Cyclic Saturation Dislocation Pattern Observation in the Vicinity of Grain Boundaries by Electron Channeling Contrast Technique

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The cyclic saturation dislocation patterns within grains and in the vicinity of low-angle grain boundaries in fatigued copper crystal were successfully observed by electron channeling contrast technique in SEM. The results show that the dislocation patterns within grains consisted of typical two-phase structure, *i.e.* persistent slip bands (PSB) and veins. With increasing plastic strain amplitude ($\gamma_{pl} \geq 1.7 \times 10^{-3}$), large amount of PSBs and regular dislocation walls were observed. The dislocation walls and PSBs could cross through the low-angle grain boundaries continuously except that the dislocation-free zone (DFZs) appeared at some local regions. Combining with the cyclic stress-strain response and dislocation patterns, the effect of low-angle grain boundaries on cyclic deformation behavior was discussed.

1. Introduction

In recent decades, cyclic deformation behaviors of copper monocrystal and polycrystal have been investigated extensively[1~6]. At present, there still exist controversial results about the plateau of cyclic stress-strain (CSS) curve in copper polycrystal[3~6]. Therefore, it is very necessary to study the effect of grain boundaries (including low-angle and large-angle grain boundaries) on cyclic deformation. Recently, Hu and his colleagues[7,8] found that the CSS curves of some copper bicrystals also exhibit plateau regions at some plastic strain range and the saturation resolved shear stresses are somewhat higher than those of single crystals due to the effect of large-angle grain boundaries. However, up to now, the cyclic deformation behavior of the crystal containing low-angle grain boundaries has not been studied yet. In addition, the electron channeling contrast (ECC) technique in scanning electron microscope (SEM) has been successfully applied to observe the dislocation patterns of deformed metals[9~11]. As compared to the TEM technique, the SEM-ECC technique has many attractive features, especially, it can obtain the information of dislocation arrangement over a large area and at some special sites, e.g. in the vicinity of grain boundary, crack or deformation band. In this paper, we will carry out cyclic deformation on the copper crystal containing some low-angle grain boundaries basically parallel to the stress axis. By ECC-technique, the dislocation patterns in the vicinity of low-angle grain boundaries and within grains can be observed. Combining with the cyclic stress- strain response and dislocation patterns, the effect of low-angle grain boundaries on the dislocation patterns and cyclic stress-strain response can be revealed.

2. Experimental

In the present study, the copper crystal with some low-angle grain boundaries was grown from OHFC copper of 99.999% purity by Bridgman method in a horizontal furnace. Four fatigue specimens (T1-T4) with some low-angle grain boundaries basically parallel to the stress axis were spark-machined from the as-grown crystal, and their total length and gauge size were 70 mm and 16×6 × 6 mm^3 respectively. By the Laue back reflection technique, the orientation of the specimen was determined as $[\bar{3}\ 7\ 11]$, and the misorientation between adjacent grains was in the range of about 5°. Push-pull fatigue tests were performed on a Schenck servo-hydraulic testing machine under constant plastic strain control at room temperature in air. A triangle wave with the frequency range of 0.05~0.3 Hz was used. Cyclic tension, compression loads and hysteresis loops were recorded. The cyclically saturated specimens were polished mechanically and electrochemically to observe the dislocation patterns within grains and in the vicinity of low-angle grain boundaries by SEM-ECC technique. The operation conditions are listed in Table 1.

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Table 1 Operation conditions of dislocation pattern observation by SEM-ECC technique

Acceleration voltage	working distance	probe	brightness	contrast	scanning rate
20 kV	15~22 mm	2~5 nA	80~98%	30~33%	TV/2K

Table 2 Deformation parameters of the copper crystal containing low-angle grain boundaries

	T1	T2	T3	T4
$\varepsilon_{pl}/(10^{-3})^{1)}$	0.30	0.80	1.60	2.26
$\gamma_{pl}/(10^{-3})^{2)}$	0.63	1.67	3.34	4.68
$\sigma_{as}/MPa^{3)}$	62.1	62.5	63.0	62.7
$\iota_{as}/MPa^{4)}$	29.8	30.0	30.2	30.1

1) ε_{pl}: axial plastic strain amplitude;
2) γ_{pl}: resolved shear strain amplitude;
3) σ_{as}: axial saturation stress;
4) ι_{as}: saturation resolved shear stress.

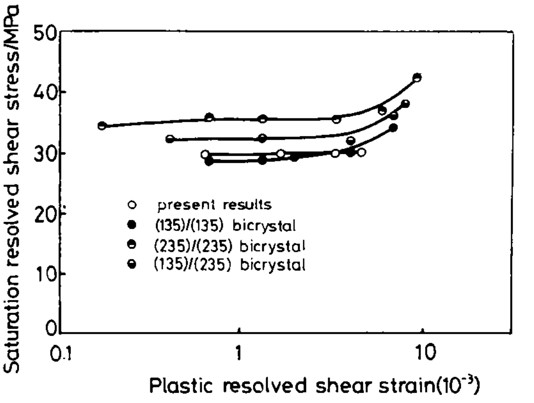

Fig.1 Cyclic stress-strain curves of the copper bicrystals[7,8] and the present crystal containing some low-angle grain boundaries

3. Results and Discussion

3.1 Cyclic stress-strain response

The results show that all the four copper crystal specimens exhibit initial cyclic hardening and saturation behavior. The cyclic saturation resolved shear stress amplitude reaches a value of about 30 MPa (see Table 2) which is similar to that of the single-slip-oriented copper monocrystal[1,2]. There also exists a plateau region in CSS curve at the plastic resolved shear strain range from 0.63×10^{-3} to 4.68×10^{-3}, as shown in Fig.1. Thus, it is indicated that the low-angle grain boundaries basically parallel to the stress axis show little effect on cyclic deformation. The result is somewhat similar to that in co-axial [$\bar{1}$35]//[$\bar{1}$35] copper bicrystal[7]. However, as reported in [$\bar{1}$35]//[$\bar{2}$35] and [$\bar{2}$35]//[$\bar{2}$35] copper bicrystals[8], their cyclic saturation stresses were higher than those of single crystals due to the effect of large-angle grain boundary.

3.2 Surface dislocation pattern observation by SEM-ECC technique

Cyclic saturation dislocation patterns in the surface of specimen became visible at a magnification of about 2000. At low strain amplitude ($\gamma_{pl} = 0.63\times10^{-3}$), the saturation dislocation patterns consisted of typical two-phase structure *i.e.* persistent slip bands (PSBs) and veins, as indicated in Fig.2(a). With increasing plastic resolved shear strain amplitude ($\gamma_{pl} \geq 1.67\times10^{-3}$), the volume fraction of PSBs increased and regular dislocation wall structures with the width of 1.5~1.7 μm can be observed throughout the whole specimen [see Fig.2(b) and (c)], which is consistent with the observation in copper monocrystal[2].

Moreover, the cyclic saturation dislocation patterns in the vicinity of low-angle grain boundaries were observed. It was found that most of dislocation walls and PSBs can transfer through the low-angle grain boundaries continuously, as shown in Fig.3(a)~(c). The result indicates that the stress and strain are compatible in the vicinity of the low-angle grain boundaries. Besides, dislocation-free zones (DFZs) were found beside low-angle grain boundary only at some local regions [see Fig.3(d)]. The width of the dislocation-free zone is about 1~2 μm. In fatigued copper polycrystals, the dislocation-free zone (DFZ) was first reported by Winter et *al.*[12]. It was found that high-angle grain boundaries were frequently associated with the zones of about 1~2.1 μm wide. They suggest that the most likely function of DFZs is to assist in the accommodation of incompatible strain in the adjacent grain during cyclic deformation. Recently, Luoh *et al.*[13] found that the formation of DFZs beside large-angle grain boundary in fatigued copper polycrystals is a very common phenomenon, however, the formation of a DFZ beside a twin boundary is very difficult[14]. In the present study, the DFZ as long as about 100 μm was observed along the low-angle grain boundaries only at some local regions.

It is generally recognized that cyclic saturation resolved shear stress ι_{as} of copper monocrystal is almost independent of plastic strain at some plastic strain range ($\gamma_{pl} = 6.0\times10^{-5} \sim 7.5\times10^{-3}$) and represents the stress required for the deformation in the PSBs[1]. In polycrystals, grain boundaries are often considered as an important factor to strengthen metallic materials and the stress-strain incompatibility at grain boundaries has been discussed extensively[15]. Thus, the low-angle grain boundaries might become an additional obstacle to the persistent slip bands (PSBs). However, in the present result, the low-angle grain boundaries seem to have little effect on the cyclic

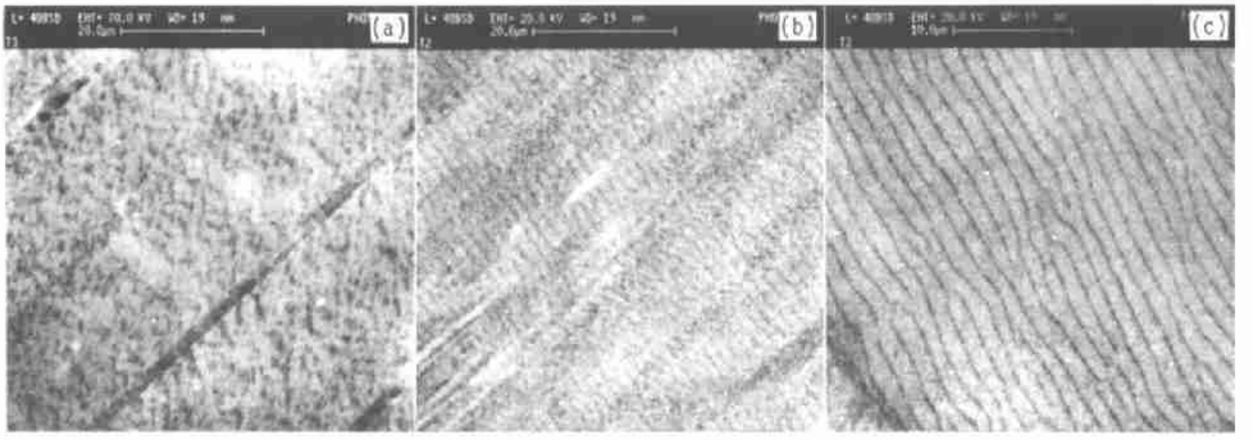

Fig.2 Cyclic saturation dislocation patterns within grains observed by SEM-ECC technique (a) persistent slip band and vein structures at low strain amplitude, (b) amounts of persistent slip bands and dislocation walls, (c) regular dislocation walls at high strain amplitude

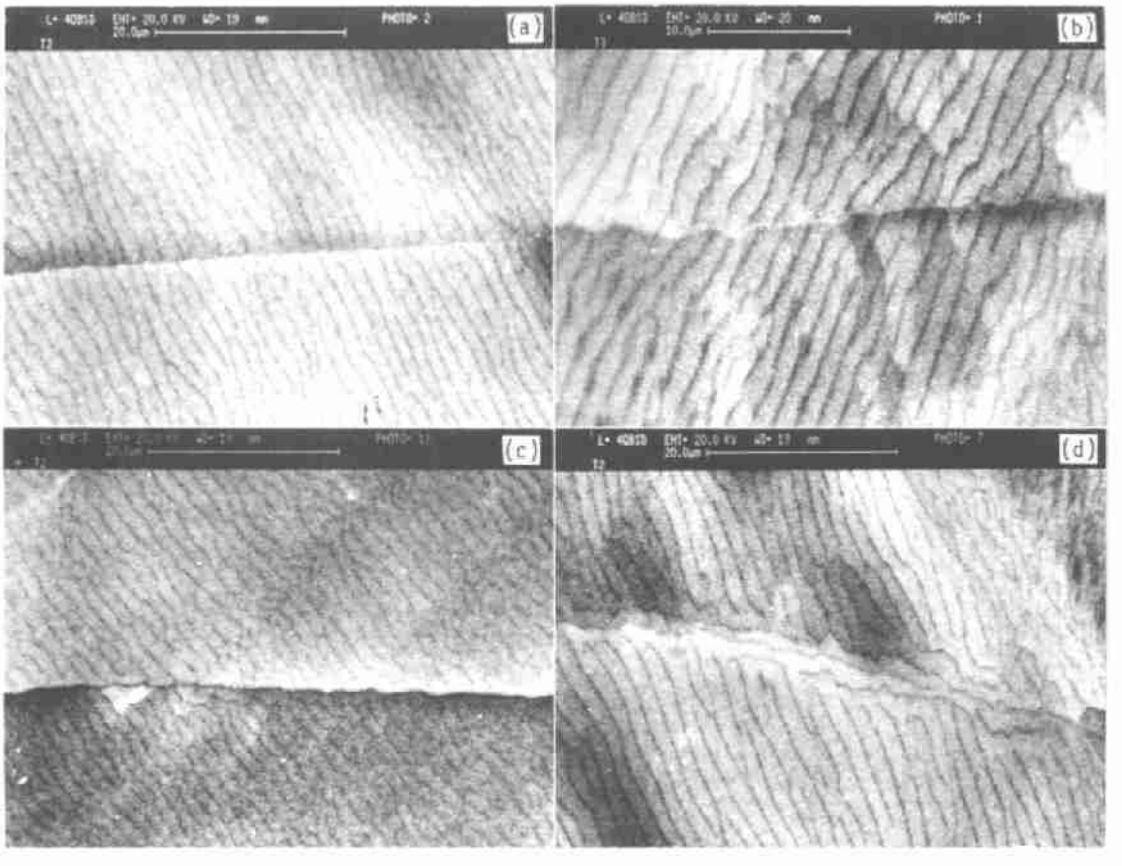

Fig.3 Cyclic saturation dislocation patterns in the vicinity of low-angle grain boundary observed by SEM-ECC technique (a) continuous dislocation walls structures, (b) continuous dislocation walls structures, (c) continuous persistent slip bands, (d) dislocation-free zone at some local regions

deformation in the copper crystal. Because the cyclic saturation resolved shear stress does not go beyond 27~30 MPa, and the cyclic saturation dislocation patterns almost continuously cross through the low-angle grain boundaries except that the DFZ was found at some local regions. In fact, the volume of the DFZ beside the low-angle grain boundaries is very little. The next reason is that the cyclic saturation dislocation patterns within grains also agree to the observation in copper monocrystal. That is to say, the resistance of these low-angle grain boundaries on PSBs may be negligible. It is reasonable to conclude that the stress and strain are nearly compatible at low-angle grain boundaries and no obvious strengthening effect exists on the copper crystal under cyclic loading. Similarly, for the copper bicrystals[7,8], the plateau saturation resolved shear stress is somewhat higher than that of single crystals, which should be associated with the effect of large-angle grain boundaries. Essentially, the cyclic stress-strain curves (CSSCs) of polycrystals are associated with the effect of grain boundaries on PSBs. The cyclic saturation stress of polycrystals increases with increasing plastic strain amplitude, which indicates that the grain boundary strengthening plays an important role. From the results of the present copper crystal and the copper bicrystals[7,8], it is suggested that there will still exist a plateau region in their CSS curves of the copper bicrystals and the crystal with low-angle grain boundaries basically parallel to the stress axis despite the different degree effect of the two kinds of grain boundaries.

4. Conclusions

(1) Copper crystal containing low-angle grain boundaries shows a cyclic saturation resolved shear stress of about 30 MPa at the plastic resolved shear strain range from 0.63×10^{-3} to 4.68×10^{-3}.

(2) The electron channeling contrast (ECC) technique has been successfully applied to observe the cyclic saturation dislocation patterns within grains and in the vicinity of low-angle grain boundaries of the copper crystal. By comparing the cyclic stress-strain response and dislocation patterns, it can be concluded that the strengthening effect of low-angle grain boundaries on the copper crystal under cyclic deformation is very limited.

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