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Effect of Fine Particle Peening Treatment prior to Nitriding on Fatigue Properties of AISI 4135 Steel*

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Abstract
In this study, a new hybrid surface modification process, fine particle peening (FPP) treatment prior to nitriding, was proposed. In order to clarify the effects of FPP treatment prior to nitriding on the fatigue strength of notched AISI 4135 steel with a stress concentration factor K_t of 2.36, fatigue tests were conducted at room temperature using a rotational bending fatigue testing machine. Hardness and residual stress distributions were measured in order to characterize the surface-modified layer. As a result, the surface hardness of the FPP-treated specimen before nitriding was higher than that of the nitrided specimen. The surface microstructures of treated specimens were characterized using scanning electron microscopy (SEM) and X-ray diffraction (XRD). The crystal structure of the compound layer of the FPP-treated specimen before nitriding was different to that of the nitrided specimen. The compound layer of the FPP-treated specimen was dense. This suggests that FPP treatment prior to nitriding is very effective for improvement of the fatigue strength of steel.

Key words: Fatigue Strength, Nitriding, Peening, Compound Layer, Residual Stress, Hardness

1. Introduction

Nitriding has been widely used as a surface modification technique to strengthen the surface of various engineering materials. The nitriding process forms a high hardness layer with a high compressive residual stress at the surface of the treated material. The nitrided layer consists of two sub-layers; one is a compound layer and the other is a diffused layer. The advantage of the compound layer is that it improves the wear resistance; however, it reduces the fatigue strength due to its brittleness. Generally the compound layer is removed using a grinding process, in order to prevent the nitrided product from decreasing the fatigue strength of the treated material. Pellizzari et al. [1] proposed that it could be possible to avoid thermal crack propagation into the diffusion layer by reducing the thickness of the compound layer. Kobayashi et al. [2] reported that the fatigue strength of gas- and ion-nitrided steel could be estimated using the $\sqrt{area}$ parameter.

When a hardened surface and compressive residual stress are both present, they are effective in improving fatigue strength. Although the case depth increases with increasing nitriding temperature and time, prolonged nitriding has a tendency to decrease the surface hardness and compressive residual stress near the surface of the specimen [3, 4]. Single

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surface treatment has some limitations in improving fatigue strength; therefore, a new surface modification combining two existing processes has been developed [5-7]. For example, Wroblewski et al. [5] showed that the compound layer porosity was decreased by introducing a shot peening process prior to nitriding.

Moreover, it was reported that the fatigue strength of a nitrided rolled specimen was higher than that of a nitrided annealed specimen [8]. Based on these results, it is effective to increase the dislocation density near the surface before nitriding. In this study, we introduced a fine particle peening (FPP) treatment prior to nitriding. The aim of this study is to characterize the compound layer generated by nitriding after FPP treatment, and to clarify the effect of the FPP treatment prior to nitriding on the fatigue properties of surface-modified steel.

2. Experimental procedures

The material used in this study was AISI 4135 chromium molybdenum steel with the chemical composition shown in Table 1. AISI 4135 steel bars 16 mm in diameter were machined into the shape and dimensions shown in Fig. 1. The stress concentration factor of the specimen was 2.36.

Figure 2 shows a flowchart illustrating the specimen preparation. Seven types of specimen were prepared with different surface-modified layers. The nitriding process was performed at 823 K for 6 h in nitrogen and ammonia atmospheres. The FPP treatment conditions are given in Table 2. The shot particles used in this study were made from a high-speed tool steel and were 55 μm in diameter with a Vickers hardness of 876HV.

In order to characterize the surface-modified layer, distributions of the micro-Vickers hardness and residual stress were measured. Residual stress was measured at the transverse section of the smallest diameter using X-ray diffraction (XRD) with a position-sensitive proportional counter (PSPC) system under the conditions shown in Table 3. The depthwise distribution was measured by electrochemically removing the local surface area. The surface microstructures of treated specimens were observed using scanning electron microscopy (SEM). The crystal structures of treated specimens (ϕ15 mm × 4 mm) were identified using XRD with Cu-Kα radiation (wavelength 0.154 nm). High-cycle fatigue tests were then performed at room temperature using a rotational bending machine (3000 rpm).

3. Results and discussion

3.1 Evaluation of compound layer

Figure 3 shows the cross-sectional microstructures of the N and FN series etched with Nital (2%). The presence of the compound layer can be clearly observed. In the case of the N series (Fig. 3(a)), cracks and pores are clearly observed at the surface of the compound layer. On the other hand, the compound layer of the FN series did not possess cracks and pores (Fig. 3(b)). Figure 4 shows field emission-SEM (FE-SEM) images of the

Table 1 Chemical composition of steel (mass%)

C	Si	Mn	P	S	Ni	Cr	Mo	V	Cu	Al
0.37	0.21	0.82	0.011	0.013	0.09	1.10	0.15	0.01	0.10	0.017

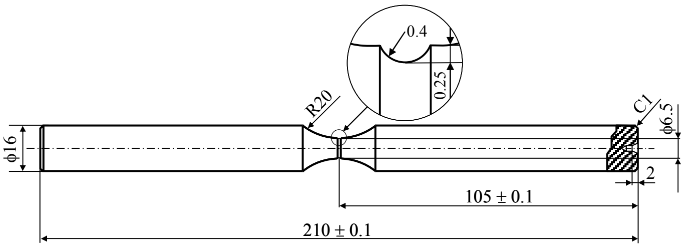

Figure 1 Specimen configuration ($K_t = 2.36$)

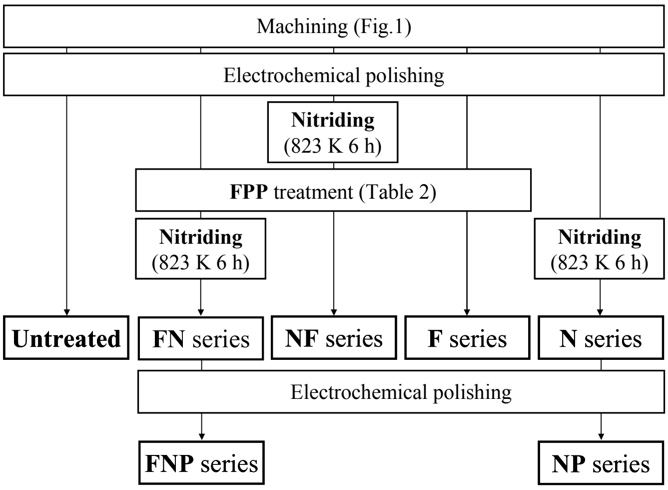

Figure 2 Flowchart illustrating the specimen preparation

Table 2 Conditions of FPP treatment

Shot particles	SKH59
Particles hardness	876HV
Particle diameter, μm	55
Air pressure, MPa	0.6
Peening time, s	60
Nozzle distance, mm	100

Table 3 Conditions of residual stress measurement

Tube voltage, kV	40
Tube current, mA	30
Diffraction angle 2θ, deg	156.4
Incident angle, deg	10, 20, 30, 35, 40
Beam diameter, mm	$\phi 2$
Stress constant, MPa / deg	-317.91

microstructures at a higher magnification. A local material stratification pattern was observed near the surface of the FN series (Fig. 4(b)). This unique structure is generated by the FPP treatment.

Figure 5 shows the micro-Vickers hardness distribution at the cross-sections. Near the surface, the hardness value for the FN series was higher than that of the nitrided specimens (N series). The depth of the hardened layer was approximately 0.4 mm for both series. The FPP treatment affects the surface hardness of the nitrided steel, and to clarify the reason for this, the amount of diffused nitrogen near the surface was measured by electron probe micro analysis (EPMA). As a result, the amount of nitrogen in the FN series was higher than that in the N series (Fig. 6). Generally, defect densities and grain size are important factors that enhance the nitrogen diffusion process [9, 10]. The FPP treatment can create high dislocation densities at the surface layer, enabling nitrogen to diffuse into the material, resulting in a harder layer at the surface of the FN series.

XRD analysis was performed for 2θ angles ranging between 20° and 90°, although only the diffraction peaks produced between 30° and 60° are shown in Fig. 7. The compound layers of the N and FN series are comprised of ε-Fe_3N and γ'-Fe_4N nitrides. Similar

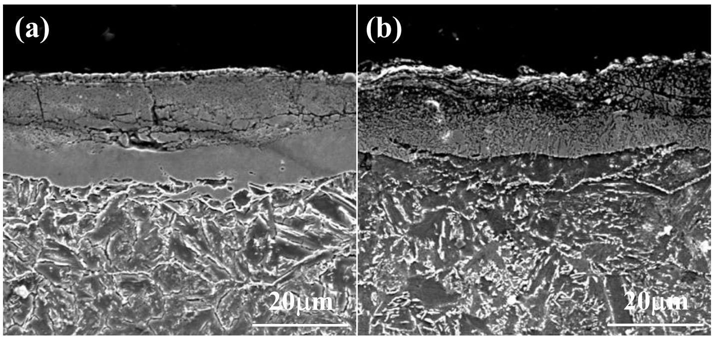

Figure 3 Cross-sectional SEM micrographs of the (a) N series and (b) FN series

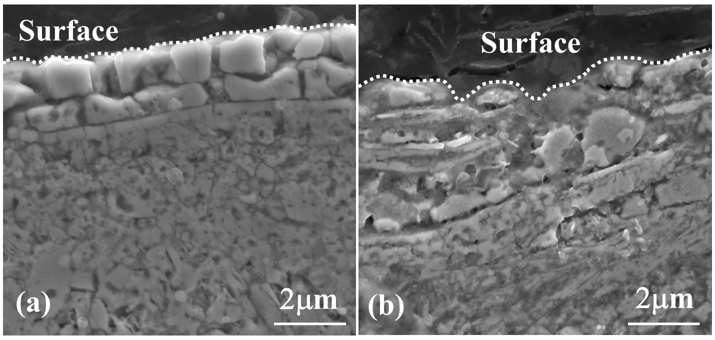

Figure 4 Cross-sectional FE-SEM micrographs of the (a) N series and (b) FN series at higher magnification

diffraction patterns were produced between the N and FN series, but the intensities of ε(002), γ'(111) and ε(112) were different. This indicates that the crystal structure of the compound layer in the FPP-treated specimen prior to nitriding is different to that of the nitrided specimen.

Figure 8 shows the residual stress distribution. Nitriding and FPP treatment generate compressive residual stress; however, the compressive residual stress of FN series, which was achieved by both FPP treatment and nitriding, was similar to that of the nitrided specimens. This result indicates that heating during the nitriding process relaxes the compressive stress previously generated by the FPP treatment. To clarify only the effect of heat on the relaxation of residual stress, the FPP-treated specimens (F series) were heated under the same conditions as in the nitriding process, but in a vacuum (FH series), and the residual stress was then measured. The value of residual stress measured for the FH series specimen was approximately 82 MPa, much lower than that of the F series surface (539 MPa).

3.2 Effect of the FPP treatment prior to nitriding on fatigue strength of steel

In order to investigate the effect of the FPP treatment prior to nitriding on fatigue strength, several rotational bending fatigue tests were conducted. Figure 9 shows the results of the high cycle fatigue tests. The vertical axis represents the nominal stress amplitude applied to the specimens. The fatigue strength of the FN series was higher than that of the N series. This indicates that the FPP treatment improves the fatigue strength of the nitrided specimens. As discussed in section 3.1, the compressive residual stress of the FN series was similar to that of the nitrided specimens (N series), implying that the compound layer can affect the fatigue strength.

To clarify the reasons for the higher fatigue strength of the FN series than the N series, the fatigue strength of electrochemically polished specimens after nitriding was evaluated. The fatigue strength of the NP series was higher than that of the N series. This means that the compound layer generated by nitriding reduces the fatigue strength of the specimen, due to the presence of cracks and pores (Fig. 3(a)); however, the fatigue strength of the FN

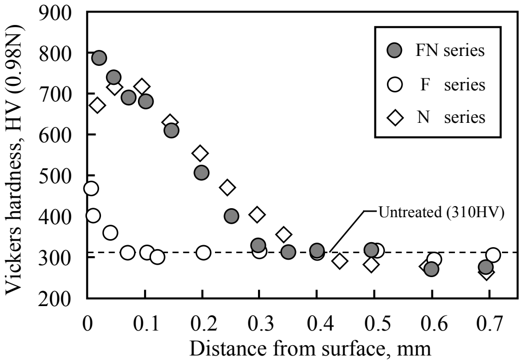

Figure 5 Distribution of Vickers hardness

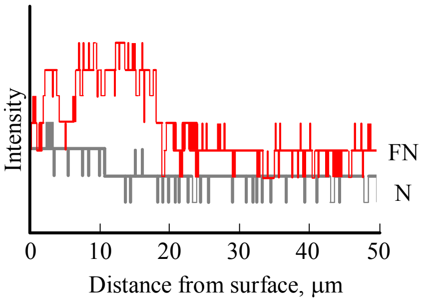

Figure 6 Nitrogen content profiles

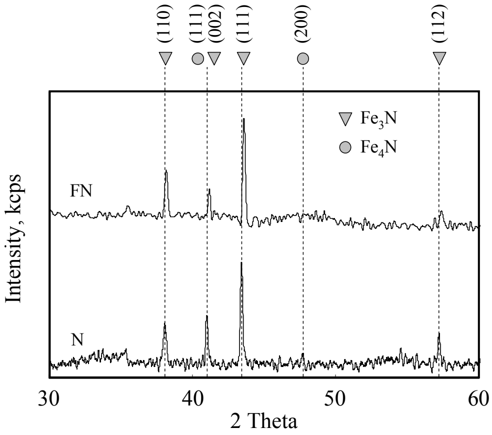

Figure 7 X-ray diffraction patterns

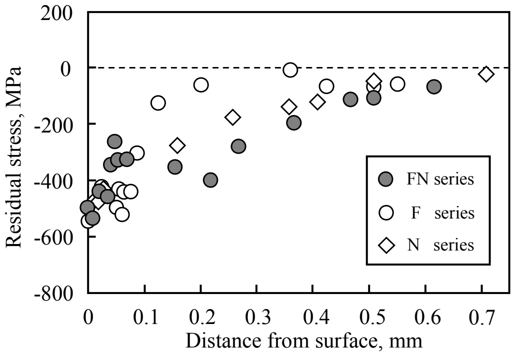

Figure 8 Distribution of residual stress

series was similar to that of the FNP series. These results imply that the FPP treatment prior to nitriding introduced in this study increases the fatigue strength of the compound layer generated during the nitriding process. This indicates that the FPP treatment prior to nitriding is very effective for improvement of the fatigue strength of steel.

The fatigue strength of nitrided steel can also be increased by removing the compound layer by FPP treatment. Figure 10 shows a cross-sectional SEM micrograph of the NF series microstructure etched with Nital (2%). It can be seen that the FPP treatment removed the porous compound layer. As a result of the fatigue test of the NF series, the FPP treatment after nitriding increased the fatigue strength of the nitrided specimens. The NF series showed the highest fatigue strength of all (Fig. 11).

3.3 Effect of FPP treatment after nitriding on fatigue strength

FPP treatment after nitriding increased the fatigue strength of nitrided steel by removing the porous component of the compound layer; however, the NF series showed higher

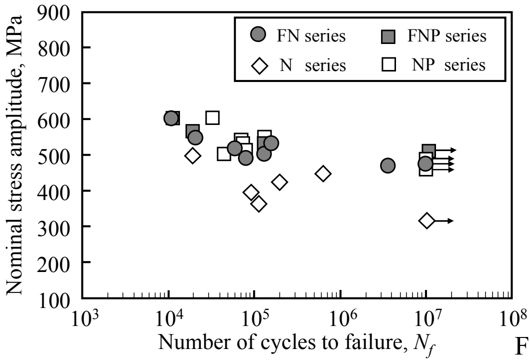

Figure 9 Results of fatigue tests

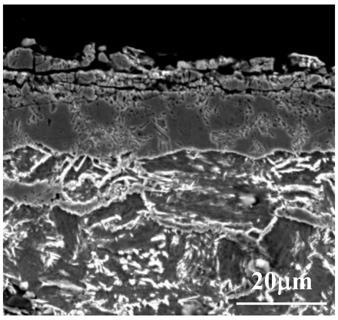

Figure 10 Cross-sectional SEM micrograph of the NF series microstructure

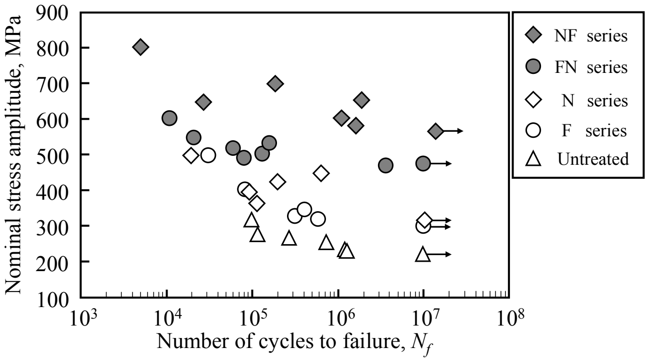

Figure 11 Results of fatigue tests

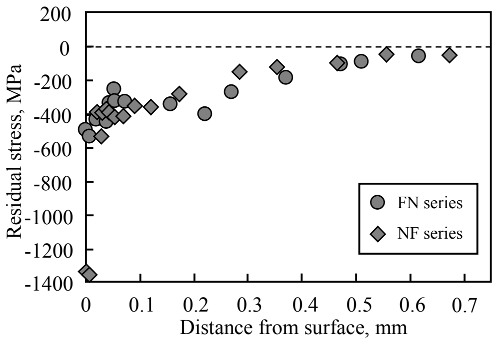

Figure 12 Distribution of residual stress

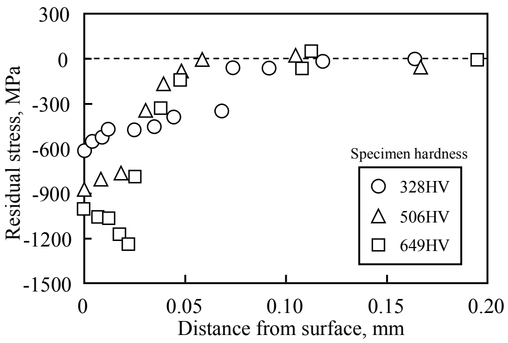

Figure 13 Distribution of residual stress

fatigue strength compared with the NP series, which lacked the porous component. To clarify the reason for this, the residual stress in the NF series was measured, as shown in Fig. 12. The compressive residual stress observed for the NF series was extremely high (approximately 1300 MPa) compared with the other series. This high compressive residual stress resulted in an increase of the fatigue strength in the NF series.

The sequence of treatment did affect the residual stress distribution of steel. To discuss the reason for this, three types of specimens with different hardness (328 HV, 506 HV, and 649 HV) were prepared, and the FPP treatment was then carried out. Figure 13 shows the residual stress distributions for these specimens. The result shows that for specimens with greater hardness, more compressive residual stress is generated, because the yield stress of harder specimens is much higher than that of the other specimens [11].

4. Conclusions

A new hybrid surface modification process, fine particle peening (FPP) treatment prior to nitriding, was proposed. To investigate the effect of such treatment, rotational bending fatigue tests of structural steels were conducted at room temperature, and the following conclusions were made.

1. The FPP treatment prior to nitriding was able to generate a harder layer than nitrided steel. This was due to the increased amount of nitrogen at the surface, as the FPP treatment enabled the diffusion of nitrogen into the material.

2. The crystal structure of the compound layer of the specimen treated with FPP before nitriding was different to that of the nitrided specimen. The compound layer of the FPP-treated specimen prior to nitriding was dense and stable. Therefore, the fatigue strength of FPP-treated steel prior to nitriding was higher than that of nitrided steel. This result implies that FPP treatment prior to nitriding is very effective for the improvement of fatigue strength in steel.

3. The fatigue strength of the FPP treated steel after nitriding showed the highest of all. This was because a high compressive residual stress was generated and the porous compound layer was removed by the FPP treatment.

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