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# **Change of Phase Structure in a Nitride Layer on Steel after Vapor-Oxidation**

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**ABSTRACT.** In the article receiving surface, the diffusion nitride-oxide layer on low-alloy structural steel with specified structures and properties.

It is established that changing the composition and structure nitrid-oxid layer depends on many factors such as the saturation temperature, the activity of a saturating atmosphere and modes of nitriding in the ammonia at the first stage of processing and oxidation in water steam at the second stage of processing.

In the process of nitrooxidation first is the process of nitriding with the formation of the nitride layer, the nitride layer and formation of zones of internal nitriding in pure iron occurs according to the diagram "Fe-N".

When oxidation of nitride layer begins partial dinitriding on the surface to form a thin surface oxide layer after reaching certain thickness prevents dinitriding and makes the diffusion of nitrogen in the depth of the metal.

**KEYWORDS.** Structure, phase structure, a diffusive layer, nitriding, oxygenating, nitric potential, the sating atmosphere, nitride - an oxidic layer, denitriding, the X-ray diffraction analysis, the ozhe-spectral analysis, corrosion resistance.

## **I. INTRODUCTION**

Nitriding - process of universal purpose by means of which metals and alloys are strengthened constructional, tool, corrosion-resistant and heat resisting became, alloys of refractory metals, the baked ceramic-metal alloys, various electroplated and diffusive coatings.

The combination of process of gas nitriding in the environment of ammonia with the subsequent oxygenating in vapors of water (nitrooxygenating) is that at the first stage of saturation carry out nitriding and at the second stage oxidizes a nitride layer in water vapors with receiving on a surface diffusive the coverings consisting of nitride, the carbonitride and the oxycarbonitride of phases [1].

Specific conditions of operation demand creation of an adjustable diffusive layer with development of these or those phase and structural components which define operability of products in an operating mode of wear, corrosion, a sign-variable bend, creep resistance at high temperatures, etc.

In the operational plan each diffusive zone of a layer bears itself certain office functions. Generally, the details working very hard at small contact loadings in the corrosion environment, require the nitrated layer with the developed nitride zone on which also corrosion resistance depends extra earnings of the rubbing surfaces. For the details working in the conditions of fatigue at the increased temperatures, the nitrated layer with the developed diffusive under layer – a zone of internal nitriding is necessary [2].

## **II. SIGNIFICANCE OF THE SYSTEM**

In the article receiving surface, the diffusion nitride-oxide layer on low-alloy structural steel with specified structures and properties. The study of literature survey is presented in section III, methodology is explained in section IV, section V covers the experimental results of the study, and section VI discusses the future study and conclusion

## **III. LITERATURE SURVEY**

When nitrooxygenating for obtaining the required effective thickness of a diffusive superficial nitride layer, nitriding is carried out at a temperature of 500-580°C with endurance of 3-5 h [1]. After such processing of a surface

the thin layer high-nitrogenous is formed (9-10% N)  $\epsilon$ -,  $\epsilon+\gamma'$ -,  $\gamma'$ -phases. Under a high-nitrogenous  $\epsilon$ -phase is located mixes ( $\epsilon \gamma'$ ) - phases and follows it thin  $\gamma'$  - a phase. Boundary advance  $\gamma'$ -phases causes substantial increase of average content of nitrogen in this part of a layer. Further a high-nitrogenous nitride zones the zone of internal nitriding in which the matrix  $\alpha$ -phase is strongly enriched with nitrogen follows. In the course of nitriding the low mobility of nitrogen in a nitride layer to some extent blocks diffusion in a zone of internal nitriding.

#### IV. METHODOLOGY

Carrying out process of gas nitriding in partially dissociated ammonia at temperatures closer than eutectoidny temperature for the "Fe-N" system accelerates process of nitriding and reduces nitrogen content in a  $\epsilon$ -phase due to its intensive migration in depth of a layer [3]. It reduces fragility of a nitride layer, allows to receive a pore-free  $\epsilon$ -phase and with other things being equal the big effective thickness determined by distance from a surface to a layer with a hardness not less than 500 HV. At the same time superficial hardness decreases a little, however does not decrease physic-mechanical properties of the blanket. Concentration of nitrogen in  $\epsilon$ -and  $\gamma'$ -phases at this temperature and phase structure of a diffusive layer is defined by the nitric potential of the atmosphere.

The intensification of the combined process of nitrooxygenating can be reached by saturation temperature increase, regulation of activity of the atmosphere, change of its structure and application of various gas environments. At the same time application of modern methods of a research it is possible to obtain a large number of information on the building nitride or nitride - oxidic layers. Detailed studying of the building diffusive nitride or nitride - the oxidic layer received after a combination of processes requires studying of a large number of parameters. One of them is temperature of saturation of a surface nitrogen when nitriding. It is known that diffusion coefficient in structure of the sating environment which defines processes of saturation through reactions of release of atomic nitrogen, oxygen and carbon.

From all known sating environments the most dynamic are the gas atmospheres which allow to receive any structural sets and various range of depths of diffusive layers. In all cases of gas nitriding ammonia is base for creation of the sating atmosphere. Under certain thermodynamic conditions (temperatures, pressure) ammonia dissociates according to reaction:



In the course of saturation, a certain nitric potential is formed and during process there is a balance of nitric potential. The relation (1) formed atomic nitrogen agrees diffuses in the surface of metal forming nitrides of MEN metals. On the surface of metals the highest nitrides of metals as it should be decrease in concentration of nitrogen deep into of the surface of saturable metal or alloy develops in the beginning. Nitric potential or the sating ability of the atmosphere is characterized by the relation of partial pressure of  $\text{NH}_3$  and  $\text{H}_2$  gases:

$$\pi_N = \frac{P_{\text{NH}_3}}{P_{\text{H}_2}^{3/2}} \tag{2}$$

where:  $P_{\text{NH}_3}$  - partial pressure of ammonia;  $P_{\text{H}_2}^{3/2}$  - partial pressure of hydrogen;

For ensuring diffusion of nitrogen in material, it is necessary that the nitric potential of the sating environment was more than nitrogen content in metal:

$$a_N^{FC} > a_N^{Me} \tag{3}$$

The equation (3) characterizes the initial stage of diffusion, on how many with formation of a nitride layer ( $\gamma'$ - and  $\epsilon$ - phase) respectively the size of nitric potential has to satisfy a condition:

$$a_N^{FC} > a_N^{\gamma'} > a_N^{\epsilon} \tag{4}$$

At short-term nitrooxygenating at first there is a process nitriding to formation of a nitride layer. In all cases formation of a nitride layer and zone of internal nitriding in pure iron happens according to the chart "Fe-N".

When oxygenating a nitride layer relation of partial pressure of  $\text{NH}_3$ ;  $\text{H}_2$  changes. Begins denitriding of a nitride layer with formation of a thin superficial oxidic layer which after obtaining a certain thickness interferes with denitriding and compels diffusions of nitrogen in the depth of metal. As a result when oxygenating thickness of a low-nitrogenous nitride layer and a zone of internal nitriding grows [1].

Formation diffusive nitride - an oxidic layer of optimum phase structure and the building can be predicted on the basis of the thermodynamic analysis. The corresponding settlement charts for the choice of the modes of processing are executed for processes of liquid and gas oxynitriding [4].

For gas processes the phase structure of the formed diffusive layer is predicted depending on composition of the atmosphere and temperature of process when calculating nitric and oxygen potentials of the atmosphere. In particular, when nitrooxygenating in ammonia with oxygen additives the conditional nitric potential of the atmosphere:

$$\pi_N = \frac{\lambda \cdot (1 - a)[0,5(1 + \lambda) + \alpha\lambda]^{1/2}}{[3/2\alpha\lambda - 0,5(1 - \lambda)]} \quad (4)$$

where:  $\alpha$  - extent of dissociation of ammonia;  $\lambda$  - an ammonia share in mix with oxygen.

In compliance with the equation (5) phase balances in the “Fe–N–O” system when oxynitriding in ammonia with oxygen additives, with increase in extent of dissociation of ammonia and temperature of process the oxidizing ability of the atmosphere decreases. Introduction of oxygen should not cause oxidation of the processed details at a nitriding stage. As appears from the generalized chart provided in work [1], at extent of dissociation of ammonia  $\alpha=30\%$  and content of oxygen of-5% oxide on a surface of details is not formed, and  $\pi_N$  and, therefore, the speed of process raise. At increase in oxygen concentration up to 10% formation of  $Fe_3O_4$  oxide at 500 is possible – 550°C and  $\alpha = 45-50\%$  [5].

Changing parameters of the atmosphere it is possible to regulate the building and phase structure of a layer, changing a ratio of nitride (carbonitride) phases in a nitride zone and forming an oxidic film of  $Fe_3O_4$  at a final stage of process.

Potential of the atmosphere is thermodynamic size and indicates a possibility of formation of a layer of the set phase structure and a certain concentration of the sating elements. However, the potential of the atmosphere does not give an assessment to the speed of formation of a layer. The assessment of speed of process can be carried out on activity of the atmosphere used for chemical heat treatment.

The activity of the atmosphere (Joint stock company) is defined by two factors: thermodynamic and kinetic. The thermodynamic factor is analyzed in the form of the difference of the current potential of the atmosphere ( $\pi_N$ ) and equilibrium ( $\pi_N^0$ ) for this phase or concentration of nitrogen. The more potential difference  $\pi_N - \pi_N^0$ , the more energy of Gibbs of reaction of interaction of iron with ammonia.

The kinetic factor is defined by a constant of speed of reaction of disintegration of ammonia (K), time ( $\tau$ ) and thinner gas presence, in particular, of oxygen containing gas ( $\alpha$ ).

Proceeding from dependence of activity of the atmosphere on thermodynamic and kinetic factors, the activity of the atmosphere is considered as the work of probabilities of thermodynamic and kinetic factors:

$$A_k = F \frac{(\beta - a)}{(1 - a)} \cdot (\pi_N - \pi_N^0) \quad (6)$$

The analysis of the equation (6) shows that the dependence of activity of the atmosphere on process parameters of process has extreme character.

On the basis of the equation (6) the activity of the atmosphere of ammonia in mix with air was calculated: estimated dependences of activity of the atmosphere of ammonia in mixes with air and experimental dependences of thickness of the diffusive zone received on steel 38H2MYuA at 570°C at various concentration of air in mix with ammonia are also received 1 hour almost full coincidence of extrema of activity of estimated composition of the atmosphere and thickness of a diffusive layer [5]. The maximum acceleration of growth of a diffusive layer is observed at 70% of air in mix with ammonia.

At nitrooxygenating process implementation under production conditions measurement of partial pressure in the furnace is very difficult. In this regard nitric potential is usually presented in the form of the equation which enter composition of the atmosphere on an entrance to the furnace, extent of dissociation of ammonia and pressure to furnaces [6].

$$\pi_N = \frac{\lambda_{NH_3} \cdot (1 - \alpha_\delta)(1 + \alpha\lambda_{NH_3})^{-1/2}}{(1,5\lambda_{NH_3}\alpha_\delta + \lambda_{H_2})^{3/2}} \cdot P^{-1/2} \quad (7)$$

where:  $\lambda_{NH_3}$  and  $\lambda_{H_2}$  volume fraction of  $NH_3$  and  $H_2$  in the atmosphere;  $\alpha_\delta$  - extent of dissociation of  $NH_3$  in the furnace; P – pressure in the furnace.

When nitriding in pure ammonia with increase in the gas atmosphere of hydrogen extent of dissociation  $\alpha_0$  decreases. Equilibrium nitric potential  $\pi^i_N$  for  $i$ -th phase is function of content of nitrogen on a surface and is equal to the nitric potential of the gas atmosphere which is in balance on nitrogen with the surface of metal.

Yu.M.Lakhtin for the first time received dependences of equilibrium potential on nitrogen content in  $\alpha$ - and  $\varepsilon$ -phases when nitriding in dissociated ammonia in production furnaces, and temperature dependences of concentration of nitrogen in a zone of internal nitriding ( $\alpha$ -phase) and  $\varepsilon$ -phases proceeding from the minimum solubility of nitrogen in a  $\varepsilon$ -phase of the nitrated layer are established [7].

## V. EXPERIMENTAL RESULTS

According to the theory of nitriding according to Yu.M.Lakhtin, regulation of phase structure of the nitrated layer is reached by change of nitric potential of the sating atmosphere. Supporting nitric potential at the level of solubility of nitrogen in this or that phase, it is possible to receive the layer consisting  $\alpha$ -solid solution, nitrides of iron and the alloying elements, to form on a surface  $\gamma'$ -phases (low nitride) or  $\varepsilon$ - a phase with the low content of nitrogen. It is recommended to regulate nitric potential dilution of ammonia with in whole or in part dissociated ammonia, oxygen, air, carbon-bearing gases, etc.

For development of production adjustable technology of conducting process and theoretical fundamentals of technology of the combined process of nitriding with the subsequent oxygenating in water vapors for superficial hardening of the low-alloyed carbon steel studied dependences of structure and the structure of the nitrated and oxidic layer on the chemical composition staly and process parameters of process. Investigated industrial steel 40X, and as model alloy used technical iron.

In Fig. 1 it is shown to dependence of change of thickness of a superficial diffusive layer depending on change of nitric potential of the sating environment at a temperature of 580°C Duration of process of saturation in all cases was 3 hours. Gas saturation was carried out with change of nitric potential in pure ammonia and addition of partially dissociated ammonia, with establishment of necessary nitric potential in the oven atmosphere.

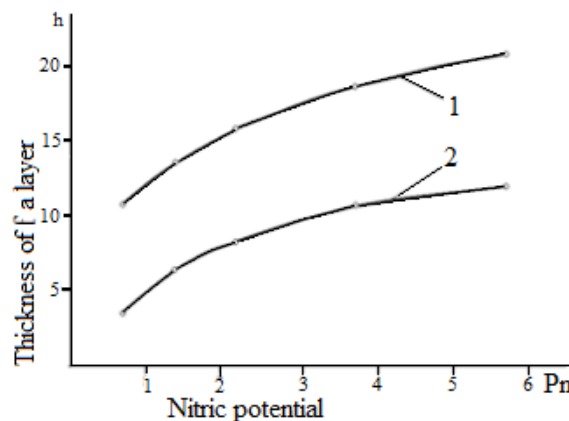


Fig. 1. Change of the general thickness of a nitride layer depending on the nitric potential of the atmosphere when nitriding in ammonia. Temperature of saturation 580°C, duration of process is 3 hours. 1 – general thickness of a nitride layer; 2 – thickness high-nitrogenous  $\varepsilon$ - phases.

From the obtained data it is visible that at increase in nitric potential thickness of the received diffusive nitride layer increases. Under the same received conditions samples investigated the X-ray diffraction analysis. Results of the X-ray diffraction analysis are given in Fig. 2.

The analysis of change of diffraction maxima of reflection of phases shows that with increase in nitric potential in a nitride layer the share  $\varepsilon$ - phases increases, and low-nitrogenous  $\gamma'$ - phases and  $\alpha$ -phases (a zone of internal nitriding) decreases.

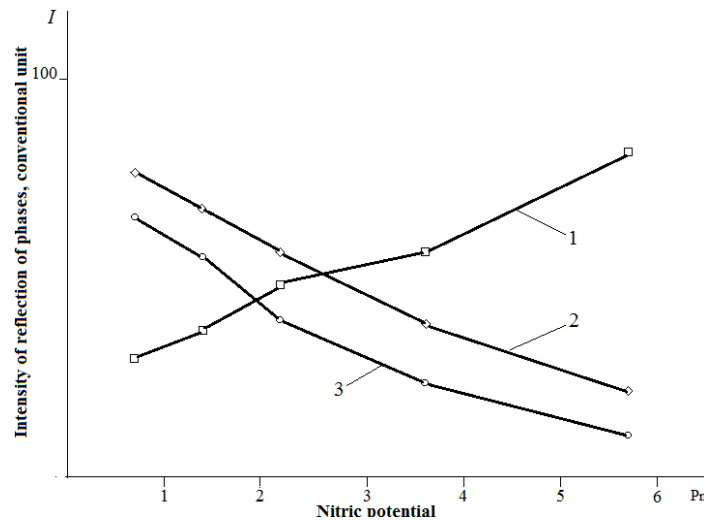


Fig. 2. Change of diffraction maxima depending on the nitric potential of the atmosphere when nitriding in ammonia. Temperature of saturation 580°C, duration of nitriding is 3 hours. 1 - ε - a phase; γ' - a phase; 3 - α-phase.

Optimum on phase structure the nitride layer can be received at values of nitric potential:  $2 < \pi_N < 3$ . At increase in nitric potential  $\pi_N > 1$  well developed nitride layer consisting from ε-and ε+γ' - phases is provided on a surface, with increase in nitric potential to  $\pi_N < 6$  the share of γ' - phases in a nitride layer decreases, and ε-phases increases. This circumstance though is followed with increase in thickness of a nitride layer, brings to an obstacle of diffusion of nitrogen through a nitride layer if necessary increase in duration of nitriding and also with increase in nitric potential  $\pi_N > 5$  for the account increase the share of a high-nitrogenous nitride layer is found fragility of a layer and porosity of a ε-phase.

When nitroxygenating at the first stage for reduction of duration of process of nitriding, economy of ammonia and decrease in content of nitrogen in a ε-phase which increases density of a nitride layer and reduces its fragility, it is possible to suggest to change nitric potential in the course of nitriding. In this case at the beginning of process the high nitric potential of the atmosphere providing on a surface of a high-nitrogenous nitride layer (the active period of saturation) is supported. Then the potential of the atmosphere decreases to some value by increase in extent of dissociation by decrease in volume of a stream of supply of ammonia in the furnace to a minimum, preliminary dissociation of ammonia or shutdown of its giving in the furnace. At implementation of process of nitriding on such mode, in the initial active stage of saturation the considerable concentration gradient on a diffusive nitride layer arises nitrogen that creates optimal conditions both for course of diffusive process, and for absorption.

For the purpose of receiving a pore-free low-nitrogenous diffusive nitride layer for further oxygenating it is desirable to receive the low-nitrogenous nitride layer consisting of a low-nitrogenous ε-phase or mix (ε+γ') - phases. At saturation by surface nitrogen in the active period the received layer having high concentration of nitrogen at the second stage of saturation in the conditions of the lowered nitric potential before achievement of balance about two directions can dissociated: process of diffusion of nitrogen of a nitride layer in depth of metal and returned back to the external environment (process of superficial denitriding).

In process of diffusion of nitrogen of a nitride layer in depth of metal the atmosphere of the furnace has no direct impact, in this case the defining factors is temperature and composition of the processed material.

Process of return of atomic nitrogen of a nitride layer back to the external environment (in the atmosphere of the furnace) can be stopped establishment in the oven atmosphere creation by a necessary condition balance of nitric potential with decrease in volume of supply of ammonia in the furnace or creation of a barrier blanket the denitridings interfering process.

Due to the above the structure of the sating atmosphere and the diffusive period can be changed over a wide range which is defined only by conditions of maintenance of high concentration of nitrogen in a nitride layer that does not demand a large amount of active atoms in the sating atmosphere.

The layer-by-layer spectral auger analysis studied distribution of nitrogen in a nitride layer after nitriding at ammonia dissociation  $\alpha = 30-45\%$  at the first stage (active saturation) and the subsequent excerpts in dissociated ammonia and also without supply of ammonia in the furnace which results are given in Fig. 3. In all cases process of nitriding at active saturation by nitrogen in the atmosphere of ammonia was carried out at a temperature of  $580^{\circ}\text{C}$  lasting 3 hours.

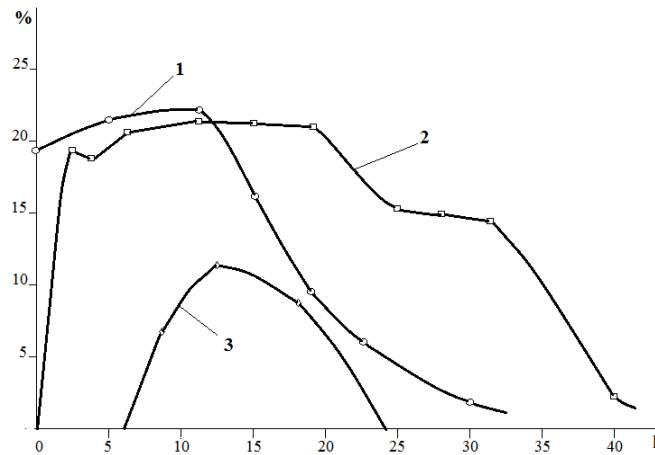


Fig. 3. Distribution of nitrogen in a nitride layer after process of gas nitriding in ammonia. 1 – nitriding  $\alpha=30-45\%$ ; 2 – nitriding with the subsequent endurance in dissociated ammonia at  $\alpha=70-85\%$  within 1 hour; 3 – nitriding with the subsequent endurance without supply of ammonia at a temperature of  $550^{\circ}\text{C}$  within 2.5 hours.

After carrying out nitriding at high nitric potential it is possible to receive high concentration of nitrogen in a nitride layer and at the following stage it is possible to dissociate a high-nitrogenous nitride layer with receiving a low-nitrogenous diffusive layer. From Fig. 3 it is visible that carrying out process at the subsequent stage of nitriding in dissociated ammonia allows to receive the developed low-nitrogenous nitride layer (Fig. 3, line 2) with big thickness of a nitride layer. At increase in duration at the second stage of process as a result of dissociation of a nitride layer in two directions of diffusion, concentration of nitrogen decreases and respectively thickness of a nitride layer decreases. This circumstance brightly is expressed at implementation of the second stage of process at a temperature of  $550^{\circ}\text{C}$  within 2.5 hours (Fig. 3, line 3). Therefore, when carrying out process of step nitriding on nitric potential, it is necessary to set the optimum modes of process for concrete material, with definition time of achievement of an equilibrium condition of a low-nitrogenous layer with obtaining effective thickness.

Decrease in concentration of nitrogen on a surface in a  $\epsilon$ -phase due to denitriding and migration of nitrogen deep into of material in process to diffusive endurance probably causes on a surface the return phase transformation with transition of a  $\epsilon$ -phase at first in mix  $(\epsilon+\gamma')$  - phases, further increase a share  $\gamma'$  - phases as a part of mix of phases and in border of a nitride layer transformation  $\gamma'$ -phases in  $(\gamma'+\alpha)$  a phase. Thickness of a nitride layer of endurance increases in the beginning because of diffusion of a concentration maximum of nitrogen in a nitride layer deep into of material, and from the beginning of transformation  $\gamma' \rightarrow (\gamma'+\alpha)$  begins reduction of thickness of a nitride layer.

Were studied by us corrosion resistance of mix  $(\epsilon+\gamma')$  - the phases received by nitriding at ammonia dissociation  $\alpha=30-45\%$  at the first stage of process and further endurance in the atmosphere of water vapor with receiving various ratios  $\epsilon$ -and  $\gamma'$ -phases.

Comparative test for corrosion resistance of the processed samples, in the environment of 3% of the number of NaCl. In the beginning (till 400 o'clock endurance) survey of samples were carried out daily, then once in three days. At the same time time of emergence of the first centers of corrosion of base metal and the area of sites of corrosion in percentage terms to all were fixed the estimated (Tab 1) Square.

Table 1  
Corrosion resistance of the nitro oxidized layers after various modes of processing in the environment of 3% of the number of NaCl

Processing conditions				Phase structure of a layer	Time of emergence of the first centers of corrosion, hour	Area of corrosion/ Test duration, (Sk/So)/hour		
Nitriding		Oxygenating					Sating atmosphere	
T <sub>а3</sub> , °C	τ <sub>а3</sub> , h	T <sub>ок</sub> , °C	τ <sub>ок</sub> , h	Nitriding	Oxygenating			
-	-	550	0,5	-	Couples waters	α, Fe <sub>3</sub> O <sub>4</sub> , Fe <sub>2</sub> O <sub>3</sub>	6	90/144
550	0,5	550	0,5	NH <sub>3</sub> +(50%N <sub>2</sub> +H <sub>2</sub> )	Couples waters	3BA+Fe <sub>3</sub> O <sub>4</sub>	18	15/144
580	3	550	1,0	NH <sub>3</sub> +(25%NH <sub>3</sub> +N <sub>2</sub> +H <sub>2</sub> )	Couples waters	ε',ε'',γ'(90%), Fe <sub>3</sub> O <sub>4</sub>	288...360	30/408
580	3	550	1,0	NH <sub>3</sub> ,α=45-65%	Couples waters	ε',ε'',γ'(50%), Fe <sub>3</sub> O <sub>4</sub>	412...480	15/912
580	3	550	1,0	NH <sub>3</sub> , α=45-65%	Couples waters	ε',ε'',γ'(75%), Fe <sub>3</sub> O <sub>4</sub>	912-960	5/1056
580	3	550	2,5	NH <sub>3</sub> , α=45-65%	Couples waters	ε',ε''-γ'(90%), Fe <sub>3</sub> O <sub>4</sub>	288...360	30/408

The most corrosion properties the nitride layer consisting of the mix ε'-,ε'', γ'(75%) with a superficial oxidic layer consisting of Fe<sub>3</sub>O<sub>4</sub> has. It claims that when oxygenating a nitride layer growth of a nitride underlayer with formation of the lowest nitride ε'-, ε''- and γ'- phases at the expense of high-nitrogenous ε- phase, received when nitriding at the first stage of nitriding is important.

It is supposed what diffusive processes in a nitride layer when receiving an oxidic layer occurs mainly on borders of γ'- phase grains. At this γ'-phase in a nitride layer will ε'-, ε''- by phases in a mesh look in the cross section of an oxy carbonate layer are captured. Dissociation of high-nitrogenous ε phase comes partial allocation of γ'- phase and formation of low-nitrogenousε'- phase at the expense of carbon from a matrix and ε'' phase from carbon of a matrix and oxygen from sating the atmospheres.

The necessary structure and phase structure nitride - an oxidic layer is reached at the optimum modes of processing, with receiving low-nitrogenous ε' phase (Fe<sub>2,3</sub>(NC)), ε''-of a phase (Fe<sub>2,3</sub> (NCO)) and γ' phase (Fe<sub>4</sub>N) of a certain ratio 25-30 microns thick and blanket of Fe<sub>3</sub>O<sub>4</sub> oxide 1-3 microns thick.

At the same time exact coordination of parameters of process of oxygenating at the second stage of processing with nitriding parameters at the first stage is necessary. Because the growth rate of an oxidic layer on a surface of a nitride layer has to be less or equally to denitriding speed at the second stage. At their not coordination can be formed intermediate Feα-φαα which leads to superficial cracking or peeling of an oxidic layer.

The dense oxidic bed during oxygenating provides aequally in dispersion of a nitride layer and by that by establishment of time of oxygenating it is reached the necessary ratios of low-nitrogenous phases in a nitride layer possessing together superficial oxidic layer necessary physical and chemical and physic-mechanical properties.

## VI. CONCLUSION AND FUTURE WORK

As a result of conducted analyses and researches it is possible to conclude that from the point of view of industrial application of low-temperature gas nitriding with the subsequent vapor-oxidationis necessary further researches on regulation of phase structure of a nitride layer with change of nitric potential at the first stage of nitriding. At the same time with receiving necessary mix of phases of a nitride layer in structure of a nitride layer it is possible to receive nitride-oxy a layer with predetermined physical and chemical or physic-mechanical properties.



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