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Effect of solution treatment after nitriding on fatigue properties in type 304 stainless steel

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ABSTRACT

The solution treatment after nitriding (STAN) was performed to improve the fatigue strength in type 304 steel by means of the solid solution of nitrogen. The static strength and the hardness were improved by the STAN. In laboratory air, the fatigue strength of the STAN specimen increased compared to that of the untreated specimen but was lower than that of the nitrided specimen. On the other hand, in 3% NaCl solution, the fatigue strength of the untreated specimen was almost similar to the result in laboratory air, while the fatigue strengths of the nitrided and the STAN specimens decreased significantly compared to those in laboratory air. This behavior is attributed to the sensitization by the formation of chromium-depleted zone resulted from the precipitation of CrN. In the STAN specimen, the strain-induced martensitic transformation in the run-out specimen at a stress level of fatigue limit was not detected by X-ray diffraction method, indicating that the γ-phase was stabilized by the solid solution of nitrogen. Similarly, also in EBSD analysis, the strain-induced martensitic transformation was not seen at crack wake in the STAN specimen.

1. Introduction

Austenitic stainless steel, type 304 (18Cr–8Ni), is widely used as a structural material under the severe conditions such as the nuclear power plants and the chemical plants, because of high corrosion resistance, good toughness and ductility. However, the strength and wear resistance of type 304 steel is relatively low as compared to other structural materials. Besides, the austenitic phase (γ-phase) in type 304 steel is metastable as can be expected from its low nickel equivalent [1–3]. Therefore, it becomes susceptible to the strain-induced martensitic transformation in cold working [4–6]. Once the strain-induced martensitic transformation occurs, high environmental sensitivity of martensitic phase (α′-phase) lowers the corrosion resistance [7,8]. Although the increase in nickel content brings on the stability of γ-phase, nickel content reflects the manufacturing costs [9]. So the special attention is paid to nitrogen (N) as an alternative element of nickel [9–13]. Nitrogen is one of the γ-phase stabilizing elements, which can improve the mechanical properties, fatigue strength and corrosion resistance [10–13].

In recent years, high nitrogen austenitic stainless steels were developed, in which γ-phase was stable in spite of considerably low content of Ni [14]. As a manner of the addition of nitrogen to stainless steel, the heat treatment in high temperature nitrogen gas was examined [14–16], and consequently high mechanical properties and corrosion resistance were obtained [16,17]. This procedure is a kind of chemical heat treatment with a diffusion process of nitrogen and is different from a conventional nitriding, that is, the heat treatment in high temperature nitrogen gas results in the solid solution of nitrogen but a conventional nitriding forms the nitrided layer. So, in this research, a solution treatment after the plasma nitriding was conducted to enhance the solid solution of nitrogen [18,19]. Since the diffusion of nitrogen atoms into substrate is carried out during heat treatment process, it is expected that the material characteristics could be improved.

In the present study, the solution treatment after nitriding (STAN) was performed to stabilize the γ-phase in type 304 steel and to improve the mechanical properties, such as static strength and fatigue behavior. Although the studies on the STAN have been conducted from a view point of surface modification [18,19], the study on fatigue behavior in corrosive environment using the STAN treated type 304 steel has been limited. Therefore the purpose of the present study is to clarify the fatigue properties of the STAN

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treated type 304 steel in laboratory air and in 3% NaCl solution. In addition, the results obtained were compared with those of the different type 304 steel annealed in high temperature nitrogen gas [20].

2. Experimental procedures

2.1. Materials and specimen configuration

The material used is austenitic stainless steel, type 304 steel (18Cr–8Ni), whose chemical composition (wt.%) is given in Table 1. In this material, Ni equivalent (Ni (eq)) is as follows [3];

$$\mathrm{Ni(eq)} = \%\mathrm{Ni} + 30 \times \%\mathrm{C} + 0.5 \times \%\mathrm{Mn} + 30 \times \%\mathrm{N} = 8.03 + 30 \times 0.06 + 0.5 \times 1.65 = 10.66(\%)$$

Since the value of Ni (eq) is less than 12%, the austenitic phase (γ-phase) of this material is seemed to be metastable.

The material was solution treated at 1080 °C for 30 min followed by water quenching, and then machined to the hourglass-shaped fatigue specimens shown in Fig. 1. Before fatigue test, the specimen surface was polished by emery paper and buff-finished. After machining, plasma nitriding was conducted at 500 °C for 8.5 h, and then re-solution treatment was performed at 1200 °C for 1 h followed by oil quenching, which treatment was designated STAN (solution treatment after nitriding) hereafter.

Another study was performed at the same time [20], in which a different type 304 steel annealed in high temperature nitrogen gas, at 1100 °C and 1200 °C, was examined. This treatment is designated ATNG (annealing treatment in nitrogen gas) in the present study. The results of the ATNG specimen were compared with those of the STAN specimen.

2.2. Experimental procedures

Fatigue tests in laboratory air and in 3% NaCl solution were conducted on three kinds of specimens, that is, the untreated, the nitrided and the STAN. Tests were performed using cantilever-type rotary bending fatigue testing machines operating at a frequency of 53 Hz. The 3% NaCl solution was kept at 30 °C and was dropped on the specimen surface in a corrosion cell attached to the testing machine.

Crack initiation and crack growth were monitored using a replication method. Stress intensity factor of small surface cracks was calculated using the analytical solution developed by Shiratori et al. assuming an aspect ratio, $a/c = 1$, where a and c are a crack depth and a half crack length at surface, respectively [21].

In order to evaluate a corrosion resistant property of the untreated and the STAN specimens, the anodic polarization was measured electrochemically in 3% NaCl solution using potentiostatic approach. In addition, the compositions of precipitate were analyzed by EDS (energy dispersive X-ray spectroscopy). The XRD (X-ray diffraction) method and the EBSD (electron backscatter diffraction) analysis were used to detect the strain-induced martensitic transformation.

Table 1
Chemical composition of type 304 (wt.%).

C	Si	Mn	P	S	Ni	Cr	Fe
0.06	0.29	1.65	0.04	0.03	8.03	18.62	Bal.

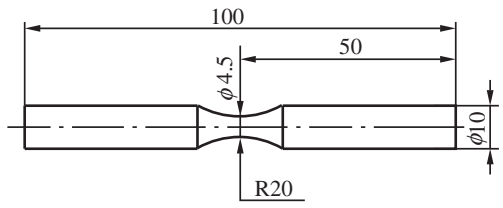

Fig. 1. Fatigue specimen configuration (in mm).

3. Results

3.1. Microstructures

Fig. 2 shows the microstructure of each specimen, (a) the untreated, (b) the nitrided layer, (c) the nitrided and (d) the STAN, in which the average grain sizes were almost similar, that is, (a) 65 μm, (c) 73 μm and (d) 69 μm, respectively. This indicates that the grain growth did not occur during nitriding and the STAN treatment. The surface of the nitrided specimen is covered by the nitrided layer which is composed of CrN. As also seen in Fig. 2(b), the nitrided layer formed on specimen surface is about 50 μm in thickness.

3.2. Hardness distributions

The Vickers hardness profiles of the nitrided, the untreated and the STAN specimens are shown in Fig. 3. Hardness on the surface of the nitrided specimen is 1360 *HV*. As compared to the untreated specimen, 153 *HV*, it becomes 8.8 times harder. However, the interior hardness decreases rapidly and corresponds with that of the untreated specimen at the depth of 0.1 mm from the surface. On the other hand, the hardness on the surface of the STAN specimens is about 280 *HV*, 1.8 times harder than that of untreated specimen and the interior hardness is harder than the untreated and the nitrided specimens.

Fig. 4 shows the hardness profiles of specimens performed three kinds of STAN treatments and also includes the results of ATNG specimens annealed in nitrogen gas at 1100 °C and 1200 °C, for comparison [20]. As seen in Fig. 4, the hardness of the STAN specimen at 1200 °C, 45 min corresponds with that of the untreated specimen at a depth of 1 mm from the surface. On the other hand, the hardness profiles of the STAN specimens at 1200 °C, 60 min and 90 min indicate a similar tendency, and the interior hardness is harder than the STAN 45 min specimen. This suggests that the nitride could be resolved by the STAN treatment and the nitrogen diffused to the deeper area of specimen. Since the hardness profiles of the STAN 60 min and 90 min specimens were almost similar, the STAN 60 min was selected as a heat treatment condition. In comparison with ATNG specimens, the interior hardness of the STAN specimens, 60 min and 90 min, is slightly higher than that of the ATNG specimens. However, as the hardness profiles of the STAN and the ATNG specimens are similar, it seems that the similar mechanism of solid solution strengthening by nitrogen would take place in the both treatments.

In order to clarify the mechanism of the increase in hardness, SEM observation and EDS analysis were performed. The microstructure near surface of the 1100 °C ATNG specimen is shown in Fig. 5. As seen in the figure, many precipitates are recognized at grain boundaries and in grains. In addition, the amount of precipitates decreases with increasing in a depth from the surface. Fig. 6 indicates the precipitate scanned by EDS. A square on the precipitate shows a scanning point of EDS. Based on the results of quantitative EDS analysis on Cr, N and Fe, the atomic concentration ratio for Cr, N and Fe was 55:40:5. Since the ratio of Cr to N is close to 1:1, it is considered that the precipitate is chromium nitride, CrN. It should be noted that the similar precipitation was also seen in the STAN specimen.