High-temperature cyclic creep and fracture behavior of Cu-SiO_2 bicrystals

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Abstract

Orientation-controlled copper bicrystals with dispersed SiO_2 particles were cyclically crept at 873 K in vacuum. Although the lives of the bicrystals first tended to increase monotonically with decreasing stress amplitude at a higher stress amplitude region, the bicrystal with higher misorientation angle showed discontinuous and abrupt live shortening at a lower stress amplitude region. This transition of the cyclic creep behavior, which depended strongly on the grain-boundary character, was brought about by the occurrence of brittle intergranular fracture (BIF). Since elongated voids were observed on the grain-boundary fracture surfaces, the occurrence of BIF in the bicrystals possessing easily-slidable grain boundaries was identified to be caused by grain-boundary sliding (GBS) and particle dragging by GBS to form the elongated voids. At the lower stress region, such voids were easily formed by GBS. At the higher stress region, however, grain-boundary steps those were densely formed by plastic deformation of the matrices effectively suppressed GBS, resulting in the absence of BIF. The mechanism of grain-boundary and stress-amplitude dependent fracture behavior will be discussed in relation with GBS.

Keywords: Cyclic creep, bicrystal, fracture, Cu-SiO_2 alloy, grain-boundary sliding, high temperature
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1. Introduction

It is well known that second phase particles lower the mobility of dislocations even at high temperatures. The particles impede also grain boundary sliding and migration. The strength of the dispersion-hardened metallic material is, therefore, maintained even at high temperatures.

From the numerous studies on dispersion-hardened alloys, a problem of grain-boundary cracking (GBC) has been focused. That is, the dispersed particles on grain boundaries sometimes hasten the GBC during static tensile deformation contrary to the expected role of strengthening by the dispersoids [1,2]. This occurs because grain-boundary sliding (GBS) causes stress concentration sites around the particles, which impede GBS, and promotes easy void formation.

Although high-temperature fatigue / cyclic creep is one of the most basic mechanical properties to be examined, studies using dispersion-hardened alloys are quite few, as far as the authors know [3,4]. Recently, Miura et al. have reported from fatigue experiments on a Cu-SiO_2 alloy at elevated temperatures that a critical stress amplitude appears where the dominant cracking and crack propagation mode change from transgranular to intergranular [5, 6]. Below the critical stress amplitude, the fatigue life was abruptly shortened by the occurrence of brittle intergranualr fracture (BIF). They attributed the fracture-mode change to the occurrence of GBS. However, their experiment to confirm the occurrence of fracture-mode change was carried out on a bicrystal with a fixed misorientation angle. In the present study, the cyclic creep / fatigue behavior of Cu-SiO_2 bicrystals having different misorientation angles is investigated at 873 K. Special emphasis was placed on the role of GBS and GBC/BIF on the cyclic creep life.

2. Experimental

Bicrystals of a Cu-0.1mass% Si alloy with [011] twist grain boundaries were grown by the Bridgman method using seed crystals. The misorientation angles were chosen to be 12^0 and 37.2^0. The sliding capability of these grain boundaries is in the order of $12^0 \ll 37.2^0$ according to the results by Monzen et al. [7]. The bicrystals were internally oxidized by the powder pack method with a mixture of Cu (1 part), Cu_2O (1 part) and Al_2O_3 (2 parts) at 1273 K for 24 h, followed by degassing treatment at 1273 K for 24 h in a graphite mold in vacuum. By these treatments, we obtained Cu-0.87vol.%SiO_2 alloy bicrystals. The yield stresses (σ_y) of these bicrystals obtained by monotonic tensile tests at 873 K and at a strain rate of 4.2×10^{-4} s^{-1} were 27.5 MPa and 19.4 MPa for the 12^0 and 37.2^0 bicrystals, respectively. Bicrystal specimens, 12 mm gage length and 4 x 2 mm^2 cross-section with boundaries inclined at 45 degrees to the loading axis, were cut by electric discharge machining (Fig. 1). After mirror-like finishing of the surfaces, cyclic creep tests at 20 Hz were carried out in a vacuum of 10^{-3} Pa at 873 K on a servo-hydraulic machine. A load ratio of R = 0.1 was chosen. The evolved microstructures and fractographs were observed in a scanning electron microscope (SEM).

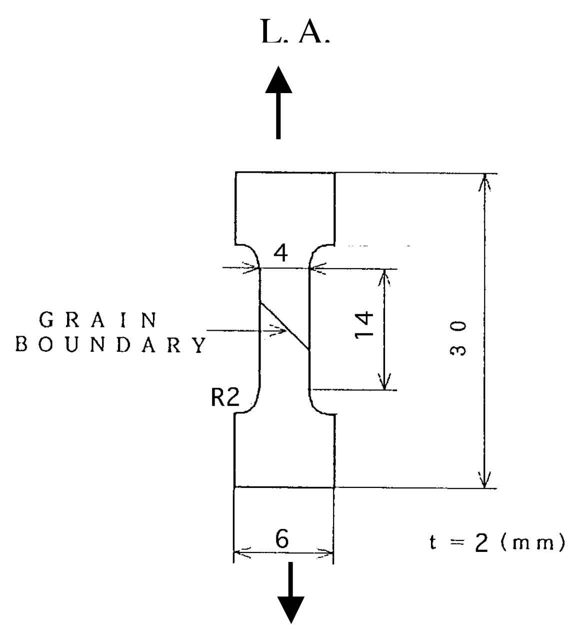

Fig. 1 Shape of a bicrystal sample for cyclic creep test. Grain boundary is inclined 45 degrees against loading axis (L.A.).

3. Results and discussion

Figure 2 shows strain (ε) vs. number of cycles (N) of the 37.2^0 bicrystals. Here, $\sigma_a = (\sigma_{max} - \sigma_{min})/2$, where σ_{max} and σ_{min} are the maximum and minimum stresses, respectively. All the ε-N curves resemble each other irrespective of stress amplitude, showing a gradual increase in strain in a steady-state stage followed by a rapid increase till rupture. However, the slope $d\varepsilon/dN$ at the steady-state stage increases with increasing stress amplitude. This is because a larger amount of creep strain per cycle occurs at higher stress amplitude. No dynamic recrystallization took place at any testing conditions in the present study. It should be noted in Fig. 2 that some of the lives cyclically crept at higher stress amplitudes are longer than those crept at lower stress amplitudes.

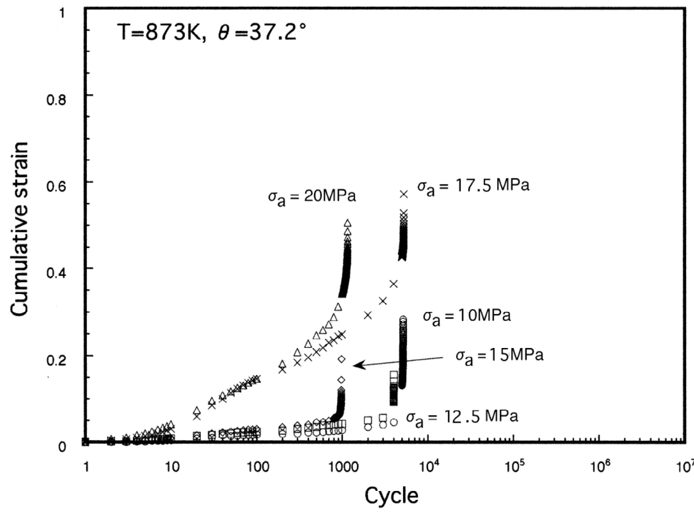

Fig. 2 Strain – cycle curves for 37.2° bicrystals cyclically crept at 873 K.

The results of the stress amplitude - cycles to fracture (S - Nf) curves for the bicrystals are summarized in Fig. 3. The open, filled and bracketed symbols indicate transgranular, intergranular and the mixed-mode fracture, respectively. For the 37.2^0 bicrystal, the life becomes longer with decreasing stress amplitude and, then, discontinuously drop at a critical stress amplitude between 15 and 20 MPa, followed by another monotonic increase. On the other hand, the life of the 12^0 bicrystal decreased monotonically showing only transgranular fracture. The change in the fracture-mode from transgranular to intergranular took place via critical stress amplitude. It is obvious, therefore, that the observed discontinuous life decrease with decreasing stress amplitude was caused by the fracture-mode change. Miura et al. have attributed the cause of the BIF to GBS that creates stress concentration sites both during monotonic tensile test and cyclic creep [2,5,6]. GBS capability of the dispersion-hardened alloys at elevated temperature is mainly controlled by inherent grain-boundary viscosity η_o [1]. The value of η_o of the 12° grain boundary is about two orders of magnitude larger than that of the 37.2° one [7]. This data suggests that GBS becomes much easier in the order of

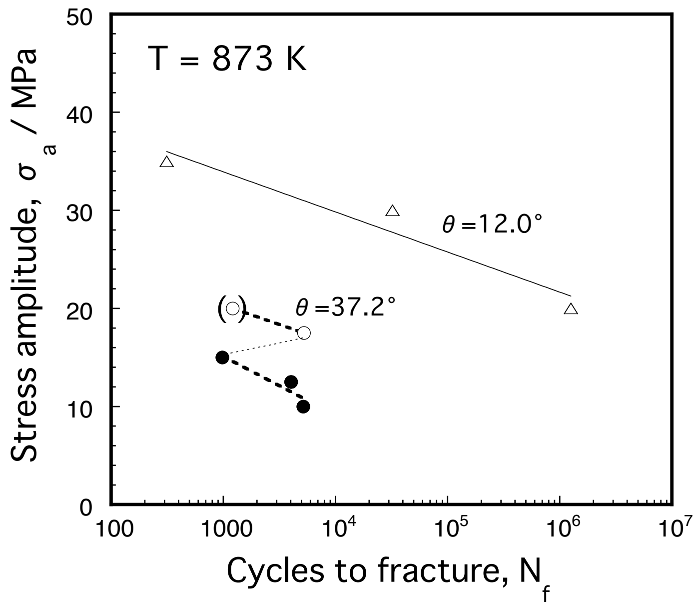

Fig. 3 The S-N_f diagrams of the bicrystals cyclically crept at 873 K. Open, solid and bracketed symbols indicate transgranular, intergranular and mixed-mode fracture, respectively.

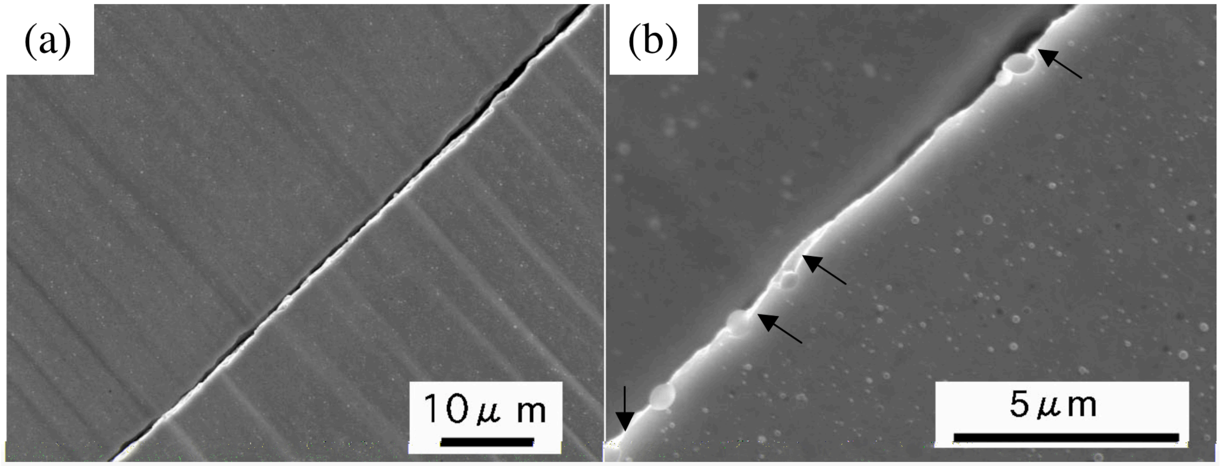

Fig. 4 Surface cracks initiated at the grain boundary of 37.2° bicrystal cyclically crept to 2000 cycles at 15 MPa. (b) is a boundary morphology in other place. Arrows indicate nucleated cavities at the particles.

$12^0 << 37.2^0$, therefore, the formation of the stress concentration sites and cracking at the grain boundary are considerably easier on the 37.2° grain boundary than the 12^0 one.

Typical SEM photographs of initiated GBC observed on the surface of the 37.2^0 bicrystal crept to 2×10^3 cycles are exhibited in Fig. 4. In Fig. 4 (a), GBC took place sharply along the grain boundary. At this cycles, no other crack was observed in matrices. It is obvious, therefore, that grain boundary is the most preferential crack nucleation site during high-temperature cyclic creep. In other part of the grain boundary where GBC was not so grown, cavities were formed only on one side of the grain-boundary particles, as shown in Fig. 4 (b). These cavities were formed by particle dragging by GBS.

Typical features of the grain-boundary fracture surface of the 37.2° bicrystal cyclically crept under a stress amplitude

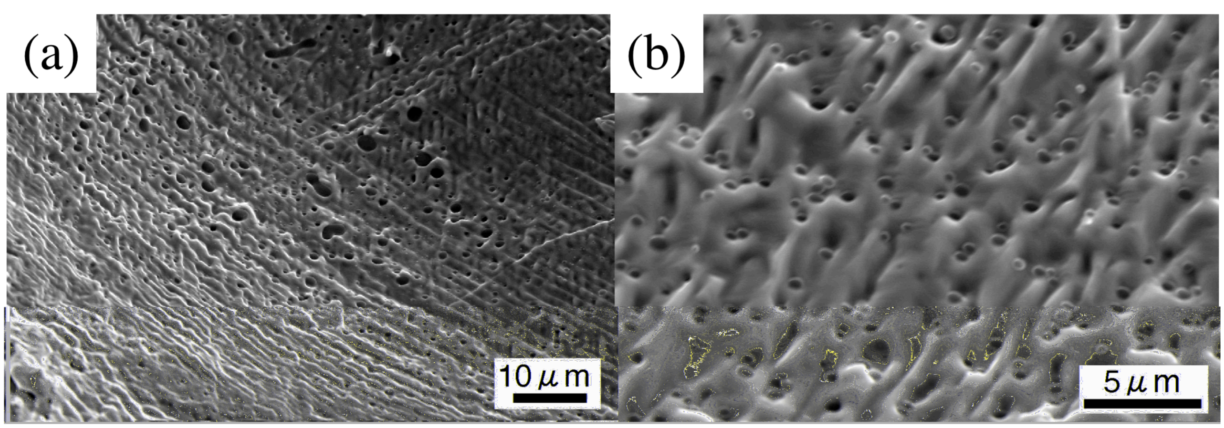

Fig. 5 Grain-boundary fracture surfaces of the 37.2°bicrystal cyclically crept at 873 K and under a stress amplitude of 15 MPa. The coverage ratio of the characteristic feature of (a) and (b) was about 0.61 and 0.19, respectively.

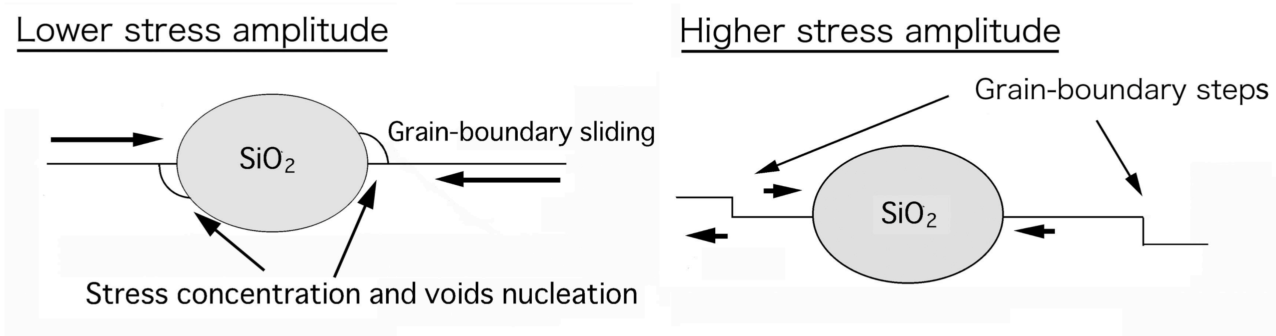

Fig. 6 Schematic illustration of the mechanism of void formation and grain-boundary strengthening under lower and higher stress amplitudes, respectively.

of 15 MPa is exhibited in Fig. 5. Fig. 5 (a) shows the most typical feature of striation with elongated voids. Some other part of the grain-boundary fracture surface was covered only by the elongated voids. They were formed by particle dragging by GBS as already mentioned in the explanation of Fig. 4 (b). The elongated voids suggest that the grain-boundary particles could not effectively suppressed GBS even under applied stress amplitude lower than the yield stress. About 80% of the grain-boundary fracture surface was composed of the above two characteristic features. This indicates the important role of the elongated voids and striation on GBC and BIF.

Miura et al. have reported that the amount of GBS during cyclic creep is comparable with the length of the elongated voids [6]. Furthermore, they found that the amount of GBS was larger when a bicrystal was deformed at a stress amplitude below the critical stress amplitude than when deformed above it. The nucleated cracks caused by GBS propagated intergranularly and combined with each other to rupture. When GBS becomes easier, the elongated voids are formed more easily and, therefore, GBC should take place more easily. This would well explain the easier occurrence of BIF and shorter life of the bicrystals of easier sliding nature.

The mechanism of the life change below and over the critical stress amplitude is schematically illustrated in Fig. 6. When cyclically deformed at a lower stress than the critical stress amplitude, voids formation by particle dragging by GBS are soon developed around the particles. This brings easy GBC and, then, BIF. On the other hand, when cyclically deformed at a higher stress than the yield stress, in-grain plastic deformation induces grain-boundary steps and they contribute to efficient suppression of GBS. The fromed grain-boundary steps contribute drastically to scatter of the stress concentration around the particles. Therefore, voids formation and intergranular fracture become much difficult, and this causes lengthening of the life.

4. Conclusions

Cyclic creep life of Cu-SiO_2 bicrystal with 37.2^0 boundary sharply drops at a critical stress amplitude due to the fracture-mode change from transgranular to intergranular with decreasing stress amplitude. On the other hand, that of 12^0 bicrystal decreased monotonically showing transgranular fracture irrespective of stress amplitude. Such cyclic creep behavior depends strongly on the grain-boundary character. Easy-sliding grain boundary shows fracture-mode transition. The impediment of GBS causes the void and crack nucleation at the grain boundary. The grain boundary with easier-sliding nature, therefore, shows easier occurrence of intergranular fracture and shorter life.

Acknowledgments

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