-16.0 GPa was detected for coatings after nitriding at the temperature of 550 °C.

4. The effect of the annealing temperature on the microhardness of the nitrided layers was determined; annealing at temperatures higher than 600 °C is accompanied by the sharp decrease in the microhardness of the nitrided layers.

5. The effect of nitriding on the wear resistance of Cr28 steel under the action of abrasive wear was established. In comparison with the wear resistance of the initial Cr28 steel, nitrided is 2,8 times higher.

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Фізичні та технологічні параметри азотування сталі Х28 в середовищі аміаку

Т.В. Лоскутова[1], І.С. Погребова[1], С.М. Котляр[1], М.М. Бобіна[1], Д.А. Каплій[1], Н.А. Харченко[2], Т.П. Говорун[2]

[1] *Національний технічний університет України «КПІ імені І. Сікорського», просп. Перемоги, 37, 03056 Київ, Україна*

[2] *Сумський державний університет, вул. Римського-Корсакова, 2, 40007 Суми, Україна*

У роботі досліджено вплив технологічних параметрів газового азотування (температури і часу) на фазовий склад, структуру, мікротвердість та зносостійкість корозійностійкої сталі феритного класу Х28. Азотування проводили в середовищі дисоційованого аміаку в інтервалі температур 550-950 °С. Встановлена залежність фазового складу сформованих покриттів від температури азотування. Рентгеноструктурним, металографічним та дюрометричним аналізами визначено, що в результаті азотування формуються наступні фази: Fe_2N, Fe_4N, Fe_α, Fe_γ, ХN. Максимальна мікротвердість 15,5-16,0 ГПа була зафіксована для покриттів на сталі Х28 після азотування при температурі 550 °С. Проаналізовано вплив температури відпалу на мікротвердість азотованого покриття. Зафіксовано, зниження мікротвердості азотованого покриття на сталі Х28 починаючи з температури відпалу 600 °С. Встановлено, що мінімальне зменшення мікротвердості при температурі відпалу в 750 °С характерне для сталі Х28, азотованої за температури 550 °С. Визначений оптимальний режим азотування (температура 550 °С, час 6 годин), який дозволяє отримати максимальну абразивну стійкість сталі Х28 після азотування. При цьому фіксується підвищення зносостійкості азотованої сталі Х28 в 2,8 рази в порівнянні з вихідною структурою.

Ключові слова: Нітридні покриття, Мікротвердість, Зносостійкість.