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Effects of grain boundary- and triple junction-character on intergranular fatigue crack nucleation in polycrystalline aluminum

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Abstract The effects of grain boundary- and triple junction-character on intergranular fatigue crack nucleation were studied in coarse-grained polycrystalline aluminum specimens whose grain boundary microstructures were analyzed by SEM-EBSD/OIM technique. Fatigue crack nucleation occurred mainly along grain boundaries and depended strongly on both the grain boundary character and grain boundary configuration with respect to the persistent slip bands. However, it was little dependent on the geometrical arrangements between the grain boundary plane and the stress axis. Particularly, random boundaries become preferential sites for fatigue crack nucleation. The fatigue cracks were also observed at CSL boundaries when the grain-boundary trace on the specimen surface was parallel to persistent slip bands. On the other hand, no intergranular fatigue cracks were observed at low-angle boundaries. The fatigue cracks were observed at triple junctions as well as grain boundaries. Their nucleation considerably occurred at triple junctions where random boundaries were interconnected. The grain boundary engineering for improvement in fatigue property was discussed on the basis of the results of the structure-dependent intergranular and triple junction fatigue crack nucleation.

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Introduction

It is well known that the grain boundaries play critical roles as preferential sites of crack nucleation and propagation in fatigue of polycrystalline materials, in addition to persistent slip bands (PSBs) [1–3]. Particularly, the fatigue fracture in fcc metals is dominated by the intergranular fatigue cracking [1–9]. The intergranular fatigue cracking has been investigated in connection with the geometrical arrangements of grain boundary plane to the stress axis [4–9], the interactions of the grain boundaries with the PSBs [6, 7, 10–12] and the dependence on grain boundary character [8–14]. Kim and Laird [5] investigated the conditions of fatigue crack nucleation at grain boundaries in the coarse-grained polycrystalline copper deformed at high strain amplitudes. According to their findings, the nucleation of intergranular fatigue crack is accelerated in the following conditions: (1) the misorientation of the adjacent grain is large, (2) the trace of the grain boundary on the specimen surface makes an angle in the range of 30°−90° with the stress axis, and (3) the dominant slip systems are directed over large slip distances at the intersection of the grain boundary with the specimen surface. More recently, Zhang and Wang [12] studied the effect of the interaction between the grain boundaries and the PSBs on the intergranular fatigue cracking in copper bicrystals containing grain boundaries with different characters. They reported that high-angle boundaries always became preferential sites for fatigue crack nucleation, irrespective of the angle between the grain boundary plane and the stress axis, and that fatigue cracks never nucleated at low-angle boundaries. Surprisingly, the coherent twin boundaries, which possess high intergranular fracture strength under static deformation, have been found to be a potential site for fatigue crack nucleation [9, 13, 14]. Heinz and Neumann [9], who investigated crack nucleation in high-cycle fatigue of austenitic stainless steel, suggested that twin boundaries in fcc materials involved a stronger strain localization than random boundaries because a twin boundary plane can play an important role as the slip plane in fatigue cracking. Until now, it has been not fully understood in polycrystalline materials whether low-Σ coincidence site lattice boundaries (CSL boundaries) other than Σ3 boundary (twin boundary) shows the higher resistance to intergranular fatigue cracking, as under a static stress condition. Moreover, it is necessary to reveal how the difference of grain boundary character affects the interactions of the grain boundaries with the PSBs and the stress axis which are associated with intergranular fatigue crack nucleation in polycrystalline materials.

On the other hand, it was found that triple junctions play an important role in the fracture of polycrystalline materials subjected to static deformation, because the compatibility stress is often generated in the vicinity of triple junctions, causing crack nucleation [15–20]. It was observed that triple junction can be a preferential site for cavitation in superplastic deformation of an Al–Li alloy. It was found that the triple junctions composed of more than two random boundaries become the most preferential sites for cavitation [18]. Furthermore, the importance of grain boundary connectivity and triple junction distribution was confirmed for crack nucleation and propagation in polycrystalline molybdenum, in connection with triple junction hardening phenomena [19, 20]. It was also found that the triple junctions with the higher connectivity of random boundaries are associated with the higher stress concentration leading to intergranular fracture under static stress condition. However, to our knowledge, the effect of triple junction character on the fatigue cracking has been little investigated, although the importance of triple junctions for fatigue fracture has been suggested in the literature [11].

Grain boundary engineering based on the concept of grain boundary design and control is useful and powerful tool to control the intergranular fracture in polycrystalline materials [21–24]. Watanabe and Tsurekawa [24] wrote an overview paper on the control of brittleness and development of desirable mechanical properties in polycrystalline systems by grain boundary engineering, on the basis of experimental and theoretical works performed up to the end of 1990's. It has been well established that the fracture stress can be increased by controlling the grain boundary microstructure, particularly the grain boundary character distribution (GBCD) and the grain boundary connectivity. The grain boundary engineering may be also applicable to the enhancement of fatigue fracture resistance of polycrystalline materials. Indeed, quite recently, an application of grain boundary engineering has been attempted for the control of high-cycle fatigue fracture in nickel-base super alloys [25, 26].

The purpose of this work is to reveal the effects of grain boundary character and triple junction character on the intergranular fatigue crack nucleation which were associated with the grain boundary configuration with respect to the PSBs and the stress axis in coarse-grained polycrystalline aluminum. Moreover, the potential of grain boundary engineering for the control of intergranular fatigue cracking in polycrystalline materials is discussed.

Experimental procedure

Specimen preparation

Pure aluminum (purity: 99.99%) was used for this work. Tensile specimens whose dimensions were 10 mm long, 4 mm wide and 3 mm thick were machined from the sheet subjected to cold rolling to 70% in strain, so as to keep the tensile direction of specimen being parallel to the rolling direction. Thereafter, the specimens were fully recrystallized by annealing at 673 K for 300 s in air. In order to observe the recrystallized microstructures in the course of fatigue test, the post-annealed specimens were mechanically polished using emery papers of 500–1,000 grade, and then electropolished in an electrolytic solution of perchloric acid–ethylene glycol–methyl alcohol with a volume fraction 1:3:6 at a current density of 1.4 A/cm^2 at 277 K for 60 s.

Characterization of grain boundary microstructure

The automated SEM-EBSD/OIM (Orientation Imaging Microscopy) from TSL inc. was applied to quantitatively analyze grain boundary microstructures, particularly, the GBCD and the grain boundary connectivity, in the whole area of pre-deformed specimens on a field emission-gun scanning electron microscope (FE-SEM HITACHI S-4200). In this work, the grain boundary character was described by Σ-value, although grain boundary plane also defines the grain boundary character and affects the properties of grain boundary as well as the Σ-value. CSL boundaries with $\Sigma \leq 29$ were defined as CSL boundaries within the maximum allowable angular deviation from the exact CSL orientation according to the Brandon's criterion, $\Delta\theta = 15/\Sigma^{1/2}$ [27]. The triple junctions are classified into four different types depending on the grain boundary connectivity of CSL boundaries and random boundaries, as discussed in Fortier et al. [28] and our former works [18–20]: (1) R0-type with no random boundaries (3-CSL), (2) R1-type with one random and two CSL boundaries (2-CSL), (3) R2-type with two random and one CSL boundaries (1-CSL), and (4) R3-type with three random boundaries (0-CSL).

Fatigue testing and observation of intergranular fatigue cracking

Fatigue tests were carried out by using a servo-hydraulic machine (Shimadzu Servopulser EHF-FB10 kN-10 LA) in air at room temperature. The sinusoidal loads were applied at the stress amplitudes between 18.4 MPa and 15.5 MPa at a stress ratio of 0.1 and at a frequency of 10 Hz. Tests were interrupted at 10, 10^2, 10^5, 10^6 cycles, in order to observe the development of fatigue cracking. The specimen surfaces were examined in detail by SEM to observe the intergranular cracks and the slip traces. The fatigue cracks were evaluated in connection with grain boundary character and triple junction character.

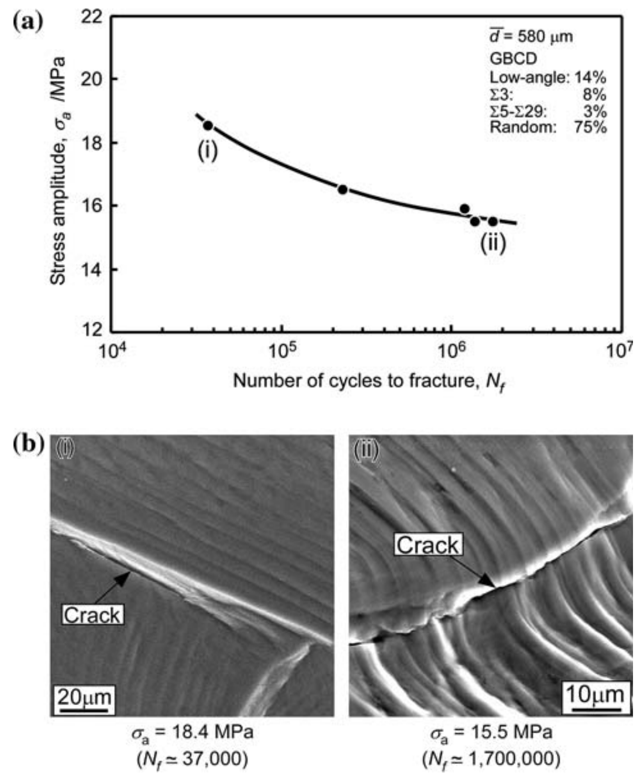

Fig. 1 (**a**) S–N curve of pure aluminum specimens and (**b**) SEM micrographs of fatigue crack nucleation and propagation at different stress amplitudes (i) $\sigma_a = 18.8$ MPa and (ii) $\sigma_a = 15.5$ MPa

Results and discussion

Fatigue properties and fatigue crack nucleation in aluminum specimens

Figure 1a shows the relationship between the stress amplitudes (σ_a) and the logarithm of the number of cycles to fracture (N_f), i.e., S–N curve, in pure aluminum specimens. The grain size and the GBCD of the specimens are given in this figure. The indicated values of grain size and GBCD are the average taken from all of the specimens. There was little difference between the data for each specimen and the average values. The specimens had a coarse-grained structure of the mean grain size of 580 µm. The number of cycles to fracture (N_f) decreases monotonically with increasing the stress amplitude (σ_a), and the S–N curve does not show any clear fatigue limit. Figure 1b shows SEM micrographs of fatigue crack in the specimens cyclic deformed at the stress amplitude of (i) 18.4 MPa ($N_f = ca.$ 37,000) and (ii) 15.5 MPa ($N_f = ca.$ 1,700,000). In both low- and high-cycle fatigue test conditions, fatigue cracks predominantly nucleated at grain boundaries and triple junctions. On the other hand, the fatigue crack nucleation along the PSBs in the grain interior was only observed in the vicinity of grain boundaries and triple junctions. It is confirmed that the grain boundaries and the triple junctions were preferential sites for fatigue crack nucleation in polycrystalline aluminum under the present test conditions.

Effect of grain boundary character on intergranular fatigue crack nucleation

Figure 2a–d are SEM micrographs showing intergranular fatigue cracks on the surface of the specimens fractured by fatigue deformation at a stress amplitude of 15.5 MPa. The characters of individual grain boundaries and triple junctions were shown by capital letters in white and black boxes in the micrographs, respectively. Low-angle, CSL and random boundaries are indicated by L, Σ plus number and R, respectively. Triple junction characters are indicated by the symbols defined in the previous section. As shown in Fig. 2a–c, although most intergranular fatigue cracks were observed at random boundaries, they were sometimes nucleated at Σ3 and Σ11 CSL boundaries (Fig. 2d). On the other hand, the fatigue cracks were never observed at low-angle boundaries. Moreover, fatigue cracks also nucleated at triple junctions (Fig. 2c). The effects of grain boundary character and triple junction character on intergranular

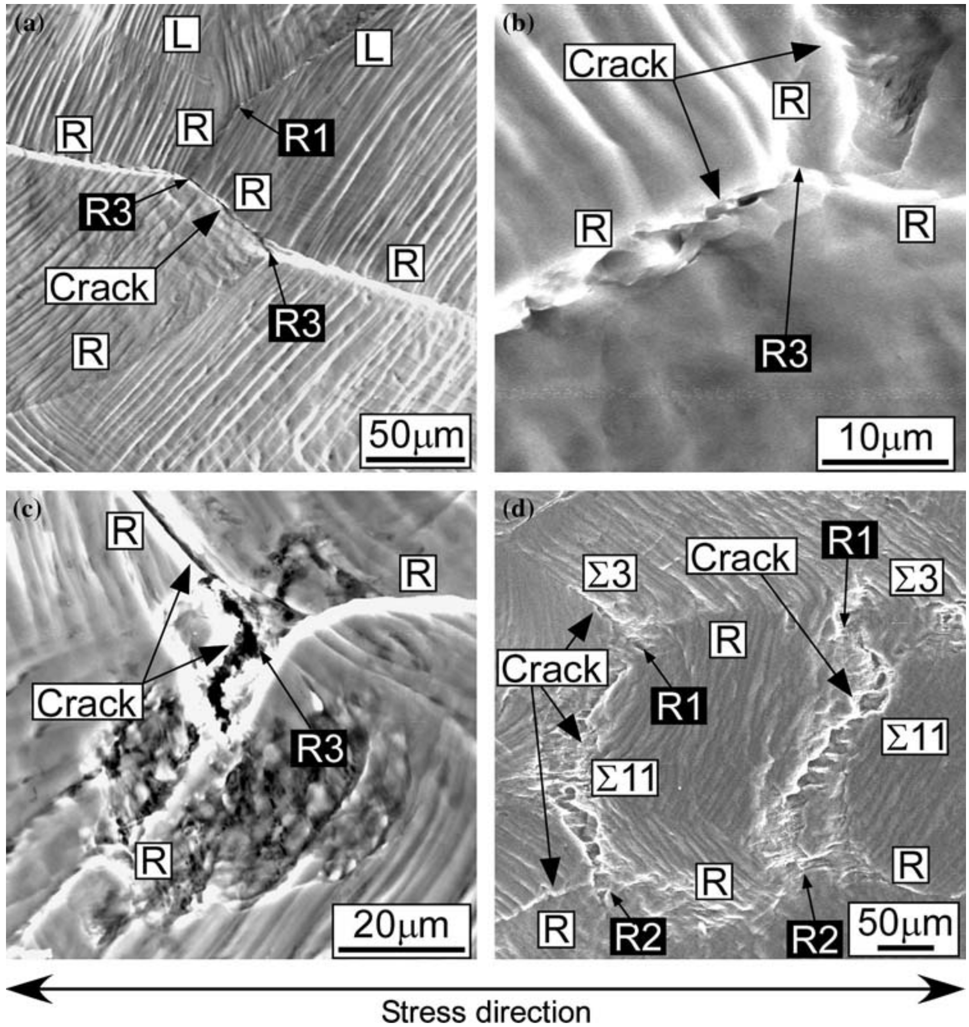

Fig. 2 Scanning electron microscopy (SEM) micrographs of intergranular fatigue cracks nucleated and propagated in the specimens cyclically deformed at $\sigma_a = 15.5$ MPa

fatigue crack nucleation are quantitatively examined in the following.

Figure 3 shows the relationship between the frequency of fatigue cracking and the grain boundary character. The data were obtained from the whole gauge area of the two specimens fatigue deformed at the same condition ($\sigma_a =$ 15.5 MPa). The fatigue cracks were observed at 53 grain boundaries for 890 total grain boundaries. Furthermore, the percentage of 83% within cracked grain boundaries was random boundaries. No intergranular fatigue cracks were observed at low-angle boundaries. As shown in Fig. 4a and b, the PSBs continuously transferred across the low-angle boundaries. Therefore, the stress concentration must be lower at low-angle boundaries than at random boundaries. These results are well consistent with the recent findings by Zhang and Wang [12], who studied the effect of the interactions between PSBs and grain boundaries on intergranular fatigue crack nucleation in copper bicrystals. Moreover, it was reported that the fatigue crack nucleation was associated with the formation of a grain-boundary step due to difference between the slip systems in adjoining grains [4]. The boundary steps were smaller at low-angle boundaries than at random boundaries as shown in Fig. 4a.

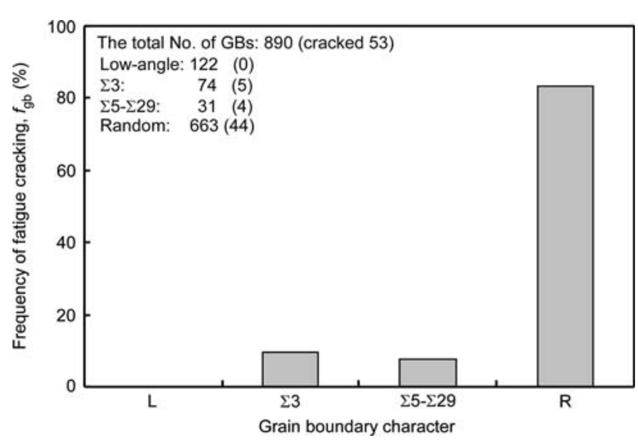

Fig. 3 Relationship between the frequency of fatigue cracking and the grain boundary character

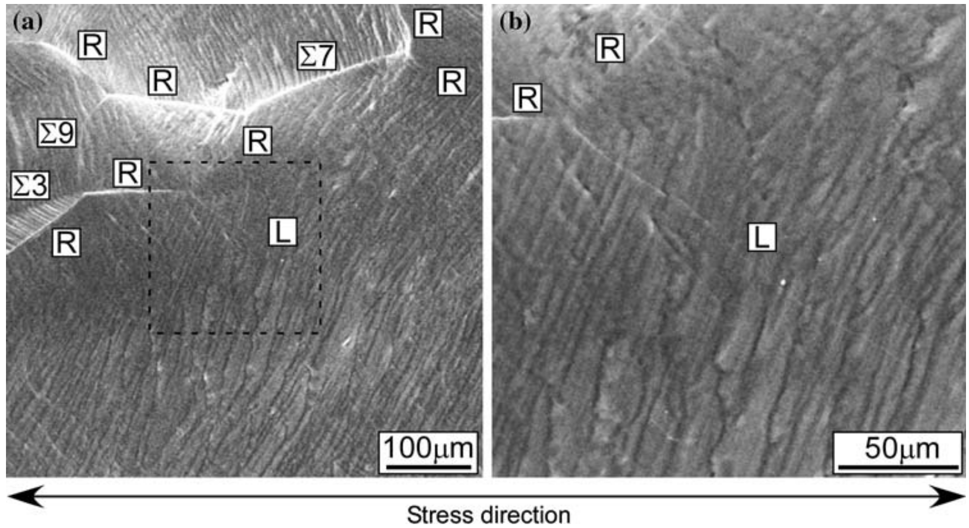

Fig. 4 (**a**) SEM micrographs taken for the surface of cyclically deformed specimen with distinct grain boundaries, (**b**) is a high magnification of the observed area including the low-angle boundary surrounded by dotted lines in (**a**)

Our observations have confirmed that the low-angle boundaries intrinsically possess superior resistance to fatigue fracture.

Moreover, intergranular fatigue cracks were also observed at CSL boundaries with low Σ values such as Σ3, Σ9, and Σ11. It was reported that a fatigue crack nucleates at (111) Σ3 coherent twin boundary, nevertheless it has the lowest boundary energy in fcc metals [9, 13, 14]. Heinz and Neumann [9] examined the nature of fatigue cracking at the twin boundary in austenitic stainless steel by high-cycle fatigue tests at room temperature. They pointed out that the twin boundaries in fcc structure involved a stronger strain localization than random boundaries because the twin boundary plane is as same as the slip plane in fcc structure. Kaneko et al. [14] also studied fatigue cracking in copper bicrystals having the (111) Σ3 coherent twin boundary, and reported the influence of the deviation angle from the exact Σ3 coincidence-relation on intergranular fatigue cracking. They found that the ratio of intergranular fatigue cracking to the primary crack length increased rapidly with increasing the deviation angle $\Delta\theta$ in the region ranging from 3° to 5°, and the Σ3 boundaries with the deviation angle less than 3° involved no intergranular fatigue cracking, while the ratio was reduced again when $\Delta\theta$ was beyond 9°, having a peak around $\Delta\theta = 4°–5°$. In the present work, most of cracked CSL grain boundaries possessed the deviation angles of less than 2° from the exact CSL relations, and we found that the fatigue cracking occurred at CSL boundaries when the boundary traces on the specimen surface were parallel to the PSBs formed in one or both of adjoining grains, as shown in Fig. 2d. Inoko et al. [6, 7] reported early that the intergranular fatigue cracking in aluminum bicrystals occurred under the following conditions; (a) grain boundary plane was parallel to the {111} orientation, and (b) it was parallel to the observably operated primary and/or secondary slip planes. In particular, intergranular fatigue fracture appears rather complicated regarding the Σ3, so called twin boundary, with different deviation angles. This may suggest structural effect on the interaction of grain boundaries with crystal slip or lattice dislocations [29].

Effects of geometrical configuration of grain boundaries on the intergranular fatigue crack nucleation

As mentioned above, it has been revealed that the geometrical configuration of grain boundary plane respective to the stress axis [3–5] and to operative slip system in the grain interior [6, 7, 10–12] play some critical role in fatigue crack nucleation and propagation in bicrystalline and polycrystalline metals. Now let us look at the result obtained from the present observations.

Figure 5 shows the relationship between the number of intergranular fatigue cracks and the angle made by the grain-boundary trace on the specimen surface and the stress axis. In this figure, the angles of 0° and 90° means that the boundary trace is parallel and perpendicular to the stress axis, respectively. There appears no significant dependence of occurrence of intergranular fatigue cracking on the angle. Thus, it can be said that the nucleation of intergranular fatigue cracks is little affected by the geometrical configuration of grain boundary plane to the stress axis from our present observation in polycrystalline aluminum.

Effect of triple junction character on fatigue crack nucleation

The triple junctions can be preferential nucleation sites for fatigue cracks, as well as grain boundaries (as seen in Fig. 2c). This section discusses the effect of triple junction

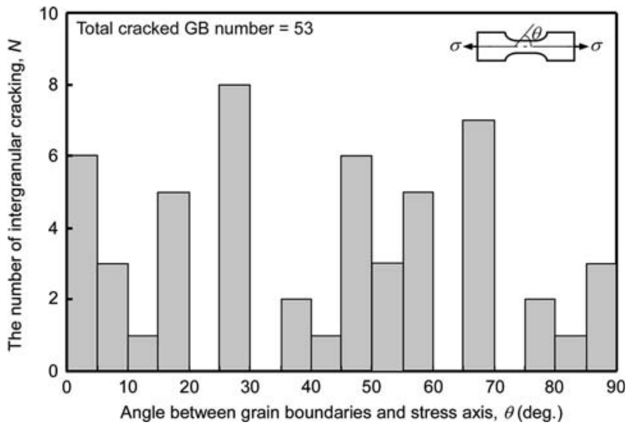

Fig. 5 The number of intergranular cracking as a function of the angle between the traces of grain boundaries on the specimen surface and the stress axis

character on fatigue crack nucleation in polycrystalline aluminum.

Figure 6 shows the relationship between the frequency of fatigue cracking and the triple junction character. The data were obtained from the whole of the gauge area of the two specimens subjected to fatigue deformation at the same condition ($\sigma_a = 15.5$ MPa). The fatigue cracks were detected at 38 triple junctions for 724 total triple junctions. The triple junctions with a higher connectivity of random boundaries became preferential sites for crack nucleation. The percentage of 71% of cracked triple junctions was R3-type triple junctions. In contrast, no intergranular fatigue cracks were observed at R0-type triple junctions. These results evidenced that fatigue crack nucleation can occur at triple junctions strongly depending on the triple junction character.

Recently, the present authors have studied the structure-dependent grain boundary hardening and triple junction

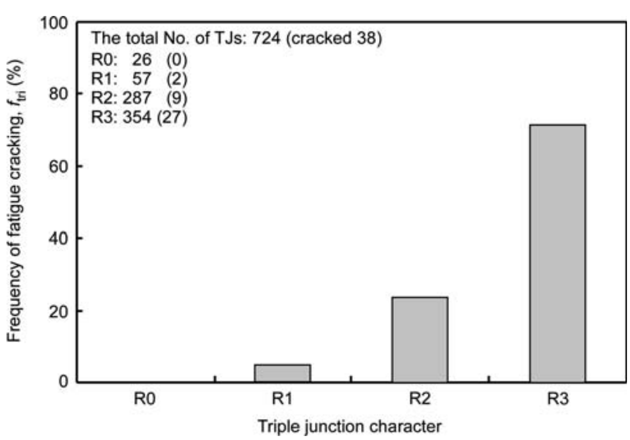

Fig. 6 Relationship between the frequency of fatigue cracking and the triple junction character

hardening in polycrystalline molybdenum in connection with plastic deformation and fracture by the microindentation tests [19, 20]. The results from these works suggest that the triple junctions, which are composed of more low-angle and CSL boundaries such as R0- and R1-type junctions, are favorable for preventing intergranular fracture under static stress condition in brittle polycrystalline materials because of their generation of a lower level stress concentration. It is likely that the stress concentration at such triple junctions would be lower level even under dynamic stress condition.

Control of intergranular fatigue crack by grain boundary engineering

Quite recently, Gao et al. have attempted an application of grain boundary engineering to the control of fatigue fracture in nickel-base superalloy [25, 26]. Here, we discuss the possibility grain boundary engineering for the control of intergranular fatigue crack nucleation in polycrystalline materials on the basis of the structure-dependent intergranular fatigue crack nucleation observed in the present work.

Figure 7a, b shows the schematic illustrations of the relationship between intergranular fatigue crack nucleation and grain boundary character and triple junction character. Figure 7a shows the condition before intergranular fatigue fracture of polycrystalline materials with grain boundaries and triple junctions subjected to deferent levels of stress concentration depending on their character, and Fig. 7b shows the condition after intergranular fatigue cracking occurred. These figures were modified from the illustration for structure-dependent intergranular fracture proposed for brittle polycrystalline materials under a static stress condition [20] on the basis of the observations on structure-dependent intergranular fracture under a dynamic stress condition. The fatigue cracks are nucleated at grain boundaries and triple junctions due to the interactions of PSBs with grain boundaries when a polycrystalline material is subjected to cyclic loading. The intergranular fatigue cracking will occur at different levels of the stress concentration according to the grain-boundary character and the triple-junction character. Intergranular fatigue cracks may preferentially occur at random boundaries and at triple junctions with more random boundary interconnected such as R3- and R2-types. On the other hand, low-angle boundaries and R0- and R1-type triple junctions possess a higher resistance against fatigue crack nucleation. The fatigue cracks can be controlled by CSL boundaries, unless they are aligned being parallel to PSBs. Therefore, it is expected that enhancement of fatigue fracture resistance in polycrystalline materials can be achieved by controlling the

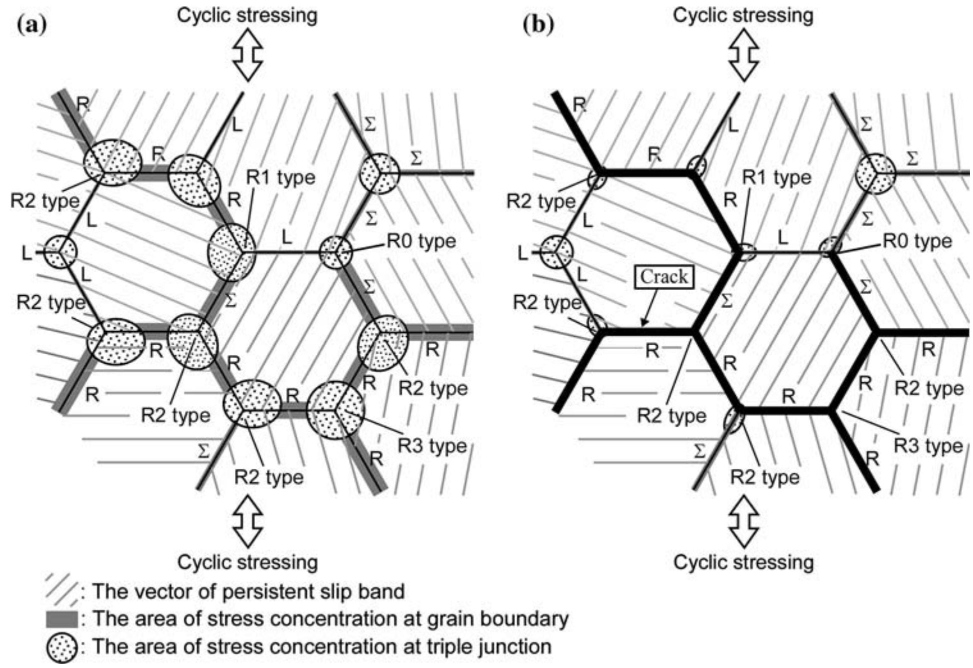

Fig. 7 Schematic illustration of structure-dependent intergranular fatigue fracture processes of polycrystalline aluminum, (**a**) before intergranular fatigue fracture for a polycrystal with grain boundaries and triple junctions of different levels of stress concentration, (**b**) after intergranular fatigue cracks nucleation and propagation

GBCD, triple junction character distribution and grain boundary geometrical configuration.

Conclusions

The effects of the grain boundary character and triple junction character on intergranular fatigue crack nucleation were studied by using the polycrystalline aluminum specimens whose grain boundary microstructures were quantitatively analyzed by SEM-EBSD/OIM. The results obtained are as follows.

1. Intergranular fatigue cracks nucleated predominantly at random boundaries, while the fatigue cracks never observed at low-angle boundaries. The fatigue cracks at CSL boundaries were observed only when the boundary trace on the specimen surface was parallel to PSBs in one or both of adjacent grains.
2. Intergranular fatigue crack nucleation strongly depended on the interaction of PSBs with grain boundaries with distinct grain boundary character, and little depended on the angle made by the grain-boundary trace and the stress axis.
3. Fatigue crack nucleation at triple junctions strongly depended on the triple junction character. The triple junctions with higher connectivity of random boundaries became preferential sites for fatigue crack nucleation.
4. The possibility of grain boundary engineering for the control of intergranular fatigue cracking in polycrystalline metallic materials has been discussed in connection with the structure-dependent fatigue crack nucleation at grain boundaries and triple junctions in polycrystalline aluminum.

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