ORALREFERENCE: ICF100301OR

FATIGUE AND FRACTURE OF Cu-SiO_2 BICRYSTAL WITH A [011] 18° TWIST BOUNDARY AT 673K

Hiromi Miura and Taku Sakai

Department of Mechanical Engineeringand Intelligent Systems,
The University of Electro-Communications, Chofu, Tokyo 182-8585, Japan

ABSTRACT

Fatigue and fracture behavior of a copper bicrystal containing dispersed SiO_2 particles and having a [011] 18° twist boundary has been investigated at a stress amplitude of 40MPa at 673K. The grain boundary was inclined 45 degrees to the loading axis to examine effect of grain-boundary sliding (GBS) on fatigue behavior. Crack nucleation took place initially at particle/matrix interface on the grain boundary. The nucleation of cracks at grain-boundary particles are caused by impediment of GBS and also by diffusional process especially near specimen surface. Because the crack seemed to propagate from the surface to the center in thickness, it was concluded that one of the cracks, nucleated on the grain boundary near the surface, propagated to rupture.

KEYWORDS

Fatigue, High temperature, Bicrystal, Copper, Dispersoids, Grain boundary, Grain-boundary sliding

INTRODUCTION

In spite of extensive use of dispersion-hardened alloys as high-temperature materials, studies on their mechanical behavior have been unfortunately limited to areas of creep and static strength. Most of studies on fatigue behavior of the dispersion-hardened alloys are carried out at ambient temperature. Recently, Miura et al. have investigated the temperature dependence of fatigue behavior of Cu-SiO_2 single crystals; easier formation of cell structures were observed with increasing temperature which contributes to fatigue hardening [1]. Furthermore, they have also reported from results using Cu-SiO_2 polycrystals that grain-boundary cracking (GBC) takes place more easily with increasing temperature and with decreasing stress amplitude [2]. The occurrence of GBC reduces the fatigue life at elevated temperatures. Therefore, the role of grain boundary in high-temperature fatigue behavior must be more significant. In fact, grain boundary seems to strongly affect on fatigue behavior even at room temperature [3].

It is reported from static tensile test of dispersion-hardened alloy at high temperatures [4,5] that grain-boundary sliding (GBS) causes stress concentration around particles to form preferential nucleation of cracks because the particles impede the GBS. Therefore, it can be expected that GBS must also affect on the high-temperature fatigue behavior. This is the motivation of the present study. For simplicity of experimental system, orientation controlled copper bicrystal with dispersed SiO_2 particles was employed as a sample.

EXPERIMENTAL

Bicrystal of a Cu-0.1mass% Si alloy having [011] 18 ° twist boundary was grown by the Bridgman method using seed crystals. The grain boundary was microscopically straight. This bicrystal was internally oxidized at 1273K for 24h with mixed powder of Cu : Cu_2O : Al_2O_3 = 1 : 1 : 2 (mass ratio) to obtain dispersed SiO_2 particles both in grains and on grain boundary. By this treatment, we obtained Cu-0.82vol.%SiO_2 bicrystal. After a degassing treatment at 1273 K for 24 h in a graphite mold in vacuum, bicrystal specimen of 12 mm gage length and 4 x 2 mm^2 cross-section with a boundary inclined at 45 degrees to loading axis were cut by electric discharge machining (Fig. 1). Its crystallographic orientation is shown in Fig. 2. The slip planes and lines in the lower crystal are 18° rotated compared to the upper crystal around [011] axis. Before fatigue tests, surfaces of the specimens were mirror-finished by careful mechanical and electritical polishing.

Fatigue tests were carried out under stress control at a stress amplitude of 40MPa in a vacuum of 10^{-3} Pa at 673 K on a servo-hydraulic machine. Cyclic loading was applied in tension at 20Hz at a load ratio of R=0.1. Some of tests were stopped after cyclic deformation to some numbers of cycles to observe surface and microstructural changes during the cyclic deformation. In that case, the samples were removed immediately from furnace after the deformation to avoid structural changes. The microstructure and fractography were observed by using an orientation image microscopy (SEM-OIM). Yield stress of the bicrystal at 673K was 27MPa, when deformed statically on an Instron-type testing machine at a strain rate of 4.2×10^{-4} s^{-1}.

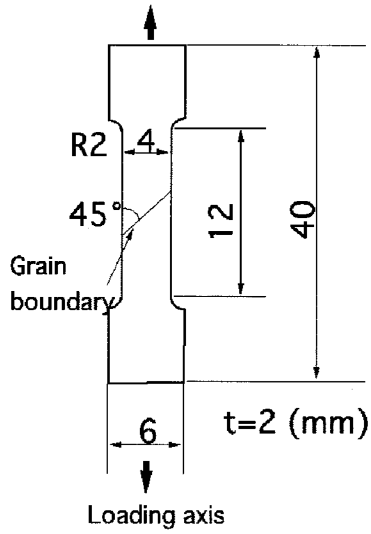

Figure 1 : Geometry of Cu-SiO_2 bicrystal fatigued.

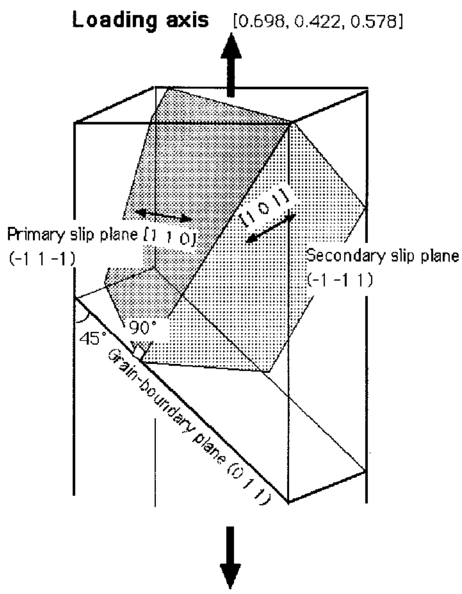

Figure 2 : Schematic illustration of crystallographical orientation of the bicrystal.

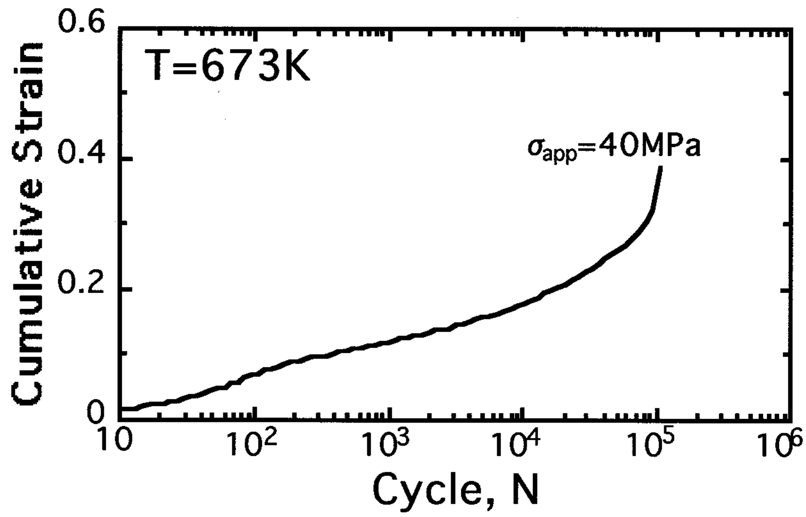

Figure 3 : Cumulative strain - number of cycle curve for Cu-SiO_2 bicrystal fatigued at 673K at a cyclic stress amplitude of 40MPa.

RESULTS AND DISCUSSION

Figure 3 shows cumulative strain against number of cycle (ε - N curve) for the Cu-SiO_2 bicrystal. The ε - N curve shows, after yielding, gradual increase in the cumulative strain at the steady-state work hardening stage and rapid increase to rupture. The ε - N curve of the Cu-SiO_2 bicrystal exhibits, therefore, typical tendency of cyclic creep.

Figure 4 shows microstructural changes with fatigue in processed SEM-OIM images scanned by 0.3μm step. The cycle, N = 10^5, is the cycle just before rupture. Image processing was carried

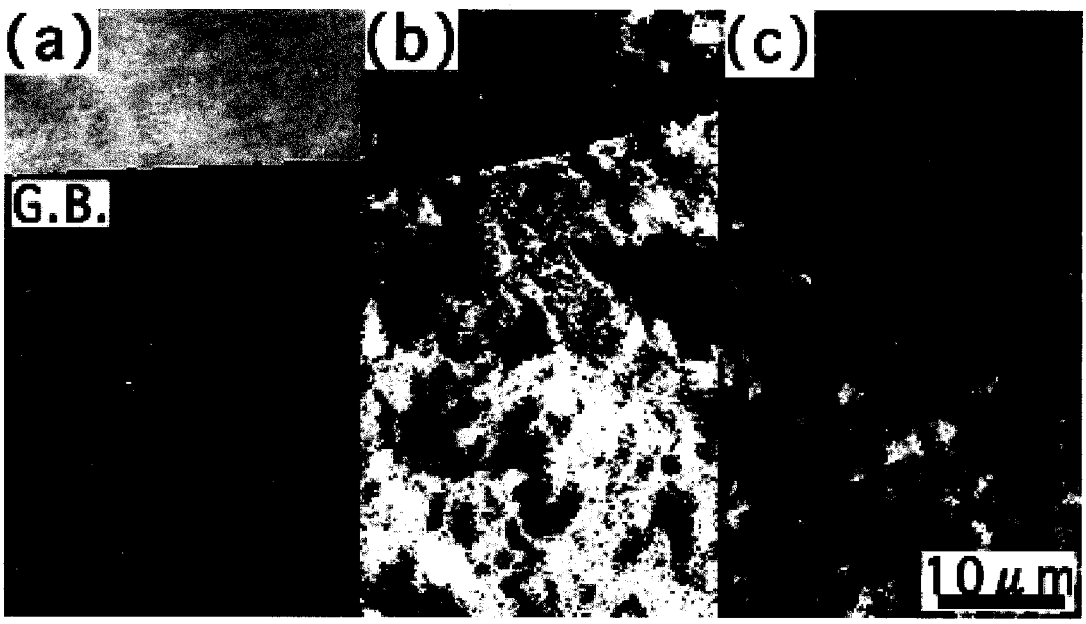

Figure 4 : Processed orientation image of the bicrystals fatigued to (a) N = 10^3, (b) N = 10^4 and (c) N = 10^5 cycles, respectively.

out to observe small change of substructures evolved in the bicrystals. The black lines seen upper sides of all the figures are the [011] 18° twist grain boundaries. Substructures develop gradually with increasing cycle. Such changes of the substructures were not detected in the original SEM-OIM images before the image processing. As can be seen in all the images, no specific structural development nor dynamic recrystallization occurred even near grain boundary while appearance of grain-boundary affected zone (GBAZ) was expected [3]. The hairlines of white contrast seen in Fig. 4 (c) are developed subboundaries. The absence of recrystallization in all the area of the samples even after deformation to a strain of $\varepsilon = 0.37$ at maximum (see Fig. 3) would be because of high-density distribution of the particles [6]. As the number of cycle increases, substructures developed more clearly and extensively. However, the substructure seems homogeneous and no GBAZ appeared to develop. This result suggests that macroscopic deformation concentration did not take place anywhere in the bicrystal under present experimental condition. This result may support the expectation that initial crack nucleates on grain boundary due to short range GBS but causes transgranular fracture, as will be shown later.

Surface morphology of the fatigued bicrystal to a number of cycle of $N = 10^3$ is shown in Fig. 5 (a). Only one slip traces perpendicular to the grain boundary seems to develop homogeneously. Slip traces of the primary and secondary slip systems are overlapped at the surface (see Fig. 2). Because the cumulative strain at this cycle is more than 0.1, both and further slip systems should work. However, change in slip morphology near the grain boundary, which corresponds to GBAZ, did not appear. What is more interesting, some small cracks are nucleated on the grain boundary. Miura et al. have reported from experimental results of static tensile tests of Cu-SiO_2 bicrystals that impediment of GBS by grain-boundary particles provides stress concentration site to promote preferential crack nucleation as schematically described in Fig. 5 (b) [4, 5]. We believe that the occurrence of GBS caused the crack nucleation also during high-temperature fatigue of dispersion-hardened alloy [7], because the [011] 18° twist boundary can slide easily at high temperature [8]. The mean particle radii in the grains and on the grain boundary measured by transmission electron microscopy were about 91 and 190 nm, respectively. The lager size of the grain-boundary particle would also attribute easier cracking around the particles due to larger stress concentration factor. The observed GBC on surface may be influenced also by diffusional process. However, rupture did not take place along the grain boundary. The crack propagated along the slip traces, as will be shown in Fig. 6. This would be due to homogeneous deformation

(b)

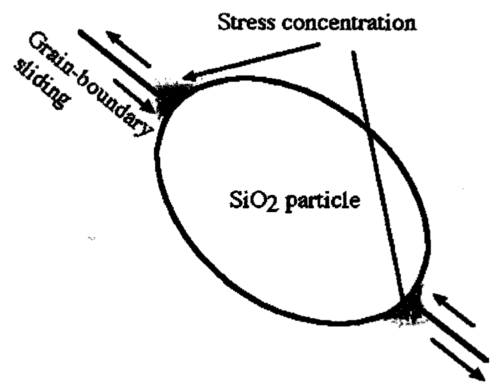

Figure 5 : (a) Surface morphology of the bicrystal fatigued to $N = 10^3$ cycles. (b) Schematic illustration of crack formation at the matrix/particle interface by stress concentration caused by grain-boundary sliding.

behavior during high-temperature fatigue of Cu-SiO_2 bicrystals. This result must not indicate that transgranular fracture occurs generally during cyclic deformation at elevated temperatures. Grain-boundary fracture would become dominant under such conditions of higher temperature and lower stress amplitude where GBS takes place more easily.

Observed typical features of fractographs are exhibited in Fig. 6. The macroscopic photograph of the fractured specimen (Fig. 6 (a)) shows that fracture occurred transgranularly. Grain boundary is pointed by arrow mark. The initiated crack on grain boundary did not propagated along the grain boundary. The SEM photograph in higher magnification shown in Fig. 6 (b) indicates that the fracture surface is composed of inhomogeneous morphologies; fine dimples around the center in thickness (Fig. 6 (c)) and relatively flat appearance of striation near the surface (Fig. 6 (d)). The existence of particles in the dimples implies that voids were formed at the particle/matrix interface. These fine dimples would be formed by ductile fracture manner just before rupture. The surface relief lines seen near the specimen's surfaces would be a striation (Fig. 6 (d)). The fractographs would imply that crack propagated from the surface to the center. It can be expected, therefore, that one of the grain-boundary cracks such as observed in Fig. 5 (a) is a starting point of crack propagation to cause rupture. This conclusion would be reasonable to think, because the crack nucleation and growth at the surface should be more acceleralated by easier and faster supply of vacancy or gas atoms, when compared with part far from the surface. If fatigue test at higher temperature is carried out, it should be expected that crack propagation along grain boundary becomes easier because of much easier occurrence of GBS and vacancy supply.

Figure 6 : Fractographs of the bicrystal; (a) whole view of the fracture surface, (b) further magnified image of a part of (a), (c) magnified image near the center of (b) and (d) magnified imageof lower side of (b), respectively.

SUMMARY

Fatigue behaviour of a copper alloy bicrystal containing SiO_2 particles and having a [011] 18° twist boundary has been studied at 673K at a cyclic stress amplitude of 40MPa. Cracks were found to nucleate preferentially at grain boundary, probably around the grain-boundary particles. This is because of easy stress concentration by impediment of grain-boundary sliding by the particles. The fracture seemed to take place by crack propagation from surface to the center in thickness. Although preferential cracking took place initially on grain boundary, grain-boundary fracture did not occur under the present fatigue condition. This would suggest that homogeneous cyclic deformation took place in the bicrystal, and then, slip trace seemed to be comparably important route for crack propagation even at elevated temperature.

ACKNOWLEDGEMENTS

The authors acknowledge the financial support by Tanikawa Found Promotion of Thermal Technology, Japan and the material supplement from Nippon Mining and Metals Co. Ltd., Kurami Works, Japan.

REFERENCES

1. Miura, H., Kotani, K. and Sakai, T. (2001) Acta materialia, in press.
2. Miura, H. and Sakai, T. (2001) Proceedings of 9th IC on Creep and Fracture of Materials and Structures, Swansea, United Kingdom, 29.
3. Wang, Z.G., Li, W.X., Zhang, F.Z. and Li, X.S. (1999) Proceedings of Fatigue '99, China, 93.
4. Miura, H., Sakai, T., Tada, N., Kato, M. and Mori, T. (1993) Acta Metall. Mater., 41, 1207.
5. Miura, H., Sakai, T., Tada, N. and Kato, M. (1996) Phil. Mag. A, 73, 871.
6. Beden, I. (1986) Scripta Metall., 20, 1.
7. Miura, H., Ooi, T. and Sakai, T. (2001) Phil. Mag. Lett., 81, 293.
8. Monzen, R., Futakuchi, M. and Suzuki, T. (1995) Scripta Metall. Mater., 8, 1277.