

Fatigue Properties of Nitrided Alloy 718 at Elevated Temperature

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Abstract: Rotating bending fatigue tests were carried out using a radical nitrided nickel base superalloy, Alloy 718, at 500 °C, to investigate the effect of radical nitriding on the initiation and propagation behavior of a fatigue crack at elevated temperature. The nitriding was conducted at 500 °C for 12h which was relatively low temperature compared with other nitriding technologies. Compound layer was formed on the specimen surface with the thickness of about 4μm. Hardness of the base alloy was hardly affected by nitriding. When stress levels were high, a crack initiated from the specimen surface in the aged alloy, and the base alloy beneath the compound layer in the nitride alloy. On the other hand, when stress levels were low, fracture origins changed to the interior of base alloy in both alloys, causing that $S - N$ curves showed a step wise shape. The crack initiation was suppressed by the compound layer. As the result, fatigue strength increased by nitriding in the horizontal region in $S - N$ curves where fatigue strength was controlled by the crack initiation and the propagation of a small crack, though the effect of nitriding on fatigue strength was very small in the region where fatigue life was occupied by the growth of a crack, i.e. in the region except for the horizontal one in $S - N$ curves.

Keywords: Fatigue, alloy 718, radical nitriding, compound layer, elevated temperature, rotating bending, fracture mechanism

1 Introduction

Ni base superalloys possesses superior static and fatigue strengths at elevated temperature. Therefore these alloys have been used as major materials for machines

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that worked under severe service conditions such as corrosive, heavy load and high temperature environment [Ghonem, Nicholas and Pineau (1993)]. On the other hand, wear resistance of the alloy is very poor [Yamane, Morino, Fukada and Kawagoishi (2010)]. In general, surface treatments have been applied to improve the wear resistance of materials. Surface treatments affect not only wear and corrosion properties, but also fatigue ones [Morita, Shimizu, Kawasaki and Chiba (1990)]. Nitriding is one of convenient surface treatment technologies, so it was widely applied to many metals. However, Ni alloys are one of metals which are difficult to nitride because of their surface covered with stable passivation film. Therefore, nitriding, e.g. ion nitriding, is usually applied at high temperature over around 700 °C [Matsuda, Nakata and Makishi (1987)]. Consequently the base alloy is softened by the nitriding due to coarsening of the grain size, over-aging and so on. These are significant problems in strength integrities, especially in fatigue. On the other hand, radical nitriding is a superior technology which can be treated at relatively low temperature around 500 °C and without damage of the base alloy [Fukada (2001)]. In the practical application of radical nitriding to these alloys, fatigue properties should be clarified, especially in long life region for reducing environment load.

In the present study, rotating bending fatigue tests were carried out using a radical nitrided nickel base superalloy, Alloy 718, at 500 °C to investigate the effect of the nitriding on initiation and propagation behavior of a fatigue crack at elevated temperature.

2 Material and experimental procedures

The material used was a Ni-base super alloy, Alloy 718, with a chemical composition (in wt. %) of 0.02C, 0.11Si, 0.12Mn, 0.009P, 0.001S, 18.67Cr, 3.09Mo, 0.09Co, 0.01Cu, 0.66Al, 0.90Ti, 18.67Fe, 0.004B, 5.12 Nb and Ta, and balance Ni. The alloy was solution treated at 982 °C for 1 h and then water quenched. After solution treatment, the alloy was heat treated under the conventional double aging, that is, it was aged at 720 °C for 8 h, and then furnace cooled to 621 °C and aged at 621 °C for 8 h followed by air-cooling. The mean grain size of the alloy was about 18 μm. Mechanical properties of the aged alloy were 0.2% proof stress of 1147 MPa, tensile strength of 1372 MPa, true breaking stress of 2073 MPa and reduction of area of 38.1 %, respectively.

Radical nitriding was performed at 500 °C for 12h in mixed gasses of nitrogen and ammonium.

Figure 1 shows shape and dimensions of specimen for fatigue test. Parts of specimens were machined a blunt and shallow notch partially at the center of specimen

shown in Fig.1 (b) to localize the crack initiation site and to make the successive observation of fatigue damage easier. However the reduction of fatigue strength by the notch was about 10%, so the specimen can be regarded as a plain specimen. All of the specimens for fatigue tests were electro-polished by $40\mu\text{m}$ in diameter from the surface after machining and emery paper grinding in order to remove the worked layer and to make the specimen surface smoother. Moreover, parts of aged specimens were nitrided at the condition stated above.

The measurement of surface damage and fracture surface in fatigued specimens were examined by using an optical microscope through plastic replica or a scanning electron microscope (SEM) directly. The length of a fatigue crack ℓ was defined as a surface length along a circumferential direction of specimen. Fatigue tests were carried out at $500\text{ }^\circ\text{C}$ using a rotating bending fatigue testing machine with an electric furnace for elevated temperature test. The loading frequency was 50Hz.

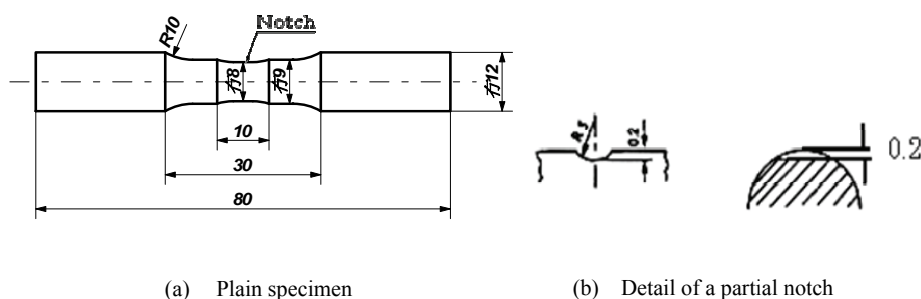


Figure 1: Shape and dimensions of specimen.

3 Results and discussion

3.1 Nitrided layer

Figure 2 shows microstructure of the nitrided specimen. Compound layer of about $4\mu\text{m}$ in thickness was formed on the specimen surface by the nitriding. The compound was confirmed as CrN by EPMA.

Figure 3 shows hardness distribution of the nitrided specimen. There is a hardened surface layer corresponding to the thickness of compound layer in the nitrided alloy. The hardness of the base alloy nitrided at $500\text{ }^\circ\text{C}$ is nearly the same as the one of the aged alloy, meaning that there is no damage of the base alloy. Actually any

difference in microstructure is not confirmed before and after the nitriding by TEM observation as seen from Figure 4. In general, compressive residual stress is yielded in surface treated metals. However, residual stress could not confirm in the nitrided alloy because of its thin compound layer.

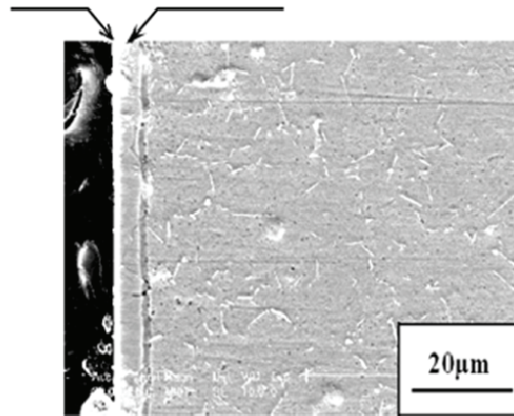


Figure 2: Microstructure of nitrided alloy.

3.2 Fatigue properties

Figure 5 shows $S-N$ curves of aged and nitrided alloys at 500 °C. In the figure, results at room temperature are also indicated by lines only. Fatigue strength at 500 °C is higher than that at room temperature except for the region at higher stress levels, about $\sigma_a=800\text{MPa}$, indicating the reverse tendency to the temperature dependence of static strength. Moreover, $S-N$ curves showed a step wise shape in both alloys at 500 °C, though fatigue life at room temperature increased with decreasing in stress levels. Fatigue strength in the nitrided alloy is higher than that in the aged alloy in the region of the horizontal line in $S-N$ curves, while there is no or little influence in fatigue strength in other life region. Thus, effect of nitriding on fatigue strength is different depending on stress levels.

Figure 6 shows crack growth curves at 800MPa, where the stress is higher level than the horizontal line in $S-N$ curves. A crack initiation is suppressed by the nitriding as seen from the slope of the growth curves in both alloys. The growth rates of a crack over the length with a few grains are not affected by the nitriding. Therefore the increase in fatigue strength by nitriding was mainly caused by the suppression of a crack initiation and the propagation of a small crack.

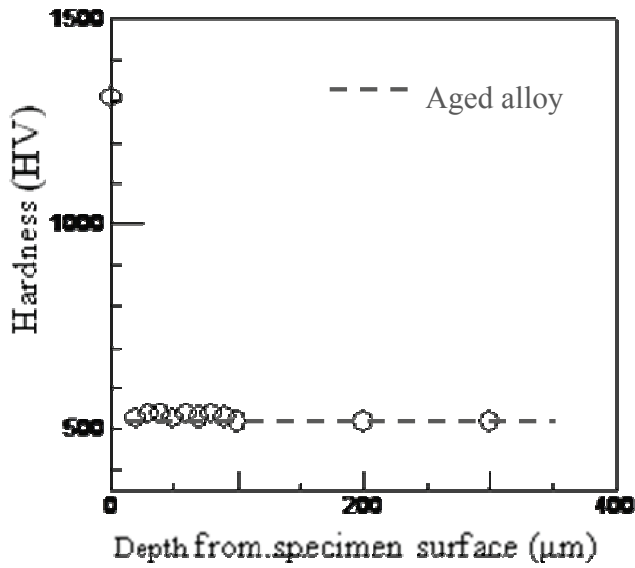
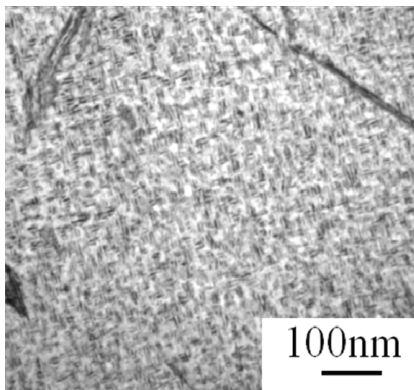
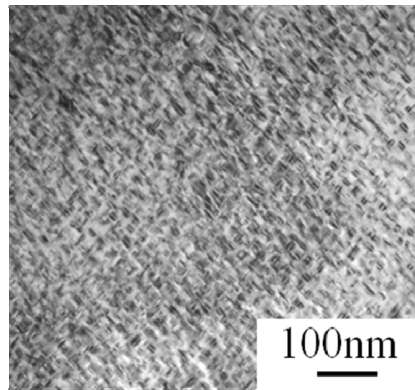


Figure 3: Hardness distribution of nitrided alloy.



(a) Aged alloy



(b) Nitrided alloy

Figure 4: Microstructure observed by TEM.

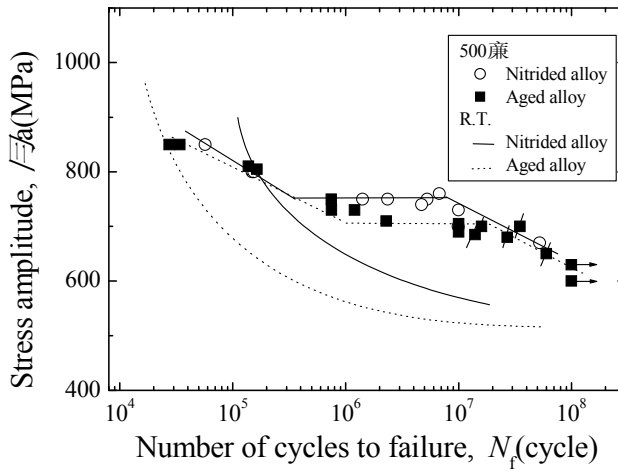


Figure 5: $S - N$ curves of nitrided and aged specimens.

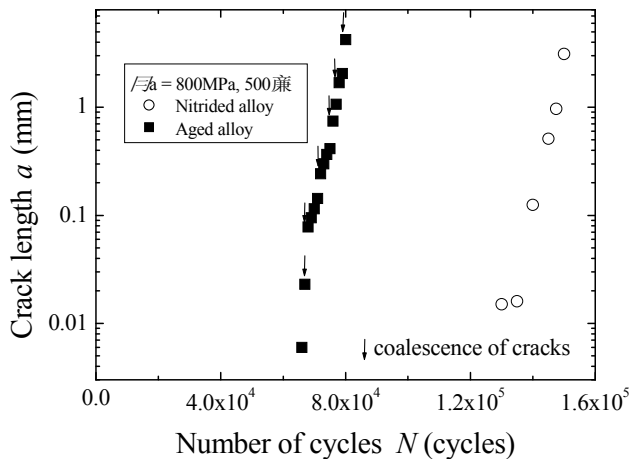


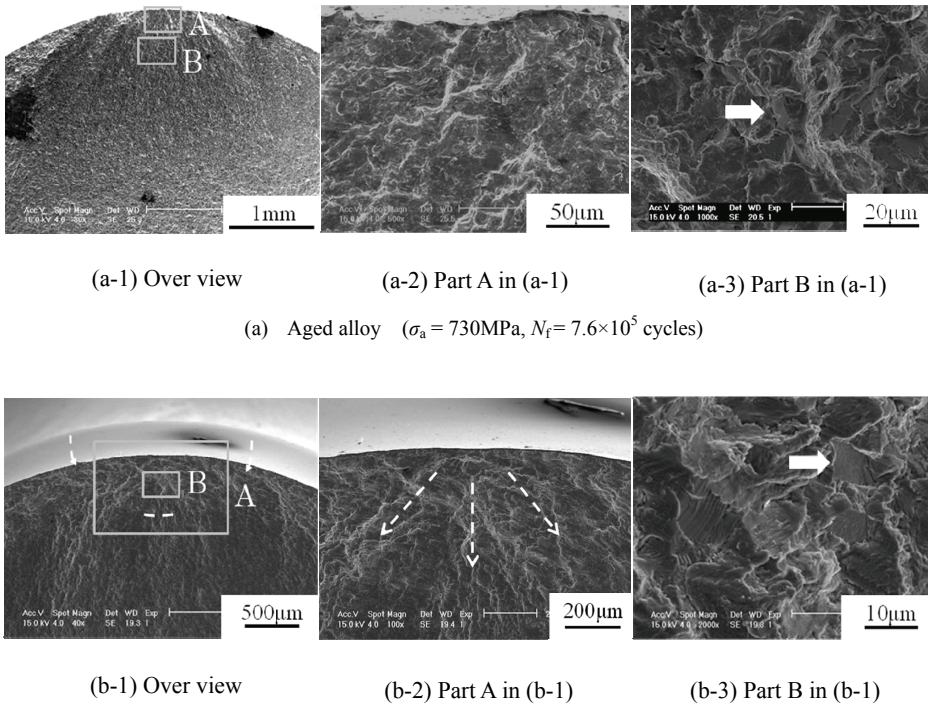
Figure 6: Crack growth curves.

Figure 7 shows fracture surfaces at high stress level in both alloys. Fracture surfaces are covered with striations and flat facets, meaning a ductile fracture in both alloys, while a flat and brittle surface layer corresponding to the compound layer is observed circumferentially in the nitride alloy. Moreover, a fracture origin is a specimen surface clearly in the aged alloy. On the other hand, it is also seemed to be a specimen surface, because there is a radial pattern developed from a point near

the compound layer in the nitrided alloy. However the compound layer is very thin, so it is difficult to clear the fracture origin from this observation. Therefore more detailed observation was conducted by means of removing the surface layer of the specimen gradually by electro-polishing at the fatigue process.

Figure 8 shows the surface states of specimen before and after electro-polishing the specimen surface at high and low stress levels of the horizontal line in $S - N$ curves in the nitrided alloy. The removed layer was about $10\mu\text{m}$ in depth. At both stress levels, a crack is confirmed clearly at the specimen surface after electro-polishing, while it is not confirmed before electro-polishing. That is, a crack initiation site in the nitrided alloy is the base alloy beneath the compound layer. By this method, the delay of crack initiation was also confirmed in the nitrided alloy.

Figure 9 shows fracture surfaces at the stress levels below the horizontal lines of $S - N$ curves in nitrided alloy. Internal cracks are observed similar to those in aged alloy, though a circumferential



(b) Nitrided alloy ($\sigma_a = 850\text{MPa}$, $N_f = 5.7 \times 10^4$ cycles)

Figure 7: Fracture surfaces at high stress.

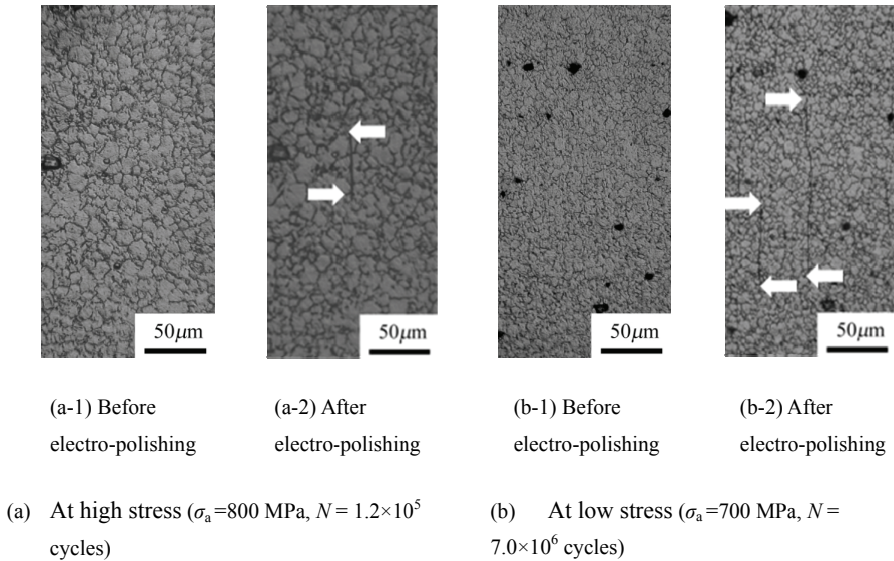


Figure 8: Surface states of specimen before and after electro-polishing.

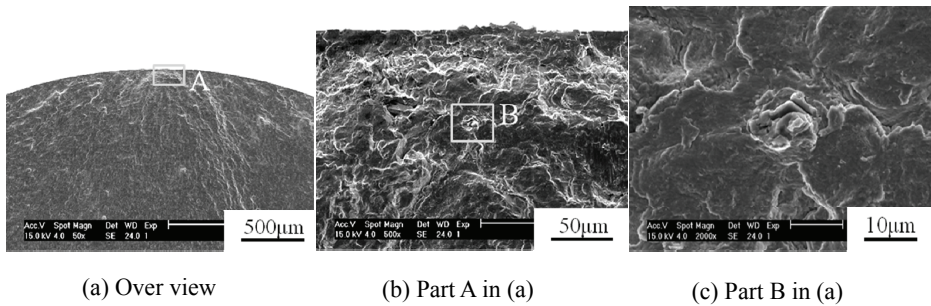


Figure 9: Fracture surfaces at low stress in nitrided alloy ($\sigma_a = 760$ MPa, $N_f=6.76 \times 10^6$ cycles).

brittle facet is also observed in the nitrided alloy. That is, when stress levels are low, fracture occurred from the subsurface, while a crack initiated at or near the specimen surface, separately, in both alloys.

4 Conclusions

When stress levels were high, a crack initiated from the specimen surface in the aged alloy, and the base alloy beneath the compound layer in the nitrided alloy. On the other hand, fracture origins changed to the interior of base alloy in both alloys with decreasing in stress level, causing that $S - N$ curves showed a step wise shape. The crack initiation was suppressed by the compound layer. As the result, fatigue strength increased by nitriding in the horizontal region of $S - N$ curves where fatigue strength was controlled by the crack initiation.

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