Cyclic deformation behavior of copper single crystals with dispersed SiO₂ particles

H. Miura*, T. Sakai, H. Mizukiri, M. Kato

*Department of Mechanical Engineering and Intelligent Systems, The University of Electro-Communications, Chofu, Tokyo 182-8585, Japan
bDepartment of Innovative and Engineered Materials, Tokyo Institute of Technology, 4259 Nagatsuta, Yokohama 226-8502, Japan

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Abstract

Cyclic deformation behavior of Cu single crystals with dispersed SiO₂ particles were investigated. Even for a small amount of 0.41 vol.% dispersion of SiO₂ particles, the cyclic deformation behavior was seen to be clearly different from that of pure Cu. There was longer life and slower crack propagation by the dispersion. This would be due to the homogeneous formation of the dislocation substructure in the Cu-SiO₂ sample to prohibit the stress concentration at a point that fastens the crack nucleation.

Keywords: Fatigue; Single crystal; Dispersed particles; Cu-SiO₂ alloy

1. Introduction

It is well known that the dispersion of fine particles into the metal matrix improves the strength because of lowering the mobility of dislocations by the Orowan mechanism. Numerous studies have been performed on the dispersion-hardened alloys to reveal their mechanical properties. Among them, cyclic deformation behavior of this class of alloys has also been of interest [1–3]. The deformable particles are often cut and destroyed during cyclic deformation to cause cyclic softening and early crack initiation [1,3]. However, as far as the authors know, there are few studies using alloys containing undeformable particles. Researches on such alloys are mainly carried out in the area of high-temperature deformation.

In the present study, the effects of small non-deformable particles in a copper matrix on the cyclic deformation behavior of a Cu-SiO₂ alloy were studied. Since grain boundaries strongly influence the cyclic deformation behavior [4,5], single crystals were employed as specimens.

2. Experimental

Single crystals of a Cu-0.05 mass% Si alloy were grown by the Bridgman method using a seed crystal. They were internally oxidized by the powder pack method at 1273 K
for 24 h. By this treatment, we obtained Cu-0.41 vol.% SiO₂ alloy single crystals containing amorphous SiO₂ oxide particles. The mean radius of the particles was 57 nm. After a degassing treatment at 1273 K for 24 h in a graphite mold in vacuum, specimens of 12 mm gage length and 4 × 2 mm² cross section were cut by electric discharge machining. For comparison, pure copper single crystal specimens were also prepared. To simulate the multiple slip situation for polycrystals, the symmetric crystallographic orientation of [001] was chosen as the loading axis (Fig. 1). Before the fatigue tests, the surfaces of the specimens were mirror-finished by careful mechanical and electrical polishing. Fatigue tests were carried out under stress control in air at room temperature in a servo-hydraulic machine. Cyclic loading was applied in tension at 20 Hz at the load ratio of $R = 0.1$. After the tests, the microstructure and fractography were examined by using an optical microscope, scanning electron microscope (SEM) and transmission electron microscope (TEM).

3. Results and discussion

Fig. 2 shows the cumulative strain against the number of cycle ($\varepsilon - N$ curves) for: (a) Cu-SiO₂ single crystals and (b) Cu single crystals. All the $\varepsilon - N$ curves resemble each other, showing gradual increase in the cumulative strain at the steady-state work hardening stage and rapid increase to rupture after the steady-state stage. The duration of the steady state depends strongly on the applied stress and on the crystals.

The steady-state slope shown as a dotted line for one
sample in Fig. 2(a), was summarized against the stress amplitude in Fig. 3. Though the steady-state region becomes shorter and less clear with increasing stress amplitude, the slope of the linear part was measured in that case. The difference in the slopes between Cu-SiO₂ and Cu single crystals becomes larger with increasing stress amplitude, though they become almost the same at lower stress amplitudes smaller than about 45 MPa. This is due to the strengthening effect of the dispersion of SiO₂ particles. That is, at lower amplitude regime, only microscopic yielding occurs for both crystals. However, at a higher stress amplitude regime, macroscopic yielding associated with Orowan-loop formation took place only for Cu-SiO₂ samples.

Fig. 4 shows the stress amplitude–life (S–N) plots. The life of the Cu-SiO₂ specimen is, at least, several times longer than that of Cu. The ratio of the yield stress ($\sigma_{y, \text{Cu-SiO}_2}/\sigma_{y, \text{Cu}}$) and that of peak stress ($\sigma_{p, \text{Cu-SiO}_2}/\sigma_{p, \text{Cu}}$) are only 1.5 and 1.2, respectively, when deformed monotonically at a strain rate of $4.2 \times 10^{-3} \text{s}^{-1}$ at room temperature. Therefore, the fatigue ratio looks almost the same. The longer life in the Cu-SiO₂ single crystal than in the Cu one may be mainly due to the increase of the tensile strength.

The propagation of a crack previously notched (1 mm in length and 0.36 mm in width) by a spark cutter was observed using an optical microscope. The crack propagation rate ($da/dN$) in Cu-SiO₂ seems to be a few orders of magnitude lower than that in Cu.
magnitude lower than that in Cu (Fig. 5). Therefore, the strengthening effect of the dispersion of SiO\textsubscript{2} particles on the life and retardation of the crack propagation is obvious. The observed abrupt drop of the crack propagation rate in the Cu-SiO\textsubscript{2} sample would be due to the higher work-hardening rate by the generated Orowan loops around the particles and the uniform dislocation substructures.

Fig. 6 shows the development of slip bands on the surfaces of Cu-SiO\textsubscript{2} and Cu samples. In both samples, the slip bands homogeneously develop with increasing cycles. However, slip bands in the Cu-SiO\textsubscript{2} samples are coarser and more inhomogenously distributed in the later stages of cycling.

Dislocation substructures of fatigued Cu-SiO\textsubscript{2} samples were observed using TEM. It is clear from Fig. 7 that dislocation walls develop to form fine cell structures with an increasing number of cycles. The resulting fine cell structure is indicative of the strengthening of the metal matrix [6]. The cell size appears to be controlled by the dispersed particles: The particles seem to be preferential node sites of the cell structures. This may restrict the dislocation and dislocation wall motion, and therefore, macroscopically homogeneous dislocation distribution was achieved. We believe that the homogeneous and persistent cell structure prohibits the stress concentration at a point in the sample to fasten the crack nucleation and the propagation. The observed homogeneous substructure is in contrast to the inhomogenous surface slip lines. Further study should be necessary to clarify the correlation of the homogeneous dislocation substructure and the inhomogeneous slip lines on the surface in the dispersion-hardened alloy.

Fig. 8 shows the fractographs. The fracture surface of the Cu-SiO\textsubscript{2} sample is composed of two different features, i.e.

![Fractographs](image-url)
typical fatigue striations and dimples. The former was formed during cyclic deformation and the latter, which implies the occurrence of ductile fracture, must have been formed in the final stage just before the rupture. On the other hand, the Cu samples fractured in a very ductile manner associated with significant reduction in the cross-sectional area. Therefore their fracture surface (Fig. 8(b)) looks like a ductile fracture surface in monotonic deformation in spite of the existence of less-clear fatigue striations.

From the above results it is concluded that the dispersion of the undeformable particles seems to enhance their fatigue resistance.

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