Ion-plasma nitriding of austenitic steel in a low-pressure low-frequency inductive discharge with ferrite core

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Abstract. An experimental investigation of ion-plasma nitriding of austenitic stainless steel AISI 321 in a low-frequency (100 kHz) nitrogen inductive discharge has been performed for the nitrogen pressure of 7 Pa, nitrogen ion densities of $10^{10} - 10^{11}$ cm$^{-3}$, sample temperatures of 440–590 °C, the densities of current on the sample surface of 1.2–3.3 mA/cm², sample biases of -500 and -750 V. The time of ion-plasma treatment was 20 and 60 min. It is shown that even for the short (20 min.) ion-plasma treatment in the low-frequency inductive discharge, formation of nitrided layers with the thickness of up to 40 μm and microhardness of up to 9 GPa is observed.

1. Introduction

New plasma nitriding technologies based on the use of “external” low-pressure, high-density plasma sources for remote generation of nitrogen ions allow to enhance the speed of nitrided layer formation increasing its thickness and give more flexibility to control processing parameters [1, 2]. Thus, increasing the lifetime and efficiency of such plasma sources is an important task. Inductively coupled plasma sources (ICP) having high lifetime and high ion density at low pressures seem to be a good choice for ion-plasma treatment but have a few disadvantages: high current frequency (~1–10 MHz), low power factor of ICP coil (cosφ<1) and thus high power losses in the ICP coil and matching network [3]. Using closed ferrite cores to improve magnetic coupling between the inductor and plasma in ICP allows to create new low-pressure, high density inductively coupled plasma sources with a low current frequency (~10–100 kHz) and a high power factor of ICP coil (cosφ≈1), thus with a good power transfer from power supply into plasma [3]. Such low-frequency inductive discharges could be used in many areas of plasma processing [3], including ion-plasma nitriding technologies. The aim of this work is to investigate the process of ion-plasma nitriding in the low-pressure, low-frequency inductive discharge improved with ferrite cores for the case of austenitic stainless steel, which is quite hard to be nitried [1, 2].

2. Experimental setup

A scheme of experimental setup is shown in figure 1. Gas discharge chamber 1 is made of stainless steel water cooled sections with the inner diameter of 230 mm. The sections are sealed and dielectrically separated with silicon rubber gaskets. The length of the chamber is 1 meter. A narrow (ID of 40 mm) U-shaped part 2 of the discharge chamber, along with the main part of the discharge
chamber 1, forms a toroidal current path for the low frequency inductive discharge. Ferrite cores 3 significantly enhance magnetic coupling between the inductive discharge and inductor 4, so a power supply with a current frequency of 50–100 kHz 5 can be used for discharge generation. The power supply is connected to the inductor through a matching network 6. Rogowsky coil 7 is used to measure the discharge current. Voltmeter 8 is used to measure the discharge voltage and to determine the electric field strength in the discharge chamber. MKS baratron 626a 9 is used to determine the nitrogen pressure. Double electric probe 10 is used to determine the electron density of plasma. Stainless steel samples 11 with the size of 20x20x1.5 mm (12X18H10T austenitic stainless steel, an analog of AISI 321) are placed inside the discharge chamber. The sample temperature is measured with a thermocouple 12. Sample bias is regulated with a DC power supply 13. To pump out the discharge chamber, a fore pump 14 is used.

To measure microhardness of nitrided layers, cross-sections of samples are prepared. Microhardness is measured with a Wolpert micro vickers tester 402MVD with a load of 10 g. To determine the microhardness, a few measurements are done in a middle part of a layer, and then an averaged value of microhardness is calculated. The microstructure analysis is carried out by X-ray diffraction (ARL X'TRA). An optical microscope (Carl Zeiss Axio Observer A1m) is used to observe the morphology and thickness of the nitrided layers.

![Experimental setup](image)

**Figure 1.** Experimental setup.

1 – Gas discharge chamber (internal diameter of 230 mm), 2 – U-shaped part of the discharge chamber (ID of 40 mm), 3 – ferrite cores, 4 – inductor (ICP coil), 5 – power supply (100 kHz, 500 V), 6 – matching network (variable LC circuit), 7 – current transformer (Rogowsky coil), 8 – voltmeter, 9 – MKS baratron 626a, 10 – double electric probe, 11 – stainless steel sample, 12 – thermocouple, 13 – DC power supply, 14 – forevacuum pump.

3. Experimental results and discussion

Ion-plasma nitriding of the stainless steel samples has been performed for the nitrogen pressure of 7 Pa. The current of the low-frequency inductive discharge was varied in the range of 7–60 A (discharge power of 1300–6400 W), which corresponds to the ion densities in the range of $10^{10}–10^{11}$ cm$^{-3}$. These ion densities are comparable with the ion density in a hot-cathode gas arc (nitrogen pressures of 0.1–0.6 Pa, discharge current of 100 A), which was used for plasma nitriding of stainless steels 12X18H10T [1] and AISI 304 [2]. It is necessary to underline that the absence of electrodes in the low-frequency inductive discharge removes limitation on the discharge current strength and allows to increase the current strength and nitrogen ion density up to significantly higher levels.

Figures 2, 3 show the optical micrographs of a cross-section of nitrided samples, treated for 20 min. under DC sample bias of -500 V and nitriding temperatures of 470–590 °C. Even for the short time of treatment, nitrided layers with the thickness of about 20–40 μm and microhardness of up to 8–9 GPa are observed. For comparison, hot-cathode arc treatment [1] for the same time under nitriding temperature of 520 °C caused only insignificant (up to 2.5 GPa) hardening of the surface layer, and the nitrided layer thickness was only 9 μm. This difference may be caused by a lower sample bias used in [1] (-300 V) and a lower current density on the sample surface respectively [4]. It is necessary to
underline that short time nitriding of austenitic stainless steel using a “standard” DC glow discharge treatment produces only very thin nitrided layers of a few micrometers [5].

**Figure 2.** Cross-section of a nitrided sample, treatment time 20 min. Nitriding temperature 470 °C, sample bias -500 V, sample current density 3.2 mA/cm², layer microhardness 6.8 GPa.

**Figure 3.** Cross-section of a nitrided sample, treatment time 20 min. Nitriding temperature 590 °C, sample bias -500 V, sample current density 3.3 mA/cm², layer microhardness 8.1 GPa.

Figures 4, 5 show the optical micrographs of a cross-section of nitrided samples treated for 60 min. under DC sample bias of -750 V and nitriding temperatures of 440–565 °C. For 1 hour time of treatment, nitrided layers with the thickness of up to 33 μm and microhardness of up to 13 GPa are observed. In this case, the sample current density was a bit lower (1.2–2.4 mA/cm²), even for a higher sample bias, due to a lower discharge current and nitrogen ion density respectively.

**Figure 4.** Cross-section of a nitrided sample, treatment time 60 min. Nitriding temperature 440 °C, sample bias -750 V, sample current density 1.2 mA/cm², layer microhardness 7.4 GPa.

**Figure 5.** Cross-section of a nitrided sample, treatment time 60 min. Nitriding temperature 565 °C, sample bias -750 V, sample current density 2.4 mA/cm², layer microhardness 10.3 GPa.

For comparison, hot-cathode arc treatment [1] for the same time under DC sample bias of -300 V and nitriding temperature of 520 °C formed a nitrided layer with the thickness of 24 μm and microhardness of about 9 GPa (stainless steel 12X18H10T). For stainless steel AISI 304 treated under nitriding temperature of 580 °C, sample bias of -700 V and sample current density of 3.2 mA/cm², the nitrided layer with the thickness of 48 μm and microhardness of about 12 GPa was observed [2]. DC glow discharge nitriding during 1 hour under the same temperatures produces layers with the thickness of about 10 μm [5] and microhardness of about 10 GPa. Thus, comparing the low-frequency ICP nitriding with the treatment in the hot-cathode gas arc [1, 2] and the “standard” DC glow discharge nitriding [5], we observe a significant enhancement in the speed of nitriding of austenitic stainless steels for the case of “external” high-density plasma sources. In the case of short-time treatment (20 min.), the best results are achieved for the low-frequency ICP nitriding, probably due to a high current
density (up to 3.3 mA/cm²) on the sample surface. The same sample current density was achieved in [2], but unfortunately the authors in [2] did not present data for 20 min. treatment.

Figure 6 shows X-ray diffraction patterns of the surface of treated samples for various treatment conditions (nitriding temperature, sample bias, current density on the sample surface, and time of treatment). Also, the resulting layer microhardness is shown.

![Figure 6](image_url)

Figure 6. The X-ray diffraction patterns of treated samples.
1 – 470 °C, -500 V, 3.2 mA/cm², 20 min., 6.8 GPa;
2 – 590 °C, -500 V, 3.3 mA/cm², 20 min., 8.1 GPa;
3 – 440 °C, -750 V, 1.2 mA/cm², 60 min., 7.4 GPa;
4 – 530 °C, -750 V, 2.0 mA/cm², 60 min., 12.9 GPa.

As it is seen from figure 6, in the case of the short-time treatment, X-ray diffraction patterns have peaks related to the austenitic phase (γ) with a face centred cubic structure, and peaks related to chromium nitride. The presence of the austenitic phase indicates that nitriding process is not completed. With increasing the treatment time up to 60 min. and nitriding temperature up to 530 °C, the peaks of austenitic phase completely disappear. For 60 min. treatment, X-ray diffraction patterns have peaks related to the nitrogen expanded austenitic phase (γN), iron nitride Fe₃N and chromium nitride CrN (the same peaks were found in [2]). It is followed by the increase in the layer microhardness (from 7–9 GPa for 20 min. treatment up to 10–13 GPa for 60 min treatment).

4. Conclusion
A new method of ion-plasma nitriding based on the use of the low-pressure low-frequency inductively coupled plasma enhanced with ferrite cores as an “external” source of nitrogen ions has been tested. Austenitic stainless steel 12X18H10T (an analog of AISI 321) was treated in the temperature range of 440–590 °C, sample biases of -500 and -750 V, current densities on the sample surface of 1.2–3.3 mA/cm², for 20 and 60 min. It is found that even the short (20 min) ion-plasma treatment results in the formation of the nitried layers with the thickness of about 20–40 µm and microhardness of about 7–9 GPa (depending on the nitriding temperature). The significant enhancement in the speed of nitriding (in comparison with other methods) can be explained by a high nitrogen ion density (~10¹¹ cm⁻³) in the low-frequency ICP, and high current density on the sample surface respectively. The further increase in the ion density (up to 10¹²–10¹³ cm⁻³) is still possible, and it may result in a further increase in the nitriding speed.

References
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Acknowledgments
This work was fulfilled in the framework of the budget project III.18.2.2.