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Structural-phase transformations in the diffusion zone nitrooxidiation of alloy steels

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Structural-Phase Transformations in the Diffusion Zone Nitrooxidiation of Alloy Steels

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Abstract. Increasing the corrosion resistance and wear resistance of cutting tools used under conditions of dynamic wear and shock loads requires the formation on their surface of a high-strength diffusion zone with damping properties. In the process of oxidation of the nitrided layer, the creation of a barrier oxide film on its surface due to the dissociation of high-nitrogen nitrides deep into the matrix, as a result of which structural and phase changes occur in the diffusion zone. In the process of oxidation of the nitrided layer, the creation of a barrier oxide film on its surface due to the dissociation of high-nitrogen nitrides deep into the matrix, as a result of which structural and phase changes occur in the diffusion zone. The process of resorption of the nitride layer at the nitride-matrix interface due to volumetric and reaction diffusion is accompanied by the formation of a transition zone with intermediate low-nitrogen nitrides and an increase in the diffusion zone in the matrix. As a result of phase changes in the transition zone with low-nitrogen nitrides with the achievement of the maximum concentration of nitrogen solubility at the "nitride-matrix" boundary, stable and metastable phases of solid solutions, both base metal and alloying elements, are released in the diffusion zone. Corrosion tests and determination of the surface gradient properties of the composite layer have proposed compositions for surface hardening of cutting tools, consisting of combined layers "oxide-nitride-diffusion zone", providing a double damping effect of the high-strength diffusion zone.

Keywords: diffusion zone, nitride(compound) layer, transition zone, metastable phase, free energy, Guinier-Preston zone, phase transformation, coherence.

INTRODUCTION

In the process of nitrooxidation of steels, multilayer diffusion nitride-oxide coatings and a diffusion sublayer are formed on the surface - zone of internal nitriding (diffusion zone), each of which is important from the point of view of the service characteristics required for the products being hardened. At the same time, for surface hardening of parts operating under conditions of corrosion and friction at low contact loads, a controlled surface diffusion layer with a developed nitride zone with certain structural and phase states is created on the surface [1-4].

For parts made of high-alloy steels operating under conditions of dynamic wear and shock loads, it is desirable to form a developed high-strength internal nitriding zone without a surface nitride layer (compound layer). Under wear conditions at elevated specific pressures, it is necessary to create a surface nitride zone resting on a sublayer of nitrogenous martensite, or a high-strength complex alloyed diffusion zone [5-9].

There are wide possibilities for combining nitriding processes with other types of chemical-thermal treatment, which allows obtaining modified both surface compound layer and diffusion zones by achieving functional structural-phase states with predetermined properties [10-14].

The nitrooxidation process, which consists of the first stage of nitriding in ammonia and oxidation of the nitride layer in water vapor, allows the creation of a composite diffusion nitride-oxide zone on the surface of hardened parts made of structural steel [4,11, 15-17], as well as on alloy steels diffusion zone with or without a compound layer [18].

Structural and phase changes in the nitride layer also occur during oxidation of the compound layer, especially when creating a barrier oxide layer on the surface, subjecting the high-nitrogen nitride layer to dissociation with a transition to low-nitrogen nitrides and further transformation of the low-nitrogen layer to diffusion zone [18,19].

During the nitrooxidation of alloy steels, depending on the duration of oxidation, the diffusion zone continues to grow due to the dissociation of the surface compound zone, and at the same time, the processes occurring at the "nitride-matrix" boundary are important, as a result of which structural and phase changes occur with the formation diffusion zone with stable or metastable structural-phase states.

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METHODS

The nitrooxidation process was carried out by a combination of gas nitriding in an atmosphere of dissociated ammonia at the first stage at a temperature of 550 °C and without removing from the furnace at the second stage of the process, oxidation was carried out at the same temperature in vapors of a 5% aqueous solution of Trilon B. The material for the study was samples with a diameter of 10 mm and a thickness of 5 mm made of R6M5 steel, used in mass production, after hardening at a temperature of 1210-1230 °C and tempering in the temperature range 550-600 °C were used. Material composition: C=0.8-0.88%; Cr=3.8-4.4%; W=5.5-6.5%; Mo=5.0-5.5%; V=1.7-2.1%.

Phase X-ray diffraction analysis of the diffusion nitride and nitride-oxide layers was carried out using the X-ray diffraction method on the DRON-3 installation in iron and cobalt filtered Kα radiation. The phase composition was determined by comparing the obtained interplanar distances (d/p) with tabulated values (d/n) for nitride and oxide phases, taking into account the ratio of line intensities of the corresponding plane [20, 21].

Metallographic analysis of processed steel samples was carried out on transverse sections using a Neophot-21 light microscope in the magnification range of 500-1000.

Microhardness along the depth of the diffusion layer was measured using a PMT-3 microhardness tester. Microhardness was measured on polished sections in the direction perpendicular to the hardened surface, by pressing a diamond indenter onto the surface of the treated samples [22].

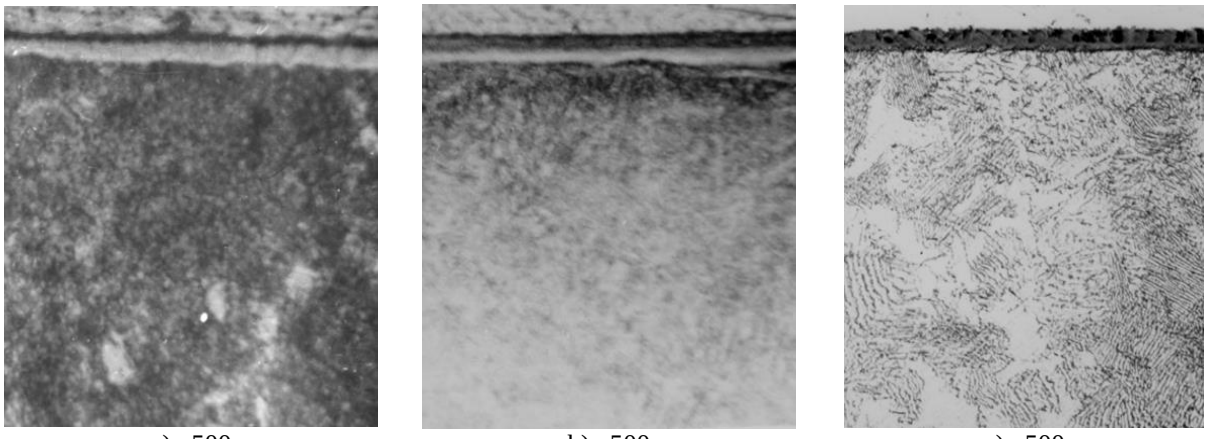
For comparative characterization of the corrosion properties of the coatings the method of exposure to neutral salt fog was used, the tests were carried out in the KTK-500 chamber, as an aggressive medium was used 3% NaCl solution in water.

RESULTS AND DISCUSSIONS

After the gas nitriding process by introducing into the atmosphere superheated steam of an aqueous solution of the Trilon B [23] at a lower eutectoid temperature for the “Fe-O” system (570 °C), a dense oxide layer is formed on the surface, consisting of almost one Fe_3O_4 magnetite with a certain thickness of the rolling layer, which prevents further deazotization of the nitride layer forcing the diffusion of nitrogen into the depth of the matrix.

The products of thermal decomposition of the Trilon B in aqueous solution at a temperature of 550 °C on the surface of a nitride layer with carbon in solid solution, resulting from decarbonization of the matrix during nitriding, leads to the creation of a heterogeneous stable environment “H_2-H_2O-CH_4-CO-C” [24] and provides carbon gasification with the formation of a dense oxide layer over the entire surface of the compound layer.

In the presence of a barrier layer, as a result of a decrease in nitrogen concentration due to volumetric and reactive diffusion, the high-nitrogen sublayer passes to low-nitrogen nitrides isomorphic to ε–nitride: carbon-doped matrix - $Fe_3(NC)$ (ε'-carbonitride) and further doped with oxygen - $Fe_3(NCO)$ (ε"-oxycarbonitride) (Fig. 1, a), and also passes to low–nitrogen nitride - Fe_4N (γ'-phase) (Fig. 1, b).



a) x500 b) x500 c) x500

Nitriding: temperature – 550 °C, time - 2 hours; oxidation: temperature – 550 °C;
time - a – 0.5 hours, b – 1.0 hours, b – 1.5 hours.

FIGURE 1. Microstructure of nitrooxidation steel samples P6M5.

During the oxidation process of the high-nitrogen nitride layer, low-nitrogen nitrides first grow in the layer and then the nitride sublayer resorbs with the formation of a mixture of (γ'+α) phases in the zone of internal nitriding with a surface oxide layer in the zone of internal nitriding (Fig. 1, c).

By forming a barrier oxide layer on the surface of the nitride layer, it becomes possible to regulate the concentration of nitrogen and, accordingly, the composition of the nitride sublayer, which is a source of nitrogen during the further formation of the internal nitriding zone during the oxidation process. Currently, the formation of an internal nitriding zone on alloy steels is achieved by a technology that provides a low nitrogen potential of the atmosphere at the level of the maximum solubility of nitrogen in the α-phase with its maximum mobility [9,10] or cyclic nitriding methods [25].

In the process of nitrooxidation, for the growth of the internal nitriding zone during the oxidation period, the nitrogen source is a nitride layer with high nitrogen contents and both volumetric and reactive diffusion in the nitride layer and at the nitride-matrix boundary is provided. During the oxidation of the nitride layer, the zone of internal nitriding increases along the depth of the matrix due to dissociation of the compound layer.

During nitrooxidation, depending on the technological modes of nitriding at the first stage and the creation of a barrier oxide layer on the surface of the nitride layer at the second stage of the process, it is possible to form various structural and phase states with a set of compositions of the nitride-oxide layer, as well as the "oxide- diffusion zone" (Fig. 1).

When the nitride layer is oxidized by the formation of a barrier oxide film on the surface, the nitride sublayer has a composition consisting of a mixture of ε (ε', ε") +γ' - phases, in the future, depending on the duration of exposure, it resolves as a result of transformations with the formation of diffusion zone (α-phase) (Fig. 2).

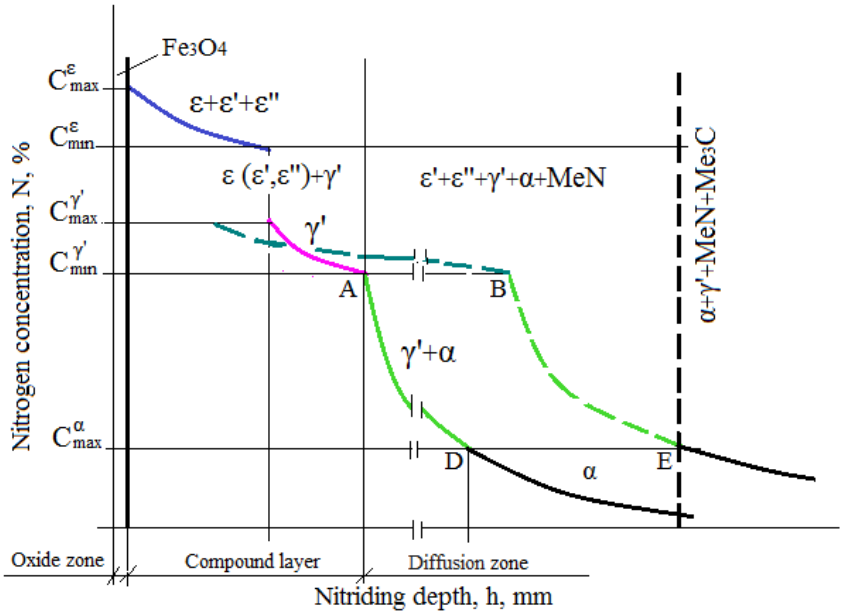


FIGURE 2. Schemes of structural-phase transformation in a diffusive nitrided layer during oxidation in water vapor.

At the first stage of nitrooxidation, the nitrided layer will consist of a nitride layer of sequentially arranged phases consisting of ε+ε'+MeN → γ', and the diffusion zone will have the structure: γ'+α+MeN → α+γ'+MeN+Me_3C.

During the oxidation process at the second stage, the low-nitrogen nitride layer has the composition of a mixture of phases: ε'+ε"+γ'+MeN, which during oxidation decomposes to form a transition zone consisting of α-phase secretions, low-nitrogen mixtures of nitrides (ε'+ε"+γ'), nitrides of alloying metals MeN.

During isothermal exposure during oxidation, the low-nitride phases in the transition zone disintegrate and after transformations, the structural-phase state of the internal nitriding zone has phase mixtures consisting of an α-phase with boundary secretions of the γ'-phase and nitrides MeN, as well as carbides Me_3C (Fig. 2).

In the process of oxidation with the transition of high-nitrogen nitrides to low-nitrogen ones, the boundary of the nitride layer moves from point A to point B (Fig. 2), such a boundary advance of the γ' phase causes a significant increase in the average nitrogen content in the transition zone. At a point In the transition zone (Fig. 2) at the "nitride-

matrix" boundary, the matrix α-phase is highly enriched in nitrogen, and at the same time, although the nitrogen diffusion coefficients in the ε- and γ' phases are significantly lower than in the α-phase, however, low nitrogen mobility in the ε- and γ' phases to a certain extent can block nitrogen diffusion in α-phase and therefore the formation of γ'-phase secretions near the boundary of the nitrogen-enriched α-phase is inevitable.

Structural and phase changes in the transition zone continue along the line (AD) until the line (BE) is reached, and at the same time, the condition of the transition zone advancement ensures the presence of low-nitrogen nitrides in the compound layer, which reaches their minimum at point E, corresponding to the maximum concentration of nitrogen in the α-phase (Fig. 2).

From a thermodynamic point of view, the phases in the transition zone are in a metastable state. Since the rate of nitrogen diffusion in the matrix is high, excess nitrides are released in micro volumes with a concentration in values of more than $C^{\alpha}_{\max}$ there are no alloying elements in the deep volumes of the diffusion layer. In general, the diffusion zone in alloyed steels, according to the activity of the alloying element Me, consists of a nitrogenous solid solution of the base metal (the diffusion zone of the I type) and nitrides of alloying elements (the diffusion zone of the II type) (Fig. 2).

The presence of oxygen in the composition of the ε"-phase in the diffusion zone, due to the metastability of the phase, passes into the composition of solid solutions of the α- and γ'-phases, and carbon in the enriched zones forms carbides of the base metal Fe_3C and alloying elements Me_3C in the diffusion zone

The phases in the internal nitriding zone can be in a stable state only under conditions when the nitrogen concentration in the transition zone is lower than, that is, beyond point E (Fig. 2), where, as a result of structural phase transformations, the formation of an internal nitriding zone (α-phase) with excessive emissions of the γ'-phase, alloying nitrides is achieved elements MeN and carbides Me_3C.

In the transition zone consisting of the α-phase and the release of the γ'-phase and the nitrides Me, the specific volumetric energy decreases, which will be equal to the difference in the specific volumetric free energies of the initial nitrogenous solid solutions and the equilibrium mixture of nitrides and a nitrogenous matrix. The decomposition of the nitrogenous solution is accompanied by an increase in free energy due to elastic deformation of the matrix and the formation of surface phase sections, and in this case the change in the free energy of the diffusion zone has the following form [26]:

$$\Delta F = -\Delta F_{vol} + \Delta F_{sur} + \Delta F_{el} \quad (1)$$

where ΔF_{vol} – the decrease in free energy during the formation of a new phase;
ΔF_{sur} – energy consumption for the formation of the interface during the separation of a new phase;
ΔF_{el} – the change in the elastic energy of the matrix during nitride release.

When obtaining diffusion layers with predetermined properties, special attention is paid to the use of phase transformations, both on the surface and on the matrix of steels and alloys. The properties of steels and alloys are closely related to their structure and composition, which are determined by the shape and relative position, as well as the degree of dispersion of heterophase structural-phase states that form after phase transformations.

In the case of obtaining modified surface diffusion layers, as well as in the zone of internal nitriding on steel and alloys, a heterophase state occurs, that is, the resulting mixture of phases differs from each other in composition and crystal structure. At the same time, establishing a connection between structure and properties is of great scientific and practical importance.

The deformation interaction of diffused atoms of insertion and substitution causes static distortions of the crystal lattice. In many cases, phase transformations in solid solutions can be considered as a result of the redistribution of atoms to the nodes of a certain crystal lattice with two possible cases:

- redistribution of atoms on scales commensurate with interatomic distances, leading to the appearance of an ordered phase;

- redistribution of atoms on scales significantly exceeding interatomic distances, accompanied by decay into two or more phases differing from each other in composition and free energy.

Due to the mismatch of the crystal lattices of the γ' phase and the released new α-phase at the "nitride-matrix" boundary, as well as the difference in the specific volumes of the initial solid solution (γ'-phase) and α-phase leads to the appearance of elastic deformation energy, hindering the formation of nitrides, creating a metastable phase state.

Metastable phases in the transition zone at the boundary with the matrix have a lower energy surface compared to stable phases and, accordingly, a lower value of the increase in ΔF_{sur} during the formation of the α-phase. A characteristic feature of the intermediate phases $Fe_{16}N_2$ (α"-phase) in the transition zone is the partial and sometimes complete coherence between the secretions and the matrix.

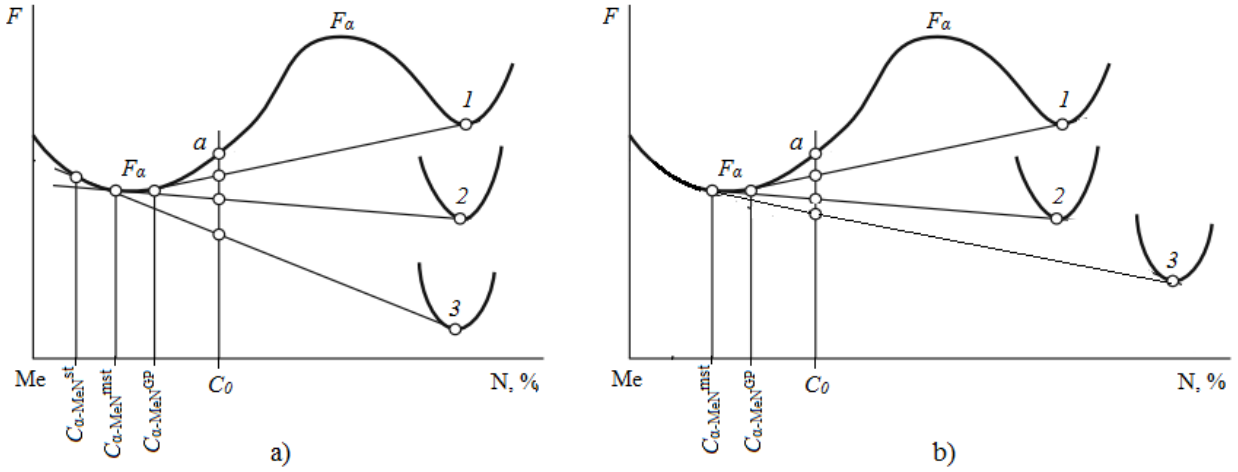
During nitriding, the formation of fully coherent Guinier-Preston zones (GP-zone), metastable phases with partial or complete coherence with the matrix, and stable phases is possible. In the transition zone, the appearance of GP-zones and intermediate nitride phases increases the differences in free energy and specific volumes of the matrix, as well as the formation of stable nitrides with crystallographic inconsistencies in the diffusion zone.

In the “nitride-matrix” diffusion layer, the GP-zone is generated homogeneously in a matrix solid solution with a high separation density, and metastable and stable phases with the same or different concentrations and free energies are generated heterogeneously at dislocations, boundaries, vacancy clusters and in the GP-zones [26].

Thermodynamically probable is the formation of secretions of different stability in the diffusion zone (Fig. 3). At the same time, the change in the free energy of the system as a function of nitrogen concentration in the diffusion zone forms metastable MeN^{mst}, stable MeN^{st}, GP- zones with the same concentrations, but having different free energies (Fig. 3, a).

The scheme of the change in free energy depending on the phase composition of the released new phases in the matrix can have different types. The change in the free energy of the GP-zone during decomposition with the release of stable and metastable nitrides may differ in concentration and the amount of free energy in the diffusion zone (Fig. 3, b).

In the schemes of dependence of the free energy of the internal nitriding zone on the nitrogen concentration, stable nitride has the minimum solubility in solid solution, and the maximum is the GP-zone.



a –separation with equal concentrations; b – separation with different concentrations and free energies.
1 - $C_{\alpha\text{-MeN}}^{GP}$; 2 - $C_{\alpha\text{-MeN}}^{mst}$; 3 - $C_{\alpha\text{-MeN}}^{st}$.

FIGURE 3. Scheme of dependence of the free energy in the diffusion zone on the nitrogen concentration.

With the emergence of a new phase, the surface energy at the interphase boundary of ΔF_{sur} and the energy of elastic deformation of the lattice ΔF_{el} increases and becomes greater than ΔF_{vol}. In this case, the formation of a metastable phase and GP-zones becomes thermodynamically preferable, for which the nucleation rate will be higher when separated from a supersaturated solid solution (Fig. 3, b).

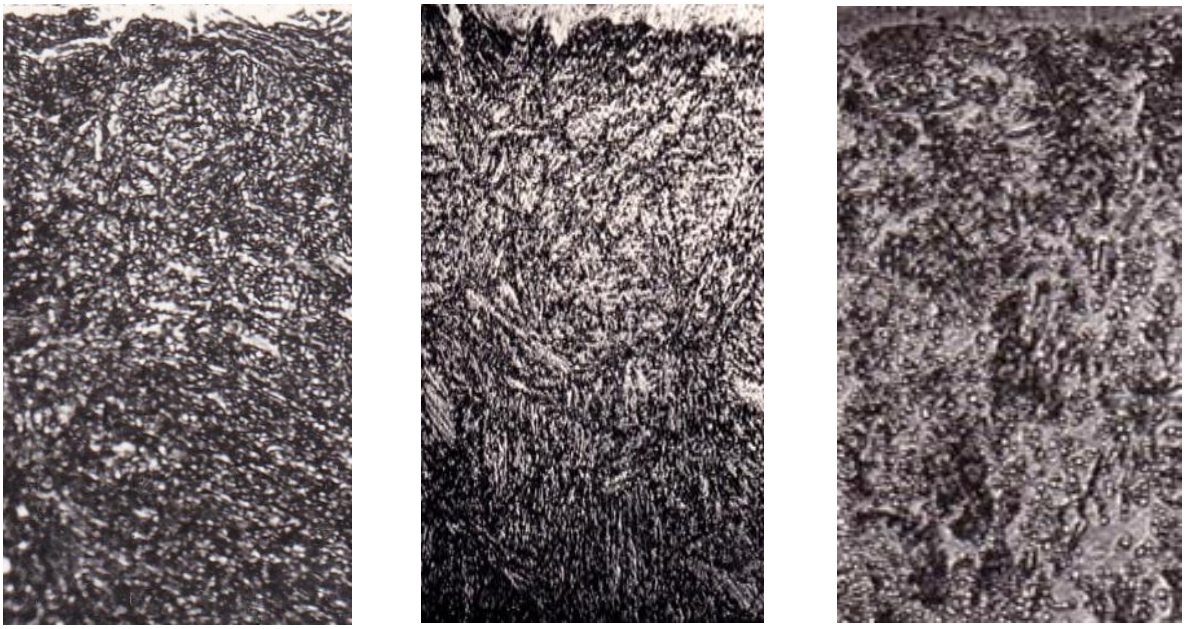
In a solid solution enriched with nitrogen at the “nitride-matrix” boundary, the formation of metastable phases up to point E is energetically advantageous (Fig. 3), the higher the super saturation of the solid solution with nitrogen relative to the stable phase, the more likely it is to appear intermediate metastable phases and GP-zones [26].

In general, during the oxidation of the nitride layer at the nitride-matrix boundary, as in the process of nitriding, the appearance of a GP-type zone continues, with an increase in the duration of oxidation, metastable nitrides with a higher free energy of critical embryo formation are formed, after which a stable nitride phase with a minimum nucleation rate is formed.

With the formation of metastable nitrides in the internal nitriding zone, the nitrogen concentration in the solid solution at the nitride boundary decreases and this concentration becomes less than the maximum limit nitrogen concentration in the GP-zone and a concentration gradient is established. Further diffusion of nitrogen in the matrix in the direction of a relatively metastable nitride makes the solution unsaturated and causes the dissolution of the previously formed GP-zones.

Fig. 4 shows the microstructures of the diffusion zone of the "nitride-matrix" diffusion layer in P6M5 steel with

the formation of diffusion zone after nitriding (Fig. 4, a) and after nitrooxidation with an oxidation duration of 0.5 hours (Fig. 4, b) and 1.5 hours (Fig. 4, c).



a) x1000 b) x1000 c) x1000

Nitriding: time - 2 hours, temperature - 550 °C; 1 – after nitriding; 2 – oxidation for 0.5 hours at 550 °C; 3 – oxidation for 1.5 hours at 550 °C.

FIGURE 4. Microstructures of the diffusion zone of the layer “nitride-matrix". Steel R6M5.

The structural transformation in the zone of internal nitriding during the nitriding of steel P6M5 occurs by the formation of disc-shaped zones on the matrix and the formation of several intermediate coherent phases, followed by coherence in the depth of the matrix with the formation of fully equilibrium secretions (Fig. 4, a).

During the oxidation of the nitride layer directly at the nitriding temperature, the region of existence of disc-shaped zones moves along the depth of the layer. In the matrix, supersaturated solutions are formed closer to the boundary of the nitride layer, even in the form of enlarged grains in the transition zone, which is characterized by the appearance of supersaturated metastable solid solutions (Fig.4, b).

With an increase in the oxidation time, more stable nitrides are isolated in the form of clusters with different, characteristic coherence of nitride coagulation, even large branched grain sizes. The formation of secretions of stable CrN phases in a uniform distribution can be seen over the entire surface of the internal nitriding zone in the form of a non-etching small sphere, characteristic of the process of spheroidization of nitrides of alloying elements. After nitrooxidation, the internal nitriding zone of P6M5 steel consists of nitrogen-doped ferrite and dispersed particles of nitrides of alloying elements, primarily chromium nitride CrN as a stable phase (Fig.4, b). In the zones of the presence of a solid solution of Fe_3N nitride, due to carbon diffusion, $Fe_3(NC)$ nitrocarbides are formed in the matrix, and stable $Fe_3(CN)$ carbonitrides are formed by the introduction of nitrogen into metastable Fe_3C carbides [27].

Alloying elements have different effects on structural and phase changes.

The alloying elements Mo and W in the composition of steel leads to the formation of clusters and subsequent ordering within clusters of atoms of substitution and introduction occurs by the mechanism of homogeneous decay. Initially, the Guinier-Preston zones consist of disc-shaped clusters of dissolved atoms located in the same places that they occupied in the initial solid solution. The process of growth and ordering occurs within clusters with the formation of one or more intermediate fully coherent phases of $Fe_{16}N_2$ (α"-phase), followed by a loss of coherence with equilibrium release in the cluster, since Mo and W expand the solubility of nitrogen in the α-phase and leads to an increase in cluster sizes.

The formation of equilibrium nitrides CrN and VN occurs without the formation of an intermediate metastable phase with the release of the limiting stoichiometric composition from the α-solution by spheroidization of nitrides, the higher their concentrations in the composition and the longer the oxidation time, the more small spheroid nitrides coherent to the α-matrix are formed (Fig. 4, c).

Comparative corrosion tests of P6M5 steel samples after nitriding and after nitrooxidation at a temperature of 550 °C, depending on the duration of oxidation, were carried out to obtain different ratios of phase compositions in the nitrided layer, the results of which are presented in Table 1.

TABLE 1. Corrosion properties of diffusion layer phase compositions on P6M5 steel samples in 3% NaCl solution. Nitriding: time - 2 hours, temperature - 550 °C.

Process	Oxidation time	Phase composition of the layer		Number of pores, 1/sm^2	The time of occurrence of corrosion foci, hour
		oxide	nitrided		
Nitriding	-	-	ε, ε′, γ′(30%), α	7	18
Nitrooxidation	1,0	Fe_3O_4	ε″, γ′(90%), α	non-porous	480
Nitrooxidation	1,5	Fe_3O_4	ε″, γ′(50%), α	non-porous	412

The results of comparative corrosion tests of P6M5 steel samples in a 3% NaCl solution showed that the most corrosion resistant zone is an internal nitriding zone with a non-porous surface oxide film of Fe_3O_4 with a nitride sublayer consisting of a mixture of (ε″+γ′)-phase with a greater proportion of γ′-phase in the composition (90%). The microhardness of the surface oxide layer is 4.5-5.0 GPa, the nitride layer is 8.5-8.9 GPa, the microhardness of the internal nitriding zone reaches up to 10.5 GPa. In general, the combined diffusion coating "oxide-nitride- diffusion zone" has non-porous and most corrosion-resistant properties and conditions for a positive property gradient are created on the surface, in which the diffusion zone has the highest hardness.

The structure of the internal nitriding zone depends on the mode of each stage of the nitrooxidation process, the nature of the alloying elements and their concentration. Depending on the nature of the alloying elements and their concentration, it is possible to isolate nitrides, both base metal and alloying elements, as well as their carbides Me_3C, carbonitrides $Me_3(CN)$ and nitrocarbides $Me_3(NC)$ both the base metal and alloying elements.

CONCLUSIONS

After nitrooxidation, the nitride oxide layer formed on the surface in any composition leads to an increase in corrosion resistance and reduces pitting formation on alloy steel products.

Cutting tools made of P6M5 steel are operated mainly under increased wear and shock loads, which require a high-strength diffusion zone consisting of a mixture of heterogeneous phases ($Fe_{\alpha[N]}$+γ'+MeN+Me_3C).

Nitrooxidation of high-speed steels makes it possible to obtain on the surface of a thin oxide layer with a stable structural and phase state with or without intermediate low-nitrogen nitride layers with an internal nitriding zone regulated in structure and composition. At the same time, an increase in the hardness of the tool is achieved and it is possible to avoid large stresses that cause an increase in the brittleness of the surface layer, as a result of which the durability of the tool increases by 2.5-3 times.

When forming an oxide zone and a low-nitrogen nitride sublayer from a mixture of (ε″+γ′)-phases on the surface of the diffusion layer, it provides a positive hardness gradient of the internal nitriding zone with a double damping effect under wear conditions.

Regulation of the phase composition of the layer by varying the parameters of each stage of the nitrooxidation process allows us to implement optimal technological schemes for obtaining the required physical and mechanical properties of steel to obtain the necessary composition of nitrides and carbides, both the base metal and the alloying element, which determines the predetermined properties of the hardened layer, taking into account their specific operating conditions.

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