**PAPER • OPEN ACCESS**

# Influence of nitriding parameters in radio-frequency inductive discharge plasma on the structural-phase state and properties of H12MF steel

View the article online for updates and enhancements.

# Influence of nitriding parameters in radio-frequency inductive discharge plasma on the structural-phase state and properties of H12MF steel

**V Y Novikov[1*], I Y Goncharov[2], M N Yapryntsev[1] and M Y Gazizova[1]**

[1] Belgorod State National Research University, Belgorod 308015, Russia.
[2] Belgorod State Technological University named after V.G. Shoukhov, Belgorod 308012, Russia.

*Corresponding author: novikov_v@bsu.edu.ru

**Abstract.** The influence of technological parameters of plasma nitriding on the structural-phase state and mechanical properties of H12MF steel has been investigated in this work. The possibility of improving the corrosion resistance of the reported material was demonstrated by changing the content of nitride phases ($Fe_3N$, $Fe_4N$, $CrN$) in the surface layer, depending on the nitriding mode.

## 1.      Introduction

The growing manufacturing of ultra-high-strength steels with tensile strength above 1500 MPa requires improvement of the life cycle of tools and dies. Die tool steel H12MF (the equivalent of the AISI D2) has become widespread in industry because of its high mechanical properties, especially due to the high abrasion resistance [1]. However, this steel has a relatively low corrosion resistance [2]. One of the most effective surface modification techniques to improve the main performance characteristics of tool steels is plasma nitriding [3, 4]. In this work, the nitriding process was performed by the high efficient plasma source based on a radio-frequency inductive discharge ("transformer coupled plasma" termed a TCP). This approach allows to create and continuously maintain a high-density gas discharge and makes it possible to independently control the energy and density of the ion flux bombardment to the substrate [5].

## 2.      Materials and methods

For this study, samples with a size of $15 \times 20 \times 3$ mm$^3$ of H12MF steel were used as the base material. These samples were ground using several grades of sandpaper, and then polished using a 1 μm diamond suspension. The nitriding process was performed with "NIKA 2012" multifunctional vacuum unit. Cleaning was carried out by a RFPG-128 radio-frequency plasma source in the atmosphere of argon, supplied at a rate of 4 l/h. The pressure in the chamber was 0.2 Pa. The power of the source was 900 W, the cleaning time was 10 min, and the sample temperature did not exceed 150 ºC, respectively. Nitriding was made in a nitrogen atmosphere, which was supplied at the rate of 4.5 l/h. The pressure in the chamber was 0.2 Pa. Nitriding time was equal to 180 min. The process was performed at a constant temperature of about 450 ºC. A pyrometer was used for the control of the temperature. The temperature was maintained by supplying a different potential to the sample, depending on the power of the RFPG-128 source. There were prepared 3 series of samples at various values of the

power source and the negative bias potential on the sample: sample 1 - 700W/300V, sample 2 - 800W/250V, sample 3 - 900W/200V.

The microstructure of the samples was studied by a FEI Quanta 200 3D scanning electron microscope (SEM). The phase-structural state was studied on a Rigaku Ultima IV diffractometer in Cu-Kα radiation. The microhardness was measured with a Vickers diamond pyramid using microindentation on an Instron microhardness test device 402MVD with a load of 100 g. The corrosion test was performed by an electrochemical method using a multichannel potentiostat-galvanostat P-20X8 and a standard three-electrode cell. A silver chloride electrode was used as a reference electrode, a carbon electrode acted as an auxiliary. All studies were performed at room temperature in the isostatic solution of 0.9% NaCl. The area of the sample surface was 1.0 cm$^2$. Studies of changes of a surface morphology before and after tests on corrosion resistance were performed using SEM.

## 3.    Results and discussion

The initial microstructure of steel H12MF is represented by ferrite (α-Fe) and $M_7C_3$ carbides. The structure of the nitride layer consists of two zones: the compound layer and the diffusion zone (Figure 1). The compound layer is a layer on the surface with a thickness of ~5-10 μm, composed of the base metal and alloying nitride elements. Further, a smooth transition from the modified layer to the base material is observed, that is, the zone of nitrogen diffusion into the depth of the material. The depth of the modified layer is observed in the SEM images for all series of samples in the range of ~60-80 μm. This spread is associated with an irregularity in the thickness of the diffusion layer caused by the irregular distribution of carbides over the volume of the initial material. In elongated colonies of carbides, diffusion of nitrogen is hindered and the thickness of the diffusion zone near their accumulations is much lower.
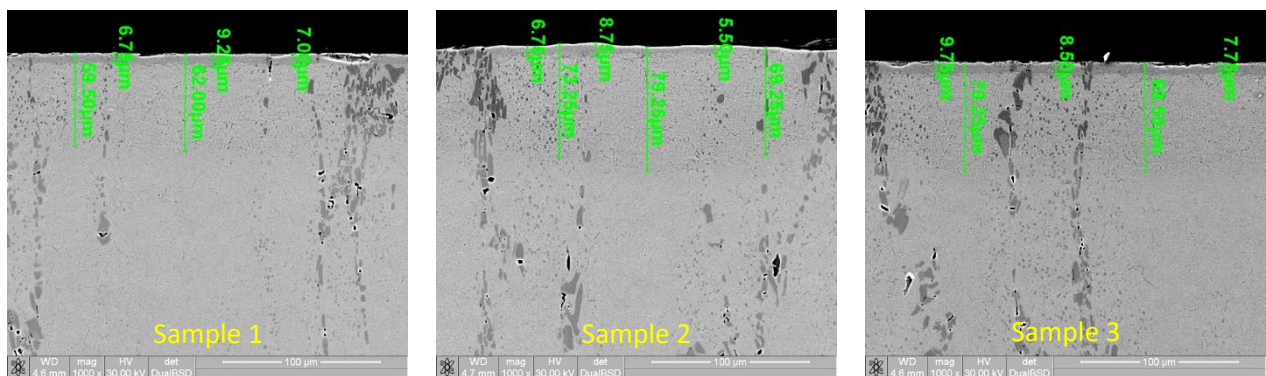


**Figure 1.** SEM micrographs of the transverse section of nitrided samples.

The depth of nitriding, determined by the microindentation method, was equal to 140 μm (Figure 2) for all modes. The value of hardness was near the surface for samples 1, 2, and 3 is 1054, 938 and 1013 HV, respectively. With increasing depth, the hardness gradually decreases to an average value of 424 HV down to 140 μm.

The phase composition of the surface layer for all samples is represented by nitrides ε-$Fe_3N$, γ´-$Fe_4N$ and CrN (Figure 3). However, depending on the mode of the process, its ratio changes over a wide range. With an increase in the RFPG power from 700 to 900 W, the content of $Fe_3N$ decreases from 82 to 51%, and the content of $Fe_4N$ and CrN increases from 10 to 33% and from 7 to 15%, respectively (Table 1).
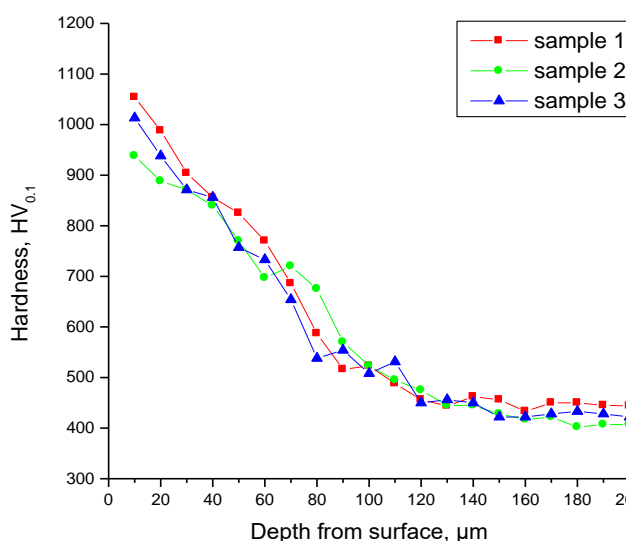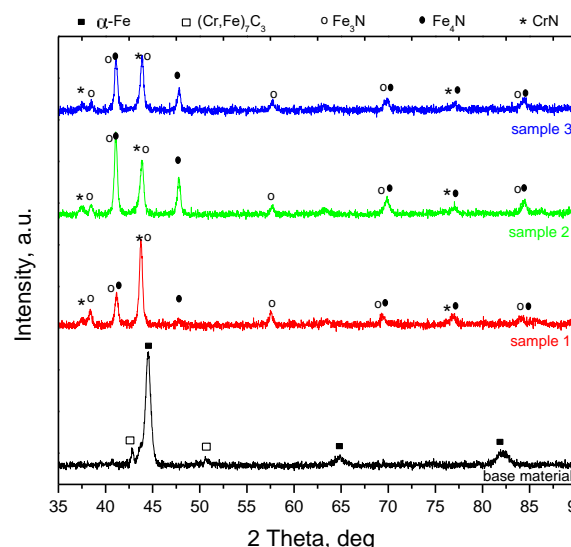
**Figure 2.** Hardness profiles of nitrided samples.



**Figure 3.** XRD of reported nitrided samples.

**Table 1.** Phase composition analysis of nitrided samples.

| | Nitriding parameters (RFPG power / bias voltage) | Phase (%) | | |
| --- | --- | --- | --- | --- |
| | | $Fe_3N$ | $Fe_4N$ | CrN |
| sample 1 | 700 W / 300 V | 82.2 | 10.6 | 7.2 |
| sample 2 | 800 W / 250 V | 62.2 | 29.2 | 8.6 |
| sample 3 | 900 W / 200 V | 51.2 | 33.2 | 15.6 |

The initial H12MF steel dissolution occurs first of all, and pitting is formed on the surface in areas with structural defects (Figure 4b). In the case of samples after nitriding, an improvement in corrosion resistance is achieved by forming a dense layer of nitrides. Analysis of the polarization curves showed that the formation of the nitride layer on the surface of H12MF steel leads to a shift in the value of the corrosion potential $E_{cor}$ to the region of positive values. So, for the state before nitriding, the corrosion potential was -90 mV after nitriding, it varies in the range from 820 mV to 960 mV depending on the process parameters (Figure 4a). This change in potential indicates an improvement in corrosion resistance (Figure 4c). The study of the surface morphology of the samples showed the presence of traces of pitting corrosion, both before and after nitriding.

It should be noted that the samples in the initial state also underwent continuous corrosion, which explains the difference in the values of the current density before and after nitriding. After surface modification, the current density decreases. The greatest potential for corrosion corresponds to the nitriding mode when the power of radio-frequency plasma generator is 900 W. The current density, which characterizes the intensity of dissolution, in this case corresponds to the minimum values, which indicates a higher corrosion resistance. Nitriding at a lower power of 700 W leads to a lower performance corrosion potential and high performance of current density. A decrease in corrosion resistance with an increase in the power of the radio-frequency plasma generator from 700 to 900 W is associated with an increase in the proportion of CrN and $\gamma'$-$Fe_4N$ in the modified layer [6].
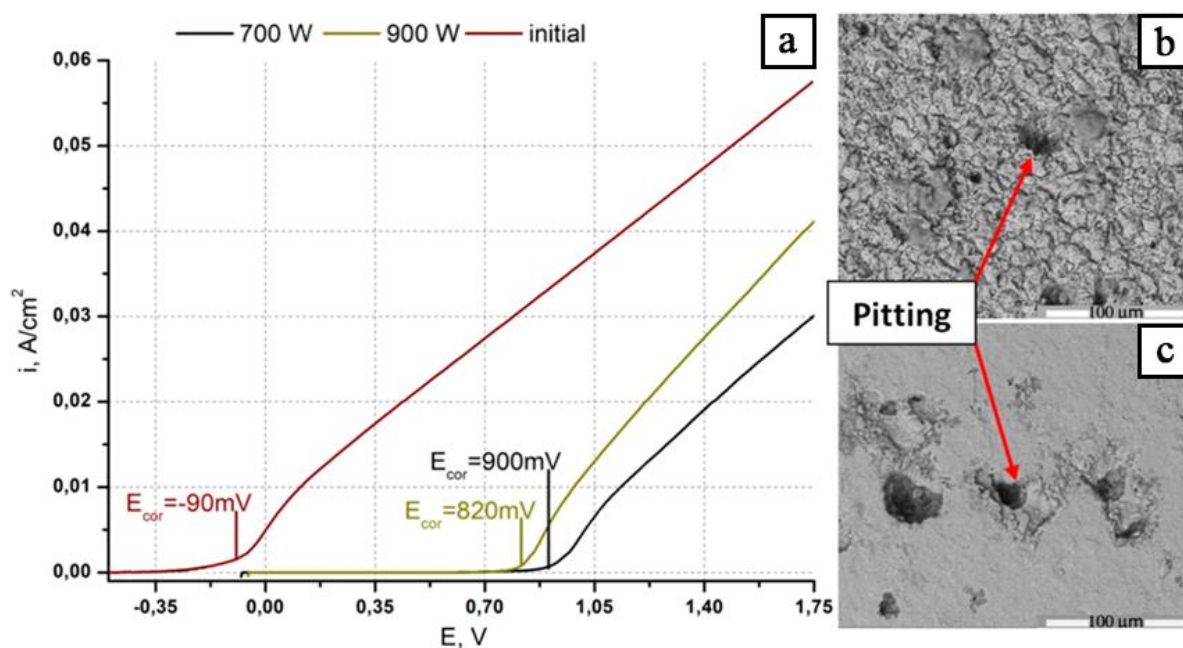
**Figure 4.** Potentiodynamic polarization curves (a) and surface SEM images of nitrided samples after corrosion test: initial (b); 700W (c).

Thus, with an increase in the power of the radio-frequency plasma source and a simultaneous decrease in the bias potential on the sample at a constant temperature of the process, an increase in the content of CrN and $\gamma'$-$Fe_4N$ in the surface layer occurs, which is accompanied by an increase in corrosion resistance while maintaining without significant changes in the thickness of the modified layer, determined mainly by diffusion processes.

**References**

[1] Ramírez G, Mestra A, Casas B, Valls I, Martínez R, Bueno R, Góez A, Mateo A and Llanes L 2012 *Surf. Coat. Tech.* **206** (13) 3069
[2] Atık E, Yunker U and Meriç C 2003 *Tribology International* **36** (3) 155
[3] Wen D C 2009 *Appl. Surf. Sci.* **256** (3) 797
[4] Díaz-Guillén J C, Naeem M, Hdz-García H M, Acevedo-Davila J L, Díaz-Guillén M R, Khan M A, Iqbal J and Mtz-Enriquez A I 2020 n *Surf. Coat. Tech.* **385** 125420
[5] Berlin E V, Grigoriev V Y and Seidman L A 2018 *Inductive Sources of High-density Plasma and their Technological Applications* (Moscow: Technosphere) p 6
[6] Hubicki R, Richert M and Wiewióra M 2020 *Materials* **13** 2311