ADJUSTABLE NITROOXIDATION TECHNOLOGY FOR LOW ALLOY STEEL.

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Abstract: The article discusses the production of a surface diffusion nitride-oxide layer on low-alloy structural steel with specified structures and properties.

To develop a controlled technology for nitro-oxidation of low-alloy carbon steel, the dependences of the composition and structure of the nitried and oxide layer on the chemical composition of the steel and the technological parameters of the process were studied. Technical iron was used as a model alloy and samples of industrial steel grades 40X, P6M5 and steel 45 were processed. Saturation temperatures were studied in the ranges above and below the eutectoid temperature for the “Fe-N” system and, accordingly, for the “Fe-O” system and it was found that the best structures are at saturation temperatures below the eutectoid.

Keywords: structure, surface layer, nitriding, oxidation, nitrogen potential, saturating atmosphere, nitrooxide layer, X-ray diffraction analysis, Auger spectral analysis, combined technology

Introduction

The ability to predict the structure and properties of the nitried layer on the surface of structural and carbon steels is of great importance for optimizing the technological process of nitriding and subsequent processing of nitried products. The structure and properties of the combined nitriding process are determined by many factors, the main ones being temperature, saturation pressure and saturation potential of the atmosphere. By changing these factors, it is possible to obtain different compositions and structures of the nitried surface layer. Simultaneously with changing the composition and structure of the nitried layer, it is possible to regulate the physical-mechanical and physical-chemical properties of the treated metal surfaces.

Analysis And Metadology Of Antiquities

Intensification of the combined nitriding process can be achieved by increasing the saturation temperature, regulating the activity of the atmosphere, changing its composition and using various gas media. At the same time, using modern research methods, it is possible to obtain a large amount of information about the structure of nitride or nitride-oxide layers.

For a detailed scientific description of the structure of a diffusion nitride or nitride-oxide layer, it is necessary to study a large number of parameters. One of them is the composition of the saturating medium, which determines the saturation processes through the reactions of release of atomic nitrogen, oxygen and carbon.

Of all known saturating media, the most dynamic are gas atmospheres, which make it possible to obtain any structural sets and a different range of depths of diffusion layers. In all cases of gas nitriding, ammonia is the basis for creating a saturating atmosphere.

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**Discussion**

Under certain thermodynamic conditions (temperature, pressure), ammonia dissociates in accordance with the reaction:

\[ \text{NH}_3 \rightarrow \text{N} + \frac{3}{2} \text{H}_2 \]  

(1)

During the saturation process, a certain nitrogen potential is formed and during the process, an equilibrium of the nitrogen potential occurs. According to relation (1), the resulting atomic nitrogen diffuses into the metal surface forming metal nitrides MeN. On the surface of metals, higher metal nitrides first develop in the order of decreasing nitrogen concentration deeper into the surface of the saturated metal or alloy.

To estimate the ratio of the partial pressure of ammonia to hydrogen, you can use the equilibrium constants (K_a):

\[ K_a = \frac{P_{\text{NH}_3}^{3/2}}{P_{\text{H}_2}} \cdot \frac{1}{a_N} \]  

(2)

where: - partial pressure of ammonia; - partial pressure of hydrogen; aN - nitrogen activity or nitrogen potential.

At constant temperature and pressure, the equilibrium constant will be constant, so equation (2) can be written as follows: and -Fe, During nitriding, a solid solution of nitrogen is formed in - phases.

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\[ a_N^2 = \frac{P_{\text{NH}_3}}{P_{\text{H}_2}^{3/2}} \]  

(3)

To ensure diffusion of nitrogen into the material, it is necessary that the nitrogen potential of the saturating medium be greater than the nitrogen content in the metal:

\[ \alpha_N^\text{Fe} > \alpha_N^\text{Me} \]  

(4)

Equation (4) characterizes the initial stage of diffusion, since with the formation of the nitride layer (-phases), respectively, the value of the nitrogen potential must satisfy the condition (- and ):

\[ a_N^\text{Fe} > a_N^\gamma > a_N^\delta \]  

(5)

During short-term nitrooxidation processes, the nitriding process first occurs with the formation of a nitride layer. In all cases, the formation of the nitride layer and the internal nitriding zone in pure iron occurs in accordance with the “Fe-N” diagram.

When the nitride layer is oxidized, the ratio of partial pressures NH3:H2 changes. Denitrogenation of the nitride layer begins with the formation of a thin surface oxide layer, which, after obtaining a certain thickness, prevents denitrogenation and forces nitrogen diffusion deep
into the metal. As a result, during oxidation, the thickness of the low-nitrogen nitride layer and the internal nitriding zone increase.

To develop a controlled technology for conducting the process and the theoretical foundations of the technology for nitro-oxidation of low-alloy carbon 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. Technical iron was used as a model alloy. Industrial steels 40X, P6M5 and steel 45 were also studied.

After saturation above the eutectoid temperature for the “Fe-N” system, a nitride zone with clearly defined columnarity is observed in the nitrided layer. This zone consists of large crystals with wide boundaries between them and a large number of pores and cracks (Fig. 1, a and b).

This layer structure prevents the subsequent formation of a uniform oxide film. After nitriding at temperatures below the eutectoid (5910C), the layers have a smaller thickness, but a uniform structure (Fig. 2).

![Image 1](https://journal.silkroad-science.com/index.php/JAIDE)

**Fig. 1. Microstructure of the nitrided layer on steel 45 (a) and steel 40X (b)**  
**Treatment mode:** Nitriding - 620°C, 3 hours.

![Image 2](https://journal.silkroad-science.com/index.php/JAIDE)

**Fig. 2. Microstructure of the nitro-oxidized layer on steel 45.**  
**Treatment mode:** Nitriding - 580°C, 3 hours; oxidation - 550°C, a - 1 hour and b - 2.5 hours in water vapor with the addition of 5% HEDP.

When a small amount of complexones is added to the composition of an oxygen-containing saturating medium, they have a beneficial effect on the formation of a dense surface oxide layer and the denitrogenation process during the oxidation of the nitride layer is sharply
reduced (Fig. 2, b). The results of studying the influence of the degree of ammonia dissociation on the thickness and composition of the diffusion layer at below the eutectoid temperature (5800C) show that the formation of a developed zone of high-nitrogen nitride (ε-phase) with a significant total layer thickness is characteristic of processes with a degree of ammonia dissociation α≤30%, however at the same time, the porosity of the nitride layer increases (Fig. 3, lines 2 and 3).

The Result
When the degree of ammonia dissociation α>50%, the thickness of the nitride zone and the amount of ε-phase on the surface of the layer decrease (Fig. 3).

Rice. 3. Dependence of the intensity of phase lines on the degree of ammonia dissociation. Nitriding in ammonia at a temperature of 5800C for 3 hours. 1- γ-phase (200); 2 - ε-phase (110) and 3 - ε'-phase (101). When carrying out nitriding at the first stage of nitro-oxidation, the formation of a nitride layer with the best qualities for further oxidation is possible when the degree of ammonia dissociation is in the range of the degree of dissociation α = 30-50%. With an increase in the degree of ammonia dissociation, the reflection intensity of the high-nitrogen nitride of the ε-phase decreases, and the intensity of the low-nitrogen nitride of the γ'-phase increases, the porosity and columnar structure of the surface nitride layer disappears.

The formation of the ε'-phase occurs only during nitriding of steel and alloys in the presence of carbon in their matrix and has a carbonitride character. Therefore, noticeable formation of this type of nitride is typical only during nitriding of carbon steel and alloys.

When developing a combined technology of nitriding with subsequent oxidation of low-alloy carbon steels in water vapor, it is necessary to know the distribution of nitrogen, carbon and oxygen along the depth of the hardened layer. One of the research methods is Auger spectral analysis of elements in metals. Let's consider its capabilities using the example of gas nitriding of 40X steel followed by oxidation in water vapor.

Conclusion
Thus, Auger spectral analysis, together with other research methods, allows us to obtain a picture of the layer-by-layer distribution of elements in nitro-oxidized steels. When studying the temperature and time dependences of the formation of a strengthening layer, based on its results, it is
possible to regulate the combined technology of nitrooxidation for surface hardening of low-alloy carbon steels.

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