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The Study of Corrosion Behavior of Active Screen Plasma Nitrided Stainless Steels

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Plasma nitriding is a surface treatment process which is increasingly used to improve wear, fatigue and corrosion resistance of industrial parts. Active screen plasma nitriding (ASPN) has both the advantages of the classic cold wall and the hot wall conventional dc plasma nitriding (DCPN) method and the parts to be nitrided are no longer directly exposed to the plasma. In this study, AS plasma nitriding has been used to nitride the UNS S31803 duplex stainless steel, AISI 304 and AISI 316 austenitic stainless steel, and AISI 420 martensitic stainless steel. Treated specimenswere characterized by means of microstructural analysis, microhardness measurements and electrochemical tests in NaCl aerated solutions. Hardness of the nitride cases of AISI 420 stainless steel by Knoop test can get up to 1300 HK0.1. From polarization tests, the corrosion current densities of AISI 420 and UNS S31803specimens ASPN at 420 °C were generally lower than those of their untreated substrates. The corrosion resistance of UNS S31803 duplex stainless steel can be enhanced by plasma nitriding at 420 °C Cowing to the formation of the S-phase.

Keywords: *Stainless steel, UNS S31803, Active screen plasma nitriding, Corrosion.*

1. Introduction

Active screen plasma nitriding is a thermochemical process to improve surface properties, such as the metallurgical, mechanical, and tribological properties, of various engineering materials. The "Active Screen" or cage is installed inside the plasma processing chamber constituted the hot wall of the vessel, and surrounds all parts to be nitrided. Enclosing inside the screen, the workload is heated up by radiation, analogous to cold wall vacuum furnace heating methods, providing a very uniform temperature throughout the entire load. Moreover, the "Active Screen" produces all necessary active and neutral particles necessary to nitride steels, titanium and its alloys.

Plasma nitriding involves the addition of nitrogen into the surface of a treated component. With typically treatment temperature between 450 °C and 590 °C, plasma nitriding is a ferritic treatment and as such produces virtually no distortion to treated components. After plasma nitriding on the stainless steels, the improvement of the wear resistances is accompanied with the reduction of its corrosion resistances, due to chromium nitrides precipitation and depletion of chromium in the substrate.[1-3] It has been observed that plasma nitriding at low temperature on austenitic stainless steel produces a hard layer with good corrosion resistance. This layer contains a super saturated solid solution of nitrogen (N) in austenite, which is formed as the expanded austenite called S-phase or γ_N.[3-6]

During active screen (AS) plasma nitriding, the work table and the parts to be treated are placed in a floating potential without sputtering on the sample surface. Active screen plasma nitriding under similar operating parameters has resulted in identical microstructure, case hardness and case depth as conventional dc plasma nitriding.[7,8] However, the surface morphologies of the AS plasma nitrided specimens have been observed to be significantly different from those of the dc plasma nitrided specimens[9].

Increasing interest in microstructure and wear resistance of plasma nitrided stainless steels has been shown in recent literatuer.[10-11] However, the corrosion behavior of the nitrided layers is not yet available. In this study, the effect of the processing temperature on the microstructure and corrosion characteristics of the plasma nitrided layers produced on the stainless steels has been investigated in detail.

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2. Experimental

Specimens in this study were taken from AISI 304 austenitic stainless steel, AISI 316 austenitic stainless steel, AISI 420 martensitic stainless steel and UNS S31803 duplex stainless steel. The chemical compositions of the specimens are listed in Table 1. AISI 420 martensitic stainless steel was heated at 1030 °C for 30 minutes, oil quenched and tempered at 510 °C for 2hrs. Cylindrical specimens were shaped to the dimension of Φ30 mm×h5 mm. Specimens were further polished by abrasive papers to 1500-grit, and ultrasonically cleaning in an acetone solution.

Active screen plasma nitriding was carried out in a dc nitriding furnace as shown in Fig. 1. The nitriding process was conducted at 420 °C and 450 °C for 10hrs and 25 hrs in the glow discharge of the plasma. The vacuum chamber was pumped down to 6 Pa and back filled with treatment atmosphere of 50%N_2+50%H_2 at a pressure of 560 Pa. The cross-sectional microstructures of the specimens were observed by optical microscopy (OM) and scanning electron microscopy. The phase of the nitrided layer on the specimen surfaces was characterized by an X-ray diffractometer using Cu-Kα radiation. Microhardness measurement was performed by using a Vickers microhardness tester. Potentiodynamic polarization measurements were performed with a conventional three electrode cell housing working electrode, the platinum counter electrode, and the saturated calomel reference electrode in a 3.5% NaCl solution at 25 °C. The potentiodynamic scan was recorded from corrosion potential 0.6 to 0.7V, at a scan rate of 1 mV/s.

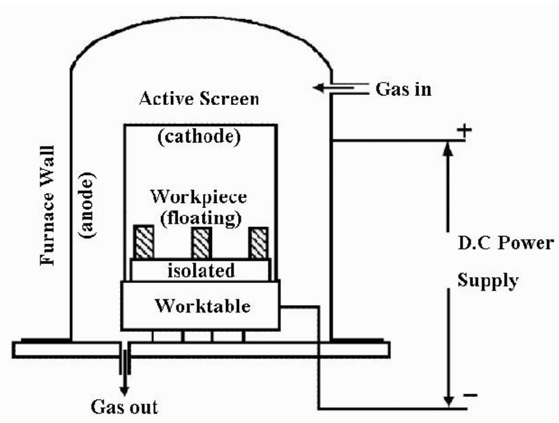

Fig. 1. Schematic diagram of ASPN facility

3. Results and Discussion

3.1 Microstructures

Fig. 2 shows the cross sectional micrographs of nitrided layers produced on UNS S31803 duplex stainless steel by the ASPN treatment. It can be observed that increasing in treatment temperature and time resulted in increment in the nitriding layer thickness from 7 μm to 18 μm. As can be seen in these figs., the α and γ phase can be easily identified. For the specimens treated at 420 °C for 10hrs, the nitrided layers were observed as a single phase formation. However, as the specimens treated for 25 hrs, it can be seen that the layers influenced by the substrates transformed to compound phases.

Fig. 3 compares the nitrided layer thickness of various AS plasma nitrided materials at 420 °C and 450 °C for 10 hrs and 25 hrs. The thickness increase of the nitrided zone was depended on the microstructure of the substrate, the nitriding time, the nitriding temperature, and the Cr content. The nitrided layer thickness of AISI 420 martensitic stainless steel specimen has achieved to 40 μm when nitriding treatment was carried out at 450 °C for 25 hrs.

3.2 XRD examinations

Fig. 4 shows the X-ray diffraction patterns of the ASPN AISI 316 stainless steel specimens. In agreement to Li et al.,[3] the result demonstrated that the S phase was detected on the specimens nitrided at 420 °C but absent at 450 °C.

Fig. 5 shows the X-ray diffraction patterns of the ASPN UNS S31803 duplex stainless steel specimens. The phases of the specimen nitrided at 420 °C for 10hrs were identified to be γ_N with small amounts of Fe_3N on the surface. The amount of the precipitates increases with increasing

Table 1. Chemical composition (wt%)

Material	C	Si	Mn	Cr	Ni	Mo
UNS S31803	0.029	0.50	1.52	22.88	5.75	2.99
AISI 316	0.049	0.13	1.95	17.58	11.89	2.53
AISI 304	0.037	0.55	1.36	17.78	8.00	0.04
AISI 420	0.22	0.65	0.93	12.30	0.33	0.02

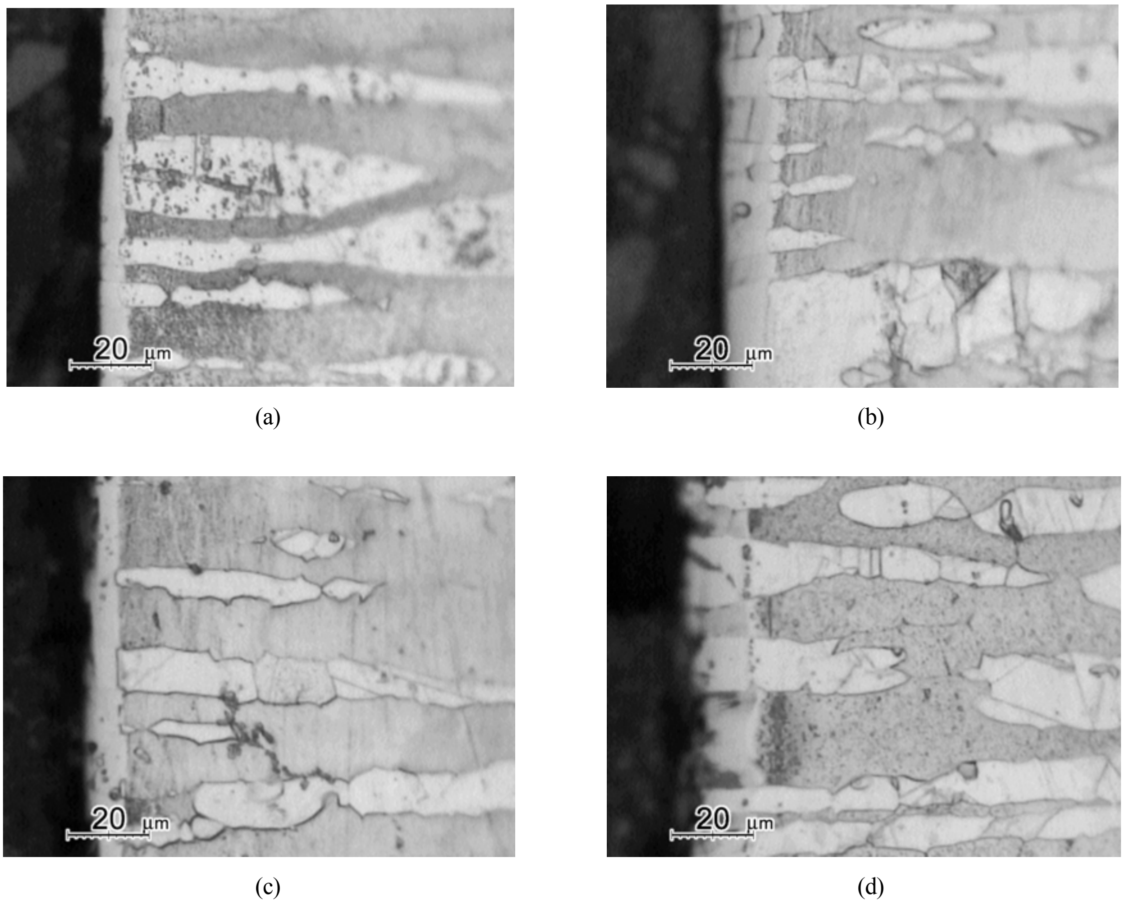

Fig. 2. Micrographs of nitrided layer of UNS S31803 specimens by plasma nitriding at various processing conditions, (a) 420°C, 10h, (b) 420°C, 25h, (c) 450°C, 10h and (d) 450°C, 25h

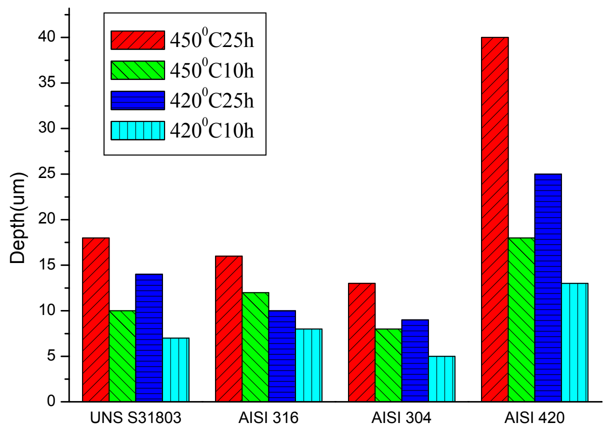

Fig. 3. Thickness of the nitrided layer in dependence on the various stainless steels and nitriding conditions

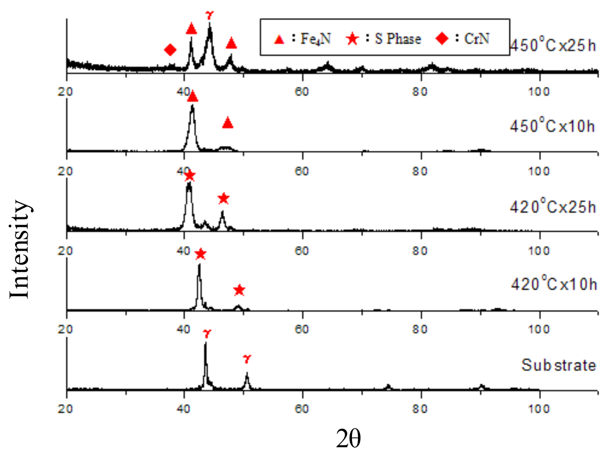

Fig. 4. X-ray diffraction patterns of ASPN AISI 316 stainless steel specimens

treatment time and temperature. CrN precipitates were observed on the specimen nitrided at 450 °C for 25 hrs. It is believed that CrN precipitate has a serious effect on the corrosion resistance of the nitrided specimens.

3.3 EPMA measurements

Fig. 6 shows the electron probe microanalysis (EPMA) image of the nitrided layer on the UNS S31803 specimen

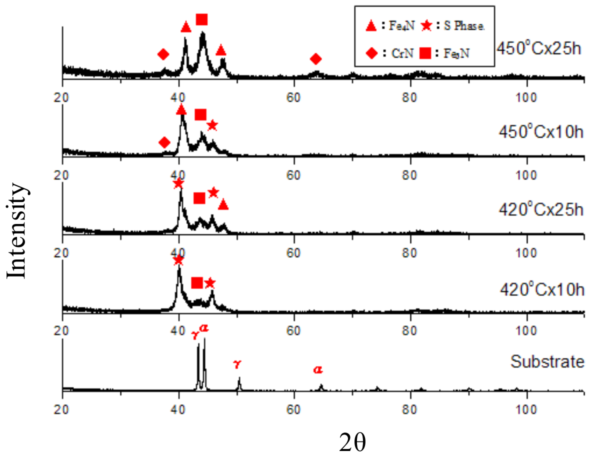

Fig. 5. X-ray diffraction patterns of ASPN UNS S31803 duplex stainless steel specimens

treated at 420 °C for 25 hrs. The amount of nitride precipitates in the ferrite phase was higher than that in the austenite phase. It is due to the nitrogen has rapid diffusion rate in ferrite (BCC) structure than austenite (FCC) structure. This can be ascribed to the nitrogen diffusion length during the plasma process.

Fig. 7 shows the EPMA image of the nitrided layer on the UNS S31803 specimen treated at 450 °C for 25 hrs. A homogenous distribution of N was present over the surface of nitrided layer. As a result of the chromium nitride formation at surface, the nitrided layer depicted a local enrichment of chromium and nitrogen concentration.

3.4 Microhardness

Fig. 8 shows the microhardness of the nitrided layers of various specimens at 420 °C and 450 °C for 10 hrs and 25 hrs. The hardness increases with increasing treatment time and temperature, as the amount of the precipitates increases. The hardness achieved above 1000 $HK_{0.1}$ for specimens nitrided at 450 °C for 25 hrs. The highest hardness was measured to 1300 $HK_{0.1}$ on AISI 420 martensitic stainless steel specimen.

3.5 Corrosion Behavior

Fig. 9 shows the polarization curves of ASPN UNS S31803 duplex stainless steel specimens at 420 °C and 450 °C for 10 hrs and 25 hrs. According to polarization

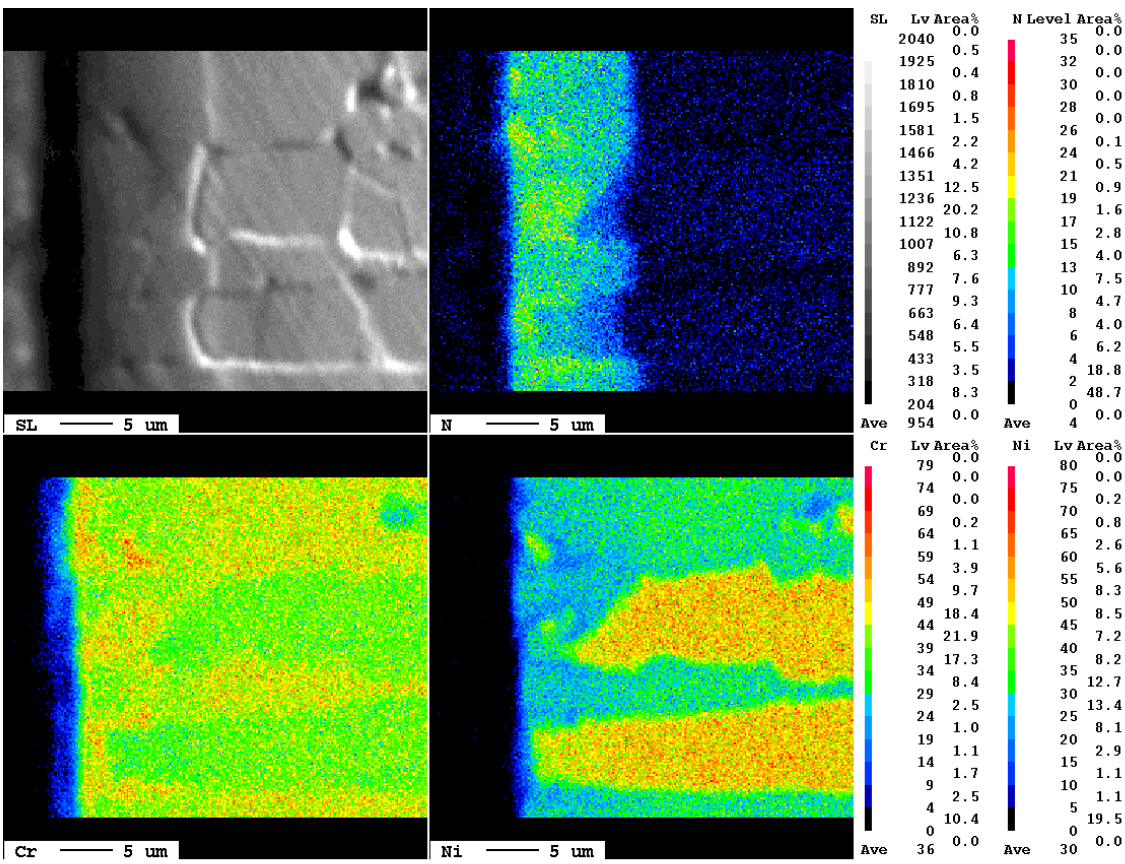

Fig. 6. EPMA image of nitrided UNS S31803 duplex stainless steel at 420°C, 25h

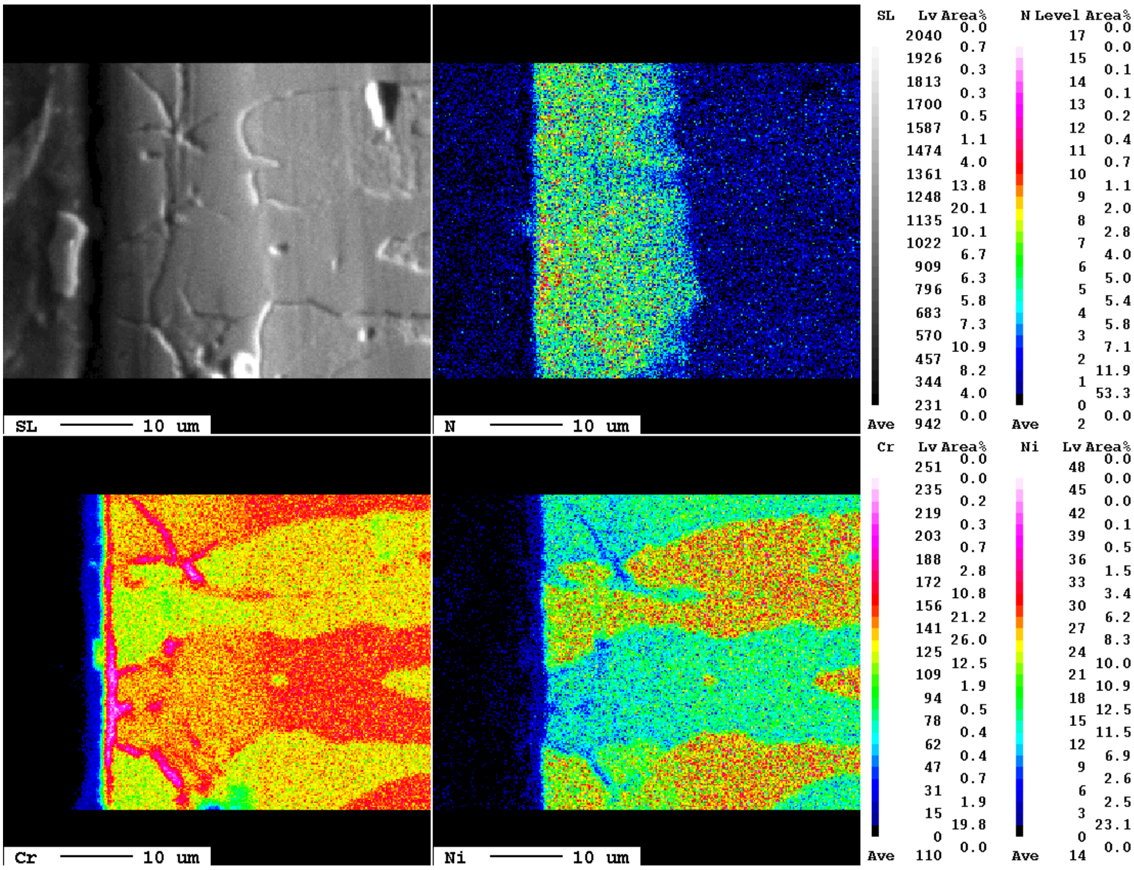

Fig. 7. EPMA image of nitrided UNS S31803 duplex stainless steel at 450°C, 25h

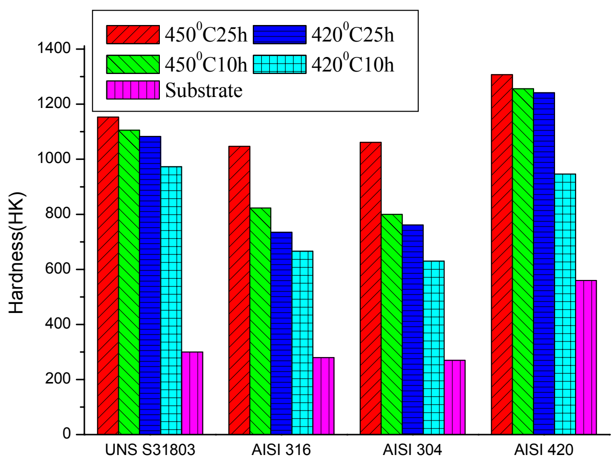

Fig. 8. Microhardness of the nitrided layer in dependence on the various stainless steels and nitriding conditions

data, corrosion potential (E_{corr}) was slightly shifted to a noble direction in specimens treated at 420 °C. Corrosion current density (i_{corr}) is slightly lower than, but close to, that for the untreated substrate. As the nitrided treatments were carried out at 450 °C, corrosion potential (E_{corr}) was shifted to a less noble direction. According to Fig. 2 and 7, chromium and iron nitride precipitates dispersed along the grain boundary, pores and surface, and the amount increased with the increase treatment time. Due to the migration of Cr from solid solution to form CrN precipitates, corrosion resistance of the specimens was deteriorated, as observed by other authors.[12,13] On the other hand, the plasma nitriding at 420 ℃ improves corrosion resistance of UNS S31803 specimens. This can be attributed to formation of γ_N phase in specimens treated at 420 ℃.

Fig. 10 shows the polarization curves of nitrided AISI 420 martensitic stainless steel specimens at 420 °C and 450 °C for 10 hrs and 25 hrs. It can be seen that corrosion potential (E_{corr}) was generally shifted to a less noble direction. Corrosion current densities (i_{corr}) of the specimens nitrided at 420 °C were slightly lower than that of the untreated substrates. The result demonstrated that the specimens nitrided at 420 °C revealed a slight improvement of the corrosion behavior over the untreated substrates. However, the corrosion resistance of AISI 420 stainless steel specimens nitrided at 450 °C was deteriorated.

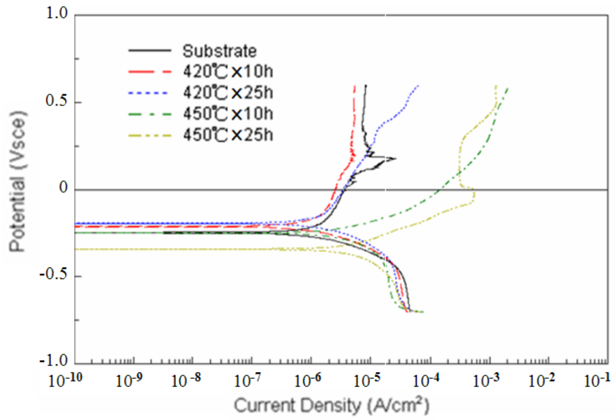

Fig. 9. Polarization curves of nitrided UNS S31803 duplex stainless steel at 420°C and 450°C for 10h and 25h

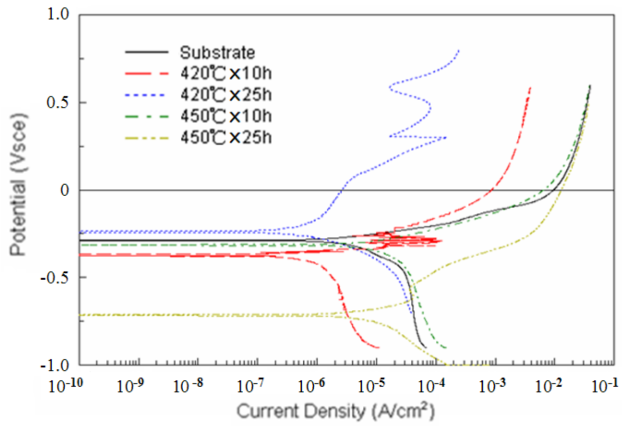

Fig. 10. Polarization curves of nitrided AISI 420 stainless steel at 420°C and 450°C for 10h and 25h

4. Conclusions

The experimental results demonstrated that the layer thickness increased with increasing treatment temperature and time. The main phase produced at 420 °C on the nitrided AISI 316 and AISI 304 stainless steel specimens were identified to be S-phase. The value of surface hardness achieved above 1000 $HK_{0.1}$ for specimens nitrided at 450 °C for 25 hours. The corrosion resistance of UNS S31803 duplex stainless steel can be enhanced by plasma nitriding at 420 °C owing to the formation of the S-phase. However, AISI 420 martensitic stainless steel specimens nitrided at 450 °C for 25 hours exhibited poor corrosion behavior. Plasma nitriding at 420 °C for 10 hours is the optimum treatment for both AISI 420 and UNS S31803 duplex stainless steels to achieve combined improvement in wear and corrosion resistance, and thus improved corrosion wear resistance is anticipated.

Acknowledgments

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