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The effects of gas nitriding on fatigue behaviour in titanium and titanium alloys

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Abstract

Fatigue behaviour has been studied on gas-nitrided smooth specimens of commercial pure titanium, alpha/beta Ti-6Al-4V alloy and beta Ti-15Mo-5Zr-3Al alloy under rotating bending, and the obtained results were compared with the fatigue behaviour of annealed or untreated specimens. It was found that the role of the nitrided layer on fatigue behaviour depended on the strength of the materials. Fatigue strength was increased by nitriding in pure titanium, while it was decreased in Ti-6Al-4V alloy and Ti-15Mo-5Zr-3Al alloy. Based on detailed observations of fatigue crack initiation, growth and fracture surfaces, the improvement and the reduction in fatigue strength by nitriding in pure titanium and both alloys were primarily attributed to enhanced crack initiation resistance and to premature crack initiation of the nitrided layer, respectively.

1 Introduction

Titanium and titanium alloys have the disadvantages of a high coefficient of friction and poor wear resistance [1]. In order to improve such tribological properties, a variety of surface engineering techniques such as gas nitriding [1-5], laser nitriding [1,6] and ion implantation [7], have been successfully applied. Of these techniques, gas nitriding is considered to be the most promising method available for engineering applications. By this method substantial improvement in wear resistance can be achieved [1-3,5]. On the other hand, it has also been reported that such surface treatments caused the decrease in the fatigue strength of titanium alloys [1,5,6]. However, the reasons for the reduction in fatigue strength and the fracture mechanisms have not been studied in detail.

In the present study, rotating bending fatigue tests were conducted using gas-nitrided smooth specimens of pure titanium, Ti-6Al-4V alloy, and Ti-15Mo-5Zr-3Al alloy. The obtained results were compared with the fatigue behaviour of annealed or untreated specimens. The effects of gas nitriding on fatigue behaviour are discussed on the basis of detailed observations on fatigue crack initiation, growth and fracture surfaces.

2 Experimental procedures

2.1 Materials and nitriding process

The materials used are commercial pure titanium, alpha/beta Ti-6Al-4V alloy, and beta Ti-15Mo-5Zr-3Al alloy. Their chemical compositions (wt.%) are Fe 0.043, O 0.095, N 0.008, H 0.0019, remainder Ti, Al 6.27, V 4.27, Fe 0.206, O 0.185, N 0.003, C 0.015, Y <0.001, H 0.0055, remainder Ti, and Al 3.01, Mo 14.51, Zr 5.05, Fe 0.042, O 0.114, N 0.0045, H <0.001, remainder Ti, respectively. Smooth specimens with 8 mm diameter and 10 mm gauge length were machined from the materials and then mechanically polished by emery paper. Subsequently, they were electropolished, and were nitrided and heat treated as follows.

In pure titanium, nitriding was performed at 850°C and 1000°C for 9 h, designated hereafter as N850 and N1000, respectively. For comparison, annealed or untreated specimens were prepared, which were annealed at the same temperature and duration as the nitrided specimens (A850 and A1000) and at 700°C for 1 h (A700).

The depth of the nitrided layer in Ti-6Al-4V alloy was controlled by varying the nitriding period, 4 h and 15 h, at 850°C, designated as N4 and N15. Specimens subjected to the same heat history as the nitrided specimens were also prepared for comparison (A4 and A15). In addition, specimens solution treated at 950°C for 1 h followed by aged at 540°C for 4 h (STA) were used to obtain a standard fatigue strength of the alloy.

In Ti-15Mo-5Zr-3Al alloy, nitriding was performed at 700°C, below the beta transus of 785°C, for 20 h and 60 h (N20 and N60). Annealed specimens experienced the same heat history as the nitrided specimens (A20 and A60) were employed.

Nitriding was performed in an electric furnace, which was evacuated before heating up to nitriding temperatures; then nitrogen gas with a purity of 99.99 % was admitted into it. Specimens were maintained for a given nitriding period under a constant nitrogen gas pressure of 0.13 MPa.

2.2 Mechanical properties

The mechanical properties are listed in Table 1. In pure titanium, the proof stress is higher and the elongation and reduction of area are lower in the nitrided specimens than in the annealed ones. In Ti-6Al-4V alloy, monotonic strengths are slightly decreased by nitriding for 4 h, but are increased by nitriding for 15 h, while in Ti-15Mo-5Zr-3Al alloy they are considerably reduced.

Table 1: Mechanical properties.

Alloy	Specimen code	0.2% proof stress	Tensile strength	Elongation	Reduction of area
		$\sigma_{0.2}$ MPa	σ_B MPa	ϕ %	ψ %
Pure titanium	A700	324	434	28	57
	A850	246	383	39	69
	N850	263	384	36	64
	A1000	317	409	34	58
	N1000	356	437	28	42
Ti-6Al-4V alloy	STA	1132	1238	9	40
	A4	927	971	7	49
	N4	890	943	11	40
	A15	900	950	7	48
	N15	925	974	13	45
Ti-15Mo-5Zr -3Al alloy	A20	957	1015	13	41
	N20	871	916	11	46
	A60	975	1001	13	40
	N60	875	901	12	47

2.3 Procedures

Experiments were conducted on a 98 N·m rotating bending fatigue testing machine operating at a frequency of 57 Hz in laboratory air at room temperature. Crack initiation and growth were monitored by replicating the specimen surface. The fracture surfaces were examined using a scanning electron microscope (SEM).

3 Results

3.1 Nitrided layer

The surface of the nitrided specimens had a golden colour and the intensity of the gold coloration tended to increase with increasing temperature or nitriding period. Thin compound TiN was seen on the specimen surface, below which the layer of nitrogen-stabilized α solid solution was formed.

Prior to fatigue testing, Vickers hardness was measured on the cross-section of the nitrided specimens. In pure titanium, hardness increased within 20-30 μm and 50-60 μm from the surface for N850 and N1000, respectively. In the titanium alloys, the nitrided layer became deeper and hardness increased, with increasing nitriding period [2,5]. Hardness increased within 25 μm from the surface for N4 and 65 μm for N15 in Ti-6Al-4V alloy, and 130 μm for N20 and 200 μm for N60 in Ti-15Mo-5Zr-3Al alloy. These depths of the increased hardness could be considered as the nitrided layer.

3.2 Fatigue strength

The *S*-*N* diagrams are shown in Figs 1, 2 and 3 for pure titanium, Ti-6Al-4V alloy and Ti-15Mo-5Zr-3Al alloy, respectively.

3.2.1 Pure titanium

The fatigue strength of the nitrided specimens, N850 and N1000, increases compared with that of the corresponding annealed specimens. In particular, the fatigue strength of N850 is remarkably improved and is superior to the standard strength of A700.

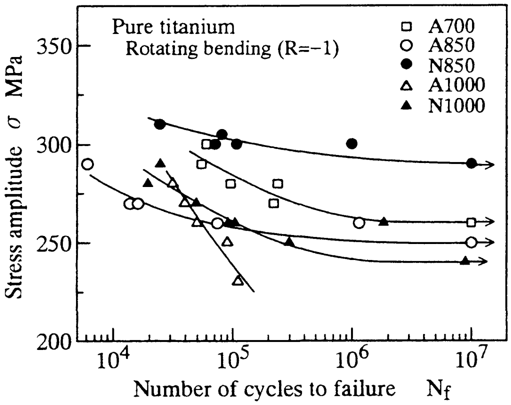

Fig.1: *S*–*N* diagram for pure titanium.

3.2.2 Ti-6Al-4V alloy

The fatigue lives of N4 are shorter than those of A4, but the fatigue limit is slightly increased. In contrast, the fatigue strength of N15 is reduced compared with A15 and is lower than that of N4, indicating that the harder and deeper nitrided layer has a detrimental effect on the fatigue strength [1,5,6].

The fatigue results of N15 whose surface was removed by electropolishing are also included in Fig.2. It is apparent that the fatigue lives gradually approach the life of A15 as the amount of removal is increased. This results suggest that the TiN layer and the harder solid solution layer just below the surface are considerd to be strongly related to the decrease in fatigue strength of the nitrided specimens.

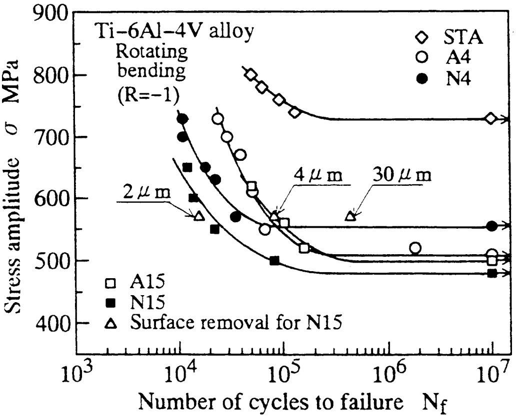

Fig.2: S–N diagram for Ti-6Al-4V alloy.

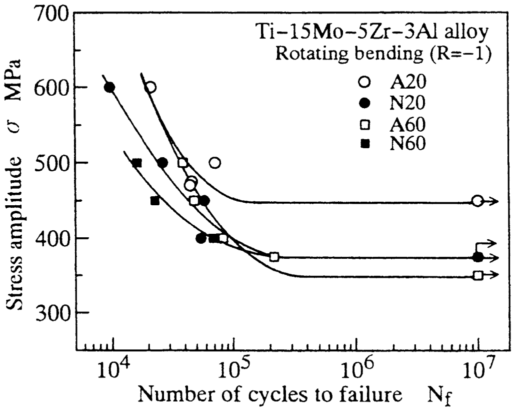

Fig.3: S–N diagram for Ti-15Mo-5Zr-3Al alloy.

3.2.3 Ti-15Mo-5Zr-3Al alloy

Similar results to Ti-6Al-4V alloy are obtained in this alloy: the fatigue strength of N20 is decreased compared with A20, while the fatigue lives of N60 are shorter than those of A60, but the fatigue limit is slightly increased.

3.3 Initiation and growth of fatigue cracks

The relationships between surface crack length, $2c$, and cycle ratio, N/N_f or number of cycles, N, are represented in Fig.4 for all the materials.

3.3.1 Pure titanium

Fatigue cracks in the nitrided specimens were initiated on the specimen surface. Figure 4(a) shows the enhanced crack initiation resistance in the nitrided specimens compared with the annealed specimens. In N850, crack initiation is relatively delayed by nitriding in spite of a higher cyclic stress applied, and no crack is initiated at σ = 260MPa, which was applied for A850. Similarly, it can be seen from the figure that crack initiation in N1000 is also delayed. The relationship between crack growth rate, $\mathrm{d}a/\mathrm{d}N$, and maximum stress intensity factor, $K_{\max}$, is represented in Fig.5. N850 shows lower $\mathrm{d}a/\mathrm{d}N$ than A850 over the entire $K_{\max}$ regime tested. In contrast, there is no noticeable difference in $\mathrm{d}a/\mathrm{d}N$ between N1000 and A1000.

3.3.2 Ti-6Al-4V alloy

As can be seen in Fig.4(b), the initiation resistance is lowered by nitriding, in particular N15 shows a considerable decrease in crack initiation resistance. It should be noted that the specimen whose surface was removed by 5 μm indicates the same initiation resistance as the corresponding annealed specimen.

Unusual crack initiation behaviour was observed in the nitrided specimens. In N15, there were no cracks at $N = 1.0\times10^4$ cycles, but a crack of $2c = 498$ μm was initiated after only 10^3 cycles. In order to confirm the reproducibility of the behaviour, the same obervation was made on a different specimen. A crack of $2c = 1390$ μm was initiated at $N = 9\times10^3$ cycles. However, no crack was observed at $N = 8\times10^3$ cycles. The crack had already grown to the core material, but its depth, a, was only 133 μm, thus the aspect ratio, a/c, is extremely small. The above observations suggest that a crack is initiated suddenly at a relatively large size or grows rapidly in the surface layer. As this crack grew into the core material and led to the final fracture, premature crack initiation is primarily responsible for the reduction in fatigue strength for the nitrided specimens.

The aspect ratio is shown in Fig.6 as a function of $2c$. The a/c values decrease with increasing $2c$ in the annealed specimens, while they are extremely small in N15 immediately after crack initiation, then increase rapidly with increasing $2c$ and coincide with those of the annealed specimens in the region of the crack length of $2c \geq 3.2$ mm.

The relationships between surface crack growth rate, $\mathrm{d}c$ / $\mathrm{d}N$, and $(K_{\max})_\mathrm{C}$, and crack growth rate into the bulk, $\mathrm{d}a/\mathrm{d}N$, and $(K_{\max})_\mathrm{A}$ are

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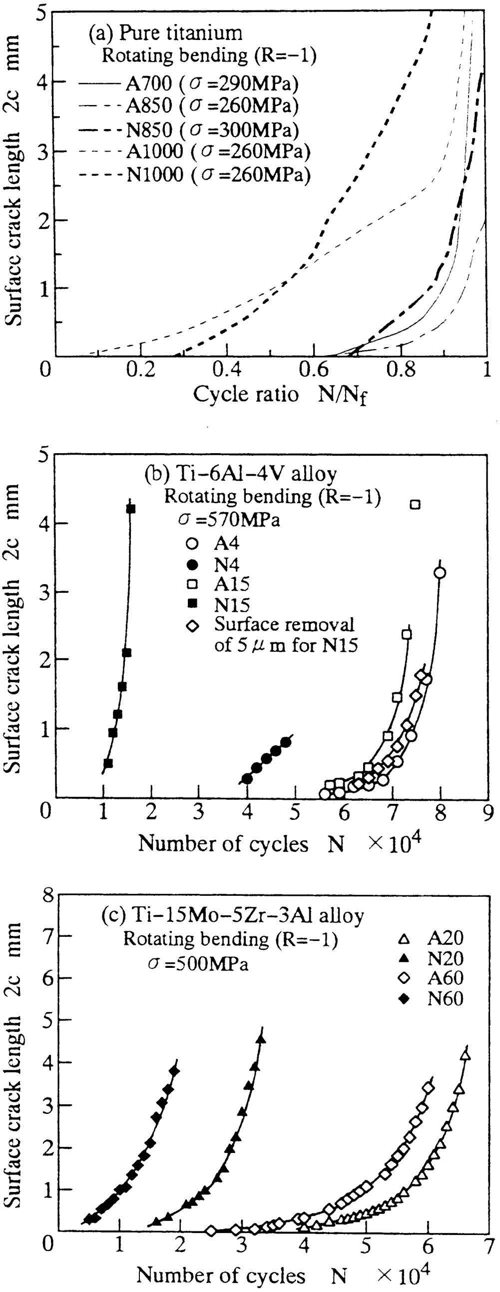

Fig.4: Crack growth curves; (a) pure titanium, (b) Ti-6Al-4V alloy, (c) Ti-15Mo-5Zr-3Al alloy.

represented in Fig.7. As can be seen in the figure, da/dN is almost the same for the nitrided and annealed specimens (Fig.7(b)), but dc/dN for the nitrided specimens is significantly faster than that for the annealed specimens (Fig.7(a)), indicating that hard nitrided layer can accelerate crack growth, which is more noticeable at smaller crack size.

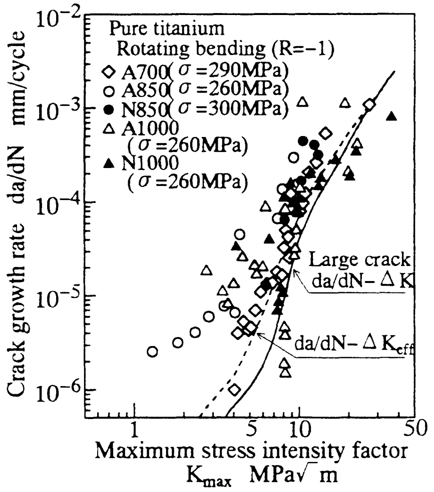

Fig.5: Relationship between crack growth rate and maximum stress intensity in pure titanium.

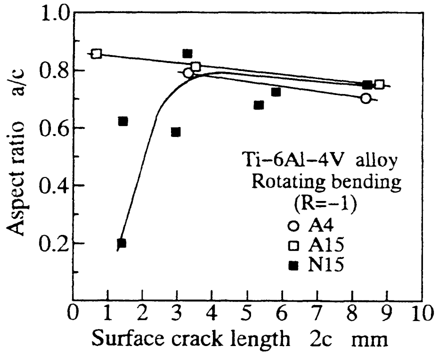

Fig.6: Variation of aspect ratio with crack growth in Ti-6Al-4V alloy.

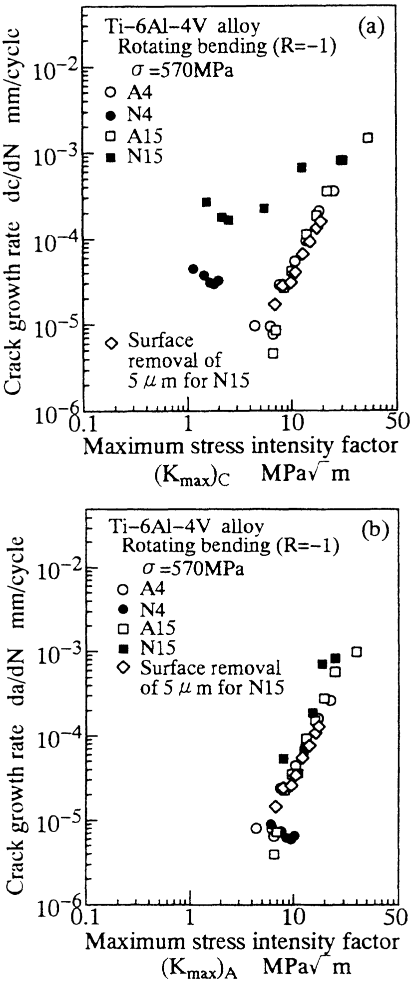

Fig.7: Relationship between crack growth rate and maximum stress intensity in Ti-6Al-4V alloy; (a) specimen surface and (b) deepest point.

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3.3.3 Ti-15Mo-5Zr-3Al alloy

The crack initiation resistance of N60 and N20 is decreased compared with A60 and A20, and N60 having a harder and deeper nitrided layer exhibits earlier crack initiation than N20 (see Fig.4(c)). In the nitrided specimens, similar crack initiation behaviour to Ti-6Al-4V alloy was observed, thus the reduction in fatigue strength of the nitrided specimens may also be attributed to premature crack initiation at a relatively large size.

4 Discussion

In pure titanium fatigue strength was increased by nitriding, which was primarily attributed to the enhanced crack initiation resistance, while it was reduced in Ti-6Al-4V alloy and Ti-15Mo-5Zr-3Al alloy due to premature crack initiation in the nitrided layer.

The contrary roles of the nitrided layer in crack initiation behaviour may be attributed to the difference in deformation between the nitrided layer and the core material. As titanium alloys have high strengths and low elastic modulus, very large elastic deformation occurs during fatigue testing. The elastic modulus of the TiN layer is two to six times that of the core materials, and thus TiN layer is under severe deformation and subjected to high stress. When localized fatigue deformation takes place at the surface of the core material, brittle cracking results in the TiN layer and the harder solid-solution layer. This is the unusual crack initiation behaviour observed in Ti-6Al-4V alloy and Ti-15Mo-5Zr-3Al alloy. Therefore, the shape of a crack, once initiated, becomes very shallow and subsequent growth into the bulk occurs rapidly.

The strength of pure titanium is considerably lower than that of the alloys (see Table 1), and thus elastic deformation during fatigue test was small. In this case, the nitrided layer can prevent the initiation of slip and consequently enhance the initiation resistance of fatigue cracks [8].

5 Conclusions

Rotating bending fatigue tests were conducted using gas-nitrided smooth specimens of commercial pure titanium, Ti-6Al-4V alloy and Ti-15Mo-5Zr-3Al alloy. The effects of gas nitriding on fatigue behaviour and the fracture mechanisms were discussed. The following conclusions can be made.

(1) The depth and hardness of the nitrided layer increased with increasing nitriding temperature and period.

(2) In pure titanium, fatigue strength was increased by nitriding, while in Ti-6Al-4V alloy and Ti-15Mo-5Zr-3Al alloy, the overall fatigue strength tended to be reduced by nitriding.

(3) In pure titanium, the crack initiation resistance of the nitrided specimens was largely enhanced compared with the annealed specimens. In Ti-6Al-4V alloy and Ti-15Mo-5Zr-3Al alloy, premature crack initiation took

place at a relatively large size in the nitrided specimens; *i.e.* the initiation resistance was significantly decreased by nitriding.

(4) There was a case where the crack growth resistance was enhanced by nitriding in pure titanium. On the contrary, the crack growth rates at the specimen surface in the nitrided specimens of Ti-6Al-4V alloy were faster than those in the annealed specimens, especially at smaller crack size.

(5) The role of the nitrided layer on fatigue behaviour depended on the strength of the materials. The improvement in fatigue strength by nitriding was attributed to the enhanced crack initiation resistance if the strength was low, as in pure titanium, while the reduction in fatigue strength was primarily due to premature crack initiation if the strength was high, as in Ti-6Al-4V alloy and Ti-15Mo-5Zr-3Al alloy.

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