Very High Cycle Fatigue Behavior of Plasma Nitrided 316 Stainless Steel

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Abstract In this study, plasma nitriding was performed for 316 stainless steel under 2 different processing time, 15hours and 25hours. Cantilever type rotational bending fatigue tests were carried out using the nitrided and non-treated samples in high cycle and very high cycle fatigue life regime in order to examine the influence of the plasma nitriding on the fatigue properties in the both fatigue life regimes of 316 stainless steel. As a result, the fatigue strength was improved by the plasma nitriding and the shape of the S-N curves was a asymptote shape with a fatigue limit up to $N = 10^8$ cycles. The fatigue cracks initiated from specimen surface in all specimen. However, the improvement of fatigue strength by plasma nitriding and the influence of nitriding time on the fatigue strength was small because the nitriding layer was quite thin, especially in the diffusion layer. For this reason, it is believed that the nitrided layer could not affect significantly the fatigue strength.

Keywords Very high cycle fatigue, Plasma nitriding, Stainless steel, Cantilever-type rotary bending fatigue test.

1. Introduction

Surface treatment is widely used to improve fatigue, wear and corrosion resistance of various industrial products. There are many surface treatment methods, such as carburizing, induction hardening, shot peening and physical vapor deposition coating etc. These treatments form a hardened surface layer with compressive residual stress, and therefore, the fatigue properties are improved by the surface layer [1-9]. Nitriding is one of the surface treatment methods. Nitriding produces a nitrogen diffused surface layer with a compound layer which is formed by reaction between nitrogen and nitride-forming elements in a material such as aluminum, chromium, molybdenum and titanium. Good fatigue and wear resistance are obtained by the nitrided surface layer. However, it is well known that the nitriding treatment is difficult to apply to stainless steel because the stainless steel has a passivation film on the surface which hinders the diffusion of nitrogen into the material. Therefore, the removal of the passivation film is required when the nitriding treatment is applied to the stainless steel.

There are three major methods to perform the nitriding process; gas nitriding, salt-bath nitriding and plasma nitriding processes. In particular, plasma nitriding process is a very attractive method to improve the surface properties for various engineering materials due to its advantages, such as low emission of toxic gases, low maintenance cost and low pollution, compared with conventional gas or salt bath nitriding process [10]. Plasma nitriding process has many parameters: the nitriding temperature, the gas mixture and the time duration [11-12]. These parameters strongly affect the material properties of nitrided samples. Many researchers have reported the influence of the parameters on the fatigue properties using various steels and have also reported that the fatigue limit can be improved by the plasma nitriding. In particular, the fatigue limit increases with increasing the processing time under cyclic bending stress due to the thick hardened layer because the fatigue cracks initiate from internal defects such as inclusions or cavities or from the interface between substrate and nitriding layer [13-18].

Since the plasma nitriding process can use the hydrogen etching effect which can remove the surface oxide layer as well as surface contamination, the plasma nitriding process can form a nitrided and diffused surface layer for the stainless steel [19-22]. The plasma nitriding will improve the fatigue strength of the stainless steel but the plasma nitriding may cause the decrease of the fatigue strength in very high cycle fatigue life regime by an internal fracture because the plasma nitriding produces thin hardened layer and the fatigue failure of some surface hardened steel occurs at stress levels below the conventional fatigue limit in the life regime greater than 10^7 cycles [23]. However few studies on the fatigue life of plasma nitrided stainless steels have been carried out in the very high cycle regime.

In the present study, SUS316 austenitic stainless steel was treated by the plasma nitriding under two nitriding times. Rotating bending fatigue tests were carried out for these nitrided specimens in order to investigate the effect of the nitriding time on the fatigue behavior in the high cycle and very high cycle fatigue regime. Fracture morphologies were observed using a scanning electron microscope (SEM) and the influence of the plasma nitriding process on the fatigue behavior of austenitic stainless steel was discussed.

2. Experimental Procedure

The material used in this study was austenitic stain less steel, SUS316. The fatigue specimens were machined into the shape and dimensions as shown in Fig. 1. The round notch surface was polished with a grinder having a mesh size of #100. Notch radius was 7 mm and the stress concentration factor of the specimen was K_t=1.06.

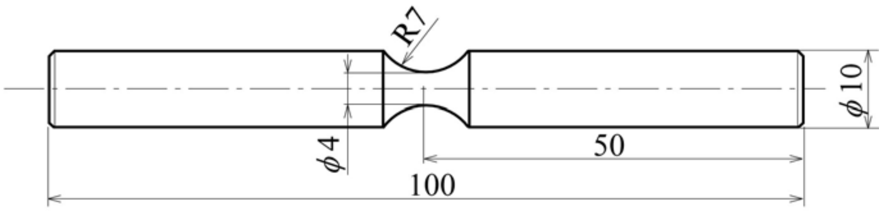

Figure 1. Shape and dimensions of specimen.

The surface of the specimen to be exposed to plasma was cleaned using an ultrasonic bath in acetone. A vacuum chamber was pumped down to 50 mTorr and then back-filled with a gas mixture of NH_3 and H_2 up to 3 Torr (NH_3:H_2 mixing ratio; 4:1). The plasma nitriding process was immediately carried out with a pulsed dc potential at the designed time in the glow discharge of the plasma. In the present study, plasma nitriding was performed at 703 K for 15 hours or 25 hours. These nitrided specimens will be called N15 and N25, respectively. After the plasma nitriding, the vacuum chamber was pumped down to 50 mTorr and the specimens were furnace cooled to room temperature.

Fatigue tests were carried out at room temperature by using a dual-spindle cantilever-type rotating bending fatigue-testing machine, which is the standard testing machine in the Research Group for Statistical Aspect of Materials Strength, Japan [24]. The stress frequency was 3150 rpm and the stress ratio R = –1. All of the fracture specimens were observed using SEM. The run-out number of cycles was N = 1.0×10^8 cycles. The fracture surface of the specimen was observed using SEM. A Vickers microhardness tester was used to measure the microhardness of samples. The parameters used in the microhardness test were 100 gf and a duration 15 s. The microstructure was observed using a cross sectional surface etched by Marble's regent (4.0 g CuSO4, 20 ml HCl, and 20 ml H_2O).

3. Experimental Results and Discussions

3.1. Characterization of hardened layer

Figure 2 shows the cross sectional surfaces subjected to the two different nitriding processes, using Marble's regent as an etchant to reveal the microstructure. The unetched surface layers, which are well-known as "white layer", were formed at both specimen's surfaces. The white layer for the N25 was slightly thicker than that for the N15. However, the white layer was thin and the thickness were about 20 µm for the N15 specimen and 30 µm for the N25 specimen, respectively. In both cases, the microstructure and the grain size below the white layers were no difference from the as-received series and the diffusion layer could not be identified by etching. So, the difference between the as-received and nitrided series was only forming of the surface white layer.

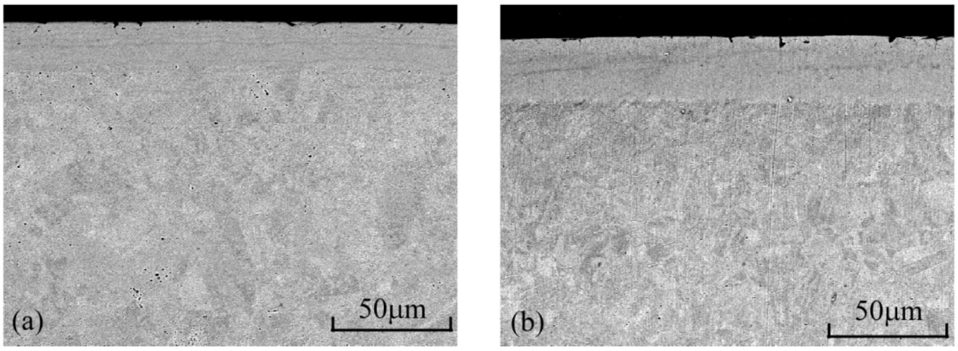

Figure 2. SEM images of plasma nitrided SUS316 stainless steel etched by Marble regent: (a) N15 and (b) N25 specimens.

Figure 3 shows the micro-Vickers hardness distribution of nitrided specimens on the cross sectional surface. The hardness of the as-received specimen was uniform from the surface to the core and the 30 points average value was about 331 HV. Compared with the virgin hardness, the hardness increased near the surface. The highest hardness values, 985 HV for the N15 specimen and 1041 HV for the N25 specimen, were measured at the surface, which is approximately three times of the virgin hardness of 331 HV. However, the hardness decreased steeply toward the center and the hardness at 50µm deep was almost same as virgin hardness for both nitrided specimens. Therefore, the thickness of the hardened layers, defined as a distance from the surface to the unhardened matrix, was less than 50 µm for both nitriding times. Since the thickness of white layer as shown in Fig.2 was about 20 - 30µm and the half size of indentation for 331 HV was about 12µm, the thickness of the diffusion layer was less than about 10µm.

In general, nitriding process produces a compound layer referred to as the white layer, and a diffusion layer below the compound layer. The former is a thin layer with high hardness but brittle [25]. As a result, the fatigue strength sometimes are degraded by the brittle layer. The latter is a relatively thick hardened layer. In the diffusion layer, the hardness gradually decreases gently toward the core. The depth of diffused zone is generally more than few hundred micrometers [13-18]. The depth of the diffused zone is important to improve the fatigue strength as well as the hardness [14]. In this study, however, no obvious diffusion layer was observed on the etched surface, and only the white layer was observed at the surface. Moreover, the hardened layer was quite shallow and the thickness was less than 50 µm which included the thickness of the compound layer. These results suggest that the depth of diffusion layer is quite shallow for stainless steel. In our previous works, plasma nitriding was carried out using a same equipment using chromium molybdenum steel (JIS SCM435) [17]. The thickness of hardened layer was about 0.1 - 0.3 mm with ~10μm compound layer for each material. The hardened layer is obviously thicker than that of present study. The difference indicates that the passivation film reforms at the stainless surface during the plasma nitriding process though the passivation film is removed intermittently by hydrogen etching effect [26, 27]. The reformation of the passivation film hinders nitrogen diffusion, and this results in a thinner diffusion layer. The thickness of the hardened layer and the value of the hardness near the surface are important parameters that influence the site of fatigue crack initiation, as discussed in detail in the following sections.

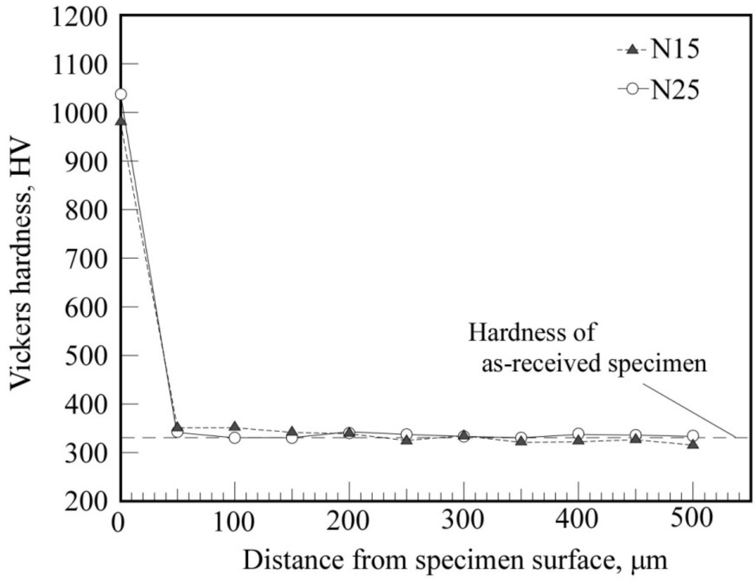

Figure 3. Distribution of Vickers-hardness in the nitrided specimens.

3.2. S-N curves

The fatigue tests to failure were carried out in air up to N= 10^8 cycles. Figure 4 shows the results of fatigue tests for as-received, N15 and N25 specimens. The *S-N* curve of the as-received specimens (represented by open square mark in Fig. 4) presented a horizontal asymptote shape, which is typical shape for low strength material [28, 29], with a fatigue limit up to N = 10^8 cycles. The fatigue limit σ_w defined as the horizontal line was about 500 MPa. The shape of two *S-N* curves of plasma nitrided specimens, represented by open triangle and circle marks in Fig. 4, showed also horizontal asymptote shape with a fatigue limit up to N = 10^8 cycles, that were similar to the *S-N* curve of the as-received specimens. The fatigue limit σ_w defined as the horizontal line was about 600 MPa for the N15 and 550 MPa for the N25 specimens, respectively. The fatigue limit of the N25 specimen with thicker hardened layer was slightly lower than that of the N15 specimen with relatively thin hardened layer as well as the fatigue strength in the life regime $N < 10^6$ cycles.

Increased fatigue strength after nitriding is a well-known phenomenon for various steels. The increase is caused by the increase of surface hardness and the formation of compressive residual stresses in the surface layer during the nitriding process. The high hardness layer hinders the fatigue crack initiation. The compressive residual stress is superimposed to the external load and leads to a reduction of the effective stress in tension. Since only a tensile stress produces fatigue cracks and contributes to crack propagation, a reduction of the effective stress in tension increases the fatigue strength. In this study, the fatigue strength was improved by the plasma nitriding process, however, the fatigue limit was clearly smaller than that predicted by surface hardness using σ_w = 1.6 HV which is well-known equation between the hardness and upper bound for fatigue limit of low and medium strength materials. In addition, the specimen with thicker hardened layer showed lower fatigue limit. The small improvement and small influence of the thickness of hardened layer mean that the nitrided layer does not act on the fatigue strength efficiently.

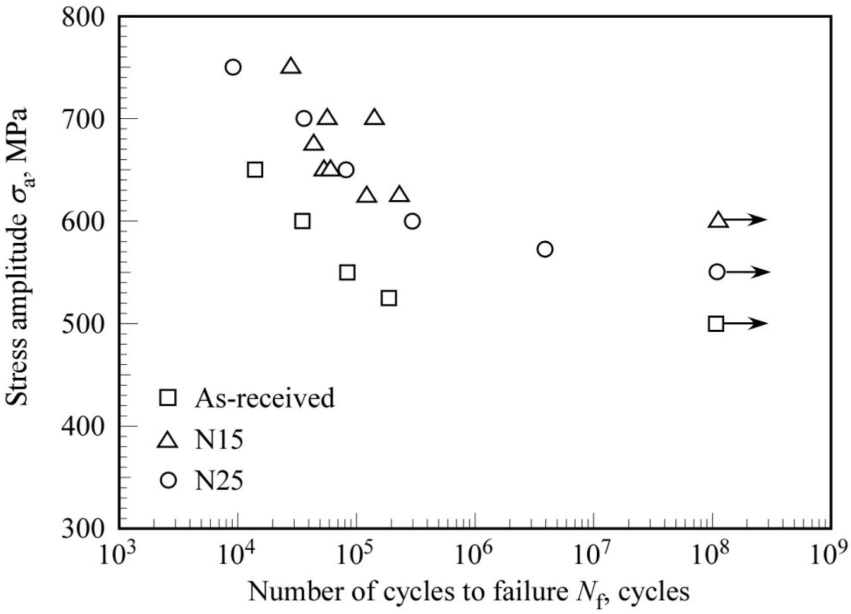

Figure 4. *S-N* curves.

3.3. Fracture surfaces

Fracture surfaces were observed using SEM. Figure 5 shows the typical SEM images of fracture surfaces for as-received and plasma nitrided specimens. Based on SEM observations of the fracture surfaces, only the surface fracture mode was observed though the hardened layer was formed on the surface of nitrided specimens. A subsurface fracture mode, fish-eye or fracture from substrate /nitriding layer interface, or cracking in the compound layer was not observed in all specimens.

The result means the nitrided layer increases the fatigue resistance on the specimen surface but the resistance is lower than the resistance for internal failure, and then the surface fracture occurs from the nitrided layer prior to internal fracture such as fish-eye failure. The nitrided layer at the stainless steel surface consists of the thin compound layer and quite thin diffusion layer as shown in Figs. 2 and 3. Since the influence of the diffusion layer on the fatigue strength can be ignored due to the small thickness, the compound layer is a dominant factor in the improvement of fatigue strength. However, the influence of compound layer is restricted because the thickness is only 20~30µm. In consequence, the small improvement of fatigue strength will be obtained for plasma nitrided specimens as shown in Fig.4. The small influence of the nitriding time will be also explained by the similar thickness of hardened layer.

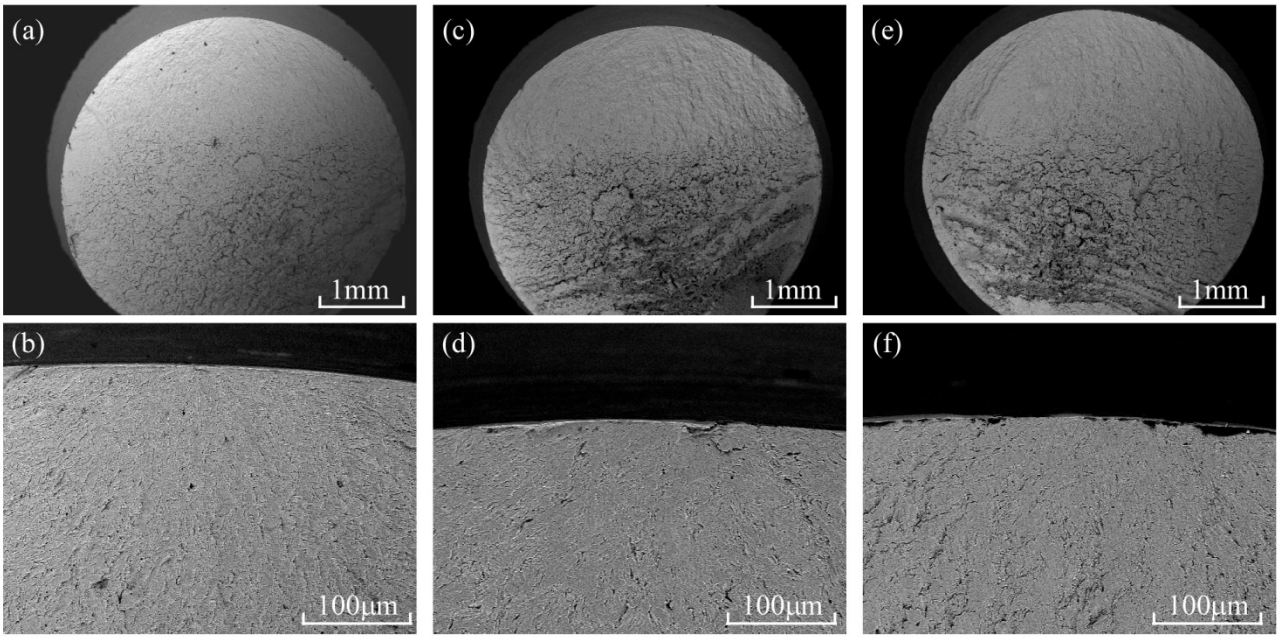

Figure 5. Typical SEM images of fracture surfaces; (a), (b) As-received, σ_a=525MPa, N_f = 1.88×10^5 cycles, (c), (d) N15, σ_a=625MPa, N_f=2.31×10^5 cycles and (e), (f) N25, σ_a=600MPa, N_f=2.96×10^5 cycles.

In conclusion, the degree of increase in the surface hardness and the thickness of the hardened layer are dominant factors determining the fatigue limit level of the plasma nitrided specimens. Therefore, the fatigue limit levels of the N15 and the N25 specimens were almost the same and thickness of the hardened layers had almost the same value. In addition, the improvement by plasma nitriding process is small because the nitrided layer, particularly diffusion layer, is quite thin. It is believed that the increase of the depth of diffused zone could improve the fatigue strength in plasma nitride stainless steels.

4. Conclusions

The following conclusion can be reached from the results of this study:

(1) A hardened surface layer was formed at the SUS316 stainless steel surface by plasma nitriding. However, the hardened layer was quite shallow. The thickness of the hardened layer was less than 50 μm for both nitriding time of 15 and 25 hours.

(2) The *S-N* curves represent a horizontal asymptote shape for all specimens.

(3) The fatigue strength was improved by plasma nitriding in all life regimes. However, the improvement of the fatigue strength was small due to the small thickness of the nitrided layer. The nitriding time also had a small influence on the fatigue strength because the thickness of the nitrided layer was similar for both nitriding times.

(4) Fatigue crack was initiated from specimen surface for all specimens.

(5) The degree of increase in the surface hardness and the depth of the hardened layer are dominant factors determining the fatigue limit level of the plasma nitrided specimens.

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