Low-pressure gas nitriding of AISI 304 austenitic stainless steel: reducing the precipitation of chromium nitrides

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Abstract

In this study, low-pressure gas nitriding (gas pressure of 0.01 MPa) was conducted to produce a thicker nitrided layer with high hardness and anti-corrosive properties on AISI 304 austenitic stainless steel. The effects of nitriding temperature and duration on the microstructure and surface property of nitrided layers were systematically evaluated by using optical microscope, X-ray diffraction, elemental analysis, microhardness test and potentiodynamic polarization tests. The samples were also treated under conventional gas pressure of 0.1 MPa for comparison. The results show that the low-pressure gas nitriding could restrain the precipitation of chromium nitrides effectively, which is beneficial for obtaining a thicker nitrided layer. Although the activation energy of nitrogen diffusion for low-pressure nitriding (220 kJ mol⁻¹) is higher than that for the atmospheric pressure nitriding (196 kJ mol⁻¹), the thickness of nitrided layers for low pressure nitriding could reach to a comparable value as that for the conventional atmospheric pressure nitriding. More importantly, the surface toughness and corrosion resistance of nitrided layers could be improved by low-pressure nitriding, which is mainly attributed to the optimized nitrogen content in nitrided layers and the reduced precipitation of chromium nitrides under low-pressure.

1. Introduction

Austenitic stainless steels (ASS) are widely used in corrosion resistant conditions. However, these materials have an inherent weakness of low hardness, which seriously restrict the applications of ASS in wear-resistant conditions. In order to improve the wear resistance of ASS, the nitriding is generally applied to achieve this purpose [1]. The nitrogen atoms diffused into the matrix of ASS and the expand austenite (denoted as S phase) with high hardness was formed [2]. Although nitriding is an effective way to improve the hardness of ASS, the anti-corrosion property of ASS starts to deteriorate when nitrided at a high temperature or for a long duration due to the inevitable precipitation of chromium nitride [3, 4]. Therefore, the precipitation-free S phase is the desired phase for the nitried ASS. The low temperature nitriding with surface activation by plasma sputtering or chemical pickling is successfully applied to achieve this purpose [5–8]. However, the S phase is always difficult to improve in thickness. For example, the S phase with thickness over 20 μm usually requires nitriding for more than 20 h at 400 °C–430 °C [9, 10]. Many researches have proved that the temperature is the most effective parameter for increasing the layer thickness [5, 11]. But the higher temperature indeed results in the precipitation of chromium nitrides. As demonstrated by the threshold temperature-time curves for ASS, the higher temperature is, the faster chromium nitrides precipitate [10, 12]. Gemma et al [13] investigated the growth kinetic of nitried layer of ASS and found an irregular nitriding behavior for ASS. A suddenly increased rate occurs at 450 °C accompanying with the precipitation of chromium nitrides. Therefore, 450 °C is
commonly considered as the critical temperature for the precipitation of chromium nitrides. In order to obtain a thicker S phase layer, many attempts have been carried out. Surface mechanical attrition treatment (SMAT) was proved to be an effective pre-treatment to increase the thickness of S phase layers \cite{14-16}. The surface grain refinement caused by SMAT contributes to the fast diffusion of nitrogen atoms. But SMAT indeed accelerate the precipitation of nitrides simultaneously \cite{17}. Many studies demonstrate that a low-pressure plasma nitriding technology could dramatically increase the thickness of S phase layer \cite{18-21}. The mechanism is summarized as strong sputtering effect at low pressure which results in higher surface activation \cite{22}. More importantly, the low pressure could restrain the precipitation of chromium nitrides effectively \cite{23}. Therefore, the gas pressure plays an important role in the nitriding processes, especially in the gas nitriding process. It is because of that the gas pressure could determine the nitrogen potential directly \cite{24}. At present, most of literature about nitriding of ASS are concentrated on plasma nitriding, while few works focus on the gas nitriding, especially on low-pressure gas nitriding. In present work, we will investigate the effect of gas pressure and nitriding parameters (temperature and duration) on the microstructure and properties of AISI 304 ASS during gas nitriding. The purpose of this work is to clarify whether the precipitation of chromium nitrides can be reduced or not at low pressure.

2. Experimental

2.1. Sample preparation

AISI 304 ASS bars with diameter of 18 mm were used as the substrate material with the composition of (wt.%) 0.188C, 0.397Si, 18.42Cr, 7.78Ni, 0.133Mn, 0.279Cu and Fe in balance. A solution treatment was conducted under 1050 °C for 1 h and followed by oil quenching to obtain a homogenous microstructure. Disk samples with thickness of 4 mm were cut from the solution-treated ASS bars. Then the samples were mechanically ground with SiC paper down to 400 grade to achieve a fine finish. Before nitriding, the surface of samples should be activated by coating a surfactant. The surfactant contains 0.06 mol sodium chloride and 0.02 mol citric acid monohydrate dissolved in 30 ml distilled water. The surfactant could reduce the intrinsic oxide film on the surface of ASS and form a gel film to prevent the further oxidation on the surface of ASS.

2.2. Gas nitriding

The gas nitriding treatments were conducted in a vacuum quartz tube furnace at different temperatures (425 °C–500 °C) and durations (4–24 h). The gas nitriding treatments can be divided into two classes. One is produced under low gas pressure conditions (0.01 MPa), the other is produced under normal atmospheric pressure conditions (0.1 MPa) for comparison. The ammonia was induced into the furnace with the flow rate of 0.5 l min$^{-1}$. After gas nitriding, all samples were slowly cooled down to room temperature in an ammonia atmosphere. The detailed parameters were listed in table 1.

2.3. Characterization

After gas nitriding, a standard procedure, including cross-sectioning, mounting, grinding and polishing, was followed to prepare the cross-sectional specimens for metallographic observations. The metallographic specimens were etched in a solution containing 50 vol% HCl + 25 vol% HNO$_3$ + 25 vol% H$_2$O \cite{3} and observed by using an optical microscopy (Olympus GX51F, Japan). In order to identify the phase compositions in the nitried layers, x-ray diffraction measurements (XRD, X'Pert Pro, Netherlands) with Cu-K$_\alpha$ radiation were carried out in a range of glancing angles 20$^\circ$–100$^\circ$ at an accelerating voltage of 40 kV. The composition distribution in nitried layers were measured by using a scanning electron microscopy (SEM, JSM-7001F, Table 1. Detailed parameters of gas nitriding.

| Samples  | Temperature (°C) | Time (h) | NH$_3$ (l min$^{-1}$) | Pressure (MPa) |
|----------|------------------|----------|-----------------------|----------------|
| GN425-8  | 425              | 8        | 0.5                   | 0.01, 0.1      |
| GN425-24 |                  |          |                       |                |
| GN450-4  | 450              | 4        |                       |                |
| GN450-8  |                  |          |                       |                |
| GN450-16 |                  |          |                       |                |
| GN450-24 |                  |          |                       |                |
| GN475-4  | 475              | 4        |                       |                |
| GN475-8  |                  |          |                       |                |
| GN475-16 |                  |          |                       |                |
| GN500-4  | 500              | 4        |                       |                |
Japan) equipped with an energy dispersive x-ray analyzer (EDS). In order to evaluate the brittleness of nitrided layers, microhardness tests were performed using a microhardness tester (HVS-1000, China) under various loads of 0.098 to 4.9 N. Microhardness profiles of nitrided layers were also obtained with load of 0.245 N for 15 s for estimating the depth of effective hardened layers. Each profile was measured at least three times to ensure the accuracy of microhardness and the average values were obtained. To evaluate the corrosion resistance of nitrided ASS, anodic polarization tests were carried out in static 3.5 wt% NaCl solution using an electrochemical workstation (Corrtest CS350350, China). During the tests, a standard three-electrode electrochemical cell was used. A saturated calomel electrode (SCE) was applied as the reference electrode and a platinum electrode was used as the counter electrode. The sample worked as the working electrode with an exposed area of 1.54 cm². The potentiodynamic polarization scanning was performed after the stability of open circuit potential reached. The scanning rate was selected as 1 mV s⁻¹ in the range of −1.5 V to +1.0 V, relative to open circuit potential. After electrochemical corrosion tests, the corroded surfaces were observed by laser scanning confocal microscope (VHK-1000C) and the depth of corrosion pits were measured and compared.

3. Results and discussion

3.1. Cross-sectional microstructure of gas nitrided layers

The cross-sectional microstructure of nitrided samples are shown in figure 1. A nitrided layer with thickness in range of 5–40 μm was observed on the surface of the AISI 304 ASS. The nitrided layers generally show a white layer appearance with dark spots. These dark spots indicate the poor corrosion resistance which results from the precipitation of chromium nitrides. The thickness of nitrided layer increases with the increase of nitriding temperature and durations both in low gas pressure and atmospheric pressure conditions. The dark precipitations begin to appear with the increase of nitriding temperature and duration as many references reported [9, 25], which corresponds to the deterioration in corrosion resistance. There is a critical value for nitriding temperature or duration regarding to precipitations in the nitrided layers. With the increase of nitriding temperature, the critical value of nitriding duration dramatically decreases, which accords to the rule of the threshold temperature-time curve for the precipitation of chromium nitrides from S phase [10, 26]. When the temperature increases to 475 °C, a suddenly precipitation occurs even in a short duration of 4 h. While for the case of 450 °C, this process will be delayed until 16 h later. It indicates that the precipitation of chromium

![Figure 1. The cross-sectional microstructures of nitrided layers of AISI 304 ASS treated under different conditions.](image-url)
nitrides is more sensitive to the nitriding temperature and the critical temperature is 450 °C. These results are consistent with the common results obtained by plasma nitriding [27]. It is worthy to note the effect of the gas pressure on the precipitation of chromium nitrides. The low-pressure gas nitrided specimens show less precipitations than the conventional gas nitrided specimens. The suppressive effect of low gas pressure on the precipitation becomes more obvious when it is nitrided at higher temperatures or for longer durations. Taking the GN475-16 and GN500-4 samples for example, the nitrided layer treated under conventional atmospheric pressure show more dense precipitates than that treated under low-pressure conditions (see figure 1). It has proved that the low pressure could restrain the precipitation effectively.

3.2. Phase composition of nitrided layers

Figure 2 displays the typical XRD patterns of surface layers of AISI 304 ASS nitrided under different conditions. The untreated surface contains the mixed phase of γ-Fe and α-Fe (see figure 2(a)). The α-Fe phase comes from the stress induced phase transition of γ-Fe to α-Fe during the mechanically grinding process [28]. The nitrided layers mainly consist of S phase, ε nitrides and chromium nitrides (CrN). The ε nitrides have a hexagonal close-packed (HCP) structure with nitrogen atoms occupying in the interstitial sites and iron atoms is partially substituted by chromium (Cr) atoms. As the nitriding temperature or duration increases, the Cr atoms gradually diffuse into the lattice of ε nitrides and then ε nitrides could transform to Cr2N nitrides without variations in structure. Both the ε nitride and Cr2N nitride could easily precipitate from the S phase due to the similar atomic arrangement and minimum misfit on the habit plane. Lei reported that the orientation relationship between S phase and ε phase would be {111}_S // {0001}_ε and <110>_S // <12-10>_ε [29]. This orientation relationship can be proved by the overlap of the diffraction peaks between the (002), and (111)_S (see figures 2(b)–(d)). With the increase of the nitriding temperature or duration, the difference in match between these two planes become more and more obvious. Then the overlapped peak splits into two parts (see figure 2(e)). On the other hand, the CrN nitride will precipitate form the S phase following the reaction of S → α-Fe + CrN, which has been mentioned in many literature [30, 31]. The reaction can be proved by the co-existed α-Fe and CrN phase when the temperature is higher than 450 °C (see figures 2(c)–(e)). An interesting phenomenon should be noted that the low-pressure presents an obvious effect on restraining the precipitation of chromium nitrides. Especially for the GN475-16 sample, the S phase has almost decomposed to the α and CrN phase when nitrided at conventional atmospheric pressure of 0.1 MPa, while there is still a large amount of residual S phase in the low-pressure nitrided layer (see figure 2(f)). It indicates that the gas pressure could effectively improve the stability of S phase and decelerate the precipitation kinetics of chromium nitrides. In fact, the gas pressure mainly affects the decomposition of S phase by changing the nitrogen (N) concentration in nitrided layers. It is obviously shown in figure 2(f) that the diffraction peaks of S phase shift to lower angle with the increase of gas pressure. It indicates that the higher nitriding pressure leads to a higher N concentration in nitrided layers. When the N concentration...
accumulates to a certain value, the decomposition of S phase occurs. Based on the results above, it can be concluded that the low-pressure nitriding is an effective way to reduce the precipitation of chromium nitrides.

3.3. Microhardness of nitrided layers

Figure 3 shows the microhardness profiles of nitrided layers treated under different conditions. The surface hardness of nitrided layers generally surpasses 1200 HV which is about four times higher than that of the substrate. With the increase of nitriding temperature, the hardness in the nitrided layer gradually increases and a high hardness platform appears, which indicates the saturation of N atoms in the nitrided layers (see figures 4(a)–(c)). When the decomposition of S phase occurs, the hardness of nitrided layers will fluctuate obviously (see red line in figure 4(c)). The similar results are also reported by Farnandes et al [25]. Compared with rapid decrease in hardness of conventional plasma nitrided specimens, the low-pressure nitriding could reduce the hardness gradient in the nitrided layers (see figures 4(b) and (d)). The adsorbed ammonia molecules on nitrided surfaces will decrease dramatically under low-pressure conditions, which results in the decrease in the diffusion flux of nitrogen atoms. According to the Fick’s first law, i.e. $J_N = -D_N \frac{dC}{dx}$, the diffusion flux ($J_N$) is proportional to concentration gradient ($dC/dx$). In addition, the hardness is also related to nitrogen content. Therefore, the low diffusion flux in low-pressure conditions indeed contributes to the smooth hardness gradient. The smooth hardness gradient is beneficial for decreasing the brittleness of nitrided layers and improving their toughness. Although the low-pressure nitriding results in the reduction of N concentration and hardness in nitrided layers, the surface hardness is not affected by the gas pressure. The low-pressure treated surface could also achieve a stiff surface with higher hardness than that for conventional atmospheric pressure treated surfaces. Figure 4 shows the indentation after hardness tests under different load on the nitrided surface of GN450-24 sample. It can be seen that for the surface nitrided under conventional atmospheric pressure, the cracks appear when the load surpasses 100 gf. While for the low-pressure nitrided surface, the cracks occur until the load reaches to 500 gf (see figures 5(a) and (b)). The hardness indentations on the cross-sections show the similar phenomenon as surfaces (see figures 5(a1) and (b1)). It indicates that the low-pressure nitriding has more advantages in improving the mechanical properties of nitrided ASS, especially the toughness which is important.

![Figure 3. Microhardness profiles of nitrided layers for (a) GN425-24, (b) GN450-24, (c) GN475-16 and (d) GN500-4 samples.](image-url)
Figure 4. Morphologies of indentations after hardness tests under different loads on the nitrided surfaces of GN450-24 samples under (a) 0.1 MPa and (b) 0.01 MPa. (a1) and (b1) show the hardness indentations on the corresponding cross-sections for (a) and (b), respectively.

Figure 5. Depth profiles of elemental distribution in the nitrided layers treated at 450 °C for 24 h under gas pressure of (a) 0.1 MPa and (b) 0.01 MPa.
in impact and wear conditions. The improved toughness is mainly attributed to the optimized N concentration and smooth hardness gradient in the low-pressure nitrided layers.

3.4. Elemental distribution in the nitrided layers

The elemental distribution in the nitrided layers treated under conventional and low-pressures conditions are compared in figure 5. It can be found obvious difference in elemental distribution between the two gas pressure conditions. For the case treated under conventional atmospheric pressure, the elemental profiles fluctuate severely in the nitrided layer and present a waved appearance (see figure 3(a)), while for the low-pressure condition, a relatively stable distribution for elements appears in the nitrided layer. It also should be noted that the Cr profile for the conventional nitrided sample exhibits a similar variation as N concentration. It can be explained by the strong attraction between N atoms and Cr atoms. The waved N and Cr profiles corresponds to the precipitation of chromium nitrides, which results from the segregation of N and Cr atoms in the nitrided layers. For ASS, the N distribution in nitrided layers is correlated with the hardness profiles. Therefore, the N distribution is usually used to evaluate the depth of nitrided layers. In our present work, the depth of nitrided layers estimated by N distribution shows a difference with that estimated by hardness profile, which indicates the homogeneity of nitrided layers. It can be seen that the low-pressure nitrided layer presents better homogeneity than the conventional nitrided layer due to its smaller difference between layer depth evaluated by N distribution and hardness profile. It can be attributed to the larger gas flow velocity obtained under low-pressure conditions, which is beneficial for the uniform adsorption of ammonia molecules on nitrided surfaces.

3.5. Corrosion properties of nitrided layers

Figure 6 shows the potentiodynamic polarization curves and corroded morphologies of nitrided surfaces treated under different conditions. It is clearly seen that the untreated sample presents a typical pitting corrosion characteristic with an obvious passivation stage and breakdown point for pitting corrosion [32]. The corroded morphology of the untreated surface also shows the corresponding deep pits (see figure 6(a)). The nitrided samples exhibit much different appearance on the polarization curve as compared with the untreated one. The corrosion mode of nitrided surfaces varies with the phase composition on the nitrided layers. The S phase shows the similar pitting corrosion characteristic as the untreated austenite with an increased breakdown potential (see figure 6(b)). The increased breakdown potential is mainly ascribed to the higher N concentration in S phase [33]. With the formation of ε nitrides or CrN nitrides, the pitting type polarization curves turn to the wave shape with a gradually increased anodic current. The waved polarization curves correspond to the alternating pitting process and fast passivation. The corroded surface also shows a relatively smooth appearance with some shallow pits (see figures 6(c)–(e)). Although the anodic current for nitrided surfaces is higher than that for the untreated surface, the slight pitting corrosion and the smooth corroded surface of the nitrided samples indicate the improved the corrosion resistance, especially for the GN425-24 and GN450-24 samples. It is also worthy to note that the low pressure nitrided surface generally presents a lower anodic current and shallower pit than the normal atmospheric pressure nitrided surface (see figures 6(c) and (d)). It indicates that the low-pressure nitrided sample has better corrosion resistance due to the reduction in precipitation of chromium nitrides.

3.6. Growth kinetic of nitrided layers

Based on the results obtained above, it has proved that the low-pressure nitriding plays an effective role on reducing the precipitation of chromium nitrides. Therefore, the low-pressure nitriding of ASS could be carried out at a higher temperature or for a longer duration, which is beneficial for obtaining a thicker and corrosion resistant nitrided layers. Figure 7 shows the thickness of nitrided layers, as a function of the root of nitriding time at 450 and 475 °C. It can be seen that the plot is close to the linear relationship, i.e. \( d \propto \sqrt{t} \), which indicates the growth of nitrided layers is mainly dominated by nitrogen diffusion process. Although the thickness of the low-pressure nitrided layers is generally thinner than that of the normal atmospheric pressure nitrided layers, the thickness gap between them becomes smaller when they have the similar phase composition. When the decomposition of S phase occurs, as shown in the plot of 475 °C nitrided sample at 0.1 MPa (green dash line), the layer thickness increases rapidly with the increase of nitriding duration. It indicates that the new formed interfaces between α-Fe and CrN phase could provide the additional diffusion channel for N diffusion. There is usually a contradiction between the thickness and corrosion resistance of nitrided ASS. Thicker nitrided layers are required at the expense of corrosion resistance. The low-pressure nitriding provides a possibility of improving the nitrided layer thickness by prolonging the nitriding time or increasing the nitriding temperature. The low-pressure nitrided sample presents a thicker nitrided layer than the normal atmospheric pressure nitrided one when treated at 475 °C for 8 h.

To identify the effect of gas pressure on the growth kinetic of nitrided layers, the plots of nitrided layer thickness (\( \ln(d) \)) against temperature (\( T^{-1} \)) for samples treated under atmospheric and low pressure conditions
for 8 h are shown in figure 8. The relationship between $\ln(d)$ and $T^{-1}$ can be described by the linear Arrhenius-type equation as follows [34]:

$$
\ln d = A - \frac{Q}{2R} \cdot \frac{1}{T}
$$

(1)

where $d$ and $T$ are the thickness of nitrided layers and absolute temperature respectively, $Q$ is the activation energy, $A$ and $R$ are both constant. The activation energy for the diffusion process can be obtained by calculating the slope of the plots. The calculated activation energy of the nitriding process in the atmospheric and low-pressure conditions are 196 and 220 kJ mol$^{-1}$ respectively. It should be noted that the calculated activation energy for gas nitriding in our present work is higher than that for other nitriding technologies, such as 42.7–73 kJ mol$^{-1}$ for ion nitriding [35, 36]. It indicates that the ion sputtering during the nitriding process can dramatically decrease the activation energy of N diffusion. Baranowska [5] introduces the ion sputtering to remove the passive film on ASS resulting in the decreased activation energy for consequently gas nitriding (98–148 kJ mol$^{-1}$). On the other hand, the low pressure further increased the activation energy of N diffusion due to the lower N concentration gradient in the nitrided layer. Thus, it can be concluded that the growth kinetic
of nitrided layers is mainly determined both by the surface status and by the N concentration on the treated surface.

4. Conclusions

In this study, the low-pressure gas nitriding has been applied on the AISI 304 ASS in order to fabricate a thick and corrosion resistant nitrided layer. The effects of nitriding parameters on the microstructure and properties of nitrided AISI 304 ASS were systematically investigated. Several conclusions were obtained as follows:

1. The low-pressure gas nitriding could evidently restrain the precipitation of chromium nitrides and improve the thermal stability of S phase, especially at higher nitriding temperature above 475 °C and for longer duration above 16 h.

2. A nitrided layer with a high surface hardness and smooth hardness gradient could be obtained by low-pressure gas nitriding, which contributed to the improvement of toughness.
The low-pressure nitried layer usually presented a lower anodic corrosion current as compared with the atmospheric pressure nitrided layer, which indicated a better corrosion resistance for the low-pressure nitrided layer.

The activation energy of the nitriding process in atmospheric and low-pressure conditions are 196 and 220 kJ mol$^{-1}$ respectively. The layer thickness for low pressure nitriding could reach to the comparable value as the atmospheric pressure nitriding when treated under a suitable parameter. The nitride layer treated at 450 °C for 24 h under the pressure of 0.01 MPa exhibited most excellent properties among all nitrided samples.

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