Fatigue crack propagation behavior and debris formation in Ti-6Al-4V alloys with different grain size

H J Kim[1, 2], J Nakahigashi[3], R Ebara[2] and M Endo[1, 2]

[1] Department of Mechanical Engineering, Fukuoka University
8-19-1, Nanakuma, Jonan-ku, Fukuoka, 814-0180, Japan
[2] Institute of Materials Science and Technology, Fukuoka University
8-19-1, Nanakuma Jonan-ku, Fukuoka, 814-0180, Japan
[3] Department of Mechanical System Engineering, Fukuyama University
1-3, Gakuen, Fukuyama, 729-0292, Japan

E-mail: hjkim@fukuoka-u.ac.jp

Abstract. Titanium alloy is widely used in applications where high specific strength as well as good heat and corrosion resistance is required. Consequently, there are a number of studies on the fatigue characteristics of titanium alloys. In recent years, grain refinement for metallic materials processed by several methods, such as severe plastic deformation, has been studied to improve the mechanical properties. Grain refinement of titanium alloy by the protium treatment is a new technology, and the fatigue properties of this material have yet to be sufficiently studied. Therefore in this study, tension-compression fatigue tests were conducted for a protium treated Ti-6Al-4V alloy with ultra-fine grains of 0.5 μm in average size as well as for an untreated alloy with conventional grains of 6 μm. Specimens had shallow, sharp notches with the depth of 50 μm and the root radius of 10 μm, which enabled successive observation of the initiation and early propagation behaviors of small fatigue cracks. Substantial amount of oxide debris was formed along the crack during crack propagation. The role of debris was discussed in association with propagation resistance.

1. Introduction

Use of titanium alloy as a structural material spreads in wide area of industry, having attractive properties such as high specific strength and good heat and chemical resistances. The fatigue properties of titanium alloys have thus been studied with great effort. Structural refinement of materials including titanium alloy has attracted attention since improvement of mechanical property is attained in many cases. To produce the fine-grained materials, the severe plastic deformation (SPD) processes such as the equal channel angular pressing (ECAP), the hot isostatic pressure (HIP) and the high pressure torsion (HPT) are commonly used. It is reported that fine-grained materials obtained by the SPD for Al alloy [1, 2], Mg alloy [3, 4] and Ti alloy [5] show an increase in monotonic and fatigue strength compared to the unprocessed materials with conventional-size grains. Another process with the hydrogen storage material for refinement is recently developed, which has mainly been applied to Mg-based and Ti-based alloys. One of the authors [6-7] developed a new method for refinement of Ti-6Al-4V alloy for medical use by means of the protium treatment. They reported that the ultra-fine grained Ti-6Al-4V alloy produced by this method, which had grains with an average grain size of 0.5 μm, had an enhanced 0.2% yield strength and exhibited a superplastic elongation of over 9000% at 1123K [6]. However, the grain refinement of titanium alloy by means of the protium treatment is a new technology, and the fatigue properties of this material are not well understood.

It is well known that oxide debris is formed due to the relative sliding of two material surfaces. There are thus many researches on debris formation in wear and fretting fatigue. For titanium alloy, there are reports that oxide debris was observed in sliding wear [8] and fretting fatigue tests [9]. Moreover, it was reported that oxide debris was also observed at a fatigue crack that propagated in shallow-notched Ti-6Al-4V alloy specimens subjected to tension-compression ($R = -1$) fatigue loading [10]. However, the process of oxide debris formation and its role in determining the fatigue properties have yet to be discussed in detail.

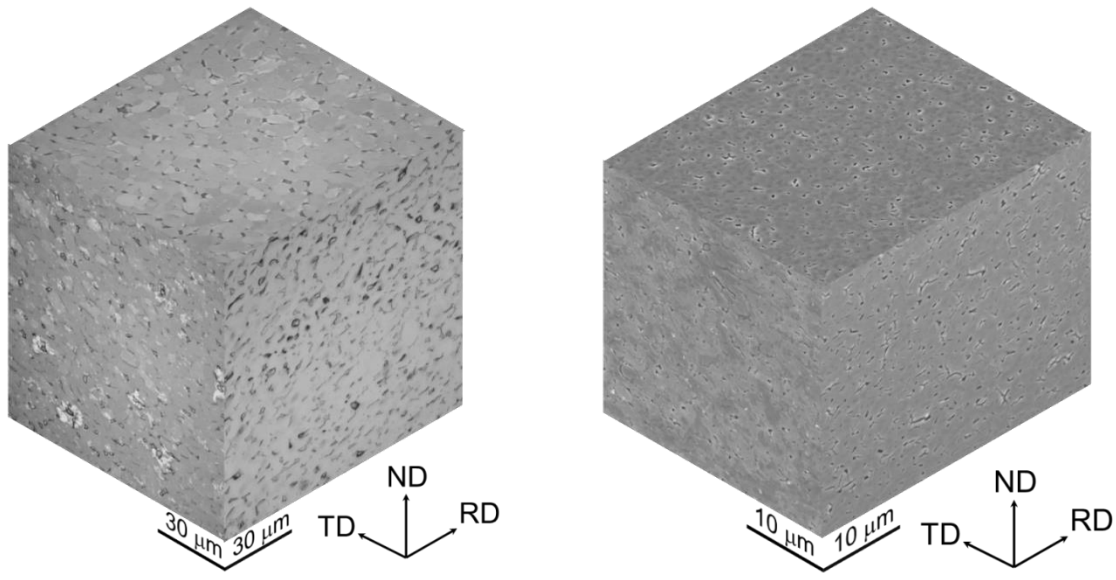
The objective of this study is to understand the fatigue crack propagation behavior of a titanium alloy, Ti-6Al-4V, refined to an ultra-fine grain level by the newly-developed protium treatment. The tension-compression ($R = -1$) fatigue tests of specimens with sharp, shallow notches were conducted for an ultra-fine grained alloy (0.5 μm in average grain size) as well as a conventional alloy (6 μm in average grain size). The behaviors of initiation and early propagation of small fatigue cracks at notch root were successively observed by the plastic replica technique. The focus of this study was also on the effect of oxide debris on the crack propagation properties.

2. Experimental procedures

The base material investigated was an $\alpha + \beta$ type Ti-6Al-4V alloy. The chemical composition is listed in Table 1. Ultra-fine grained Ti-6Al-4V alloy was obtained by the protium treatment described in the literature [6-7, 11]. The Ti-6Al-4V alloy with grains of conventional, regular size was obtained by the same process by using argon gas, instead of hydrogen gas. Figure 1 shows the microstructures of these Ti-6Al-4V alloys. The average grain sizes of the conventional and the ultra-fine grained Ti-6Al-4V alloys were 6 and 0.5 μm, respectively. For alloys with regular-sized and ultra-fine grains, the tensile strengths: 1026 MPa and 1266 MPa; the yield strengths: 955 MPa and 1125 MPa; the percent elongations: 21 % and 9.0 %; and the Vickers hardness values measured under a 9.8 N load: 314 and 351, respectively.

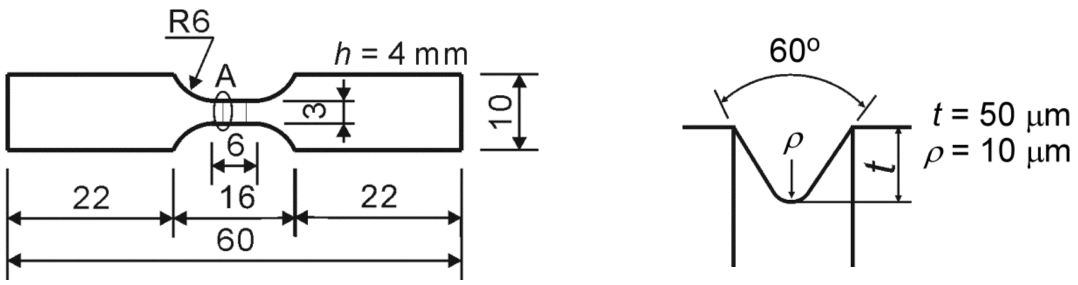
Table 1. Chemical composition (mass %)

Al	V	Fe	O	N	C	H
6.15	3.18	0.16	0.14	0.01	0.01	0.001



(a) Conventional alloy (b) Ultra-fine grained alloy

Figure 1. Microstructure.



(a) Tension-compression plate specimen (b) Cross sectional shape of notch

Figure 2. Shapes and dimensions of specimen and notch.

The shapes and dimensions of 4 mm thick plate specimens are shown in Figure 2(a). After surface finish with an emery paper of #2000, the stress relief annealing was performed at 600°C for 4 h [12], and about a 50 μm of surface layer was removed by electro-polishing. Two notches normal to the loading direction were introduced at both sides of a specimen, i.e. a specimen had four notches in total.

The cross-sectional shape and dimensions of notch is shown in Figure 2(b). The root radius, ρ, was 10 μm and the depth, t, was 50 μm, giving a stress concentration factor of about 5.5 under tension. After introducing the notches, about a 10 μm deep surface layer was removed by electro-polishing. In practice, by observing these notches, the crack initiation and propagation processes were studied.

The tension-compression fatigue tests were carried out using a uniaxial hydraulic fatigue testing machine at a frequency of 30 Hz and at a stress ratio, R, of -1. The fatigue limit was defined by the maximum stress amplitude under which a specimen did not break for $N = 2\times10^7$ cycles. To avoid the effect of bending, for every fatigue test, the strain at smooth surfaces of both sides were monitored with four strain gages, and then the alignment was adjusted using an alignment fixture until bending stresses were in the range of ±10 MPa. The plastic replicas were taken at appointed numbers of cycles by interrupting fatigue test. Gold was evaporated on the surface of these replicas before microscopic observation.

3. Results and discussion

3.1. S-N characteristics of notched specimens and fatigue process at notch root

In the case of $\alpha + \beta$ type Ti-6Al-4V alloy, the α-phase that plays a dominant role for fatigue crack initiation has a HCP crystal structure with very few slip systems. In the case of smooth specimen, the number of cracks that can initiate at the surface is quite few because of the small number of α-grains with a preferred crystallographic orientation with respect to loading direction. Therefore, it is hard to systematically investigate a typical fatigue process from crack initiation to failure by using the smooth specimens. In this study, thus, four notches with the notch root radius of 10 μm and the notch depth of 50 μm were introduced into the surface of a specimen to make a successive observation of fatigue process easier.

Figure 3 shows the *S-N* data for notched specimens of ultra-fine grained and conventional alloys. The fatigue limit of ultra-fine grained Ti-6Al-4V alloy was 270 MPa and that of conventional alloy was 240 MPa. Many researchers reported that the smooth specimen of the grain refined materials obtained by the SPD process for carbon steel [13, 14], stainless steel [15] and etc. [16-17] showed a significantly enhanced fatigue limit due to the grain size refinement. The smooth specimens of grain-refined pure titanium [18-19] and titanium alloy [5, 11, 20-21] showed similarly that the fatigue limit increased with the grain size refinement. In this study, however, the fatigue limit of ultra-fine grained Ti-6Al-4V alloy specimen with sharp, shallow notches did not show such a significant enhancement compared to that of conventional Ti-6Al-4V alloy specimen with identical notches. The ratio of fatigue of these alloys is calculated to be 270 MPa/240 MPa = 1.13, which would rather be a reasonable value, given the ratio of Vickers hardness of 351/314 = 1.12 is taken into account.

IOP Conf. Series: Journal of Physics: Conf. Series **843** (2017) 012039 doi:10.1088/1742-6596/843/1/012039

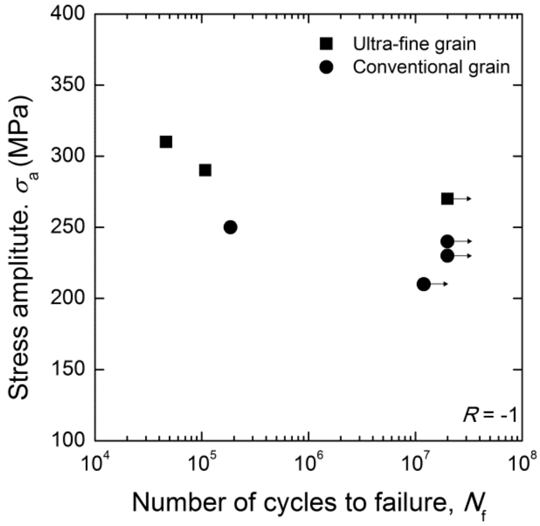
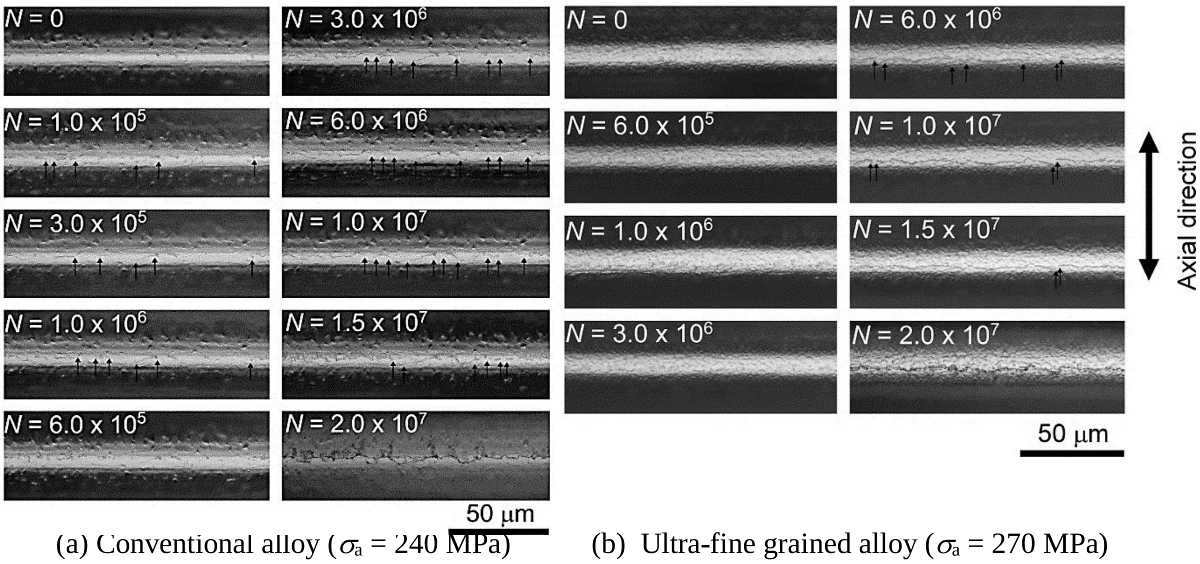


Figure 3. *S-N* data.

Figures 4 shows the fatigue crack initiation and propagation processes for conventional alloy (Figure 4(a)) and ultra-fine grained alloy (Figure 4(b)), which were successively observed at notch root at fatigue limit by the plastic replica technique. Only the micrographs taken at $N = 2.0\times10^7$ cycles show the real feature of notch roots, which were directly observed for specimens dismounted from the testing machine after the test. For both the conventional and ultra-fine grained alloys, numerous hairline cracks were observed at notch root, but slip bands could not be detected at crack initiation sites. Many cracks initiated before $N = 10^5$ cycles at notch root in the case of conventional-sized grain (Figure 4(a)), while in the case of ultra-fine grain (Figure 4(b)), the crack initiation was observed between $N = 3.0\times10^6$ and 6.0×10^6 cycles. Namely, more than 30 times of load cycles were required to give rise to crack initiation for ultra-fine grained alloy specimen subjected to an even higher stress of σ_a = 270 MPa. This result suggests that grain refinement would work to enhance the crack initiation threshold. This is presumably because of a decrease in the amount of cyclic slip resulting from an increase in yield strength associated with an increase in grain boundary density. Conversely, no marked difference of total number of cycles to failure in the finite life region (cf. Figure 3) was observed between two alloys though the number of data is not enough.

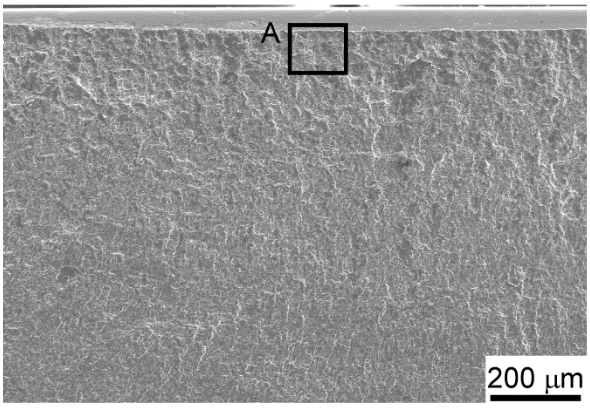


(a) Conventional alloy (σ_a = 240 MPa) (b) Ultra-fine grained alloy (σ_a = 270 MPa)

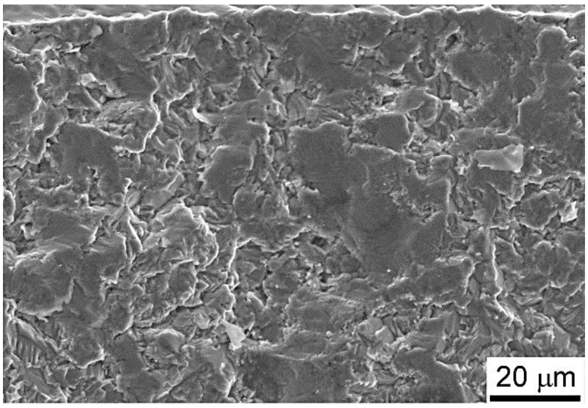
Figure 4. Fatigue process observed at notch root of Ti-6Al-4V alloy specimens.

3.2. Roughness of fracture surface and oxide debris formation

Owing to the notch, several fatigue cracks initiated simultaneously at the notch root, and they coalesced to each other. At fatigue limit, the crack path at notch root was more tortuous for conventional alloy (Figure 4(a)) than for ultra-fine grain alloy (Figure 4(b)) but the difference was small. The fracture surfaces originated from notches of specimens under the stress above fatigue limit are shown for conventional alloy in Figure 5 and for fine-grained alloys in Figure 6. The fracture surface very close to the notch root was relatively smooth for both alloys but at the place apart from the notch root, it was much rougher for conventional alloy than for ultra-fine grain alloy.

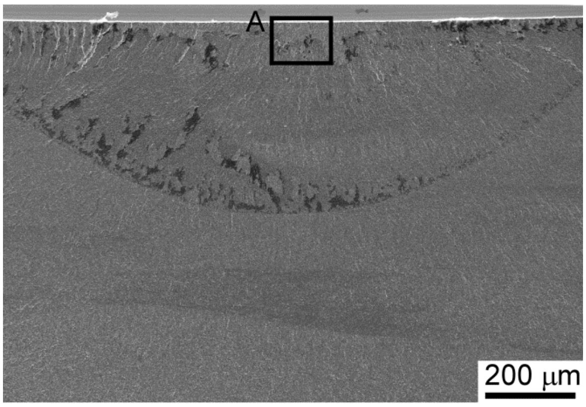


(a) Low-power field

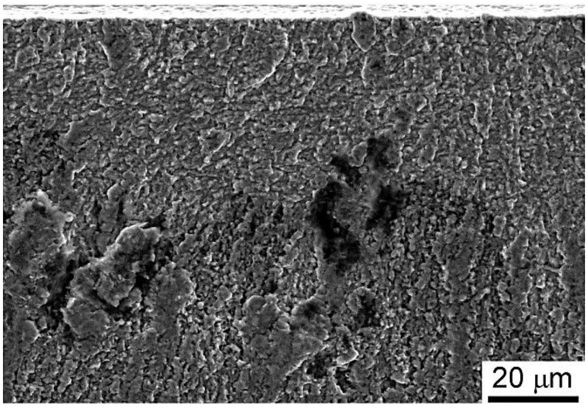


(b) Magnified image at A

Figure 5. Fracture surface of conventional alloy (σ_a = 250 MPa, N_f = 1.9×10^5 cycles).



(a) Low-powered field



(b) Magnified image at A

Figure 6. Fracture surface of ultra-fine grained alloy (σ_a = 290 MPa, N_f = 1.1×10^5 cycles)

Figure 7 shows the debris (the color is reddish brown) observed along the crack path at notch root, which is considered to be oxide particles emerged out from the inside of cracks as a result of repeated contact of crack face asperities. The occurrence of oxide debris is an evidence that the crack surfaces interfere to each other. The amount of debris was larger for conventional alloy than fine-grained alloy as seen in Figure 7. This may be because more debris is likely to be produced by contact of rougher surfaces. It is reported that in titanium alloys, the crack growth rate and the threshold stress intensity factor range, ΔK_{th}, is primarily influenced by the roughness-induced crack closure especially for low stress ratio [22] but in this literature, no report about debris is provided. The debris produced within the crack would possibly develop the oxide-induced crack closure. It is thus expected that the effect of crack

(a) Conventional alloy (b) Ultra-fine grained alloy

Figure 7. Oxide debris observed along crack path at notch root at fatigue limit.

closure is relatively larger for coarser-grained alloy that tends to form a rougher crack plane and thereby produces more amount of debris.

The fatigue life is the sum of crack initiation life and crack propagation life. As mentioned, with regard to crack initiation life, for the same stress level, more number of loading cycles was necessary for ultra-fine grain alloy than for conventional grain alloy. Conversely, with regard to crack propagation life, less contribution of crack closure is expected for ultra-fine grain alloy than for conventional grain alloy. As a result, it is considered that the total fatigue life had no marked difference because of these opposing effects. For the threshold condition, the effect of crack closure may be smaller since the crack surface roughness is smaller in the vicinity of notch root and the debris may possibly be easily ejected from shallow cracks. Consequently, the threshold levels are supposed to be mainly determined by the material factor, i.e. the Vickers hardness.

4. Conclusions

The influences of oxide debris and crack surface roughness on the fatigue crack propagation behavior were investigated by conducting high-cycle tension-compression fatigue tests using both conventional grain and ultra-fine grain Ti-6Al-4V alloy specimens containing sharp, shallow notches. The results of the present study are summarized as follows:

(1) The fatigue limit of notched specimen for ultra-fine grained alloy was increased by 12% compared to that of the conventional alloy. This is mainly because of the difference of material hardness.
(2) The fatigue crack initiation was delayed for ultra-fine grain alloy compared to conventional alloy. Inversely, the difference in total fatigue life was small.
(3) The oxide debris was observed at notch root of both conventional and the ultra-fine grained alloy specimens. The amount of the oxide debris was smaller for ultra-fine grained alloy than for conventional alloy.
(4) The crack closure effect was larger for conventional grain alloy than ultra-fine grain.

References

[1] Valiev R Z, Enikeev N A, Murashkin M Y, Kazykhanov V U and Sauvage X 2010 *Scr. Mater.* **63** 949
[2] Höppel H W, May L, Prell M and Göken M 2011 *Int. J. Fatigue* **33** 10
[3] Zhu R, Wu Y J, Ji W Q and Wang J T 2011 *Mater. Lett.* **65** 3593
[4] Jahadi R, Sedighi M and Jahed H, 2014 *Mater. Sci. Eng.* A **593** 178
[5] Saitova L R, Höppel H W, Göken M, Semenova I P, Raab G I and Valiev R Z 2009 *Mater. Sci. Eng.* A **503** 145
[6] Nakahigashi J and Yoshimura H 2002 *Mater. Trans.* **43** 2768
[7] Yoshimura H and Nakahigashi J 2002 *Int. J. Hydrog. Energy* **27** 769
[8] Rastkar A R Bloyce A and Bell T 2000 *Wear* **204** 19
[9] Antoniou R A and Radtke T C 1997 *Mater. Sci. Eng.* A **237** 229
[10] Shojima K, Weldle S, Okazaki S, Endo M, Eifler D and Balle F 2013 *Mater. Sci. Forum* **750** 232
[11] Ebara R Endo M Kim H J and Nakahigashi J 2014 *Fatigue of Materials III: Advances and*

IOP Conf. Series: Journal of Physics: Conf. Series **843** (2017) 012039 doi:10.1088/1742-6596/843/1/012039

Emergences in Understanding (Edited by Srivatsan T S Ashraf Imam M and Srinivasan R) TMS (The Mineral Metals & Material Society) 33
[12] Donachie M J 2000 *Titanium: A Technical Guide* (ASM International)
[13] Hirukawa H and Furuya Y 2009 *Tetsu to Hagane* **95** 79
[14] Chapetti M D Miyata H Tagawa T Miyata T and Fujioka M 2004 *Mater. Sci. Eng.* A **381** 331
[15] Matsumura Y Kishi K Ishibashi Y and Ebara R 2008 *J. Soc. Mater. Sci., Japan* **57** 134
[16] Vinogradov A Patlan V Suzuki Y Kitagawa K and Kopylov V I 2002 *Acta Mater.* **50** 1639
[17] Vinogradov A Hashimoto S and Kopylov V I 2003 *Mater. Sci. Eng.* A **355** 277
[18] Vinogradov A Y Stolyarov V V Hasimoto S and Vailev R Z 2001 *Mater. Sci. Eng.* A **318** 163
[19] Kim W J Hyun C Y and Kim H K 2006 *Scr. Mater.* **54** 1745
[20] Saitova L R Höppel H W Göken M Semenova I P and Vailev R Z 2009 *Int. J. Fatigue* **31** 322
[21] Nakaiso T 2005 Master thesis of Kagawa University
[22] Halliday M D Beevers C J 1981 *J. Test. Eval.* **9** 195