not adjusted, with the temperature and time conditions

The Effect of Ion Nitriding on the Residual Stress of the Nitrided Layer on Austenitic Stainless

Yi-Tsung Hsiang1*[1], Chung-Chun Wu[1]

1 *Department of Mechanical Engineering , Southern Taiwan University of Science and Technolgy., Tainan City 710-301, TAIWAN*

Low-temperature ion nitriding has excellent performance in improving the surface hardness of stainless steel, with a wear-resistant nitriding layer formed on the surface, which can strike a balance between corrosion resistance and wear resistance. In the case of a common treatment temperature of 430°C, for example, after applying ion nitriding for 24 hours in this experiment, the surface hardness of stainless steel was observed to increase from 330-360 HV to 1250-1330 HV, and the thickness of the nitrided layer was approximately 11-15 µm. Since the nitriding process generated crushing stress on the surface of the material, the residual stress value was measured using the Pulstec µ-X360n Residual Stress Analyzer from the surface to the substrate, and the residual stress value was reduced from -913MPa to -124MPa. When the nitriding temperature exceeded 450°C, the lattice expansion caused by structural changes would result in the generation of chromium nitride on the surface, and the residual stress value would be greatly raised, leading to the rupture of the nitrided layer on the surface and affecting its corrosion resistance. In addition, this study also delves into the effect of the nitrided layer formed on the surface of 304 and 316 austenitic stainless under different temperatures and treatment times with fixed gas ratios on the abrasion and residual stresses.

Keywords: *Ion nitriding, Austenitic stainless, Nitrided layer, Residual stress*

1. Introduction

300 series stainless steel features the austenitic structure at room temperature and is characterized by its good processing and anti-corrosion properties, which is widely applied in the food, healthcare and sanitation sectors. Since austenitic stainless steel is austenitic at both normal and high temperatures, it cannot be strengthened by quenching and tempering, so we have developed the martensitic stainless steel. Since the corrosion resistance of martensitic stainless is weaker than that of austenitic stainless, we conducted surface hardening heat treatment of martensitic stainless in order to retain the good corrosion resistance and surface strengthening of austenitic stainless. However, if the treatment temperature was too high, chromium precipitation would occur and the original corrosion resistance would be compromised. In this study, ion nitriding equipment was employed so that the treatment environment could be operated at a lower temperature and the nitriding effect could be achieved. In terms of materials, we observed the changes in the surface microstructure and mechanical structure of the commonly used 304 [1-7] and 316 [8-17] stainless steels following the surface ion nitriding.

2. Experimental Procedure

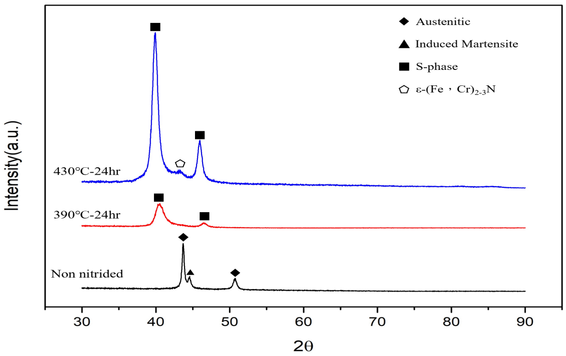
Cut the 304 test bars into 10*10*20mm rectangles and the 316 test bars into diameter 15mm*20mm cylinders, and carried out Glow Discharge Spectrometry (Table 1.) to confirm their composition. The side of the body was drilled with a 3mm tungsten drill, the surface was ground by 80→150→400→800→1200 waterproof sandpaper without polishing, wiped by 95% alcohol, and then put into ELTROPULS Plasma Heat Treatment D-52499 Ion Nitriding Furnace in a hanging way. In this experiment, five sets of parameters were set for comparison(Table 2). Except for the changes in temperature and time, the pre-cleaning parameters, gas ratio 1H:3N, discharge treatment time, voltage, current, and internal pressure were listed as follows (Table 2.).Finished products were analyzed as follows: surface hardness was measured using Future-Tech FM-110 100g load weight, XRD was gauged using X-ray diffractometer Bruker D2 PHASER, microstructural composition was observed using HITACHI S-3000N scanning electron microscope, depth of nitriding layer on the surface was observed using Olympus BX-53M, and corrosion resistance was tested by salt spray tester SC-450 Weiss Umwelttechnik Salt Spray Chamber SSC, and the residual stress was measured by Pulstec µ-X360n Residual Stress Analyzer to gauge the residual stress after nitriding.

3. Results and Discussion

3.1 XRD Analysis

As Figure 1.can be observed from the chart, 304 stainless steel at 43.6° and 50.7° [4)]exhibits an obvious austenitic signal, in which 304 stainless steel at 44.5° displays an apparent signal, which is induced martensite [2,7)], the reason for which is that stable austenite at room temperature undergoing the stress of processing, forging and molding produces induced martensite. After nitriding treatment, due to the entry of nitrogen atoms, the lattice angle was shifted, resulting in the formation of expanded austenite (S phase) with high hardness. Specimens nitrided for 24 hours at 390°C and 430°C exhibited obvious S-phase signals at 40° and 46°.

Figure 1.



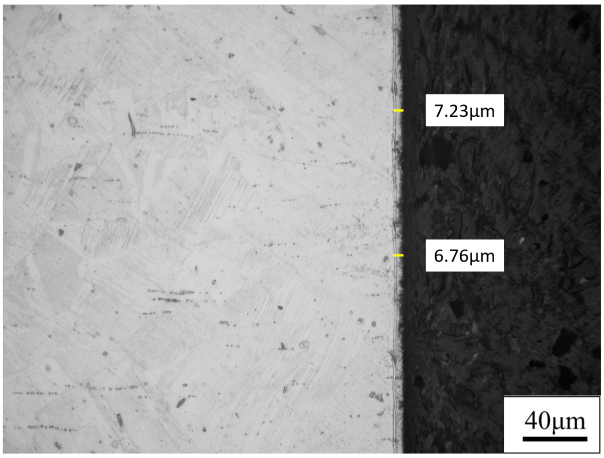
X-ray diffractometer for AISI 304

1

3.2 Microstructural Analysis

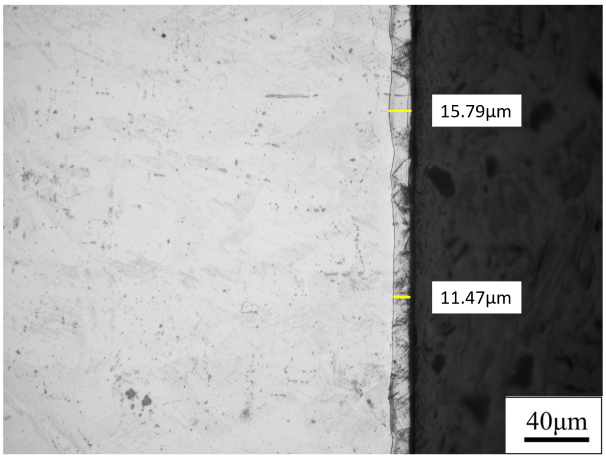
Observed the changes of nitrided layers after nitriding treatment by using metallographic microscopes. Under the condition of 24 hours treatment at 390°C, the low treatment temperature and short treatment time slowed down the formation of nitrided layer, and the thickness of nitrided layer formed was around 6μm(Figure 2.). When the treatment time remained unchanged and the temperature was raised to 430°C, the thickness of the nitrided layer could go up to 15μm(Figure 3.). When the temperature was elevated to 470°C, the expanded austenite was decomposed into CrN by the chromium precipitated from the high temperature, which made the nitrided layer thinner, and the nitrided layer averaged 10μm(Figure 4.) in thickness. Due to the precipitation of chromium nitride, the corrosion resistance would be compromised. In this experiment, the treatment temperature was maintained at 430°C, and the treatment time was extended to double and triple the original treatment time to observe the formation of the nitrided layer.

Figure 2.



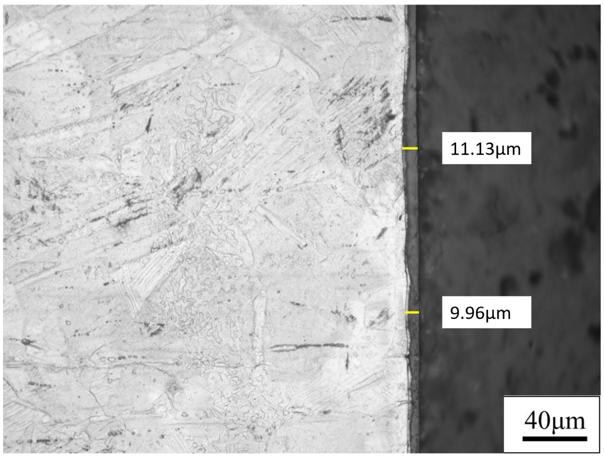
AISI 304 with 390℃ 24hr nitrided

Figure 3.



AISI 304 with 430℃ 24hr nitrided

Figure 4.

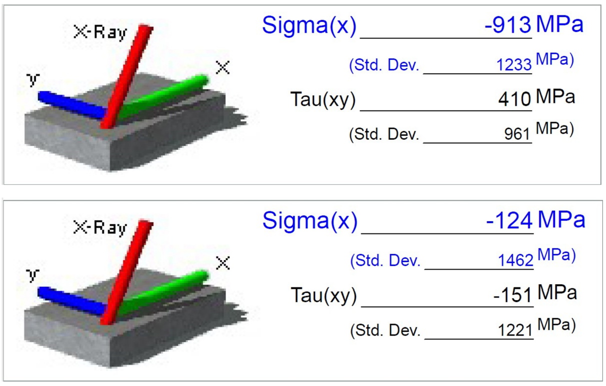


AISI 304 with 470℃ 24hr nitrided

3.3 Residual Stress Analysis

The changes in the XRD graphs show that the lattice expansion due to nitrogen atom infiltration leads to a shift in the angle, and this variation results in residual stress on the surface. The residual stress on the surface was analyzed by Pulstec μ-X360n Residual Stress Analyzer and it was found that the residual stress of 304 stainless steel was between -913 Mpa and -124 Mpa (Figure 5.)at 470°C for 24 hours.

Figure 5.



Residual Stress with AISI 304 after 470℃ 24hr nitride

4. Conclusion

AISI 304 is processed to induced martensite, which is conducive to the formation of nitrided layer.

When the temperature exceeds 450℃,CrN is produced, which affects the performance of nitride layer, and affects wear resistance, corrosion, and hardness.

Table

Table 1. Chemical composition of stainless steel used in the experiment (mass %)

	C	Si	Mn	P	S	Cr	Ni
304	0.034	0.346	0.996	0.04	0.022	19.2	8.56
316	0.033	0.392	1.59	0.035	0.02	17.8	10.2

Table 2. Temperature and time conditions

Material	Temperature	Time
AISI 304 AISI 316	390°C	24Hr
	430°C	24Hr
	470°C	24Hr
	430°C	48Hr
	430°C	72Hr

References

1) Jun Wang, Ji Xiong, Qian Peng, Hongyuan Fan, Ying Wang, Guijiang Li, Baoluo Shen: MATERIALS CHARACTERIZATION **60** (2009) 197–203
2) J. Biehler, H. Hoche, M. Oechsner: Surface & Coatings Technology (2017)
3) X. Tao, X. Liu, A. Matthews, A. Leyland: Acta Materialia (2018)
4) Paula Cisquini, Simão Vervloet Ramos, Pedro Rupf Pereira Viana, Vanessa de Freitas Cunha Lins, Adonias Ribeiro Franco Jr. , Estéfano Aparecido Vieira: j m a t e r r e s t e c h n o l . 2 0 1 9;8(2):1897–1906
5) J. Feugeas, B. Go´mez, A. Craievich: Surface and Coatings Technology **154** (2002) 167–175
6) H.T. Chen, M.F. Yan, S.S. Fu: Vacuum **105** (2014) 33-38
7) Wenzhi Xie , Yunxia Chen, Dazhi Chen, Yang Yang, Chengsong Zhang, Guodong Cui and Yixue Wang: Mater. Res. Express **7** (2020) 066406
8) L. Nosei, S. Farina, M. Ávalos, L. Náchez, B.J. Gómez, J. Feugeas: Thin Solid Films **516** (2008) 1044–1050
9) Gui-jiang Li, Qian Peng, Cong Li, Ying Wang, Jian Gao, Shu-yuan Chen, Jun Wang, Bao-luo Shen: Surface & Coatings Technology **202** (2008) 2749–2754
10) O. Oztur, S. Okur, J.P. Riviere: Nuclear Instruments and Methods in Physics Research B **267** (2009) 1540–1545
11) M. Chemkhi, D. Retraint, A. Roos, C. Garnier, L. Waltz, C. Demangel, G. Proust: Surface & Coatings Technology **221** (2013) 191-195
12) Yang Li, Zhuo Wang, Liang Wang: Applied Surface Science **298** (2014) 243-250
13) M. Yazıcı, O. Çomaklı, T. Yetim, A.F. Yetim, A. Çelik: Surface & Coatings Technology **261** (2015) 181-188
14) Insup Lee, Abhik Barua: Surface & Coatings Technology **307** (2016) 1045-1052
15) M. Chemkhi, D. Retraint, A. Roos, C. Demangel: Surface & Coatings Technology (2017)
16) Andr´e Paulo Tschiptschin, Arthur Seiji Nishikawa, Luis Bernardo Varela, Carlos Eduardo Pinedo: Thin Solid Films (2017)
17) Ali Fatih Yetim, Mustafa Yazıcı: Journal of Bionic Engineering **11** (2014) 620–629