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RESULTS OF APPROBATION OF THE INNOVATIVE METHOD OF ION NITRIDING FOR STEELS WITH LOW TEMPERATURES OF TEMPERING

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Апробований інноваційний спосіб комплексної обробки для сталей з низькою температурою відпустку, який включає попереднє азотування в вакуумному газовому розряді перед загартуванням і низьким відпуском. В цьому випадку при азотуванні температура нагріву мало впливає на процес високотемпературної обробки, а процес азотування значно прискорюється (оскільки атоми азоту легше проникають в незагартовану сталь). Після остаточної термообробки це призводить до збільшення до 2000 мкм глибини проникнення атомів азоту і товщини формування області з підвищеною твердістю

Ключові слова: іонне азотування, комплексна обробка, дифузія, глибина впливу, дифракційні спектри

Апробирован инновационный способ комплексной обработки для сталей с низкой температурой отпуска, который включает предварительное азотирование в вакуумном газовом разряде перед закалкой и низким отпуском. В этом случае при азотировании температура нагрева мало влияет на процесс высокотемпературной обработки, а процесс азотирования значительно ускоряется (поскольку атомы азота легче проникают в незакаленную сталь). Это приводит после окончательной термообработки к увеличению до 2000 мкм глубины проникновения атомов азота и толщины формирования области с повышенной твердостью

Ключевые слова: ионное азотирование, комплексная обработка, диффузия, глубина воздействия, дифракционные спектры

1. Introduction

Practically all criteria of working ability are determined by the properties of surface layers – they in particular must be improved first of all [1, 2]. In this case, high surface hardness in the presence of large tensile stresses leads to brittle failure and crumbling, thus enhancing abrasive wear [3], and large compressive stresses – to plastic deformation of the base and its flow [4].

It was established [5] that in the case of surface modification by applying coatings, the destruction of the system coating-base begins essentially from the plastic deformation of substrate layer near the interface boundary [6]. In order to improve functional properties, they use in such cases creation of special phase [7] or structural [8] states, composite materials [9] and strengthening of boundaries through the distribution of "useful additives" [10]. Although this ensures, in a number of cases, improvement in the operational characteristics, it has, however, low efficiency at work under conditions of multicycle impacts and high loads.

The modification of structure and surface properties by nitriding makes it possible to considerably improve their hardness, wear resistance, thermal stability and other functional characteristics, including at operation under conditions of multicycle impacts and large loads. That is why studies directed to the development of innovation technologies of nitriding are relevant and required by industry.

2. Literature review and problem statement

Depending on the operating pressure, they use at nitriding: high-vacuum technologies (for the high-voltage sources of gas ions [11]), low-vacuum conditions (for the nitriding in the glow discharge [12]) and technologies of nitriding under atmospheric pressure (“furnace nitriding” [13]).

The most widely used in industry at present is the method of “furnace nitriding” of steels with the application of ammonia at temperatures from 500 to 600 °C. The use of this technology makes it possible to attain a thickness of the nitrided layer in the range of 0.2–0.6 mm over a period from 10 to 90 the hours of treatment [14]. The application of catalytic nitriding employing special catalysts for the ammonia disassociation makes it possible to decrease duration of treatment in furnace to two times [15].

To considerably increase intensity of the process of diffusion saturation of the surface with nitrogen is possible through ionic nitriding with the use of the low-temperature plasma [16]. In this case, energy of the diffusion activation is greatly reduced (in comparison with the thermal “furnace nitriding” [17]), while a diffusion coefficient increases [18].

Ionic nitriding in the gas plasma of vacuum-arc installations at temperatures from 500 to 600 °C makes it possible not to reduce duration of nitriding to 1 hour only. The ionic nitriding (in comparison with the furnace nitriding) also allows improving relative wear resistance of the nitrided layer [19]; it enlarges the possibilities to regulate phase composition [20, 21] (by changing energy characteristics of the process or changing the ratio of working gases in the mixture (nitrogen and argon) [22]); as well as it makes it possible to eliminate harmful effect of ammonia [23]. In the process of saturation, under the action of ionic bombardment, excess concentration of tructural defects occurs in the material [24]. This contributes to an increase in the intensity of diffusion processes (including the mass transfer of nitrogen [18]) and creates a possibility for the occurrence of new phases (based on the compounds with nitrogen, as an S-phase and others) [25, 26]. Such phases do not form undeer conditions of usual saturation with nitrogen at an elevated temperature.

Such nitriding is typically conducted after high-temperature treatment and mechanical machining [27]. In this case, in the process of nitriding, the articles are exposed to negative potential from 800 to 1300 V [16], while regulating its magnitude so that to support the temperature of articles within the range from 500 to 600 °C at arc currents from 50 to 100 A. Hardness of article surface after nitriding is high and reaches the level to 11–17 GPa depending on the composition of steel.

However, by using the methods described above, it is not possible to nitride the articles made of steels with low temperatures of tempering, for example, 40KH, 9KHS, KH12MF and others, since the temperature of nitriding exceeds the temperature of tempering.

Furthermore, an analysis of the results of operational tests of nitrided steels, which work under conditions of impact-cyclic loads, revealed that the formation of nitrided high-hard layer with a sharp boundary on the surface leads to the crumbling of the surface. This is linked to the fact that formation of brittle nitride zone significantly decreases plasticity of the nitrided layer [28]. That is why exploratory research has been conducted in recent years into technologies that would yield the process of nitriding with the larger uniformity of nitrogen distribution and at the larger depth [29].

3. The aim and objectives of the study

The aim of present study is to verify an innovation technique of nitriding steel articles with a low temperature of tempering. Innovation technique is based on a change in the sequence of regimes and number of cycles of thermochemical treatment of articles: preliminary nitriding in the vacuum gas discharge and subsequent hardening and tempering.

To accomplish the aim, the following tasks have been set:

– to conduct comparative study into influence of the innovation technology of ionic nitriding on the structure and hardness of steels of different class;

– to determine a phase-structural state, critical for an increase in the hardness of a near-surface region.

4. Materials, methods of their processing and research

We employed as the source materials of nitriding samples made of steels KH12MF, 9KHS and 40KH whose chemical composition is given in Table 1.

Table 1

Chemical composition of steels KH12MF, 9KHS and 40KH

Steel grade	Chemical composition, %							
	C	Si	Mn	Cr	Ni	Cu	S	P
					not higher than			
KH12MF	1.45–1.65	0.1–0.4	0.2–0.5	11.0–12.5	0.35	0.3	0.03	0.03
9KHS	0.85–0.95	1.20–1.60	0.30–0.60	0.95–1.25	0.35	0.30	0.030	0.030
40KH	0.36–0.44	0.17–0.37	0.50–0.80	0.80–1.0	0.30	0.30	0.035	0.035

Field of application of steel of grade KH12MF is the dies of complex shape, gears, knurl screw dies, portages, matrices and punches of cutting dinking dies with complex configuration of working parts. Steel of grade 9KHS is used in the production of screw dies, markers, punches. Steel of grade 40KH is related to the pearlitic class and is used for manufacturing different gears, axes, critical machine parts, connecting rods, bushings and other components with enhanced strength.

In the present study, we applied a technique for nitriding steel articles in the vacuum-arc gas discharge [16], according to which the nitriding is also conducted after high-temperature treatment and mechanical machining. This technique is executed in the installation with a vacuum chamber, equipped with vacuum-arc vaporizers. Fig. 1 shows a duagram of the installation “Bulat-6” (Ukraine), designed for the nitriding in the arc gas discharge.

Vacuum-arc vaporizer 1, whose housing is electrically connected to the housing of vacuum chamber 2, is equipped with slit screen 3, which does not pass the ions of the evaporated metal of cathode but which does not impede the passage of gas particles and electrons. The housings of other two vacuum-arc vaporizers 4 and 5 are isolated from the chamber and serve as anodes of the two-stage vacuum-arc discharge in the working volume of the chamber.

When vaporizer 1 is enabled (it serves as electron emitter), in the presence of nitrogen, supplied to the chamber with flow regulator 11, in the vacuum chamber there occur gas arc discharges when we feed positive potentials from power units 6 and 7 to the housings of vaporizers 4 and 5. Steel articles 8 receive negative voltage of 800...1300 V from high-voltage source 9, the magnitude of voltage is changed depending on the required temperature of nitriding, which occurs as a result of the bombardment of the surface of steel articles with nitrogen ions from the arc discharges. In order to ensure uniformity in the heating of articles, they are positioned on the revolving rotation device 10.

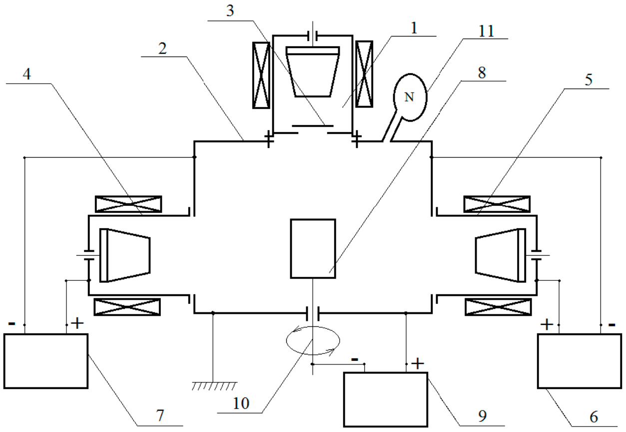

Fig. 1. Schematic diagram of the vacuum-arc installation of the type "BULAT-6" for ionic nitriding: 1 — vacuum-arc vaporizer; 2 — vacuum chamber; 3 — slit screen; 4, 5 — vacuum-arc vaporizers; 6, 7 — power sources of gas discharge; 8 — nitrided articles; 9 — high-voltage source; 10 — rotation device; 11 — flow regulator

Nitriding was applied to the batches of punches made of steels KH12MF, 9KHS and 40KH (Fig. 2) in as-received condition. Nitriding was conducted after preliminary evacuation up to a pressure of 0.003 Pa with subsequent suppply of nitrogen up to a pressure of 0.65 Pa. At negative potential on the punches of 950 V, we maintained temperature of nitriding at 550 °C for one hour.

Heat treatment after nitriding was performed in the muffle furnace and comprised hardening and low temperature tempering (executed in one- or double cycle).

Fig. 2. General view of the punch made of steel 40KH

Metallographic examinations of microstructure were carried out by the method of optical microscopy of the cross-sections of samples.

Etching was carried out with the solution of nitric acid (HNO_3), chloric iron ($FeCl_2$) and adding special components for the etching of base steels for 15–20 seconds.

We used the microscope Optika XDS-3 med to investigate the microstructure of samples at magnification 50, 100, 500 times.

Phase composition and structural state were investigated by the method of X-ray diffraction on the diffractometers DRON-3M and DRON-4 (Russia) in the emission Cu-K_α with the use in the second beam of graphite monochromator and a high degree of collimation [30]. We took images under pointwise regime with the step of scanning $\Delta(2\theta)=0.05\div0{,}2^\circ$ and the duration of the accumulation of pulses at each point $20\div40$ s.

The measurement of microhardness was carried out on the instrument PMT-3 with the load of 200 grams and aperture of 15 seconds according to GOST 9450-76.

In order to determine the magnitude of microhardness, we measured the magnitude of the imprint of indentor by the method, with which the intersection moves along the bisector of the angle between diagonals. This method provides a possibility to measure both diagonals of the imprint with the constant position of eyepiece micrometer.

5. Investigation of the effect of complex treatment on the microstructure and microhardness of different types steels

Microstructure of the sample made of steel KH12MF after complex treatment (under the mode: ionic nitriding – hardening – low temperature tempering) is shown in Fig. 3, *a*; the distribution of microhardness by depth – in Fig. 4, *a*. Metallographic analysis shows that microstructure has different morphology by depth, which means different properties, also confirmed by data on microhardness. Thus, a surface layer with depth of up to 130–150 μm has a pronounced interface with the base metal. The structure of heterogeneous surface layer can be divided into three zones: surface zone 1, with depth of up to 15–20 μm, with low hardness to 2990 MPa, this is a zone of maximal decarbonization (dark structure). Zone 2 is the zone of decarbonization, with depth of 25–30 μm of microhardness at 5235 MPa, the structure corresponds to alloyed ferrite with dendritic grains whose direction corresponds to the character of heat withdrawal. Zone 3 of the surface layer has a mixed type structure: from alloyed α-Fe (whose degree of alloying grows by depth) to the high-carbon alloyed α-solid solution with carbides of iron and chromium. The microhardness of this transition zone by depth grows from 5250 to 8000 MPa. Beyond the surface layer is the structure of the basic nitrided matrix with a hardness of 8800–9150 MPa whose maximum hardness corresponds to a depth of about 500 μm. An increase in the hardness, from the near-surface layer to the basic structure, at depth from 200 to 500 μm, is almost 500 MPa, which is explained by the release of not only alloyed (special) carbides, but also carbonitrides (at depth from 250 μm).

In the articles made of steel 9KHS, nitriding with subsequent heat treatment also led to the formation of surface layer with decreased hardness (Fig. 4, *b*). High-hard state in this case is attained at depth larger than 600 μm.

Analogous result, which testifies to the generality of diffusion processes, which lead to the reduction in surface properties and their increase at depth, was obtained also on the parts made of steel 40KH. Fig. 3, *b*, 4, *c*) show that after complex treatment (similar to the previous types of steels), surface layer of ≈200 μm has lowered hardness. At larger depth, after complex treatment, the magnitude of hardness is at the level of 7900–8000 MPa, which is somewhat higher than the hardness of steel 40KH after standard high-temperature treatment (Fig. 4*c*, dependence 2).

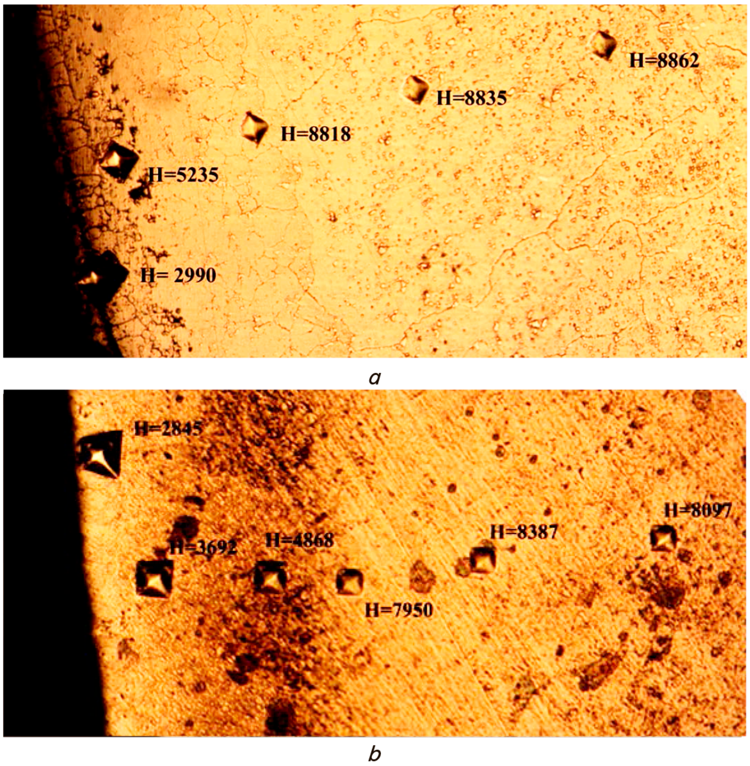

Fig. 3. Microstructure of steels after complex treatment: *a* – KH12MF; *b* – 40KH (×500)

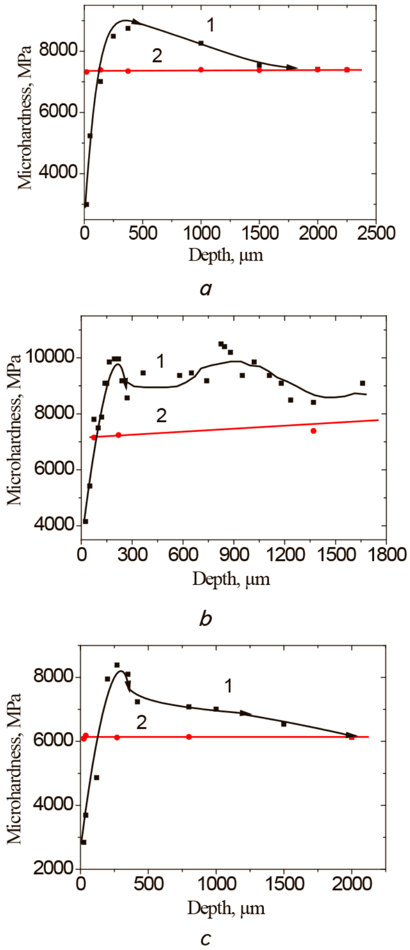

Fig. 4. Microhardness of steels after complex (nitriding and heat treatment) (1) and standard heat treatment (2): *a* – KH12MF; *b* – 9KHS; *c* – 40KH

Thus, the allowance predetermined in the production makes it possible, after nitriding and heat treatments, to polish to the finished size, clearing away the defective layer together with the allowance with a thickness of about 0.2 mm.

For the punches made of steel 40KH, removal of the surface technical layer yields to the surface of operating layer with hardness exceeding 8000 MPa (hardness of steel 40KH after a standard high-temperature treatment (hardening and tempering) is 6000 MPa).

According to data of X-ray diffractometry, after removal of the technological decarbonized layer with a thickness of about 200 μm, the structure of high-hard layer represents a two-phase composition from the mixture of α-Fe phase grains and the grains of lower nitride Fe_4N (Fig. 5, spectrum 1).

At larger depth (100–400 μm from the high-hard surface) where the hardness is 7900–8000 MPa, a solid solution of α-Fe, supersaturated with nitrogen atoms, forms (Fig. 5, spectrum 2). The supersaturation is evidenced by the enlarged period of lattice from 0.2914 nm to 0.2353 nm; in this case, a finely dispersed structure with the size of regions of coherent scattering about 10 nm is formed.

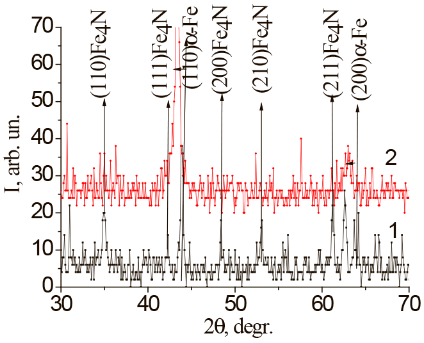

Fig. 5. XRD-spectrums of the sample made of steel 40KH after complex treatment and removal of the technological layer with low hardness: 1– high-hard surface; 2 – near-surface layer (100–400 μm) with enhanced hardness

6. Discussion of results of complex treatment of steels

At classical nitriding, it is possible to attain very high hardness of the nitrided layer (12–14 GPa), but its depth is only 20–60 μm. In this case, the material of this layer is characterized by high heterogeneity and brittleness. For the parts that work under conditions of impact loads, such layer is critical and is subjected to destruction. In order to make the process of nitriding effective for the parts of the type of dies, punches, which operate under conditions of multicycle, alternating, impact load, we proposed and tested conducting, after the nitriding, of heat treatment. Such procedure of treatment made it possible to extend the strengthened layer to larger depth, while the surface layer becomes less rigid and easily workable at the last stage of finishing. The thickness of surface layer (150–200 μm) with relatively low hardness is the optimal thickness, which is actually the allowance for finishing.

Deeper located is the operating layer of thickness to 2000 μm, which enables to carry out finishing of a part without additional strengthening, since the reinforced layer is sufficiently large. Thus, resource of the part increases considerably.

Thus, the technique verified under actual conditions is applicable for the nitriding of steels with a low temperature of tempering and consists of the ionic nitriding in the vacuum gas discharge before hardening and tempering. That is why the heating temperature during ionic nitriding influences little the process of high-temperature treatment. Hardness of the surface of articles after this sequence of operations for steels with the low temperature of tempering is at the level of 8–10 GPa.

In this case, the process of nitriding is accelerated considerably, since nitrogen atoms penetrate untempered steel more easily, and thus the duration of nitriding does not exceed 1 hour. Only one-time mechanical machining will suffice after the completion of the process of thermochemical treatment, removing a layer of about 200 μm with lowered hardness.

Results obtained in the course of study formed a basis for the directed surface modification of the industrial parties of punches of different purposes. At present, their production tests are conducted with the accumulation of statistical data.

7. Conclusions

The complex innovation treatment, which includes the process of ionic nitriding with the subsequent heat treatment in the form of hardening with subsequent tempering, makes it possible to substantially increase the depth of structure modification and enhance hardness, and, therefore, to improve working ability of parts made of steels with the low temperature of tempering.

1. An analysis of microstructure that we performed for different types of steels, exposed to complex treatment, revealed the generality of an increase to 2000 μm of thickness of the penetration of nitrogen atoms and the formation of region with the changed structure and enhanced hardness. In addition, general is the formation of the surface decarbonized (technological) layer. Such layer is the allowance for finishing in order to obtain the required accuracy of dimensions and surface finish.

2. We determined phase composition of the nitrided layer with high hardness, which consists of lower nitride Fe_4N and the solution of nitrogen in α-Fe.

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