Ionic nitriding of R6M5 steel with ultrafine-grained structure in a pulse glow discharge

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Abstract. The article presents comparative data on the hardness of steel R6M5, rolled in helical rolls (BB) with different number of passes and smooth rolls (HS). It is shown that severe plastic deformation (SPD) in HR with different passes and hardening of the surface of steel samples by nitriding in a glow discharge makes it possible to increase the hardness and length of the nitrided layer of tool steel. The effect of nitriding in a glow discharge on the structure and mechanical properties of steel R6M5 obtained by quenching at 1200°C and single tempering at 560°C with preliminary SPD in HR and SR has been investigated. It was found that for a specimen rolled with twelve passes in HR and one pass in SR, the increase in microhardness to the surface is comparatively small (1.5-1.8) than for specimens deformed with four or eight passes in HR and one pass in SR (2 - 2.2 times). It is shown that the microhardness along the depth of the diffusion zone for samples deformed with twelve passes in HR is distributed smoothly, and the thickness of the diffusion layer for these samples is 2.6 times greater than the thickness of the diffusion layer of samples previously deformed by a small number of passes in HR.

Keywords: glow discharge nitriding, temperature, nitrides, hardness, structure, helical rolls.

Today, steel nitriding is used in various industries to increase the operational durability of a wide range of products: discs, crankshafts and camshafts, spindles of metal-cutting machines, stamping and cutting tools, etc. [1].

The search for sources of active nitrogen led to the development of a nitriding process in a glow discharge [1-3]. At present, a series of units with a capacity of up to 150 kW have been developed, which can process large-sized parts up to 12 m long. The use of ion nitriding made it possible to reduce the duration of the technological cycle by 2-5 times, optimize the composition of the diffusion layer, provide a technologically simple process automation scheme, improve the quality of nitrided coatings.

Various types of electrical discharges are used to activate the gas phase: arc, glow, spark and corona; magnetic and electrostatic fields; irradiation with ultraviolet and γ-rays [4–7].

The most widespread at present is the process of nitriding with ionized nitrogen in the plasma of a glow discharge (ion nitriding) [1]. In a rarefied nitrogen-containing atmosphere between the cathode (part) and the anode, a glow discharge is initiated, gas ions, bombarding the cathode surface, heat it to the saturation temperature. The nitriding temperature is 470–580°C, the vacuum is 1–10 mm Hg, the operating voltage ranges from 400 to 1100 V, the process duration is from several minutes to 24 hours.

The intensification of the process during ion nitriding is explained by the effect of a glow discharge on all elementary processes responsible for the formation of a diffusion layer: activation of the gas phase, adsorption and diffusion [1, 2]. At present, installations for nitriding, operating on a glow discharge, have almost completely replaced installations for gas and liquid nitriding. This is primarily due to the fact that the process of ionic nitriding in them is the fastest and most efficient.

Thus, at present, one of the popular technological methods for changing the performance characteristics of steels and alloys is the diffusion saturation of the surface layers with nitrogen. Nitriding increases fatigue resistance, corrosion resistance, heat resistance, hardness and wear resistance. However, the properties of the nitrided layer in most cases depend on the chemical composition and the initial structure of the material. In particular, this concerns such parameters as hardness and length of the hardened area.

Analysis of existing nitriding methods shows a rather noticeable efficiency of this method for hardening steels containing alloying nitride-forming elements, such as titanium, chromium, vanadium, aluminum, etc. [3-5, 8]. Alloyed steels after nitriding have a surface hardness of HV 850 - 1200 kg/mm^2, while the hardness of the nitrided surface of parts made of carbon and low-alloy steels does not exceed HV 350 - 500 kg/mm^2. The presence of alloying materials increases the solubility of nitrogen in iron and contributes to a significant increase in hardness and strength due to the formation of ultradispersed nitrides of these elements in the saturable layer. However, high-alloy steels used for the manufacture of parts for nitriding have a complex smelting technology and contain expensive and scarce alloying materials. Most often, for the structural strength of the mating units of various parts, alloying of their base throughout the entire volume is not required; it is enough to introduce alloying elements into the surface layer.

It is known [9] that the rate of nitrogen diffusion in steels depends on the area of grain boundaries. It can be noted here that transcrystalline processes undergo significantly slower in coarse-grained steels at temperatures below 600°C. Therefore, to intensify the formation of the nitrided layer, it is necessary to choose materials with a finer structure, or to bring their initial coarse-grained state to a submicrocrystalline one as a result of preliminary treatment. In particular, the refinement of the average grain size of steels of the ferrite-pearlite class can be achieved as a result of quenching with the formation of a martensitic structure [10]. Quenching itself also leads to an increase in the hardness of the steel, and not only on the surface, but throughout the entire volume of the part. However, quenched structures are prone to softening as a result of tempering at elevated operating temperatures, have low corrosion resistance even in normal environments, and also tend to diffusely set in contact with the surfaces of many materials in tribological processes.

In our opinion, severe plastic deformation (SPD) can be used to improve the performance properties of the surface layers of tool steel. It is known that in recent years it is SPDs that are increasingly used to improve the structural homogeneity and quality of metallic materials [11].

The aim of the work is to experimentally confirm the feasibility of using SPD of the surface zones of tool steel to intensify the nitriding process of tool steel.

Equipment, materials and technique of the experiment

In work [12], a tool was developed with rolls with helical working surfaces. In these rolls, the helical protrusions and valleys of the upper and lower rolls are located oppositely. Helical rolls (HR) are designed to produce semi-finished products with a fine-grained structure. The developed tool implements SPD without significant changes in the original shape and dimensions of the workpiece. It should be noted that smooth rolls (SR) installed on any laboratory mill can be used to level the undulating surface of semi-finished products with a fine-grained structure and obtain strips of the required size.

A series of experiments were carried out in laboratory conditions. Steel R6M5 with a size of 6×150×400 mm was chosen as the workpiece material. Rolling of this billet on a mill with HR and SR was carried out in the following modes:

- heating to a temperature of 850 °C, holding for 2 h, rolling in two passes in HR to a thickness of 5.8 mm, heating at a temperature of 850 °C, holding for 30 minutes, rolling in two passes in HR to a thickness of 5.5 mm, heating at a temperature of 850 °C, exposure 30 min, rolling in SR to a thickness of 4.0 mm;
- heating to a temperature of 850 °C, holding for 2 h, rolling in four passes in HR to a thickness of 5.8 mm, heating at a temperature of 850 °C, holding for 30 minutes, rolling in four passes in HR to a thickness of 5.5 mm, heating at a temperature of 850 °C, exposure 30 min, rolling in SR to a thickness of 4.0 mm.
- heating to a temperature of 850 °C, holding for 2 h, rolling in six passes in HR to a thickness of 5.8 mm, heating at a temperature of 850 °C, holding for 30 minutes, rolling in six passes in HR to a thickness of 5.5 mm, heating at a temperature of 850 °C, exposure 30 min, rolling in SR to a thickness of 4.0 mm.

To study the effects of ion nitriding on the structural-phase composition and hardness of strips from R6M5 high-speed tool steel, rolled according to the above-described technology, model specimens 50×20×4 mm in size were cut out. The test samples were first ground and then polished with diamond pastes to a mirror finish. Before loading into the nitriding chamber, the model samples were washed in gasoline, then in acetone, and dried in air. Glow discharge nitriding was carried out in a JON-600 device. The following process parameters were used: temperature T = 520 °C, vacuum pressure (reactive atmosphere) p = 150 Pa, time t = 15 hours, the composition of the reaction atmosphere was a mixture consisting of 90% N_2, 5% Ar and 5% H_2, the flow rate of gaseous media was as follows: 900 ml/min N_2 + 50 ml/min Ar + 50 ml/min H_2. After nitriding, the samples were subjected to heat treatment, which consisted of quenching at 1200 °C and a single high tempering at 560 °C.

Measurements of the microhardness along the depth of the diffusion zone were carried out on a Struers Duramin-1/-2 microhardness tester. The static load applied to the indenter for 10 s was 980.7 mN (100 g).

Metallographic analysis was carried out using an energy-dispersive spectrometer JNCAENERGY (England) installed on an electron probe microanalyzer JEOL (Jeol) at an accelerating voltage of 25 kV. The magnification range of the JEOL device is from 40 to 40,000 times. The structural features of the samples were also studied using a JEM-2100CX transmission electron microscope (TEM) at accelerating voltages of 200 kV. To reveal the structure of the steel, the section was etched in a solution of 5% HNO_3 and 95% C_2H_5OH.

Research results and their discussion

Analysis of the photograph of the initial structure of the material showed that in the cross section of the sample there are coarse lines and carbide banding. The structure contains inclusions of both large and medium and small carbides. This orientation of the carbide phases in the cross section leads to anisotropy of volumetric changes during quenching and an increase in the level of thermal stresses caused by the phenomenon of phase hardening. In our opinion, this structural heterogeneity affects the performance of the tool. It should be noted that the surface hardness of the initial samples was 60 ... 62 HRC.

The study of the structural state of steel R6M5 after rolling in HR with four and eight passes shows that a relatively fine-grained structure is formed in the surface zone of the section. In this case, the density of intragranular dislocations increases, and a strip structure is formed.

It should be noted that an increase in the number of passes leads to a decrease in the width of the microbands, while thinner shear bands are formed at the boundaries of the original wide microbands. An increase in the number of passes to eight leads to the formation of a fine-grained grain-subgrain structure in steel R6M5. The sizes of individual grains in the peripheral zone of the workpiece reach 12-19 microns.

Deformation by twelve passes in HR of billets heated at a temperature of 850 °C led to the formation of a homogeneous and equiaxial structure in the peripheral zone of the longitudinal and cross-section of the billet. It can be seen that further refinement of the grain-subgrain structure occurs. As a result of softening processes in the metal of the workpiece, a recrystallization structure is formed in the surface zone of the rolled strips with an average grain size of about 6 - 9 μm.

It can be assumed that with an increase in the degree of deformation in subsequent rolling passes in HR, structure refinement occurs not only by twinning, but also by the formation of cellular substructures as a result of the development of dislocation slip processes. At high degrees of accumulated deformation, the boundaries of former twins and subgrains transform into high-angle boundaries.

Thus, during rolling in HR, the action of alternating deformation mechanisms provides fragmentation and reorientation of the crystal lattice. At the same time, high-angle boundaries are formed in the peripheral zones of the workpiece with a high density.

Analysis of the data obtained shows that rolling at the last stage in SR at a temperature of 850 °C significantly affects the microstructure of the alloy. The microstructure of the surface zones of the R6M5 steel strips, after rolling on HR with eight passes and one pass in SR, is characterized by the presence of subgrains formed inside the former deformation bands. The average subgrain size is 1 - 4 μm.

SR rolling of billets deformed with twelve passes in HR results in the formation of an ultrafine-grained (UFG) structure on the surface of the strips. As a result of softening processes, a structure with a size of 0.6 to 0.9 μm is formed on the surface of the rolled strips. In the images of the microstructure after rolling in SR, a clear image of grain boundaries was observed. The type of microstructure indicated the formation of grains with predominantly high-angle boundaries.

The experiments have shown that the main parameters of the nitrided layers treated in a glow discharge are the hardness of the surface layer and the depth of nitrogen penetration. It should be noted that the depth of the nitrided layer is 0.03–0.8 mm.

The study of the results of measuring the microhardness showed that samples deformed with twelve passes in HR and subsequently processed by nitriding in a glow discharge have a comparatively small increase in microhardness on the surface (1.5-1.8) than samples deformed with four or eight passes in HR (2 - 2.2 times). This increase in microhardness is due to the formation of nitride phases with a high nitrogen content in the near-surface layer. The high nitrogen content in the near-surface layers is due to the low nitrogen diffusion coefficient in steel that has undergone SPD with a small number of passes in HR.

Evaluation of the microhardness over the cross section of the sample showed that the strip deformed with twelve passes in HR and then nitrided in a glow discharge has a smoother distribution of microhardness over the depth of the diffusion zone. In our opinion, this is due to the formation of the UFG structure in these samples and the appearance of crystal lattice defects in the material structure as a result of such SPD. All this creates more favorable conditions for the diffusion of nitrogen deep into the material. It should be noted that the thickness of the diffusion layer in specimens deformed with twelve passes in HR is 2.6 times greater than the thickness of the diffusion layer in specimens deformed with four or eight passes in HR. It can be seen from the results obtained that nitriding in a glow discharge at a given temperature does not lead to a decrease in the mechanical properties of the base material.

Studies have found that the rate of increase in microhardness is maximum at the beginning of the nitriding process. In our opinion, during ion nitriding, a high concentration gradient, which is the driving force of the process, is established in the initial stages of the nitriding process in a glow discharge.

The creation of a UFG structure at SPD and a limiting concentration gradient at the early stages of ion nitriding, activation of the UFG surface and the escalation of defects in the surface zone are accompanied by intense absorption of nitrogen and the completion of the formation of a relatively thick layer in terms of hardness during the first hour of the nitriding process.

A coarser-grained structure accelerates the nitriding process of the surface layers of the samples, while a nitrided layer, small in size, is formed. This is manifested in higher values of microhardness in these layers in comparison with nitrided layers obtained in samples with a fine-grained structure.

The thickness of the diffusion layer, in the samples rolled with eight and twelve passes in HR, reaches an almost maximum value during 7 and 4 hours of nitriding, respectively. An increase in the nitriding time over 7 hours has a relatively weak effect on the depth of the diffusion layer. At the same time, the size of the diffusion layer obtained in the samples with a fine-grained structure on the surface increases even more in comparison with the samples with a coarse-grained structure on the surface.

With a nitriding time of the order of 10 h, the maximum values of the microhardness and the depth of penetration of the nitrided layer in the samples obtained, rolled with eight and twelve passes in HR, practically do not differ. However, it should be noted that when nitriding samples deformed with twelve passes in HR and nitrided in a glow discharge, the limiting values of hardness are reached at a nitriding time of 3-4 hours. The same parameters in the samples deformed with eight passes in HR and nitrided in a glow discharge are realized during the nitriding time of 6–7 hours.

Conclusions

1. It was found that for the specimen after SPD, rolled by twelve passes in HR, the increase in microhardness to the surface is comparatively small (1.5-1.8) than for specimens deformed with four or eight passes in HR (2-2.2 times).

2. The distribution of microhardness over the depth of the diffusion zone in deformed specimens with developed SPD is smoother, and the thickness of the diffusion layer is 2.6 times greater than the thickness of the diffusion layer in specimens deformed with a small SPD.

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