Structural quality criteria for a surface layer formed by gas pressure nitriding of austenitic steels

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Abstract. The tribotechnical efficiency of gas pressure nitriding of austenitic class steels is shown. The characteristics of the structure and operational properties (contact endurance and wear resistance) are given in comparison with cemented steels.

1. Introduction

Nitriding along with cementation are the main methods of surface hardening of the most critical machine parts. An important advantage of nitriding in comparison with cementation is the provision of a higher extreme pressure resistance of steel, cyclic strength, and corrosion resistance. The advantage of nitriding is a slight change in the volume of parts, which can be taken into account in their manufacture. Grinding volumes are incomparably smaller than with cementation. The noted advantages explain the continuous growth in the use of nitriding in various engineering industries. Along with this, gas or ion nitriding has significant drawbacks, the main of which are a long saturation time and a limited (up to 0.5 mm) thickness of the diffusion layer.

The prospect of eliminating these shortcomings and a technological breakthrough in the field of increasing the load capacity of steel parts reinforced with nitrogen is associated with the development and application of gas-pressure nitriding [1]. Gas-pressure nitriding is a process that implements the simultaneous impact on the surface of steel of high nitrogen gas pressure and temperature, which provide a high rate of nitrogen supply to the saturated surface and a high rate of its diffusion removal into the metal depth. These features make it possible to form diffusion layers of large thickness, commensurable with the thickness of the cemented layer in a relatively short time - 3-5 hours.

Austenitic structural steels have a valuable combination of mechanical and corrosive properties. Defect of these steels is the low resistance to fatigue and wear, which limits the scope of their application. The main way to increase the operational characteristics of austenitic steels is nitriding. At the same time, high-chromium steels of the austenitic class are hardly nitrided. Therefore, the task arose of finding ways to improve nitriding and intensification of the process of diffusion saturation of austenitic steels. In this paper, we consider the use of gas pressure nitriding and the influence of
technological process factors on the structural characteristics and operational properties of austenitic steels is shown.

2. Materials and research methods
Experimental samples of high-chromium austenitic steels 12Kh18N10T, 08Kh16G15N5MAF and high-manganese steel 40G14N8Kh3B1 were subjected to gas-pressure nitriding. The pressure of the gaseous medium was varied from 30 to 150 MPa, the process temperature was in the range of 950 - 1150°C, the saturation time was 3 hours. Bench tests to assess the resistance of the nitrided layer of contact fatigue were performed on a Sh-17 two-pin roller machine of the IMASH RAS design, which provided tests according to the rolling pattern with 6% slip and imitated the operation of the nitrided layer of gears [2]. Tests for wear resistance were carried out on a Skoda-Savin machine with a load of 150N. The structure of the nitrided layer was analyzed by x-ray and electron microscopy methods.

3. Research results and discussion
The effect of process pressure on the microhardness distribution of 12Kh18N10T steel is shown in figure 1.

![Figure 1](image.png)

**Figure 1.** Microhardness distribution over the thickness of the nitrided layer of 12Kh18N10T steel saturated at 950°C, 3 h and pressure: 1 - 30 MPa; 2 - 100 MPa; 3 - 150 MPa.

It is seen that there is a flowing distribution of hardness over the thickness of the layer. A noticeable increase in surface hardening is observed with increasing nitrogen pressure in the working chamber. This dependence indicates that an increase in pressure increases the saturating ability of the technological atmosphere and, as a result, a more intensive supply of nitrogen to the surface layer. In the presence of a high chromium content in steel, the incoming nitrogen binds to highly hard chromium nitrides. The density of nitride particles, their size, and their relation to the matrix lattice largely determine the level of surface hardening. Meanwhile, the outflow of atomic nitrogen to the growing nitride particles inhibits the growth of the diffusion layer, the thickness of which turned out to be practically independent of pressure at a temperature of 950°C and amounted to about 0.5 mm. According to transmission electron microscopy, the main components of the structure of the nitrided layer are martensite, austenite, and CrN nitrides, the volume fraction of which is not less than 20%. Nitride particles are densely located in the martensitic matrix, have an equiaxial shape and a size of ~ 10 15 nm, figure 2, and orientation in the region of martensite plates, which illustrates the dark-field image in the (111) CrN reflection. The structure of the studied steels 40G14N8Kh3MF1 and 08Kh16G15N5MAU is similar to the structure of the layer of steel 12Kh18N10T considered, while the nitride formation intensity increases in proportion to the concentration of chromium in the steel.
Figure 2. The structure of the nitrided layer of steel 12Kh18N10T after diffusion saturation at 150 MPa, 1050°C, 3 h: (a) - microstructure of the layer (transverse section), x250; (b) - electron microscopic dark-field image in the reflection of nitride CrN, x50 000.

The effect of nitriding temperature is illustrated by the data in table 1.

Table 1. Characteristics of the diffusion layer.

| Steel grade     | Pressure, MPa | t, °C | Layer thickness, mm | Hardness | Phase comp. a |
|-----------------|---------------|------|---------------------|----------|---------------|
| 12Kh18N10T      | 30            | 950  | 0.35                | 380      | M, A, CrN     |
|                 | 100           | 950  | 0.50                | 550      |               |
|                 | 150           | 950  | 0.60                | 730      |               |
|                 | 150           | 1050 | 0.90                | 790      |               |
| 40GL14N8Kh3MF1  | 150           | 950  | 0.35                | 450      | M, A, CrN, VN |
|                 | 150           | 1050 | 0.85                | 540      |               |
| 08Kh16G15N5MAU  | 150           | 950  | 1.10                | 590      | M, A, CrN, AlN|
|                 | 150           | 1050 | 1.60                | 680      |               |

a M-martensite, A-austenite.

It is shown that an increase in the pressure and temperature of the process allows reaching the thickness of the hardened layer of 1.5 mm, which significantly (3-4 times) exceeds the capabilities of traditional processes of gas and ion nitriding. The higher the chromium content, the greater the surface hardening. This result confirms the position that chromium nitrides make the main contribution to austenite hardening.

In this regard, the results of bench tests for contact endurance and wear resistance were analyzed from the standpoint of structural changes. Examples of test results in the form of contact fatigue curves are shown in figure 3. It can be seen that the greatest contact endurance limit is ensured by gas pressure nitriding at 150 MPa, 1000°C, with quenching and subsequent tempering at a temperature of 500 °C, curve 1.

Figure 3. Contact strength curves of steel: 1 - gas pressure nitriding; 2 - cementation; 3 - gas nitriding
Wear resistance was studied on steels martensitic 16Kh2N3MF-BAY-Sh and austenitic 12Kh18N10T classes. Similar data were obtained for steels and unified laws obtained.

| Pressure, MPa | Temperature, °C | Tempering temperature, °C | Relative wear resistance |
|--------------|-----------------|---------------------------|--------------------------|
| 30           | 950             | -                         | 1.17                     |
| 100          | 950             | -                         | 1.28                     |
| 150          | 950             | -                         | 1.39                     |
| 150          | 1050            | -                         | 1.71                     |
| 150          | 1100            | -                         | 1.76                     |
| 150          | 1000            | -                         | 2.13                     |
| 150          | 1000            | -                         | 2.19                     |
| Gas cementation, 930°C, 8h |                  |                           | 1.56                     |
| Gas nitriding, 540°C, 48h |                  |                           | 2.83                     |

An analysis of the results of metallophysical studies showed that the effect of the sizes of nitrides on contact endurance and wear resistance is associated with the level of microdeformation of the crystal lattice of the matrix, which depends on the coherent or incoherent release of nitrides [3]. Based on the analysis of the broadening of the martensite lines, it was found that with coherent nitrides the magnitude of microdistortions is about $10.3 \times 10^{-3}$ with incoherent particles it is 2.5 times smaller. With incoherent precipitates, the density of the nitrides of the particles is lower, the fragility is less, and as a result, the fatigue resistance is higher. A decrease in the magnitude of microdeformation leads to an increase in the plasticity in the microvolumes of the layer, which contributes to the relaxation of local microstresses and to an increase in resistance to the nucleation of a fatigue crack and generally to an increase in the operational characteristics of steel.

4. Conclusion

The advantages of gas pressure nitriding over existing processes of alloying the surface of steel with nitrogen are established. Particularly great is the advantage of this process when hardening austenitic steels belong to hard nitriding. For these steels, gas pressure nitriding is the only process that makes it possible to obtain layers of high hardness and wear resistance with a thickness exceeding 1 mm. The characteristics of diffusion layers — their structural state, saturation, phase composition, and, as a result, properties are controlled by a small group of technological factors — pressure, temperature and time parameters.

References

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