Fatigue strength of ion-nitrided Ti-6Al-4V alloy in high cycle region
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Abstract

Though various kinds of surface treatment methods were applied to improve the wear resistant properties of Ti-6Al-4V alloy, it has been reported that the brittle surface layer of this alloy deteriorated their fatigue properties. Furthermore, when this alloy is nitrided by gas and salt bath, the fatigue strength of nitrided one decreases as compared with that of non-nitrided one. On the other hand, as the ion-nitriding method is treated in clean environment, the surface of the material could become free from hydrogen and oxygen. There are few reports about the fatigue strength of ion-nitrided titanium alloys.

Fatigue tests have been accomplished by rotating bending fatigue testing machine using a partial shallow notch specimen after different temperatures of heat treatment. In addition, the specimen surface has been successively observed by replica method during tests and it seems clear that the fatigue strength is improved by removing the chemical compound surface layer, because this chemical compound layer deteriorates the fatigue strength of nitrided specimen. From the above results, the surface layer would be requested to have the equivalent toughness of the matrix to improve its fatigue strength according to the view point of practical use.

1 Introduction

Generally speaking, titanium alloys have been widely used in the aerospace industries because of their superior properties such as high specific strength and high corrosion resistance etc.. In addition, not only high fatigue resistance but
also high wear resistance are essentially required for the many kinds of critical use of structures. Though these kinds of alloys especially show the low wear resistance, various kinds of surface hardening methods had been applied to improve these problems. When this material is nitrided by gas or salt bath; e.g. Morita et al [1,2], Tokagi et al [3,4], the abrasion resistance increases remarkably. On the other hand, as the material absorbs hydrogen and oxygen, the toughness of materials is deteriorated. Furthermore, it has been reported that fatigue strength of nitrided materials decreases as compared with that of non-nitrided one. The authors have considered that the ion-nitriding process would be one of the important methods to accomplish the above requirements. As the ion-nitriding process is treated in clean atmosphere, the surface of material is free from hydrogen and oxygen. However, there are few reports about the fatigue properties of ion-nitrided titanium alloy [5,6,7,8,9].

In this study, the fatigue properties, especially, the fatigue strength, fatigue crack initiation and propagation behavior have been investigated using the specimen ion-nitrided Ti-6Al-4V alloy which is one of the most representative titanium alloys.

2 Experimental procedure

The material used in this test is the ion-nitrided Ti-6Al-4V alloy. Tables 1 and 2 list the chemical composition and the heat-treatment conditions of specimens, respectively.

Figure 1 shows the shape and dimensions of specimens. Though the authors had started the experiments using the specimen of type A, they have faced troubles to remove the specimen's warp during ion-nitriding and have changed from the specimen of type A to type B afterward. That is, the specimen of type B is smaller than type A, type B is less affected than type A about the specimen's warp during ion-nitriding process.

After machining, all of the specimens are polished, and annealed in vacuum at the temperature of 600 °C for 30 minutes afterwards. Therefore, ion-nitriding process has been adopted to the titanium specimen. The fatigue tests have been performed by both Ono-type (capacity; 98 N-m, 3,400 rpm) and small-Ono-type (capacity; 14.7 N-m, 3,000 rpm) rotating bending fatigue testing machines. Each specimen used in fatigue test has the partial shallow notch, which does not affect for its fatigue strength at all, in order to limit the fatigue damaged part. Fatigue crack initiation and propagation behavior are continuously observed using replica method at the partial shallow notch.

3 Result and discussion

Figure 2 shows an example of micro-structure of the longitudinal section of the specimen nitrided at 780°C for 1 hour (T8N-1). In this case, the chemical compound layer of titanium and nitrogen is approximately 2 μm in the thickness.
Although it is clear that the surface hardness is larger than that of matrix, it was difficult to measure the hardness in the surface layer itself for the thickness of a few micron meter. The hardness of this part was evaluated by another method. That is, Figure 3 shows the relation between surface hardness and indentation depth. The indentation depth becomes deeper according to larger load weight. As shown in this figure, the surface hardness of the specimen, T8N-1, has exceeded over Hv 1000. In addition, as ion-nitriding temperature becomes lower, the surface hardness becomes also lower.

Figure 4 shows the effect of heat treatment conditions on fatigue strength. When considering the fatigue limit is the limit stress in which the specimen can stand for the stress amplitude by $10^7$ cycles, the fatigue limit of the specimen, T8N-1, is smaller than that of the specimen, TNo, by 20 MPa. On the other hand, fatigue limit of the specimen, T8N-1, is larger than that of the specimen, T8V-1, by 80 MPa. Namely, though the fatigue limit of ion-nitrided specimen (T8N-1) is slightly smaller than that of the specimens (T8V-1) which is only annealed, that of ion-nitrided specimen becomes considerably larger than that of the specimen which is treated with the same heat treatment history.

Figure 5 shows the effect of ion-nitriding hours on fatigue strength. According to this figure, the fatigue limit of the specimen, T8N-1, becomes higher than that of the specimen, T8N-5, by 30 MPa. That is, the fatigue limit is deteriorated, when ion-nitriding hours becomes larger. Though the ion-nitriding temperature changes from 780 °C (T8N-5) to 680 °C (T7N-5) by keeping constant for another condition, there is no effect on the fatigue strength. These data is smaller than that of the specimen nitrided by 1 hour (T8N-1). The ion-nitriding temperature changes 580°C (T6N-5) and 480 °C (T5N-5). The same tendency as before would be concluded about the specimens T6N and T5N etc..

Figure 6 shows the relation between the fatigue limit and heat treatment temperature including ion-nitriding temperature. When the temperature becomes lower, the fatigue limit becomes higher. Though the fatigue limit of the ion-nitrided specimen, T8N-5, is higher than that of the vacuum annealed specimen in the same temperature, T8V-1, by 50MPa, that of the ion-nitrided specimen, T8N-5, becomes increased by removing the nitrided surface layer by 30MPa and this value is smaller than that of the no heat treatment specimen, TNo, by 20MPa. This is considered that the fatigue limit of Ti-6Al-4V specimen will be deteriorated by 100MPa under heat treatment in the temperature of 780 °C and increased by 50MPa under ion-nitriding. When the surface brittle layer was removed after ion-nitriding, its fatigue limit also becomes increased by 30MPa (see Figures 5 and 6). Even if the surface brittle layer was removed, nitrogen diffusion layer exists at the surface of the specimen and this diffusion layer raises the fatigue limit of the specimen by 80MPa. Static tensile tests had been already performed using the ion-nitrided specimen, T8N-1, and annealed one in vacuum, T8V-1, for the purpose of investigating the effect of surface layer on crack initiation.
Figure 7 shows the successive observation results of surface state during static tensile test, where $\varepsilon$, means the total strain (elastic strain + plastic strain) [6,7]. The black parts are $\alpha$ phase and white parts here and there are $\beta$ phase in the matrix. Figures 7 show the ion-nitrided specimen (T8N-1) and the annealed specimen in vacuum, T8V-1, in the same heat treatment condition. In Figure 7, when the total strain becomes about 0.7%, which is within elastic range, micro cracks have already initiated at $\alpha$ phase in perpendicular to loading direction. The length and number of cracks become increase according to increasing strain.

From the above results, even if there exists the very thin surface layer, i.e., about $2\mu$m in the thickness, micro-cracks initiate at less than 0.7% of the total strain and initiate in the perpendicular to the loading direction.

Figure 8 shows the representative successive observation results obtained by replica method for the fatigue crack initiation behavior of the specimen, T8N-1, under the stress amplitude of 450MPa. Fatigue cracks initiate at $4\times10^4$ cycles, and the cracks propagate and reach to be a final fracture at $5.34\times10^4$ cycles. That is, as the fatigue cracks initiate at about 75% of fatigue life ratio, the specimen breaks as soon as the fatigue cracks initiate. However, fatigue cracks not always initiate from the partial notch, because of severe limitation of the slip system in this material. Therefore, it is not certain whether this crack in Figure 8 would be the main crack for fracture or not.

Figure 9 shows the representative successive observation results obtained by replica method for the fatigue crack initiation behavior of the specimen, T7N-5, under the stress amplitude of 400MPa. From this figure, the fatigue crack initiates at the small surface defect, which was made during ion-nitriding process by $1\times10^4$ cycles. The fatigue cracks initiate at about 10% of the fatigue life ratio, they propagate and reach to be a final fracture by $1.33\times10^5$ cycles. When comparing Figures 8 and 9, there is much difference about the cycle ratio for fatigue crack initiation. The cycle ratio for fatigue crack initiation becomes rather small, when fatigue cracks initiate at some surface defects. While, it becomes larger as shown in Figure 10, when fatigue cracks initiate in $\alpha$ phase itself.

In addition, according to the observation results of surface states of the specimens, T7N-5 and T8N-5, subjected to the stress amplitude of fatigue limit by $1\times10^7$ cycles. It is found that the non-propagating micro-cracks are not existed in these specimens. The fatigue strength of nitrided specimen becomes slightly smaller than that of non-nitrided one and becomes considerably larger than that of the specimen with the same heat treatment history as the above.

**Conclusion**

Fatigue tests had been accomplished by rotating bending fatigue testing machine using a partial shallow notch of ion-nitrided Ti-6Al-4V alloys after the different
temperatures of heat treatment. In addition, the specimen’s surface has been successively observed by replica method during tests.

The main results obtained in this study are as follows:
(1) There exists a chemical compounds layer, which consists of Ti and nitrogen of 2 μ m in the thickness, and the diffusion layer of about 10 μ m. The micro-Vickers hardness number of the surface becomes over 1,000.
(2) The fatigue strength of ion-nitrided specimens are inferior to those of non-ion-nitried specimens. On the other hand, that of ion-nitrided specimens are superior to those of annealed specimens in the same temperature condition as the nitriding.
(3) Though the fatigue limit of the specimens nitrided at 780 °C for 5 hours roughly becomes the same as that of the specimen nitrided at 680 °C for 5 hours, the former’s fatigue strength is inferior to that of the specimen nitrided at 780 °C for 1 hour.
(4) As the fatigue strength becomes improved by removing the chemical compound surface layer, this chemical compound layer deteriorates the fatigue strength of nitrided specimen. From the above results, the surface layer would be requested to have the equivalent toughness to the matrix for improving its fatigue strength from the view point of practical use.

Reference


Table 1. Chemical composition. [mass %]

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Table 2. Heat treatment condition.

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<td>TNo</td>
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</tr>
<tr>
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<td>A</td>
<td>ⓐ</td>
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<tr>
<td>T8V-1</td>
<td>780°C×1hr Vacuum annealing*</td>
<td>A</td>
<td>×</td>
</tr>
<tr>
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<td>780°C×5hrs Ion-nitriding</td>
<td>B</td>
<td>ⓠ Ⓝ</td>
</tr>
<tr>
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<td>ⓠ</td>
</tr>
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<td>B</td>
<td>ⓡ</td>
</tr>
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<td>580°C×5hrs Ion-nitriding</td>
<td>B</td>
<td>ⓡ</td>
</tr>
<tr>
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<td>ⓠ</td>
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<tr>
<td>T5V-5</td>
<td>480°C×5hrs Vacuum annealing*</td>
<td>B</td>
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T6NS-5 and T5NS-5 were nitrided with steel plate.  
* ; No Ion-nitriding  
⊕ : Nitrided surface layer removed
Figure 1: Shape and dimensions of specimens.

(a) Type A

(b) Type B

Figure 2: Microstructure of nitrided Ti-6Al-4V alloy.
The number in parenthesis means loading weight; unit N

Figure 3: Micro Vickers hardness distribution of nitrided Ti-6Al-4V alloy.

Figure 4: S-N curves.
Figure 5: S-N curves.

Figure 6: Relationship between fatigue limit and heat treatment temperature.
Figure 7: Successive surface observation of crack initiation [6].

Figure 8: Successive observation of fatigue crack initiation (T8N-1).

Figure 9: Successive observation of fatigue crack initiation (T7N-5).