

Microstructural And X-Ray Structural Analysis Of Nitro-Oxidized Annealed Steel

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Annotation: The article examines the structural and phase changes of surface diffusion nitride-oxide coatings obtained in the process of nitriding in a gas medium followed by oxidation in water vapor of ferrite-pearlite steels, as well as the effect of phase changes on corrosion properties.

Keywords: ferrite, pearlite, nitriding, oxidation, diffusion coating, nitride layer, oxide layer, corrosion resistance

Introduction

When nitriding metals and alloys, multiphase surface diffusion coatings are formed on the surface of the workpiece, consisting of a nitride zone and an internal nitriding zone, which provide a wide range of physical-mechanical and physical-chemical characteristics of nitrided materials. In order to expand the scope of nitriding, combinations of the process with other surface hardening methods are used. In this case, intensification of the process is achieved, regulation of the structure and structure, as well as modification of the phase composition of the surface nitride layer with the achievement of the required properties, taking into account the specific operating conditions of the processed product [1-3].

When nitriding according to the “iron-nitrogen” phase diagram, a nitride layer is formed on the surface of the workpiece, consisting of the ϵ -phase, $(\epsilon+\gamma')$ -phase and γ' -phase. The mixture is located under the high-nitrogen ϵ -phase and is followed by a thin γ' -phase. The boundary advancement of the γ' -phase significantly increases the average nitrogen content in the internal nitriding zone [4-7].

As a result of oxidation of the nitride layer, nitride, carbonitride, oxycarbonitride layers and a zone of internal nitriding with an enriched γ' -phase are obtained, each of which is responsible for certain corrosion or wear-resistant properties [7-8].

According to the theory of nitriding according to Yu.M.Lakhtin, regulation of the phase composition of the nitrided layer is achieved by changing the nitrogen potential of the saturating atmosphere. By maintaining the nitrogen potential at the level of nitrogen solubility in one or another phase, a layer consisting of iron nitrides and alloying elements can be formed on the surface of metals and alloys, and it is also possible to form a γ' -phase (low nitride) or a γ -phase with a low nitrogen content on the surface of products.

METHODS.

To develop a production controlled technology for conducting the process and the theoretical foundations of the technology of the combined process of nitriding with subsequent oxidation in water vapor for surface hardening of low-alloy steel, the dependences of the composition and structure of the nitrided and oxide layer on the chemical composition of steels and

technological parameters of the process were studied. Industrial steels 20, 45 and 40X were studied after annealing with a ferrite-pearlite structure.

When nitro-oxidation, in order to obtain the required effective thickness of the diffusion surface nitride layer, nitriding is carried out at a temperature of 500-580⁰C with a holding time of 3-5 hours. Metallographic analysis of the treated steel samples was carried out on transverse sections using a Neophot-21 light microscope.

Qualitative phase X-ray diffraction analysis of the diffusion nitride and nitride-oxide layers was studied using a Dron-3 diffractometer using filtered cobalt K_α radiation. The general corrosion resistance of the treated samples was studied in a 3% NaCl solution.

RESULTS AND ITS DISCUSSION

During nitriding followed by oxidation, a combined diffusion layer is formed, consisting of a surface oxide zone, carbonitride and oxycarbonitride zones, followed by a diffusion sublayer - internal nitriding zones (INA). In all cases, at the first stage of saturation, preferential diffusion of nitrogen occurs, and the structure and phase composition of the diffusion nitride layer is determined by the phase diagram for the “Fe-N” system.

During gas nitriding in dissociated ammonia at saturation above the eutectoid temperature (591⁰C) for the “iron-nitrogen” system, the resulting diffusion nitride layer, depending on the cooling method, consists of a porous $\xi+\varepsilon$ -phase, $\varepsilon+\gamma'$ -phase, γ -phase and it is followed by an internal nitriding zone (Figure 1, a). The high-nitrogen $\xi+\varepsilon$ -phase has a columnar structure and, when cooled, is easily oxidized by atmospheric oxygen and always forms a thin porous oxide film consisting of Fe₂O₃ on its surface. A mixture of $\varepsilon+\gamma'$ -phase in the nitride layer is formed during the cooling period, due to the difference in nitrogen concentration along the depth of the layer (Figure 1, a).

In low-carbon steels, the internal nitriding zone formed in a mixture with the γ -phase, during the saturation process, has virtually no significant effect on any properties of the material being processed (Figure 1, a).

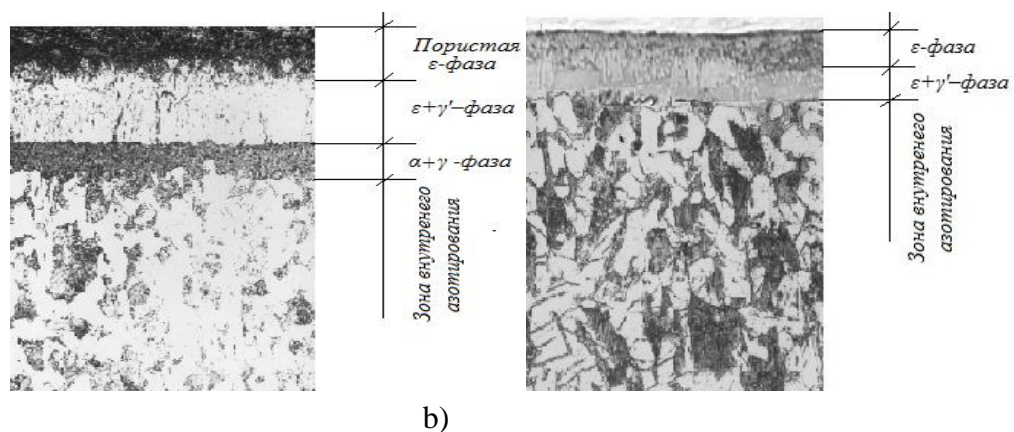


Figure 1. Microstructure of steel 20 after nitriding at temperatures of 620⁰C (a) and 580⁰C (b) in a dissociated atmosphere for 3 hours.

When nitriding below the eutectoid temperature (580⁰C), a more dense structure is formed on the surface of steel 20 than the nitride layer obtained at a higher eutectoid temperature (Figure 1, b).

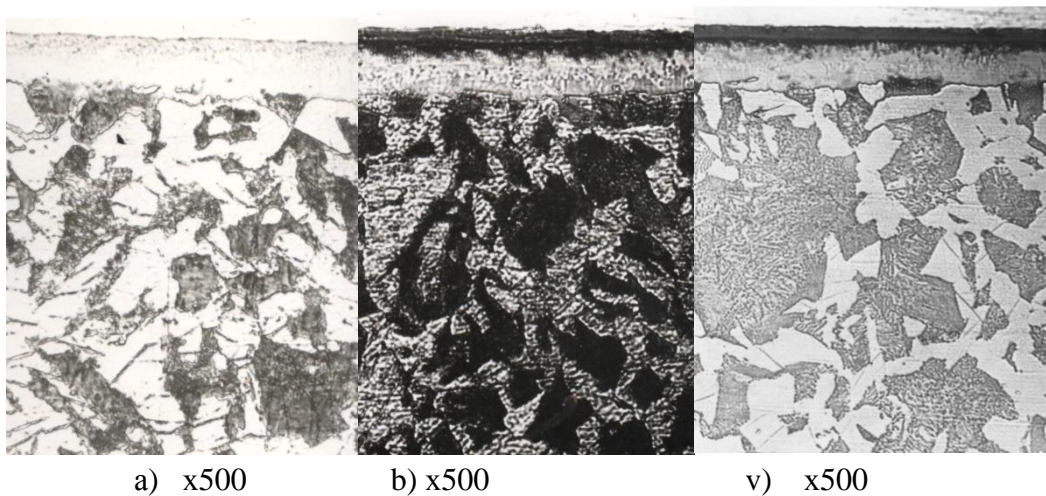
Forming a mixture of $\varepsilon+\gamma'$ -phases in the nitride layer behind the high-nitrogen ε -phase, the formation during the cooling period has dispersed structures due to the uneven distribution of carbon in the steel matrix. (Figure 1, b).

During the subsequent oxidation in water vapor of the nitride layer obtained at below

eutectoid temperatures (Figure 2, a), a thin oxide layer is formed on the surface of the nitride layer due to denitrogenation of the nitride layer (Figure 2, a and b).

As a result of this change in the nitrogen potential of the saturating atmosphere during nitriding, it is possible to obtain a more dense structure of the mixture of ϵ -phase nitride and ϵ' -phase carbonitride in the surface layer for its further oxidation (Figure 2, a). When nitriding is combined with subsequent oxidation in water vapor during the period of saturation of the nitride layer with carbon in the steel matrix, a carbonitride ϵ -phase is formed; subsequently, due to the diffusion of oxygen in the carbonitride phase during oxidation, an oxycarbonitride ϵ -phase is formed [5,7].

During oxidation, with the achievement of the required density of the oxide layer, which counteracts denitrogenation and decarbonization of the matrix under the oxide layer, depending on the concentration of nitrogen, carbon and oxygen, mixtures are formed when the nitride layer is cooled ϵ -, ϵ' - and ϵ'' -phase and is followed by $\epsilon''+\gamma'$ -phases (Figure 2, b and v).



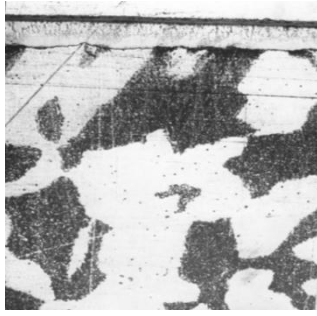
a-nitriding at a temperature of 580°C for 3 hours; b-nitriding at a temperature of 580°C for 3 hours; subsequent oxidation at a temperature of 580°C for 0.5 hours; b-nitriding at a temperature of 580°C for 3 hours; subsequent oxidation at a temperature of 550°C for 1.0 hour.

The oxide film obtained on the surface of the nitride at above the eutectoid temperature for the iron-oxygen system (570°C) has a microporous structure, and wustite (FeO) is detected in x-ray diffraction patterns during rapid cooling (Figure 3, a).

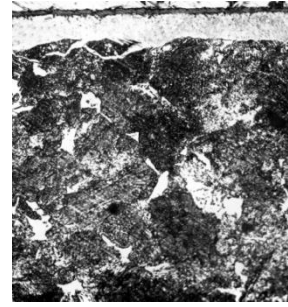
In this case, the resulting oxide layer has a dense structure and consists of one oxide Fe_3O_4 , and the resulting oxide layer also acts as a barrier layer that prevents denitrogenation, since due to the dissociation of the high-nitrogen ϵ -phase, a more uniform distribution of nitrogen, carbon and oxygen in the nitrided layer with uniform formation of oxycarbonitride layer of the ϵ'' phase at different durations of the oxidation process (Figure 2, b).

The study of the microstructure and X-ray diffraction analyzes of the nitro-oxidation process on steels 45 and 40X also confirms the obtaining of a uniform oxide layer consisting of Fe_3O_4 monoxide and a mixture of the surface oxide film of the nitride layer in a mixture of the oxycarbonitride ϵ'' -phase and γ' -nitride, it is possible to obtain a uniform nitride layer on the surface of the ferrite and pearlite on annealed steels (Figure 3, a and b).

A low-nitride layer consisting of ϵ' , ϵ'' and γ' -phase, therefore, to form a carbonitride layer on the ferrite surface, it is necessary to set the oxidation time of the nitride layer based on the diffusion of carbon from the pearlite part to the ferrite, obtaining a uniform carbonitride or oxycarbonitride layer under the oxide layer.



a) x500



b) x500

a - nitriding at a temperature of 580⁰C in an ammonia atmosphere with the degree of ammonia dissociation $\alpha = 45-60\%$ and followed by oxidation at a temperature of 550⁰C for 0.5 hours. b - nitriding at a temperature of 580⁰C in a mixture of 0.75% NH₃ + 0.25% H₂ for 3 hours and subsequent oxidation at a temperature of 550⁰C for 0.5 hours. Figure 1. Microstructure of steel 45 (a) and 40X (i) after nitrooxidation.

The time dependences of the appearance of the first foci of corrosion in a 3% aqueous solution of NaCl for samples with a nitride-oxide coating were studied. With an increase in the amount of γ' -phase in the nitride zone, the time until the first foci of corrosion increases, reaching up to 450 hours. A further increase in the amount of γ' -phase is accompanied by a decrease in corrosion resistance. The presence of an oxide layer has a positive effect on the corrosion properties of the layer. It should be noted that in the experiments performed, the thickness of the oxide zone was 1-5 microns.

Subsequent oxidation of the γ' - phase in water vapor at a temperature of 540-550⁰C for 0.5-1 hour on the surface can produce a uniform oxide layer 1-3 microns thick. The resulting nitrooxide layer has better corrosion properties, the time of appearance of the first foci of corrosion on which is 1056 hours.

The nitride layer, consisting of a mixture of ϵ' , ϵ'' and γ' - phase with a surface oxide layer consisting of Fe₃O₄, has the most corrosive properties. This states that during the oxidation of the nitride layer, the growth of the nitride sublayer with the formation of lower nitride ϵ' , ϵ'' and γ' - phases due to the high-nitrogen γ' -phase obtained during nitriding at the first stage of nitriding is important.

The results of the studies show that:

- the required structure and phase composition of the nitride-oxide layer is achieved under optimal processing conditions, obtaining low-nitrogen ϵ' , ϵ'' and γ' -phases and a surface layer of Fe₃O₄ oxide 1-5 microns thick.

- by nitrooxidation of annealed steels with a ferrite-pearlite structure, it is possible to obtain a uniform surface diffusion layer with the necessary ratios of nitrogen phases in the nitride layer;

- corrosion resistance depends on the ratio of nitride phases; the oxide layer consisting of Fe₃O₄ has better adhesive strength.

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