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EFFECT OF GAS PRESSURE ON ACTIVE SCREEN PLASMA NITRIDING RESPONSE[1]

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Abstract

An austenitic stainless steel AISI 304 was active screen plasma nitrided using a 304 steel screen to investigate the effect of the gas pressure on the ASPN response. The sample was treated for 18 ks at 723 K in 25% N_2 + 75% H_2 gases. The gas pressure was changed to 100, 600 and 1200 Pa. The distance between screen and sample was also changed to 10, 30 and 50 mm. The nitrided samples were characterized by appearance observation, surface roughness, optical microscopy, X-ray diffraction, and microhardness testing. After nitriding, polygonal particles with a normal distribution were observed at the center and edges of all the ASPN-treated sample surfaces. Particles on the sample surfaces were finer with an increase in the gas pressure. The nitrided layer with a greater and homogeneous thickness was obtained at a low gas pressure of 100 Pa.

Key words: Surface engineering; Cathodic cage; Edging effect; Active screen plasma nitriding; Stainless steel.

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1 INTRODUCTION

The nitriding process is widely used to improve the tribological properties and wear resistance of steels and titanium alloys. Compared with the conventional nitriding processes such as gas nitriding and salt bath nitriding, the glow-discharge plasma nitriding process offers the following advantages: no pollution, high nitrogen potential, short treatment time, clean environment, and low energy consumption.[1,2] The components to be treated are subjected to a high cathodic potential to produce plasma directly on their surfaces. An “edging effect” occurs due to distortions of the electric field around the corners and edges of the components although the components are well heated. This results in nonuniformity in properties such as the hardness and thickness of the surface layer.[3]

Recently, there has been considerable interest in active-screen plasma nitriding (ASPN), through-cage plasma nitriding (TCPN), and cathodic-cage plasma nitriding (CCPN).[4-15] In these processes, the edging effect is completely eliminated because the plasma is produced on the cage and not directly on the samples.[4] This process can be used to treat polymers that are nonconductive materials.[7,11] However, little information has been reported on the effect of the gas pressure on ASPN properties such as surface morphology, microstructure, and nitrogen depth.

In this study, an austenitic stainless steel sample was nitrided by ASPN to investigate the effect of the gas pressure on ASPN properties.

2 MATERIALS AND METHODS

The sample material used in this study was an austenitic stainless steel AISI 304 (nominal composition (mass%): <0.08% C, <1.00% Si, <2.00% Mn, <0.045% P, <0.03% S, 18.00–20.00% Cr, 8.00–10.50% Ni, balance Fe). The sample disc was 13 mm in diameter and 5 mm in thickness. The sample surface was mechanically ground with 150- to 1500-grit SiC, finely polished with 0.05-μm alumina in suspension, degreased ultrasonically in acetone, and dried in air before placing the sample in the nitriding furnace.

ASPN experiments were carried out using a DC plasma-nitriding unit (NDK, Inc. Japan, JIN-S1). A quartz (SiO_2) rod was placed on the cathodic stage in order to construct an isolated sample stage. The sample was placed on the sample stage in a floating potential and isolated from the cathodic screen and the anode. The screen, which was an expanded metal of AISI 304 with a thickness of 1.5 mm, a diameter of 150 mm, and a height of 155 mm, was mounted on the cathodic stage around the sample stage. The AISI 304 rods were placed on the sample stage so that the distance between the screen and the sample could be changed to 10, 30, and 50 mm. The screen was thoroughly degreased ultrasonically in acetone.

Nitriding was performed in a nitrogen-hydrogen atmosphere with 25% N_2 + 75% H_2 for 18 ks at 723 K under 100, 600, and 1200 Pa by the ASPN process. After placing the sample on the sample stage, the chamber was evacuated at ~3 Pa. Then, nitrogen and hydrogen were introduced into the chamber, and a DC bias voltage was supplied. After nitriding, the DC supply was switched off, and the sample was cooled to room temperature in the furnace.

After nitriding, cross sections of each sample were first cut using a low-speed saw and then polished and chemically etched. The nitrided microstructure was examined with a scanning electron microscope (SEM). The phase structures on the nitrided surface were determined by X-ray diffraction (XRD) studies. In addition, the hardness of the surface and the cross sections of the nitrided sample were measured using a Vickers microhardness tester under a 0.1-N load.

3 RESULTS AND DISCUSSION

AISI 304 samples were plasma nitrided for 18 ks at 723 K under 100, 600, and 1200 Pa pressure in 25% N_2 + 75% H_2 using the ASPN process. After treatment, the appearance of the samples was examined visually. Edging effect was not observed in each sample. This shows that glow discharge did not occur on the sample surface because the sample was isolated by placing SiO_2 ceramics between the sample and the cathodic stage. Furthermore, in the ASPN process, the samples were heated to the treatment temperature by the heat radiated from the active screen, which promoted a higher homogeneity of temperature in the treated samples.[4]

XRD results of the samples treated by the ASPN process are shown in Figure 1. γ-austenite and S phase, which is considered to be a supersaturated solid solution of nitrogen in the austenitic phase, were identified on the sample surface in each condition. The layers formed on each sample were of the same phase irrespective of the gas pressure and the distance between the screen and the sample.

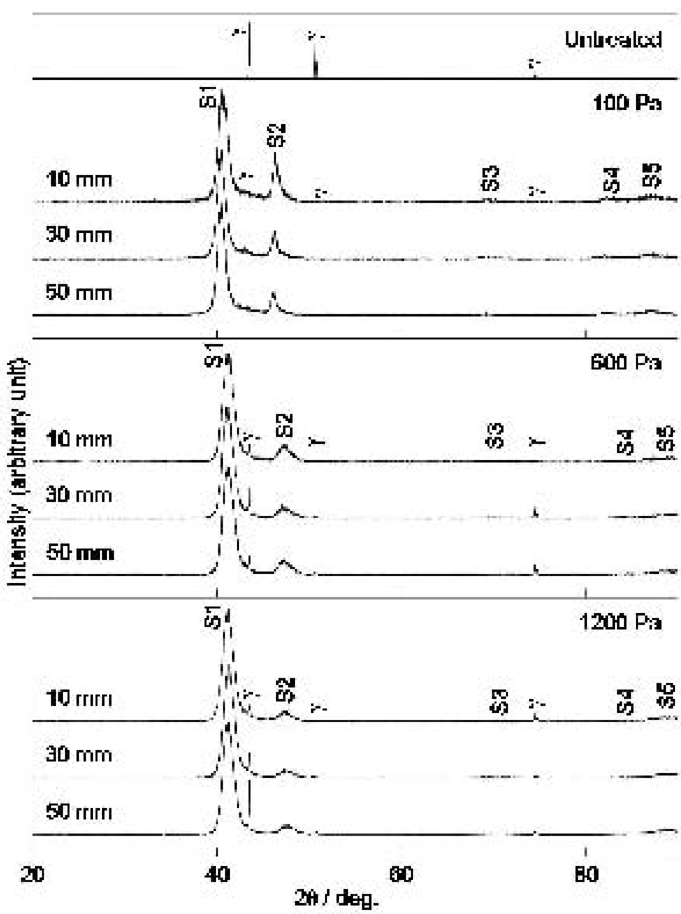

Figure 1. XRD pattern of AISI 304 steel sample treated at 723 K by ASPN.

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Figure 2 shows SEM micrographs of the sample surfaces treated by the ASPN process. The surface morphologies of samples treated at different pressures are different. When the distance between the screen and the sample was 10 mm, a uniform, normal distribution of polygonal particles was observed at the center and edges of the sample surface irrespective of the distance between the screen and the sample. The particle size becomes finer as the gas pressure increases. These results can be attributed to the FeN particles present in the plasma; these particles were formed on the active screen and deposited on the sample surface during the ASPN process. Therefore, FeN deposition increased as the gas pressure decreased, and these deposited particles grew.

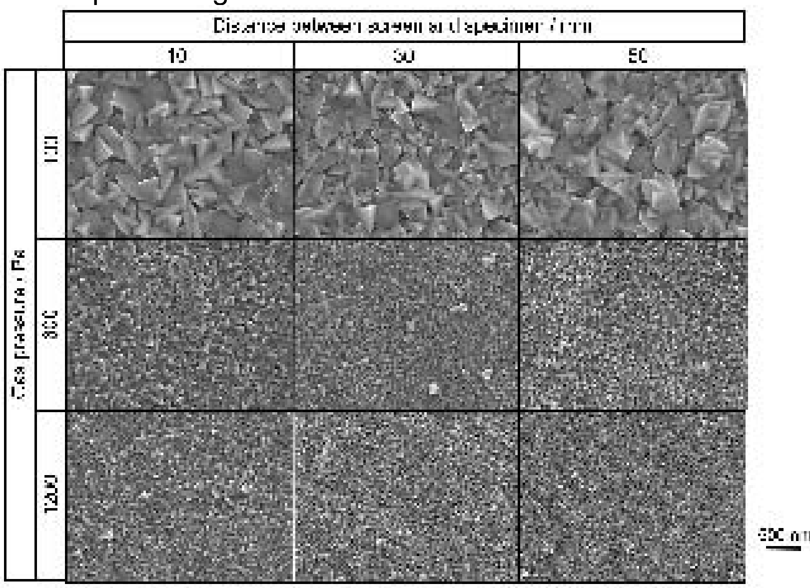

Figure 2. SEM micrographs of AISI 304 steel sample treated at 723 K by ASPN.

The microstructures of the cross sections of the samples treated by the ASPN process are shown in Figure 3. An S-phase layer, which was very slightly corroded by oxalic acid, was observed in the sample surfaces. Figure 4 shows the effect of the gas pressure and the distance between the screen and the sample on the thickness of the nitrided layer. When the samples were treated at 600 and 1200 Pa, the thickness of the nitrided layer decreased as the distance between the screen and the sample increased. This result indicates that the amount of the active species available for nitriding depends on the distance between the cathodic screen and the sample because the sample is isolated by placing SiO_2 ceramics between the sample and the stage. On the other hand, the nitrided layer treated at 100 Pa had the homogeneous thickness irrespective of the distance between the screen and the sample. Moreover, the thickness of the nitrided layer treated at 100 Pa was greater than that treated at 600 and 1200 Pa. These results suggest that the homogeneous temperature distribution expanded with a decrease of the gas pressure. As a result, the nitrided layer with a greater and homogeneous thickness was obtained at a low pressure of 100 Pa.

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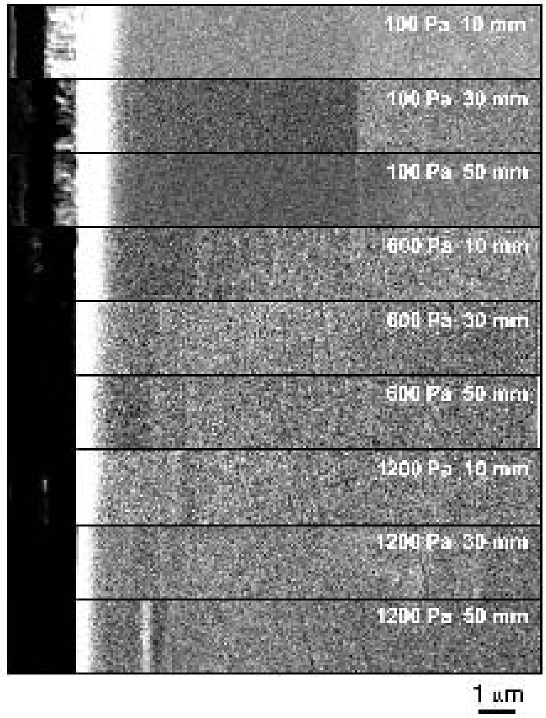

Figure 3. Cross sectional microstructures of AISI 304 steel sample treated at 723 K by ASPN.

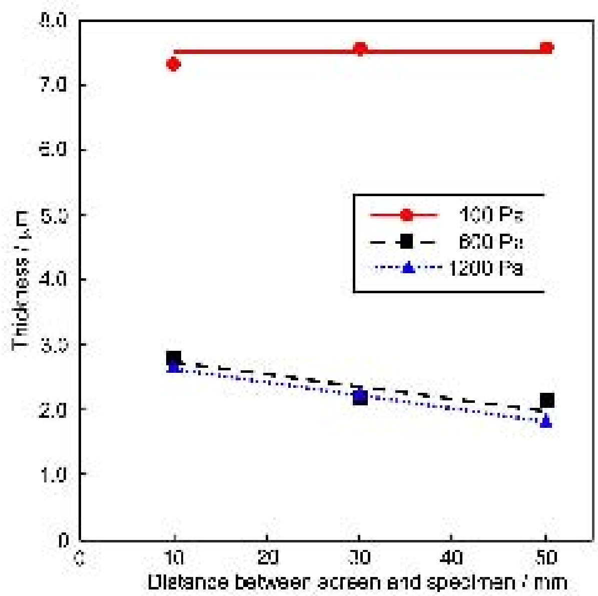

Figure 4. Effect of the gas pressure and the distance between the screen and the sample on the thickness of AISI 304 steel sample treated at 723 K by ASPN.

Figures 5 shows the cross-sectional hardness distribution of the samples treated by the ASPN process. The hardness decreased considerably toward the core of the substrate irrespective of the gas pressure and the distance between the screen and the sample. In addition, the hardening effect of the sample treated at 100 Pa was greater than that treated at 600 and 1200 Pa. The dependence of the distance on the surface hardness is a result of the thickness of the S-phase layer on the surface, as shown in Figure 4.

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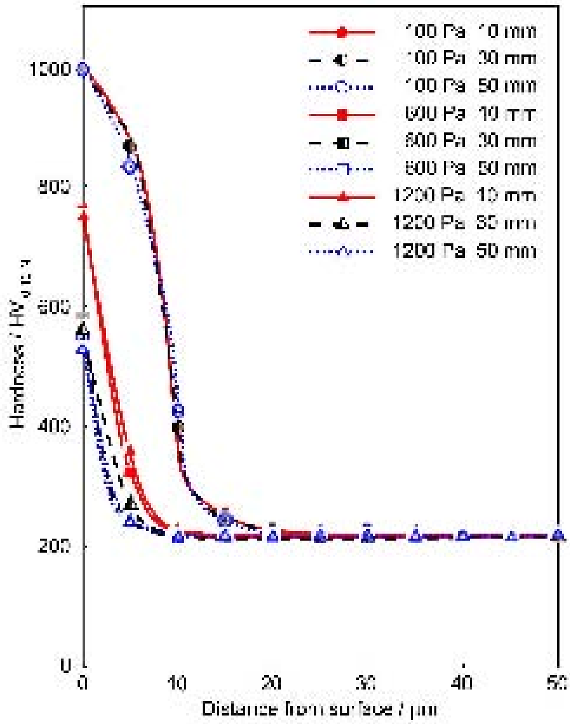

Figure 5. Hardness distribution from surface of AISI 304 steel sample treated at 723 K by ASPN.

4 CONCLUSIONS

Austenitic stainless steel AISI 304 samples were nitrided by the ASPN process using an austenitic stainless steel screen to investigate the effect of the gas pressure on the ASPN properties. After nitriding, polygonal particles with a normal distribution were observed at the center and edges of all the sample surfaces. Particles on the sample surfaces became finer as the gas pressure increased. Furthermore, under the gas pressure of 100 Pa, the thickness of the nitrided layer was almost same regardless the distance between screen and sample. In contrast, the thickness of the nitrided layer under the gas pressure of 600 and 1200 Pa was greater with decrease in the distance between screen and sample.

REFERENCES

1 SUN, Y.; BELL, T. Plasma surface engineering of low alloy steel. *Mater. Sci. Eng. A*, v. 140, p.419-34, 1991.
2 SAMANDI, M.; SHEDDEN, B.A.; SMITH, D.I.; COLLINS, G.A.; HUTCHINGS, R.; TENDYS, J. Microstructure, corrosion and tribological behaviour of plasma immersion ion-implanted austenitic stainless steel. *Surf. Coat. Technol.*, v. 59, p.261-66, 1993.
3 ALVES JR., C.; SILVA, E.F. da; MARTINELLI, A.E. Effect of workpiece geometry on the uniformity of nitrided layers. *Surf. Coat. Technol.*, v. 139, p.1-5, 2001.
4 LI, C.X.; BELL, T.; DONG, H. A study of active screen plasma nitriding. *Surf. Eng.*, v. 18, p.174-81, 2002.
5 LI, C.X.; Georges, J.; LI, X.Y. Active screen plasma nitriding of austenitic stainless steel. *Surf. Eng.*, v. 18, p.453-58, 2002.
6 LI, C.X.; BELL, T. Corrosion properties of active screen plasma nitrided 316 austenitic stainless steel. *Corros. Sci.*, v. 46, p.1527-47, 2004.

7 LI, C.X.; BELL, T. Potential of plasma nitriding of polymer for improved hardness and wear resistance. *J. Mater. Process. Technol.*, v. 168, p.219-24, 2005.
8 HUBBARD, P.; DOWEY, S.J.; DOYLE, E.D.; MCCULLOCH, D.G. Influence of bias and *in situ* cleaning on through cage (TC) or active screen plasma nitrided (ASPN) steels. *Surf. Eng.*, v. 22, p.243-47, 2006.
9 ZHAO, C.; LI, C.X.; DONG, H.; BELL, T. Study on the active screen plasma nitriding and its nitriding mechanism. *Surf. Coat. Technol.*, v. 201, p.2320-25, 2006.
10 ALVES JR., C.; de ARAUJO, F.O.; RIBEIRO, K.J.B.; da COSTA, J.A.P.; SOUSA, R.R.M.; de SOUSA, R.S.; Use of cathodic cage in plasma nitriding. *Surf. Coat. Technol.*, v. 201, p.2450-54, 2006.
11 KAULING, A.P.; SOARES, G.V.; FIGUEROA, C.A.; de OLIVEIRA, R.V.B.; BAUMVOL, I.J.R.; GIACOMELLI, C.; MIOTTI, L. Polypropylene surface modification by active screen plasma nitriding. *Mater. Sci. Eng. C*, v. 29, p.363-66, 2009.
12 NISHIMOTO, A.; TOKUDA, A.; AKAMATSU, K. Effect of through cage on active screen plasma nitriding properties. *Mater. Trans.*, v. 50, p.1169-73, 2009.
13 GALLO, S.C.; DONG, H. Study of active screen plasma processing conditions for carburising and nitriding austenitic stainless steel. *Surf. Coat. Technol.*, v. 203, p.3669-75, 2009.
14 NISHIMOTO, A; BELL, T.E.; BELL, T. Feasibility study of active screen plasma nitriding of titanium alloy. *Surf. Eng.*, v. 26, n. 1-2, p.74-79, 2010.
15 LI, C.X. Active screen plasma nitriding - an overview. *Surf. Eng.*, v. 26, n. 1-2, p.135-41, 2010.