



# Characteristics of the nitrated layer formed on AISI 304 austenitic stainless steel by high temperature nitriding assisted hollow cathode discharge



Yang Li<sup>a,\*</sup>, Shangzhou Zhang<sup>a</sup>, Yongyong He<sup>b</sup>, Lei Zhang<sup>a</sup>, Liang Wang<sup>c</sup>

<sup>a</sup> Department of Materials Science and Engineering, Yantai University, Yantai 264005, PR China

<sup>b</sup> State Key Laboratory of Tribology, Tsinghua University, Beijing 100084, PR China

<sup>c</sup> Department of Materials Science and Engineering, Dalian Maritime University, Dalian 116026, PR China

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## ABSTRACT

A series of experiments have been conducted on AISI 304 stainless steel using a hollow cathode discharge assisted plasma nitriding apparatus. Specimens were nitrated at high temperatures (520–560 °C) in order to produce nitrogen expanded austenite phase within a short time. The nitrated specimen was characterized by scanning electron microscopy, X-ray diffraction, X-ray photoelectron spectroscopy, potentiodynamic polarization and microhardness tester. The corrosion properties of nitrated samples were evaluated using anodic polarization tests in 3.5% NaCl solution. The nitrated layer was shown to consist of nitrogen expanded austenite and possibly a small amount of CrN precipitates and iron nitrides. The results indicated that rapid nitriding assisted hollow cathode discharge not only increased the surface hardness but also improved the corrosion resistance of the untreated substrate.

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## 1. Introduction

Austenitic stainless steels offer good resistance to general corrosion due to the formation of a passive surface film [1]. They are widely used in the food and chemical processing industries as well as in biomaterial applications [2]. However, they can suffer from pitting corrosion in chloride ion containing solutions [3]. Nevertheless, at the same time they have found little use in mechanical engineering applications because of their low hardness and poor wear resistance [4,5]. For these reasons, surface hardening by nitriding has long been a well-known thermo-chemical treatment for improving the surface properties of austenitic stainless steels [6,7]. Low temperature plasma nitriding can produce a new phase with high hardness and good corrosion resistance on austenitic stainless steel surfaces [8]. It does so by the formation of a non-equilibrium supersaturated layer, what is called ‘S-phase’ or expanded austenite ‘ $\gamma_N$ -phase’ [7]. Zhang and Bell [9] described this surface layer as being hard and corrosion resistance with anticorrosion properties equivalent to the original material. Nitrogen remains in solid solution inside the  $\gamma_N$  phase without removing chromium from the austenitic structure by precipitation of Cr nitrides. Since then, many researchers have produced such

modified layer on austenitic stainless steels by various nitriding processes at low temperatures (<450 °C) [8]. These production methods include glow discharge plasma nitriding [10], plasma immersion ion implantation [11], plasma source ion nitriding [12], active screen plasma nitriding [13], and reactive magnetron sputtering [14].

It is generally thought that higher temperatures may cause precipitation of CrN, thus removing Cr from the solid solution and degrading the corrosion resistance of the materials [15]. Li [16] stated that the precipitation of CrN occurs, above the nitriding treatment temperatures of 420–440 °C for the AISI 304L austenitic stainless steel. In plasma immersion ion implantation, Collins et al. [17] found that the  $\gamma_N$  phase layer was formed on the 450 °C implanted sample whereas at 520 °C, the precipitation of CrN and  $\alpha$ -ferrite dominated. In the investigation of Lei [18], austenitic stainless steels were plasma source ion nitrated at various process temperatures from 230 to 480 °C for 4 h. The  $\gamma_N$  phase with the different nitrogen contents was formed in the range of 300–450 °C, however, the  $\gamma_N$  phase decomposed with precipitation of chromium nitride CrN above 450 °C. Therefore, in order to avoid the detriment to the corrosion resistance of austenitic stainless steels, these nitriding treatments performed at temperatures lower than 450 °C. However, a long nitriding time is necessary to obtain a sufficiently thick  $\gamma_N$  phase layer by low temperature nitriding techniques.

\* Corresponding author. Tel.: +86 535 6706050; fax: +86 535 6706038.

E-mail addresses: [liyang@ytu.edu.cn](mailto:liyang@ytu.edu.cn), [metalytu@163.com](mailto:metalytu@163.com) (Y. Li).

Hollow cathode discharges (HCD) are used in a wide variety of applications in plasma processing (ion etching, thin film deposition, surface treatment), where the hollow cathode is used as an emission source [19]. In this research, a double cylinders active screen was designed. Using hollow cathode discharge effect exiting between inner and outer cylinders is easily to be heated to higher temperature and increase the working space to required temperature rapidly.

In this study, we reported the results of high temperature (520–560 °C) plasma nitriding of AISI 304 austenitic stainless steel within short time. It showed that high temperature plasma nitriding with hollow cathode discharge can also produces a phase layer without precipitation of chromium nitride for a short time (0.5 h). However, most publications reported that the long nitriding times (>2 h) is necessary to obtain a similar thick  $\gamma_N$  phase layer by low temperature (<450 °C) nitriding techniques. The aim of this work is to study the influence of high temperature nitriding assisted hollow cathode discharge on the microstructure, chemical composition and the corrosion behavior of the AISI 304 austenitic stainless steel.

## 2. Experiments

The samples used in this work was AISI 304 austenitic stainless steel with the following chemical compositions (wt.%): C < 0.05, Cr 18.0–18.9, Ni 8.80–9.20, Si 0.80–1.00, Mn 1.60–2.00, S < 0.02, P < 0.02, and Fe balance. Samples (15 × 15 × 4 mm<sup>3</sup>) were cut from a hot rolling plate, ground and mirror polished then cleaned with acetone before nitriding.

Fig. 1 shows a schematic diagram of the ion nitriding setup with multiple hollow cathodes. The active screen consists of two cylinders with different diameter. The diameter of inner cylinder is 460 mm and that of the outer cylinder is 488 mm. The cylinders were placed on the worktable and connected to the cathodic potential. The space between two cylinders is 8 mm. These two cylinders made up a hollow cathode in a discharge system [20]. They can also be heated rapidly to the required temperature for treatment. The screen was made of 6 mm thick perforated austenitic stainless steel plate, which had uniformly distributed round holes of 15 mm in diameter. The samples were set on a plate of cathodic potential. The temperature was obtained by an electrically isolated thermocouple attached to the sample holder. The nitrided samples were cooled in vacuum conditions after finishing the nitriding process. Table 1 lists the parameters for the plasma nitriding treatments investigated in this study. Some samples were nitrided at different temperatures for 30 min, which were to study the influence of the treatment temperature on the microstructure, the morphology, the hardness properties and the corrosion

**Table 1**  
Processing parameters of plasma nitriding of stainless steel.

| Condition           | Parameter         |
|---------------------|-------------------|
| Temperature (°C)    | 520, 540, 560     |
| Time (min)          | 5, 15, 30, 40, 60 |
| Gas                 | NH <sub>3</sub>   |
| Pressure (Pa)       | 500               |
| Voltage (V)         | 550–700           |
| Current density (A) | 4–8               |

behavior of the austenitic stainless steel nitrided by hollow cathode discharge-assisted process. Other samples were nitrided at temperature 540 °C for the different times, it is to evaluate the effect of time of nitrided behavior of austenitic stainless steel at high temperature.

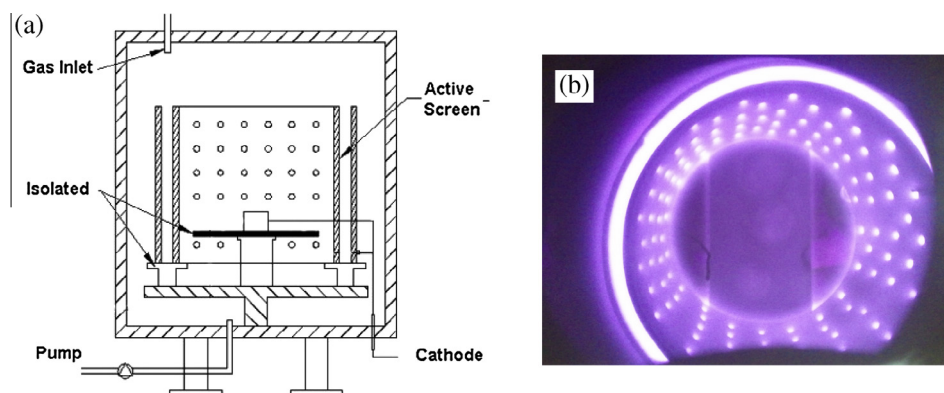
The samples were etched using a chemical solution of (CuSO<sub>4</sub> · 5H<sub>2</sub>O 5 g + HCl 20 ml + H<sub>2</sub>O 20 ml) to reveal the nitrided layer. The cross-sectional micrographs were observed by scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDX). The phase compositions of the nitrided layer were determined using a D/MAX-Ultima<sup>+</sup> X-ray diffractometer (XRD) in the  $\theta$ - $2\theta$  geometry, with Cu K $\alpha$  radiation. The surface hardness was measured by a Vickers' indenter with the load of 25 gf.

The surface chemical characterization of the nitrided samples was determined by X-ray photoelectron spectroscopy (XPS, Escalab250, Thermo Corp). XPS measurements were performed using an X-ray source of AlK $\alpha$  (1486.6 eV). Measured binding energies were corrected by referring to the binding energy of the C1s neutral carbon peak at 284.6 eV. The etching rate was approximately 0.02 nm/s. The specimen was sputtered by Ar ions. The experimental data were analyzed by XPSPEAK4.1 software.

The corrosion resistance of untreated and nitrided samples was evaluated by measuring polarization curves in 3.5% NaCl solution using ZAHNER IM6e electrochemical working station. The flat cell which is a three-electrode set-up consists of the specimen as the working electrode, a saturated calomel electrode (SCE) as the reference electrode, and a platinum sheet used as the counter electrode. The scan potential was in the range of –800 to 1000 mV (vs. SCE), and the scan rate was 1 mV/s. Each test was repeated at least three times in order to assure its reproducibility. At the end of each type of corrosion test microscopy analysis was carried out in order to evaluate the corrosion morphology and the damaging amount.

## 3. Results and discussion

Fig. 2 presents the cross-section optical micrographs of the samples nitrided at 520, 540, and 560 °C for 30 min. It could be



**Fig. 1.** Schematic diagram of the nitriding apparatus (a) and digital photograph of the hollow cathodic discharge (b).

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