Isothermal Heat Treatment of AISI 430 Ferritic Stainless Steel after High Temperature Gas Nitriding

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Abstract It has been known that the ferritic stainless steel can be changed to martensitic stainless steel when nitrogen is added. However the high hardness of martensitic stainless steel prevents the plastic deformation. In this study, instead of martensite, the surface microstructure was changed into nitrogen pearlite to increase the plastic deformation easily by isothermal heat treatment after high temperature gas nitriding (HTGN) the AISI 430 ferritic stainless steel. The isothermal treatment was carried out at 780°C for 4, 6, and 10 hrs, respectively, after HTGN treatment at 1100°C for 10 hrs. The surface layer of isothermal-treated steel appeared nitrogen pearlite composed with fine chromium nitride and ferrite. Hence, the interior region that was not affected by nitrogen permeation exhibited ferrite phase. When quenching the isothermal treated steel at 1100°C, martensitic phase formed at the surface layer. The hardness of surface layer of isothermal-treated steel and quenched steel measured the value of 150~240 Hv and 630 Hv, respectively.

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1. Introduction

High temperature gas nitriding (HTGN) is a nitrogen permeation process by heat treating in an atmosphere of nitrogen gas at high temperature [1, 2]. The depth of nitrogen permeation is dependent on the amount of alloying elements, the pressure of nitrogen gas, HTGN treatment temperature and time [3]. HTGN treatment has usually been applied to the austenitic, martensitic and duplex stainless steels, which have high Cr contents [4-7]. However, ferrite stainless steels have not been applied the HTGN treatment due to the low nitrogen solubility in the ferrite phase. Recently, a study on the HTGN treatment has progressed for ferrite stainless steel [8, 9]. After cooling the HTGN-treated ferritic stainless steel, even if air cooling, the surface phase is transformed into hard martensitic phase [9, 10]. This prevents the plastic deformation after HTGN treatment.

Therefore, it is difficult to deform the finishing products that requires deformation or machining. In order to improve the ability of plastic deformation, instead of hard martensite case, if we fabricate the soft surface layer by adjusting the cooling rate from HTGN treatment temperature, plastic deformations such as forging, rolling etc. may be facilitated. After plastic deformation, the end-user could be achieved a hard martensitic surface layer simply by cooling the solution-annealed ferritic stainless steel.

This study, using AISI 430 ferritic stainless steel, was to investigate the formation of soft surface layer by isothermal transformation heat treatment (IT) after HTGN treatment to facilitate the plastic deformation. After IT treatment, formation of martensitic phase at the surface layer were examined by solution annealing (SA). Thus the precipitates appeared after IT treatment and formation of hard martensitic phase were examined.

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Table 1. Chemical composition of the AISI 430 steel (wt.%)

Specimen	C	Si	Mn	P	S	Cr	Ni	Fe
AISI 430	0.04	0.26	0.40	-	-	16.26	0.1	Bal.

2. Experimental Procedure

The 3 mm thick plate of AISI 430 ferritic stainless steel was prepared. Table 1 shows the chemical composition of the ferrite stainless steel. The plate was cut into dimensions of 15 mm × 60 mm × 3 mm. The steel was HTGN heat-treated using a pressure changeable furnace in an atmosphere containing 1 kg/cm² nitrogen gas at 1100°C for 10 hrs, followed by isothermal heat-treated (IT) at 780°C for 4, 6 and 10 hrs (HTGN-IT). Cooling rate from HTGN temperature to IT temperature was 10°C/min. After IT treatment, the steel was solution annealed (SA) at 1100°C for 1 hr, and then quenched in the water. After heat treatment at each stage, the microstructure was observed by using optical microscope and SEM-EDS analysis after polishing and etching the steel in the solution of 15 ml hydrochloric acid, 10 ml nitric acid, 10ml acetic acid and 30 ml ethanol. Identification of phases was analyzed by using X-ray diffractometer (XRD) with Cu-K α radiation. Nitrogen, carbon and Cr contents from the surface to the interior were analyzed by using GDA after HTGN-IT treatment. Hardness measurement was carried out under the load of 100 g on cross-section from the surface to the interior by using micro-Vickers hardness tester.

3. Results and Discussion

3.1 Effect of High Temperature Gas Nitriding-isothermal Heat Treatment on the Phase Change.

Fig. 1 shows OM image of as-received AISI 430 ferritic stainless steel. The ferrite grains were arranged to the direction of rolling.

Fig. 2. shows Fe-Cr-N phase diagram at 1100°C.

Fig. 1. Optical micrograph of AISI 430 ferritic stainless steel. **Fig. 2.** Fe-Cr-N phase diagram at 1100°C derived by ThermoClac [3].

With increasing nitrogen and Cr contents, the region of austenite phase field is increased. And the austenite single phase field is shown from 0.3 to 0.7%N at the 16.3% Cr. Austenite plus M₂N phases can be seen above 0.7%N.

Fig. 3 shows the cross-sectional view of OM micrographs of isothermal heat treated steel at 780°C for 1, 4 and 10 hrs after cooling to 10°C/min

Fig. 3. Optical micrographs of isothermal transformation heat treated steel at 780°C after high temperature gas nitriding at 1100°C in AISI 430 ferritic steel. IT treatment time: a) 1 hr b) 4 hrs c) 10 hrs.

for the HTGN-treated steel at 1100°C for 10 hrs.

Layered nitrogen pearlite [11] with the 800 μm depth and small quantity of martensite between nitrogen pearlite was formed at the surface layer for 1 hour IT treatment. On the other hand, the interior region appeared ferrite and martensite (a). For the isothermal heat-treated steels for 4 and 10 hours, nitrogen pearlite without martensite phase was formed with the depth of 800 μm from the surface, and the inside regions showed only ferrite phases. Thus, $\gamma + \text{M}_2\text{N}$ phases existed at the HTGN temperature, as shown in Fig. 2, was changed into $\alpha + \text{M}_2\text{N}$ nitrogen pearlite during IT treatment at 780°C.

To identify the phase appeared at the surface of HTGN-IT treated steel, X-ray diffraction analysis was applied as shown in Fig. 4. Strong peaks and weak Cr_2N peaks were detected. This implies that

Fig. 4. XRD analysis result of HTGN-IT treated AISI 430 steel.

the Cr_2N was formed in the matrix of ferrite during IT treatment.

Fig. 5 shows SEM micrographs and EDS analysis

Fig. 5. SEM micrographs and EDS analysis results of the surface region after HTGN-IT treatment. IT-treated at 780°C for 10 hrs : a) surface b) near surface.

Fig. 6. GDS analysis result of depth below the surface after HTGN-IT treatment.

result of the surface layer after HTGN-IT treatment. Pearlite like structure, which is called nitrogen pearlite, composed with nitrides and ferrite, was observed [11]. EDS analysis showed that the precipitates were chromium nitrides.

Fig. 6 shows the variation of nitrogen, carbon and Cr contents with increasing depth below the surface. Nitrogen content decreased steeply at the outmost surface layer, and decreased gradually, reaching 0.96%N at the 20 μm and 0.84%N at the 80 μm from the surface. Also, in Fig. 2, austenite + M_2N phases can be seen above 0.7%N at the

16.3%Cr. On considering the nitrogen content of HTGN-IT treated specimen as shown in Fig. 5, it is sure that the nitrogen content may be over 0.7% at the HTGN temperature. Therefore, it is said that the austenite + M_2N phases at HTGN temperature was changed to ferrite + M_2N phases during cooling from HTGN temperature to 780°C. During nitrogen permeates from the surface to the interior, a substitutional element Cr also moves from the interior to the surface due to strong affinity between nitrogen atoms and Cr atoms [12]. This made a Cr depletion zone at the near surface region as shown in the upper part of the Figure.

Fig. 7 shows the hardness variation with depth below the surface after HTGN-IT treatment. The hardness decreased with depth, and the maximum value of ~ 240 Hv appeared after isothermal treatment for 4 hrs and 6 hrs. Also, minimum hardness value measured ~ 130 Hv at the distance of 1500 μm from the surface. From the surface hardness and microstructure of HTGN-IT treated AISI 430 steel, these hardness values are enough to deform plastically [13]. In this figure, the depth of nitrogen permeation can be deduced from the hardness as ~ 1200 μm .

Fig. 7. Hardness as a function of depth below the surface after HTGN-IT treatment.

Fig. 8. Optical micrographs of SA-treated steels at the surface region after HTGN-IT treatment.

3.2 Effect of Solution Annealing on the Phase Change for the HTGN-IT Treated AISI430 Steel.

Fig. 8 shows OM images of the solution-annealed (SA) steel at 1100°C for 1 h after HTGN-IT treatment. Martensite phase appeared at the surface layer, however ferrite phase was shown in the interior region. White phase that could not distinguish the phase with OM were also observed in the martensite matrix.

Fig. 9 shows x-ray diffraction analysis result to identify the phase appeared at the surface layer of SA-treated steel. The strong α' peaks and weak γ peaks were observed. That is, martensite and

Fig. 9. XRD analysis result of SA-treated steels after HTGN-IT treatment.

Fig. 10. Hardness as function of the depth below the surface of SA-treated steels after HTGN-IT treatment.

retained austenite were formed after SA treatment. The white phase observed in the matrix from OM (Fig. 8) might be a retained austenite.

Fig. 10 shows the hardness variation of SA-treated steel after HTGN-IT treatment. Hardness appeared high at near surface, were showed nearly constant with the value of ~ 570 Hv up to 800 μm . After that, the hardness decreased steeply. The effect of IT-treatment time on the hardness value was small.

4. Conclusions

Isothermal heat treatment (IT) after high temperature gas nitriding (HTGN) was carried out

to find a soft surface by using AISI 430 ferritic stainless steel. After IT treatment, solution annealing (SA) was performed to obtain a hard martensitic phase at the surface layer. The results obtained are summarized as follows.

1. HTGN-IT treatment showed nitrogen pearlite with the alternative structure between ferrite and nitrides (Cr_2N). The hardness of HTGN-IT treated steel decreased gradually after showing the maximum value of 240 Hv.

2. Variation of nitrogen content after HTGN-IT treatment appeared the value of 0.96%N at the 20 μm and 0.84%N at the 80 μm .

3. Solution annealing after the HTGN-IT treatment showed the microstructure of martensite and retained austenite at the surface layer. The hardness of this martensitic phase region measured the value of ~ 570 Hv.

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