High-temperature internal nitriding of heavy-section steel products

V. Khatkevich, S. Nikulin, S. Rogachev, A. Sergeychev
The National University of Science and Technology “MISIS”, Leninsky pr. 4, 119049 Moscow, Russia
csaap@mail.ru

Abstract. A method for the internal nitriding of 0.08C-17Cr-0.5Ti corrosion-resistant steel sheets more than 2.5 mm thick has been suggested. The procedure includes the high-temperature nitriding of thin sheets, their hot pressing, and annealings. After such a treatment, the microhardness magnitude demonstrates the uniform distribution across the sheet section and reaches 280 HV. The formed structure of the material is characterized by a grain size of 0.5 μm and the presence of chromium-nitride particle precipitates. Compression tests showed that the prepared samples are deformed as a monolithic material; no cracks and laminations are formed on the surface.

1. Introduction
The high-temperature internal nitriding process is the saturation of a material with nitrogen. In the course of the process, in contrast to the common surface nitriding process during which nitrides are formed in the surface layer, the deep diffusion of nitrogen (up to through diffusion) takes place, which results in the formation of nitrides over the whole section of a sample. The structure of a material subjected to internal nitriding contains disperse particles distributed over the entire volume of the material, which are thermodynamically stable nitrides of alloying elements and ensure the efficient hardening of the material [1-4].

The internal nitriding is successfully used to uniformly harden thin-wall products, over their volume, made from ferritic corrosion resistant steels [5-8]. In this case, the substantial increase in the static and cyclic strength and high relative elongation at test temperatures of from room temperature to 700 °C take place.

However, the internal nitriding of steel products more than 0.5 mm in thickness is related to the substantial increase in the process time and nonuniform saturation of material with nitrogen [7]. In the present study, a method for the internal nitriding of 0.08C-17Cr-0.5Ti corrosion-resistant steel sheets more than 2.5 mm thick has been suggested. The procedure includes the high-temperature nitriding of thin sheets, their hot pressing, and annealing.

2. Experimental Procedure
Flat 0.08C-17Cr-0.5Ti steel samples 0.5 mm thick in the initially recrystallized state were subjected to nitriding at 1000-1100 °C in pure nitrogen and subsequent vacuum tempering at 700 °C. Such a treatment leads to the formation of nitried-ferrite structure over the whole section of steel sample,
precipitation of second-phase \( \text{Cr}_2\text{N} \) mainly lamellar particles, and increase in the microhardness from 180 to 320 HV. The average grain size was 7±1 µm.

For hot pressing, a sheet pile consisting of 11 alternating non-nitrided and nitrided steel sheets was formed; the thickness of each sheet is 0.5 mm. Subsequently, the sheet pile was vacuumized and subjected to plastic deformation at 800 °C using a Gleeble System 3800 thermal mechanical simulator. Metallographic analysis of the structure of samples was performed with magnifications of ×100 and ×500 using a Buehler optical microscope and sections etched in an aqueous solution of hydrochloric acid (50 ml concentrated HCl + 50 ml H\(_2\)O).

Electron-microscopic studies of the cross-section structure were performed using a JEM-2100 (JEOL) transmission electron microscope and thin films. The Vickers microhardness was measured using the 100-g load applied for 10 s and a MICROMET 5101 tester equipped with a digital camera and ImageExpert MicroHardness 2 software.

Compression tests were performed using spark-cut rectangular samples 2×2 mm in section and 3 mm in height and an Instron 5966 testing machine. Samples were subjected to compression to a degree of deformation of 50%; the compression direction coincided with the plane of samples layers.

3. Results and Discussion
After pressing, sheets 2.7 mm in thickness, which consists of firmly jointed layers, were obtained; no interfaces are observed on the polished section. Laminar structure of the resulting material was revealed after chemical etching (Fig. 1). The average thickness of individual layers after pressing was 250 µm.

![Figure 1. Structure of a cross-section of a pressed sample](image)

Fig. 2 shows the distribution of microhardnes across the thickness of a prepared steel pack subjected to pressing and subsequent heat treatment. The microhardness of nitrogen-free and nitrided steel layers is 180 HV and 340…420 HV, respectively. The short-time heating of the pack at 1000-1100 °C and cooling in air lead to the nitrogen redistribution in nitrogen-free and nitride layers; in this case, after hardening, the microhardness increases to 550…630 HV. The subsequent vacuum annealing at 700 °C leads to the decrease in microhardness to 280 HV and uniform macrohardness magnitude over the material section. The decrease in the microhardness from 320 to 280 HV (i.e., by 12%) as compared to that for the initial steel state (before pressing) is related to the decrease in the mass fraction of nitrogen in the material because of the nitrogen redistribution between layers during heating.
According to TEM data, the pressing and subsequent short-time heating at 1075 °C and tempering at 700 °C result in the formation of fragmented structure (in the cross section of a sample) with an average grain size of 0.50±0.05 µm, fine chromium-nitride particles within the grains, and elongated chromium-nitride particles at grain boundaries (Fig. 3).

**Figure 2.** Microhardness distribution across the steel-pack thickness

**Figure 3.** Microstructure (a-c) and (d) interface of steel layers of a sample subjected to pressing, short-time heating at 1075 °C and tempering at 700 °C
The decrease in the average grain size from 7 to 0.5 µm is likely to in part compensate for the softening associated with a decrease in the mass fraction of nitrogen in the material. Compression tests showed that, after hot pressing, laminations are observed on the surface of samples. Such laminations were also observed in a sample cross section (Fig. 4a). The depth of laminations reaches 30% of the total thickness of the sample.

![Figure 4. Cross section of a sample after compression tests (the compression direction is shown by an arrow): (a) sample subjected to pressing; (b) sample subjected to pressing and short-time annealing at 1075 °C at tempering at 700 °C](image)

At the same time, samples subjected to hot pressing, short-time annealing at 1075 °C and subsequent tempering at 700 °C were deformed as a monolithic material without the formation of cracks and laminations on the surface. Cross sections of such samples demonstrated only individual laminations no more than 50 µm deep (Fig. 4b).

Thus, in the present study, the possibility of internal nitriding of steel products more than 2.5 mm thick and their substantial hardening was demonstrated.

4. Conclusions
A method for the internal nitriding of 0.08C-17Cr-0.5Ti corrosion-resistant steel sheets more than 2.5 mm thick has been suggested. The procedure includes the high-temperature nitriding of thin sheets, their hot pressing, and annealing. After such a treatment, the microhardness magnitude exhibits a uniform distribution across the sheet section and reaches 280 HV. The formed structure of the material is characterized by a grain size of 0.5 µm and the presence of fine chromium-nitride particle precipitates and elongated chromium nitride particles at grain boundaries. Compression tests showed that the prepared samples deform as a monolithic material, no cracks and laminations being formed on the surface.

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