Influence of Pre-FPP (Fine Particle Peening) on Fatigue Fracture Mechanism of Nitrided AISI 4135 Steel

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Abstract. A new hybrid surface modification process: combination of a Fine Particle Peening (FPP) treatment and nitriding was proposed. In order to clarify the effects of the FPP treatment before nitriding (pre-FPP treatment) on fatigue strength and mechanism of notched AISI 4135 steel with stress concentration factor of 2.36, fatigue tests were carried out with a rotational bending fatigue testing machine at room temperature. Crack propagation rate, hardness and residual stress distributions were measured in order to investigate the high cycle fatigue properties. Surface hardness of the FPP treated specimen before nitriding was higher than that of the nitrided specimen. Surface microstructures of treated specimens were then characterized by a Scanning electron microscope (SEM), X-ray diffraction (XRD) and electron probe micro analyzer (EPMA). The compound layer of FPP treated specimen before nitriding was dense and stable. Therefore, the fatigue strength of the pre-FPP treated specimen was higher than that of the nitrided specimen. This suggests that the pre-FPP treatment process improves the fatigue strength of the nitrided specimen and also means that the FPP treatment before nitriding is very effective in improving the fatigue strength of steel.

Introduction

Surface modification techniques are used for various mechanical components in order to improve the fatigue strength, wear and corrosion resistances. Gas nitriding, which is generally performed at a relatively lower temperature; around 800K, than other heat treatments, has been widely used in various fields of engineering. The process has several advantages. The main advantages of this process are low volume expansion and form a higher hardness layer with a high compressive residual stress at the surface of treated materials.

The nitrided layer consists of two layers; one is compound at top surface and the other is diffusion layers. An advantage of the compound layer is higher wear resistance, but the compound layer decreases the fatigue strength of materials due to its brittleness. Pellizzari et al. [1] suggested that it might 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 can be estimated by regarding the compound layer as defective. Therefore, in general, a compound layer is removed by using a grinding process in order to prevent the reduction of the fatigue strength of nitrided products.

When a hardened surface and compressive residual stress are 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 surface hardness and compressive residual stress near the surface of the specimen [3], [4]. Since single surface treatment has some limitations in improving fatigue strength, 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 the shot peening process before nitriding.

Moreover, it was reported that the fatigue strength of the nitrided rolled specimen was higher than that of the nitrided annealed specimens [8]. In general, dislocation densities and grain size are important factors that enhance the nitrogen diffusion process [9], [10]. Therefore, we introduced a Fine Particle Peening (FPP) treatment before nitriding (pre-FPP treatment). The aims of this study are to characterize the compound layer generated by pre-FPP treated nitriding (hybrid surface treatment) and to clarify the effect of the hybrid surface treatment on fatigue properties of structural steel.

Experimental Procedures

Material used in this study was AISI 4135 chromium molybdenum steel which has the chemical composition shown in Table 1. AISI 4135 steel bars with 16mm diameter were machined into the shape and dimensions shown in Fig.1. The stress concentration factor of the specimen for rotational bending fatigue test was 2.36 (Fig.1 (a)).

Fig. 2 shows a flowchart illustrating the specimen preparation; 6 types of specimens with different surface modified layer were prepared.

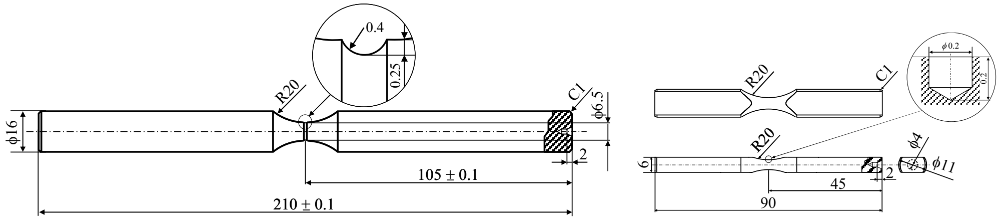
FPP treatment was performed under the conditions shown in Table 2. Shot particles used in this study were high-speed tool steel of 55μm with hardness of 876HV. The gas nitriding was then performed at 823K for 6 hours in a nitrogen and ammonia environment.

To observe the crack propagation behavior, micro-hole (ϕ200μm with a depth of 200μm) was formed at the smallest diameter of the specimen (see Fig.1 (b)) after surface treatments. Crack lengths were measured by interrupting the fatigue test at a fixed number of cycles using the replica method.

In order to characterize the surface-modified layer, the distributions of the Micro-Vickers hardness and the residual stress were measured. Residual stress was also measured at the transverse section of the smallest diameter by the X-ray diffraction technique using a position-sensitive proportional counter (PSPC) system under the conditions shown in Table 3. Depthwise distribution was measured by electrochemically removing the local surface area. Since residual stress measurement was difficult for the notched specimen, we measured the residual stress of the smooth specimen with same surface modification.

Table 1 Chemical composition [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



(a) Rotational bending fatigue test

(b) Crack propagation test

Fig.1 Specimen configurations

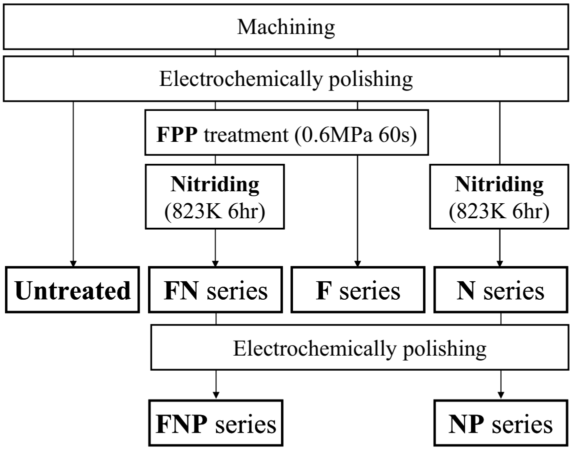


Fig.2 Flowchart illustrating the specimen preparation

Table 2 Conditions of FPP treatment

Shot particles	SKH59
Particles hardness	876HV
Particles diameter	#300 (Mean value:55μm)
Air pressure, MPa	0.6
Peening time, sec.	60
Nozzle distance, mm	100

Table 3 Conditions of measuring residual stress

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

Surface microstructures of treated specimens were observed by a Scanning electron microscope (SEM). The crystal structures of treated specimens (ϕ15mm with a thickness of 4mm) were identified using X-ray diffraction (XRD) with a Cu-Kα radiation (wavelength 0.154nm).

High cycle fatigue tests were carried out with a rotational bending machine (3000 rpm) at room temperature. In order to discuss the fracture mechanism, fracture surfaces were observed using an SEM.

Results

Compound layer evaluation. Fig. 3 shows microstructures of the N and FN series observed at cross-section after etched by 2% Nital. This figure shows the presence of the compound layer. By X-ray diffraction analysis, it was revealed that the compound layer generated at both N and FN series was comprised of ε-Fe_3N and γ'-Fe_4N nitrides; however, in the case of the N series (Fig.3 (a)), cracks and pores were clearly observed in the compound layer. On the other hand, the compound layer of the FN series did not possess any cracks and pores (Fig.3 (b)). The same tendency was observed in the compound layer at top surface (see Fig.4). And a local stratification pattern was observed near the surface of the FN series. This unique structure may be generated by the FPP treatment before nitriding.

Fig. 5 shows a higher magnification SEM image of the FPP treated specimen (F series) etched by a Glow Discharge Optical Emission Spectrometry (GD-OES). Extremely fine grains less than 1μm were clearly observed. Moreover, grains inside the local stratification structure were much finer than those of outside. These nanocrystalline structures near the treated surface may be generated by a concentration of plastic deformation [11]. It is expected that this structure accelerates the diffusion of nitrogen into the materials.

Fig.6 shows the Vickers hardness distribution at cross-sections. Depth of the hardened layer was approximately 0.4mm in both series. Near the surface, the hardness value in the FN series was higher than that of the nitrided specimens. This means that the pre-FPP treatment increases the surface hardness of nitrided steel.

To clarify the reason for this, the amount of the diffused nitrogen element near the surface was measured by EPMA. As a result, the amount of nitrogen in the FN series was higher than that in the N series near the surface. This was because the fine grains generated by the pre-FPP treatment accelerated the diffusion of nitrogen into the material. Therefore, the compound layer of the FN series showed higher hardness and became denser than that of the N series.

Fig.7 shows the residual stress distribution. Nitriding and FPP treatment generated compressive residual stress; however, compressive residual stress of the FN series, which was performed by both FPP treatment and nitriding, was similar to that of the nitrided specimens. To verify the test results, FPP treated specimens (F series) were heated under the same conditions as in the nitriding process, but in vacuum environment (F-Heating series). Then the residual stress of the F-Heating specimen was measured. As a result, the value of compressive residual stress measured at the F-Heating series was approximately 82MPa, and this was much lower than that of the F series surface of 539MPa. This means that the high temperature generated during the nitriding process releases the compressive residual stress.

Effect of the pre-FPP treatment on fatigue strength of nitrided steel. In order to investigate the effect of the pre-FPP treatment on fatigue strength of the nitrided steel, rotational bending fatigue tests were carried out. Fig.8 shows the results of high cycle fatigue tests. In this figure, the vertical axis

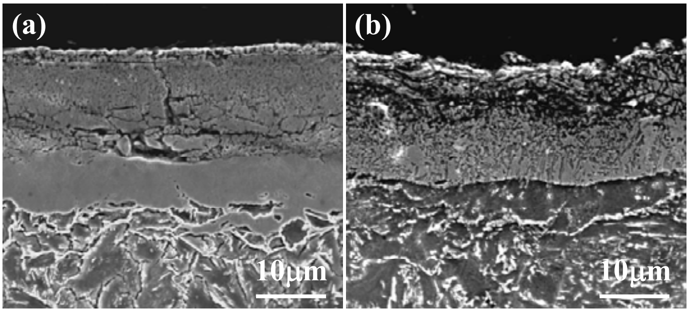


Fig.3 Microstructures observed at cross-section after etching by 2% Nital ((a) N series (b) FN series)

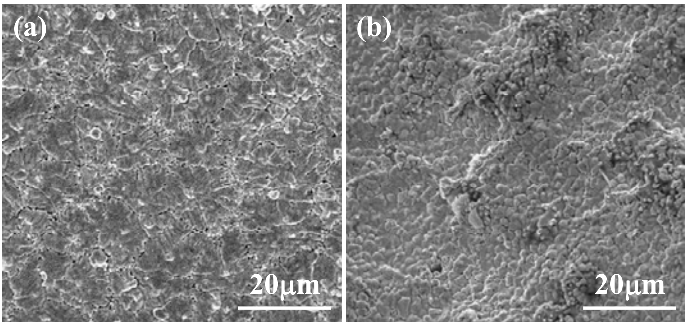


Fig.4 SEM observations of the compound layer at top surface ((a) N series (b) FN series)

represents the nominal stress amplitude applied to the specimens. The fatigue strength of the FN series (●) was higher than that of the N series (◇), although compressive residual stress of the FN series was similar to that of nitrided specimens (N series). This means that the pre-FPP treatment improves the fatigue strength of the nitrided specimens.

To clarify the reason for the higher fatigue strength of the FN series, the fatigue strength of electrochemical polished specimens after nitriding was evaluated (Fig.9). The fatigue strength of the NP series (□) was higher than that of the N series. This means that the compound layer generated bynitriding reduces the fatigue strength of the specimen due to the existence of cracks and pores (Fig.3 (a)); however, the fatigue strength of the FN series was similar to that of the FNP series (■). This means that the fatigue strength of the compound layer on the pre-FPP treated specimen is much higher than that on the N series specimen.

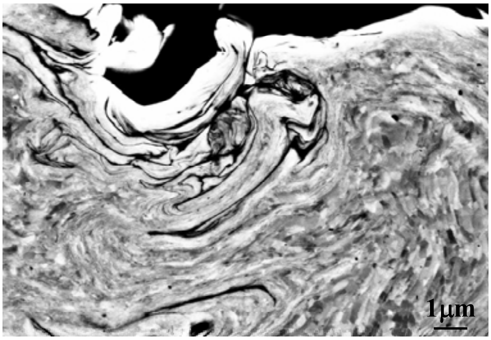


Fig.5 SEM observations of microstructure of F series at cross section

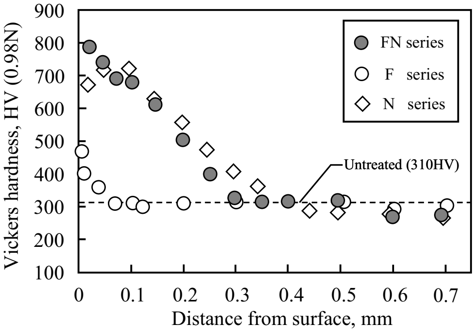


Fig.6 Vickers hardness distribution

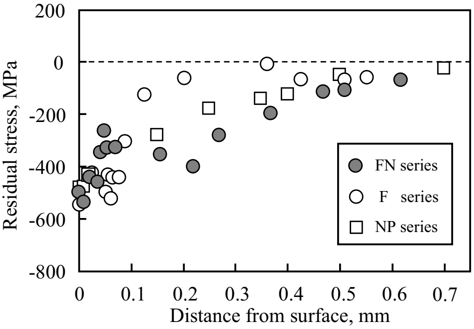


Fig.7 Residual stress distribution

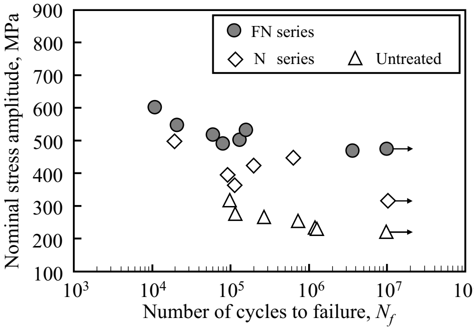


Fig.8 Results of fatigue tests

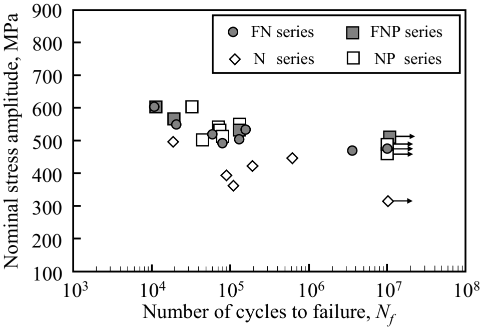


Fig.9 Results of fatigue tests

Discussion

Fracture mechanism of pre-FPP treated and nitrided specimen. In order to improve the fatigue strength of AISI 4135 steel, a new hybrid surface modification process combining nitriding and FPP treatment was proposed. The effects of the hybrid surface modification on high cycle fatigue properties were investigated. The results showed that the hybrid surface treatment increased the fatigue strength of the nitrided specimen.

In this section, in order to investigate the fatigue fracture mechanism of both N and FN series, the crack propagation behaviors were discussed. Fig.10 shows the relationship between crack propagation rate *da/dN* and the stress intensity factor range ΔK, calculated from the surface crack propagation curve. No noticeable differences were observed in the crack propagation rate between the N and FN series. This was because a value of compressive residual stress of the FN series was the same as that of the N series, shown in Fig.7; however, crack length of the FN series was shorter than that of the N series at the same number of cycles (see Fig.11). These results revealed that the dense compound layer of the FN series did not delay the crack propagation rate but the crack initiation life.

The fracture surfaces were observed with an SEM with special attention focused on the crack initiation sites. Fig. 12 shows typical features of fracture surfaces of the N and FN series. In the case of the N series, steps were observed at the fracture surface. These results indicate that multiple cracks initiate and propagate inside the material. On the other hand, in the case of the FN series, no steps were observed in the FN series.

Fig.13 shows microstructures of the N and FN series observed at the longitudinal section using an optical microscope after fatigue tests. The compound layer of the FN series remained unchanging; however, removed compound layer was observed in the N series during the fatigue process. These results show that there are differences in fracture mechanisms of the N and FN series.

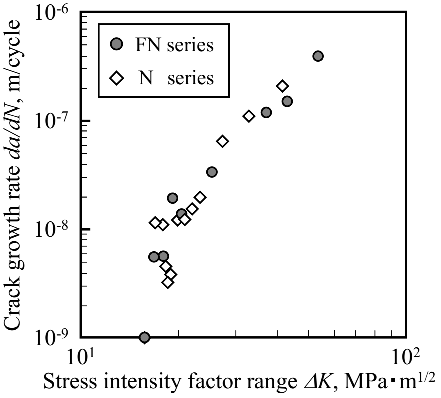


Fig.10 Relationship between crack propagation rate *da/dN* and stress intensity factor range ΔK

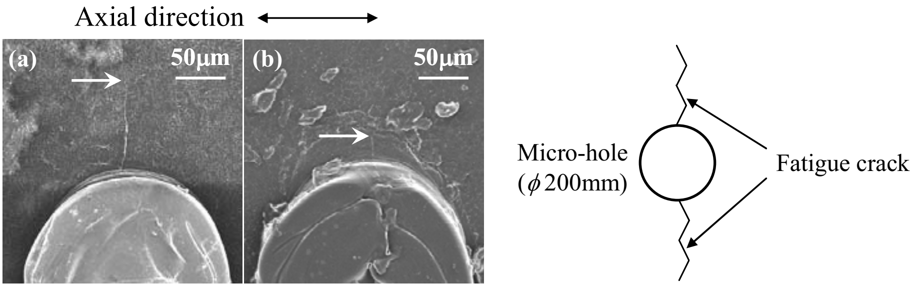


Fig.11 SEM observations of cracks (Arrow mark represents crack tip) ((a) N series (b) FN series)

To clarify the reason for this, crack propagation behavior was observed using the replica method. Fig.14 shows the crack propagation behavior of N series specimen. It was observed that fatigue cracks propagated along the *crack like defects* existing in the compound layer. This result implies that, in the case of the N series, fatigue cracks which initiated at *crack like defects* in the compound layer coalesce and then propagate inside the material. *Crack like defects* which existed in the compound layer caused a reduction in the fatigue strength of the N series.

Consequently, the pre-FPP treatment prevents the crack nucleation due to the dense compound layer, resulting in improving the fatigue strength of the nitrided steel.

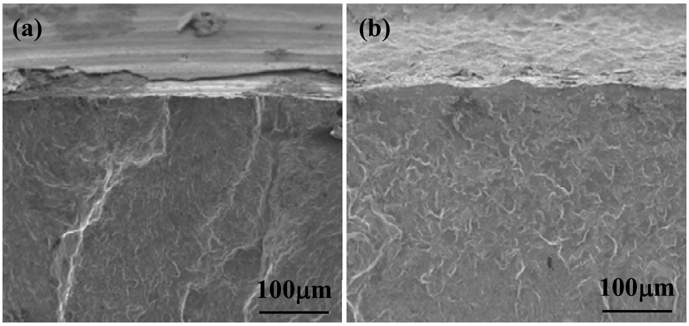


Fig.12 Typical features of fracture surfaces ((a) N series (b) FN series)

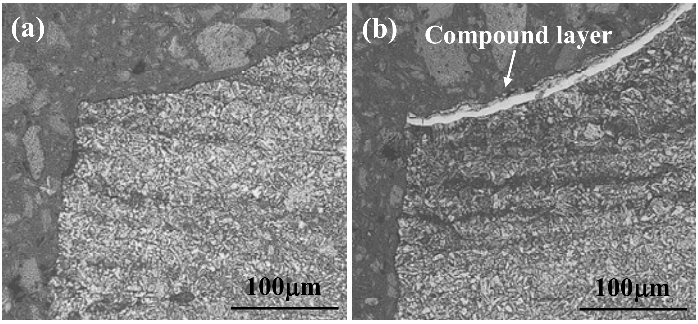


Fig.13 Microstructures observed at the longitudinal section using an optical microscope after fatigue tests ((a) N series (b) FN series)

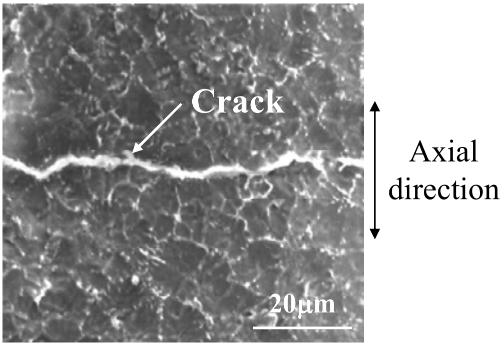


Fig.14 Crack propagation behavior of N series

Summary

In this study, we focused on a new hybrid surface modification process: a Fine Particle Peening (FPP) treatment before nitriding (pre-FPP treatment) was proposed. Rotational bending fatigue tests were carried out at room temperature on surface modified structural steel. The influence of the proposed treatment on fatigue properties of structural steel was investigated.

1. The pre-FPP treatment was able to generate a harder layer than the nitrided steel. This was because fine grains generated by the pre-FPP treatment accelerated the diffusion of nitrogen into the material.
2. The hybrid surface treatment increased the fatigue strength of structural steel. This was because the compound layer of the pre-FPP treated specimen was dense and stable.
3. There were differences in fracture mechanism between the nitrided specimen and hybrid surface treated specimen. This was because of the nitrided specimen, fatigue cracks initiated at several *crack like defects* in the compound layer; however, the pre-FPP treatment increases the crack initiation life.

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