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MODIFICATION OF AISI 347 STAINLESS STEEL SURFACE BY THE LOW TEMPERATURE SALT BATH NITRIDING PROCESS: A REVIEW

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ABSTRACT

Salt bath nitriding of type 347 austenite stainless steels conducted at low temperature, 430°C, using a complex salt bath heat-treatment. Microstructure and the properties of the nitrided surface were systematically evaluated. Experimental results revealed that when 347 stainless steel subjected to complex salt bathing nitrided at such temperature less than 4 hours, the main phase of the nitrided modify layer was the expanded austenite (S phase) generally. When the treatment times prolonged up to 8 hours and more, S phase was formed and subsequently transformed partially into CrN, and then the secondary CrN phase precipitated. All treatments can effectively increase the surface hardness. And the nitrided layer depth thickened intensively with the increasing nitriding time. The growth of the nitrided layers took place mainly by nitrogen diffusion according to the expected parabolic rate law. Low temperature nitriding can improve the corrosion resistance against diluted hydrochloric acid. After nitriding for 16 hours, the sample has the best corrosion resistance than others though there are many CrN transformed in the layer.

Keywords: 347 stainless steel; salt bath nitriding; low temperature; corrosion property; microstructure

I. INTRODUCTION

Austenitic stainless steels (ASS) characterize with their excellent corrosion resistance higher ductility, nonmagnetic properties and hygiene properties enable a very wide application range. However, the major disadvantage is low hardness, which leads to very poor tribological properties. Increasing the hardness without losing corrosion resistance could significantly broaden their applications.

In order to improve the alloy's tribological properties, significant surface hardening was achieved, which is in line with most research on plasma and gas nitriding of stainless steel. Moreover, the previous study suggests that the performance of surface corrosion-resistance provided by complex salt bath chemical heat-treatment is better than which provided by hard chrome plating or other galvanic layers. Consequently, Li and Yeung claimed that the complex salt bath nitriding process was considered as an effective engineering technology to improve corrosion properties. It is an environment-friendly process that originates from salt bath nitriding technology where a combination of high fatigue resistance and good wear and corrosion resistance can be achieved. Salt bath process technology can solve environmental problems and can be applicable to the hardening of stainless and high alloy steels with high reaction efficiency. Actually, this process is a nitro carburizing process, since the environment of molten salt contains both carbon and nitrogen. The two elements generally diffuse into the surface of steel parts, simultaneously.

But austenitic stainless steel is known as a material difficult to nitride. Some standard nitriding techniques such as gas nitriding or ion nitriding, which are widely used in industry, fail to improve mechanical properties without losing corrosion resistance. Recently, Hiroyuki Tsujimura nitrided an ASS347 stainless steel tube in a molten salt (LiCl–KCl–Li₃N) at 773 K with different applied potentials and this suggested that the molten salt electrochemical process could be applied to specimens of various shapes. Abdel conducted the 316 stainless steel samples nitrided in a closed system containing KNO₃ salt bath (the nitrate bath) in presence of ultra pure N₂ gas atmosphere at 450°C. And nitriding makes the stainless steel gain higher electrochemical corrosion resistance to chloride ions attack after two weeks of immersion in 3.5% NaCl solution.

But insufficient knowledge exists about the effects of nitriding factor on microstructure and properties when complex salt bath nitriding is done on AISI 347 stainless steel at low temperature. Therefore, the aim of the study is to make an attempt to investigate the influence of salt bath process on surface microstructure of 347 stainless steels, by using vicker-hardness, scanning electron microscopy (SEM) and corrosion resistance experiments.

II. EXPERIMENT

The authors have taken substrates made of AISI 347 stainless steel with the composition (wt.%): C 0.08, Cr 18, Ni 11.0, Si 1.00, Mn 2.00, S 0.03, P 0.045 and Fe balance. Every flat surface of each sample was ground by sand paper and then ultrasonically cleaned. The samples of 347 steel nitriding were dipped in the molten salt at 430°C for 1, 2, 4, 8, 16 hours and then cooled in air to room temperature. After the heat chemical treatment, samples were ultrasonically cleaned in alcohol bath for 15 min.

The salt medium for nitro carburizing 347 austenitic stainless steel samples was mainly composed of M_2CO_3 (M denotes elements of K, Na, Li), $CO(NH_2)_2$ and some trace components. CNO– concentration in the salt was above 40%. The pre treatment to remove passive film of the steel samples was not necessary because the CNO– in salt bath has a strong reducing potential.

The structural changes in the modified layer were investigated using cross-sections for optical microscopy and the Type JSM5910-LV scanning electron microscopy with the Oxford EDS tester. Corrosion tests were performed by immersing the untreated and nitride samples in a 10% HCl water solution at room temperature for up to 120 h. The back and the side of the disc samples were masked with several layers of lacquer, leaving only one flat surface, with a theoretical area of 565 mm², in contact with the testing solution. After certain time interval, samples were removed from the solution. They were cleaned and dried, and then measured with a balance to an accuracy of 0.1 mg, so that the weight loss due to corrosion was obtained. Two batches of tests were performed, and each data reported in this paper represents the average value of two tests.

III. RESULTS AND DISCUSSION

Metallography Analysis

It was observed that the microstructure produced during salt bath nitro carburised of

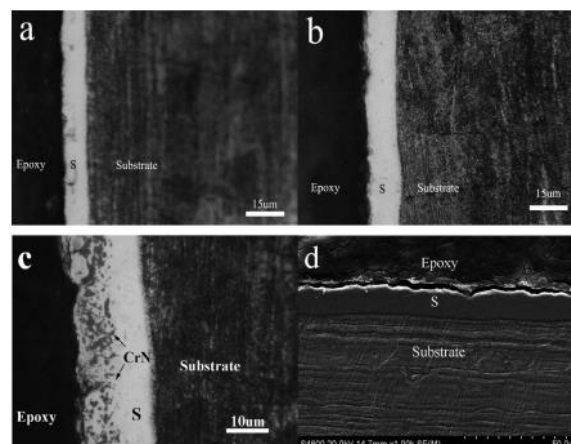


Fig.1. Cross-sectional microstructure of sample nitrided at temperature 430°C a:4 hrs; b:8 hrs; c:16 hrs; d:4 hrs.

347 type stainless steel changed according to the treatment time at 430°C, as shown in Fig. 1. A typical cross-sectional micrograph of the nitride layer in Figs. 1(a) and 1(b) is exemplified to show a bright white layer. It appears to be “bright” under optical microscope (Fig. 1(a)) and “featureless” at a higher magnification under SEM (Fig. 1(d)). This phenomenon would indicate that a possible enhancement in corrosion resistance of the modified layer

was obtained by nitrocarburized to the harsh etchants (here is Marble's reagent). But the substrate has not. So the nitrided layers distinguished from the substrate due to the etching degree are different. The depths of nitrided layers were increased with the treated time. It can be seen that the depth of the total nitrocarburized layer is approximately 18 μm after salt nitriding treatment for 16 hours at 430°C. And when the stainless steel specimen treated for 16 hours, some secondary precipitate transformed. The corrosion resistances of precipitate zone became worse at 430°C. The dark areas can be easily observed under the microscope after reagent etched (Figs. 1(c) and 1(e)).

The microstructure scanned by SEM and EDS of nitrided layer of a cross-section of the complex nitrided sample in 430°C for 16 h are shown in Fig. 6. From the pictures, it can be obviously observed that there are some dark zones in the nitriding layer. And EDS indicated that the dark zone is rich in chromium, while poor in Fe and Ni.

Hardness and Corrosion Behaviour

Figure 2 shows the micro-hardness of nitrided layers as a function of treatment time at 430°C. As shown in picture, a very steep micro-hardness increasing was found on the sample surface and it came to a gradual steady when temperature rises beyond 1 hour.

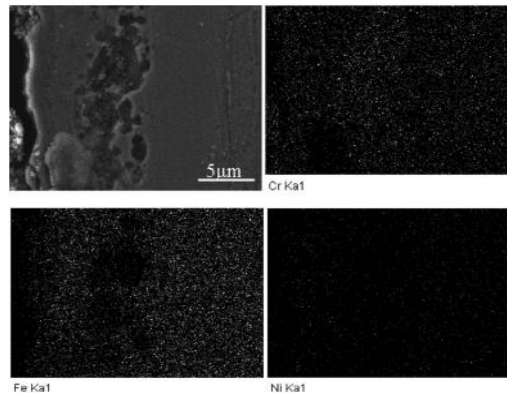


Fig. 2. Cross-sectional microstructure and EDS of sample nitrided for 16 hours at 430°C

1 150 HV0.1 for 8 hours, which is about 3.5 times as hard as the untreated material (328 HV0.1). The relatively high hardness of these nitrided layers could be caused by the great misfit-induced stress fields associated with supersaturation of nitrogen in Fe–Cr–Ni solid solution and fine dispersed CrN precipitates in following stage.

Micrographs of the corroded surfaces on untreated and nitrided 347 steel after immersion in 10% HCl for 120 h are shown in Fig. 9. SEM examinations indicated that nitriding has changed the dominant corrosion mechanisms of the 347 steel from localised selectivity corrosion to general corrosion in the diluted hydrochloric acid. For non-treated sample, some selectivity corrosion zones had penetrated to a very deep depth. In comparison, corrosion of sample 16 h was more likely to be in a general form, although dissolution preferably occurred along the grain boundaries and some crystallographic directions (Fig. 9(c)). Some selectivity corrosion zones can indeed observed, but the amount and depth of them were much smaller. In this sense, it may be concluded that the long time nitriding has modified the corrosion properties of the 347 stainless steel in diluted hydrochloric acid.

It is common knowledge that the beneficial effect of nitrogen can only be realised when nitrogen is in solid solution. Once CrN transformed, as that occurred in nitriding at a relative higher temperature or longer treat period, chromium in the matrix will be depleted, particularly around the nitrided precipitates. In this case, the longtime (16 hours) low temperature nitrided samples have a pretty thick modified layer in surface. But many CrN are transformed in the outer layer, which can be seen under SEM. The relative thin S phase sub-layer exists between the CrN zones and the substrate (Figs. 1 and 6). From the immersing test results, it is obvious that the 16 h sample, with the thickest modify layer, has the best corrosion resistance in all samples though it has many CrN precipitate. And the 4 h sample has better corrosion resistance property than non-treatment sample. This indicated that the thickness of the surface modified file after nitrided play a great role in corrosion resistance property. The thin deposition layer on top of the

nitrided case appeared to have mitigated the corrosion attack to a certain extent during immersing tests. This could have related to the different compositions and structure in the deposition layer. Further investigations are needed to verify the mechanism involved.

IV. CONCLUSION

From the above-mentioned investigations, The authors has concluded that when 347 stainless steel subjected to complex salt bathing nitriding at such temperature less than 4 hours, the main phase of the nitrided coating layer was the expanded austenite (S phase) generally. When the treatment time prolonged up to 8 hours and more, S phase is formed and subsequently transforms partially into CrN, and then the secondary CrN phase precipitated. All treatments can effectively improve the surface hardness. And the nitrided layer depth thickened changed intensively with the increasing nitriding time. The growth of the nitride layer takes place mainly by nitrogen diffusion according to the expected parabolic rate law. Low temperature nitriding can improve the corrosion resistance against diluted hydrochloric acid. After nitrided for 16 hours, the sample has the best corrosion resistance than others, though there are many CrN transformed in the layer.

REFERENCES

1. Yetim, A. F.; Alasaran, A.; Çelik, (2010), A. *Investigation of tribological and electrochemical properties of dual layer after low temperature plasma carbo-nitriding*. Surf. Eng., 26, 178–184
2. Mohamad Alabdullah, Ashwin Polishetty, and Guy Littlefair,(2016), *Impacts of Wear and Geometry Response of the Cutting Tool on Machinability of Super Austenitic Stainless Steel*,International Journal of Manufacturing Engineering.
3. Basso R. L. O., Pastore H. O., Schmidt V., Baumvol I. J. R, Abarca S. A. C., De Souza F. S., Spinelli A., Fiqueroa C. A. and Giacomelli C., (2010) *Microstructure and corrosion behavior of pulsed plasma-nitrided AISI H13 tool steel*, Corrosion Science,
4. E. Haruman, Y. Sun, H. Malik, AG.E. Sutjipto, K. Widi. (2006)*Low Temperature Nitriding of Austenitic Stainless Steel, the 3Asian Conference on Heat Treatment of Materials, Nov. 10-12*
5. Sueyoshi, H.; Hamaishi, K.; Nakamura, Y.; Kiyofuji, J. (1996),*Effect of mechanical pre-treatment on gas nitriding behaviour in austenitic stainless steels*. Mater. Trans. JIM 37, 150–156.
6. K. A. Widi, I. Wardana, W. Suprpto, Y. S, Irawan, (2016), *The Role of Diffusion Media in Nitriding Process on Surface Layers Characteristics of AISI 4140 with and without Hard Chrome Coatings*, Tribology in Industry Vol. 38, No. 3 308-317.
7. Yuan, X.; Zhao, Y.; Li X. and Chen, L.; (2017), *Effects of Gas Nitriding Temperature on the Surface Properties of a High Manganese TWIP Steel*, Metals, 7, 102.
8. R. W. Reynoldson, *Quality Heat Technologies PTY LTD, Operating Instructions, Fluid heat Laboratory Fluidised Bed Furnace Model FH12M, 2001.*
9. Figueroa, C. A., Ferlauto, A. S., Alvarez, F., "Identification of the mechanism limiting nitrogen diffusion in the metallic alloys by in situ photoemission electron spectroscopy", *Journal of Applied Physics*, v. 94, n. 8, pp. 5435-5437, 2003.
10. E. J. Mittemeijer, J. T. Slycke. (1996) *Thermodynamical activities of nitrogen and carbon imposed by gaseous nitriding and carburizing atmospheres*, *Proceedings of the Second International Conference on Carburizing and Nitriding with Atmospheres*, 6 – 8 December 1995, Cleveland, Ohio (eds. J. Grosch, J. Morral and M. Schneider), ASM, Materials Park Ohio, USA, 1995, 247-254.
11. Seyda Polat, S. Hakan Atapek, and Fatih Gumus, (2012) *Gas nitriding of a hot work tool steel and its characterization*.