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Influence of time on the microstructure of AISI 321 austenitic stainless steel in salt bath nitriding

Jun Wang [a,b], Yuanhua Lin [b,*], Jing Yan [c], Dezhi Zen [b], Qiang Zhang [c], Runbo Huang [a], Hongyuan Fan [a]

[a] School of Manufacturing Science and Engineering, Sichuan University, Chengdu, 610065, PR China
[b] State Key Lab of Oil and Gas Reservoir Geology and Exploitation, Southwest Petroleum University, Chengdu, 610500, PR China
[c] Southwest Oil and Gasfield Company Research Institute of Natural Gas, Chengdu 610023, PR China

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ABSTRACT

Influence of nitriding time on the microstructure and microhardness of AISI 321 austenite stainless steel was investigated, using a complex salt bath heat-treatment at low temperature, 430 °C. Experimental results revealed that after salt bath nitriding, a modified layer was formed on the surface of substrate with the thickness ranging from 2 μm to 30 μm with changing treating time. The nitrided layer depth thickened extensively with increasing nitriding time. The growth of the nitrided layer takes place mainly by nitrogen diffusion according to the expected parabolic rate law. Scanning electron microscopy and X-ray diffraction showed that in 321 stainless steel subjected to complex salt bathing nitrided at such temperature for less than 8 hours, the main phase of the nitrided layer was expanded austenite (S phase) by large. When the treatment time is prolonged up to 8 hours and more, S phase is formed and subsequently transforms partially into CrN, and then the secondary CrN phase precipitated. With treating time prolonged, more CrN precipitates formed along the grain boundaries in the outer part. In the inside part between the some CrN and the substrate, there is still a broad single S phase layer. All treatments can effectively improve the surface hardness.

1. Introduction

Austenitic stainless steel (ASS), with excellent corrosion resistance and high ductility, is attractive in a wide range of applications, such for outdoor machines in the chemical, coal and oil industries. However, a major disadvantage is its low hardness, which leads to very poor tribological properties [1–6]. To elevate the alloy's tribological properties, thermal–chemical surface hardening techniques such as nitriding, carburizing, and nitrocarburizing [7–9], with or without plasma actions, are extensively studied to improve surface mechanical and tribological properties of austenitic stainless steels [10–16].

Salt bath nitriding is developed as an industrial process especially for surface modification of iron-based steels and this process technology has solved environmental problems and can be applied to harden stainless and high alloy steels with high reaction efficiency [17–21]. It is an environment-friendly process in which a combination of high fatigue resistance and good wear and corrosion resistance can be achieved [22,23]. Actually, this process is actually a nitrocarburizing process. Since the environment of molten salt contains both carbon and nitrogen, two elements generally diffuse into the surface of steel parts, simultaneously [24]. In comparison with plasma nitrocarburizing methods, salt bath treatment has the advantages of easy operation, low cost, energy efficiency, and stability [22–24]. But the common salt bath nitriding technology carried out 580–650 °C. At these temperatures, the great mass precipitation of CrN results in a depletion of Cr from the matrix. This induces a strong decrease in corrosion resistance that degrades the beneficial effect of increased hardness [24–28]. So, the traditional nitriding processes are not apt for surface hardening of stainless steel without loss of corrosion resistance.

Recently, Hiroyuki Tsujimura [20] successfully nitrided an SUS 304 stainless steel tube in a molten salt (LiCl–KCl–Li3N) at 500 °C with different applied potentials and suggested that the molten salt electrochemical process can be applied to specimens of various shapes. More recently, Abdel [29] nitrided the 316 stainless steel samples by the KNO_3 salt bath (the nitrate bath) under the protection of ultra pure N_2 gas atmosphere at 450 °C, and demonstrated that this type of nitriding conferred the stainless steel with higher electrochemical corrosion resistance against chloride ions attack with 2 weeks of immersion in 3.5% NaCl solution.

However, there is insufficient knowledge about the effects of nitriding factor on microstructure and properties when complex salt bath nitriding is done on AISI 321 stainless steel at low temperature. Therefore, the aim of the study is to make an attempt to investigate the influence of processing time on the microstructure, the phase

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* Corresponding author Tel.: +86 13880643313.
E-mail address: Scutg@163.com (Y. Lin).

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composition and the microhardness in the nitrided layer by using X-ray diffraction, scanning electron microscopy and energy dispersive X-ray.

2. Experimental details

The specimens were made of AISI 321 ASS with the composition (wt.%): C 0.04, Si 0.54, Ti 0.48, Mn 1.33, Cr 17.55, Ni 9.00 and Fe balance. All of the flat surfaces of each sample were ground with sand paper. The pretreatment to remove passive film of the steel samples was not necessary because the CNO^- in salt bath has a strong reducing potential. The samples were dipped into molten salt to nitride at 430 °C for 1, 2, 4, 8, 16, and 40 hours and then cooled in air to room temperature. After the chemical treatment, samples were ultrasonically cleaned in alcohol bath for 15 min.

The salt medium for nitrocarburizing AISI 321 ASS sample was mainly composed of M_2CO_3 (M denotes some elements of halogen), $CO(NH_2)_2$ and some trace components. CNO^- concentration in the salt was above 40%.

The nascent nitrogen utilized for nitriding reaction comes from the dissociation of CNO^-: $4CNO^- \rightarrow CO_3^{-2} + 2CN^- + CO + 2[N]$ [19]. Concentration gradient between sample surface and nitriding salt bath constitutes the driving force for active nitrogen atom permeating into austenite structure, leading to surface nitride layer formation. In the meantime, a few nascent carbons which come from the dissociation of CO: $2CO \rightarrow CO^2 + [C]$ also permeate into austenite structure together with nitrogen.

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 energy dispersive X-ray tester. X-ray diffractometer type Dmax-1400 with Cu K alpha radiation and a nickel filter were used to determine the phases present in the modified layer.

3. Results and discussion

3.1. Metallography analysis

It was observed that the microstructure produced during 430 °C salt bath nitriding of AISI 321 ASS changed with the treatment time, as shown in Fig. 1. A typical cross-sectional micrograph of the nitride layer in Fig. 1a and b is exemplified to show a bright white layer. This

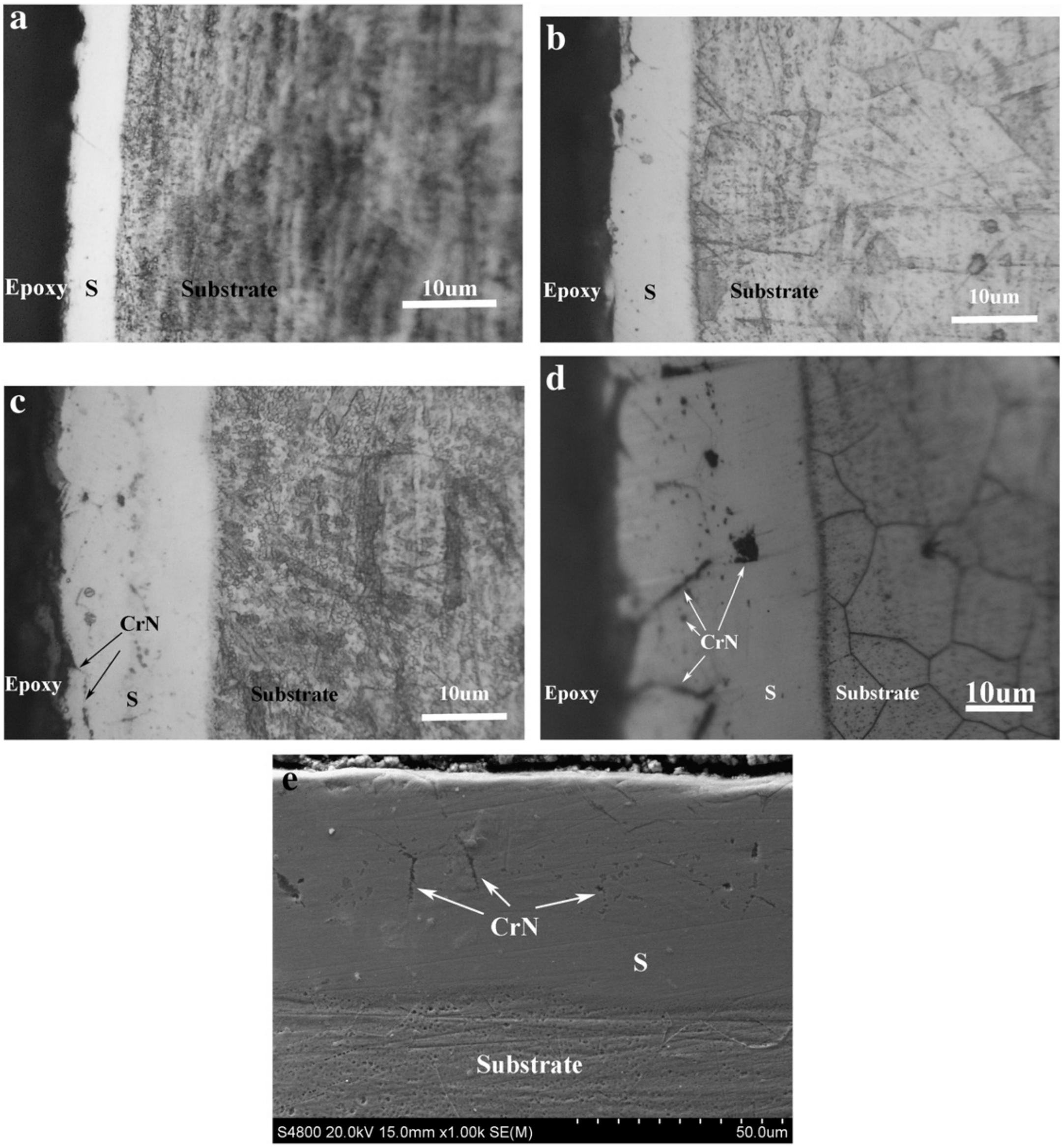

Fig. 1. Cross-sectional view to show microstructure of sample nitrided at temperature 430 °C. (a: 4 hours; b: 8 hours; c: 16 hours; d: 40 hours; e: 40 hours in SEM.)