SEM Electron Channeling Contrast Imaging of Dislocation Structures in Fatigued [017] Cu Single Crystals Oriented for Critical Double Slip

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Dislocation structures in fatigued [017] critical double-slip-oriented Cu single crystals were studied using the electron channeling contrast technique in scanning electron microscopy. It was found that the dislocation structures are strongly dependent upon the applied plastic strain amplitude $\gamma_{pl}$. As $\gamma_{pl}$ falls into the quasi-plateau region in the cyclic stress-strain curve of the [017] crystal, a special “two-phase” dislocation structure, i.e., persistent slip band ladder-like structures and matrix labyrinth structures, was observed. When $\gamma_{pl} \geq 3.0 \times 10^{-5}$, which is beyond the quasi-plateau region, two types of deformation bands denoted DBI and DBII were observed. The dislocation microstructures in DBI and DBII consist mainly of well-developed regular labyrinth structures and densely-aligned dislocation walls, respectively, and the microstructures at the intersection region of DBI and DBII comprise dislocation walls together with a number of dislocation cells. The orientation dependence of microstructures in DBI and DBII is further summarized and discussed.

(Received December 28, 2009; Accepted February 5, 2010; Published April 25, 2010)

Keywords: copper single crystal, dislocation structure, scanning electron microscopy (SEM), electron channeling contrast (ECC), cyclic deformation, double slip, deformation band

1. Introduction

Over the past four decades, significant contributions have been made on the cyclic deformation behavior and dislocation microstructures of Cu single crystals,¹⁻⁴ and the cyclic stress-strain (CSS) curves of many typical single-, double- and multiple-slip-oriented Cu single crystals have been well established.⁵ It has been recognized that those crystals with double-slip orientations located on different sides of the standard stereographic triangle exhibit an obvious orientation dependence of fatigue dislocation structures, which can well interpret the corresponding orientation dependence of their CSS behaviors.³⁻⁴ For example, concerning the critical double-slip orientations located on the [001]-[011] side of the standard triangle, the persistent slip band (PSB) ladder or ladder-like structure is a common dislocation feature, but such a structure changes generally to the labyrinth structure as the orientation moves from the [011]-orientation to the [001]-orientation along the [001]-[011] side of the standard triangle, causing that the corresponding CSS behavior varies from more like that of single-slip-oriented crystals to polycrystals.⁶ Even then, there is still lack of systematic knowledge on the dislocation microstructures in cyclically deformed Cu single crystals with double-slip orientations, especially on the dislocation structures directly linked to some special sites like deformation bands (DBs). In the present work, the dislocation structures in a fatigued [017] critical double-slip-oriented Cu single crystal are investigated using the electron channeling contrast (ECC) technique in scanning electron microscopy (SEM),⁶ focusing on the effect of the applied plastic strain amplitude $\gamma_{pl}$ on the dislocation structures as well as the observation of dislocation microstructures in fatigue-induced DBs.

2. Experimental Procedures

[017]-oriented single crystals were grown from OFHC copper of 99.999% purity by the Bridgman method. The dimensions of the fatigue specimen are $7 \times 7 \times 70$ mm³, with a gauge section of $7 \times 5 \times 16$ mm³. The Laue back-reflection technique was used to determine the orientation of the specimen. Before the fatigue test, all specimens were electro-polished to produce a strain-free and mirror-like surface for microscopic observations. Push-pull cyclic deformation tests were performed at room temperature in air using a Shimadzu servo-hydraulic testing machine. A triangular waveform signal with a frequency range of 0.05–0.4 Hz was used for the constant plastic strain control. Cyclic stress-strain hysteresis loops were registered with an X-Y plotter, and the loop width $(\Delta \epsilon_p)$ at zero stress was kept constant during testing. The plastic resolved shear strain amplitude $\gamma_{pl}$ and shear stress $\tau$ were calculated by $\gamma_{pl} = \Delta \epsilon_p / 2 \Omega$ and $\tau = \sigma \Omega$, where $\Omega$ is the Schmid factor of primary slip system and $\sigma$ is an average value of the peak stresses in push and pull. The crystal specimens were deformed cyclically up to the occurrence of saturation. After the fatigue test, one of the deformed surfaces, e.g., (271) or (271), was polished mechanically and then electrolytically for the observation of dislocation structures by the SEM-ECC technique. The ECC technique was employed in a Cambridge S360 SEM using an inverted imaging mode. Dislocation-poor regions appear as bright areas in the micrograph, whereas dislocation-dense regions are seen as dark ones; this is in accord with a transmission electron microscopy (TEM) micrograph under bright field imaging conditions. The parameters of SEM working conditions were carefully set at those listed in Table 1.

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3. Results and Discussion

3.1 Cyclic stress-strain curve

The cyclic deformation test data of [017] Cu single crystals have been reported in Ref. 7). Here, the CSS curve of the [017] crystal is reproduced in Fig. 1, where the results obtained with single-slip Cu crystals are also included for comparison. It is well known that Cu single crystals oriented for single slip exhibit a quite wide stress plateau region within the range of $6.0 \times 10^{-2}$–$7.5 \times 10^{-3}$, which is attributed to the formation and continuous increase of PSB structures. In contrast, the CSS curve of the [017] double-slip crystal does not show a clear plateau; however, its saturation stress does not increase markedly with increasing plastic strain amplitude ($\gamma_{pl}$). Apparently, the stress level (35–39 MPa) of the quasi-plateau is comparatively higher than the plateau stress level of single-slip crystals.

3.2 Dislocation structures

The $\gamma_{pl}$-dependent dislocation structures of cyclically deformed [017] crystals were examined by the SEM-ECC technique at four different plastic strain amplitudes, which correspond to the distinctive regions of the CSS curve, as seen in the circled data points in Fig. 1.

At a lower $\gamma_{pl}$ of $1.2 \times 10^{-4}$, which is below the quasi-plateau region in the CSS curve, a basically observed feature of the dislocation structure is irregular labyrinths, as shown in Fig. 2, which is reproduced from Ref. 4). At a higher $\gamma_{pl}$ over the range of $3.5 \times 10^{-4}$–$1.5 \times 10^{-3}$, which can be considered approximately as a quasi-plateau. However, its saturation stress does not increase markedly with increasing $\gamma_{pl}$: $5.0 \times 10^{-4}$–$1.5 \times 10^{-3}$, which can be considered approximately as a quasi-plateau. In another hand, with increasing $\gamma_{pl}$, the decrease in the scale of the labyrinth structure would lead to a gradual increase in the saturation stress. As a result, the occurrence of a quasi-plateau region in the CSS curve of [017] crystals should be due to the joint effects of the formation of PSB ladder-like structures and the scale change of labyrinth structures.

As $\gamma_{pl}$ is further increased to a high value of $3.0 \times 10^{-3}$, which is beyond the quasi-plateau region in the CSS curve, the [017] crystal exhibits complex deformation characteristics and dislocation structures, and PSB ladder-like structures disappear completely, as shown in Fig. 4. Two types of DBs denoted DBI and DBII can be clearly seen on the crystal surface (Fig. 4(a)). DBII develops roughly along the primary PSB, and DBII makes a certain angle with the primary PSBs. The dislocation microstructures in DBI are composed of well-developed regular labyrinth structures, as seen in Fig. 4(b), and the width of labyrinth channels has an identical relationship with the scale change of labyrinth structures.

### Table 1 Parameters of the SEM working conditions.

<table>
<thead>
<tr>
<th>Acceleration voltage</th>
<th>Working distance</th>
<th>Filament current</th>
<th>Probe current</th>
<th>Brightness</th>
<th>Contrast</th>
<th>Scanning rate</th>
</tr>
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<tbody>
<tr>
<td>20 kV</td>
<td>15–22 mm</td>
<td>$I = 2–3$ A</td>
<td>$I = 2–5$ nA</td>
<td>80–95%</td>
<td>30–33%</td>
<td>TV/2 k</td>
</tr>
</tbody>
</table>

Fig. 1 The cyclic stress-strain curve of the [017] Cu single crystal. Note that the results of single-slip-oriented crystals taken from Ref. 1) is also given by the dotted line in this figure for comparison, and that the dislocation structures of [017] crystal specimens cycled at some selected $\gamma_{pl}$, corresponding to the circled data points in this figure, were observed.

Fig. 2 Irregular labyrinth structures formed in the [017] crystal cyclically deformed at $\gamma_{pl} = 1.2 \times 10^{-4}$. Note that the direction of stress axis is horizontal in this figure, as is the case for the following Figs. 3, 4 and 5. As $\gamma_{pl}$ increases to $7.0 \times 10^{-4}$, which is exactly within the quasi-plateau region, the most areas of crystal surface are covered by regular labyrinth structures, as shown in Fig. 3(a), and the average width of channels is reduced to about 0.7 µm (measured from a magnified image of Fig. 3(b)). In addition, some narrow PSB ladder-like structures are embedded in the labyrinths, as shown in Figs. 3(c) and (d), constituting a distinctive “two-phase” structure, i.e., PSB ladder-like structures and matrix labyrinth structures, which is obviously different from the well-known “two-phase” structure, PSB ladders and matrix veins, obtained with single-slip crystals. It is believed that the formation of a narrow quasi-plateau region in the CSS curve of [017] crystals is primarily related to the formation of such PSB ladder-like structures, which can carry a considerable part of the cyclic plastic strain. The formation of this kind of PSB ladder-like structures was thought to be promoted by the involvement of a critical (secondary) slip. In another hand, with increasing $\gamma_{pl}$, the decrease in the scale of the labyrinth structure would lead to a gradual increase in the saturation stress. As a result, the occurrence of a quasi-plateau region in the CSS curve of [017] crystals should be due to the joint effects of the formation of PSB ladder-like structures and the scale change of labyrinth structures.

As $\gamma_{pl}$ is further increased to a high value of $3.0 \times 10^{-3}$, which is beyond the quasi-plateau region in the CSS curve, the [017] crystal exhibits complex deformation characteristics and dislocation structures, and PSB ladder-like structures disappear completely, as shown in Fig. 4. Two types of DBs denoted DBI and DBII can be clearly seen on the crystal surface (Fig. 4(a)). DBI develops roughly along the primary PSB, and DBII makes a certain angle with the primary PSBs. The dislocation microstructures in DBI are composed of well-developed regular labyrinth structures, as seen in Fig. 4(b), and the width of labyrinth channels has an identical relationship with the scale change of labyrinth structures.
value of $\gamma_{pl} = 7.0 \times 10^{-4}$ with the one at $\gamma_{pl} = 7.0 \times 10^{-4}$. However, the microstructures in DBII consist of densely-aligned dislocation walls with an average channel width of $\sim 0.5 \mu m$, as shown in Fig. 4(c), and these walls extend almost along a direction perpendicular to the loading axis [017]. Quite similar wall structures were also found in fatigued [111] multiple-slip-oriented Cu single crystals.\textsuperscript{10,11} At the intersection region of DBI and DBII, the boundary between the labyrinth structure of DBI and the wall structure of DBII is discernible, as marked by the dashed line in Fig. 4(d). The wall direction in DBII seems to be parallel to one of labyrinth walls in DBI, but the channel widths of these two kinds of walls are somewhat different, inevitably leading to microstructural discontinuity at the intersection of two kinds of DBs, and thereby to a serious local plastic deformation concentration. That is why slip bands are often observed to be disrupted at the intersection between DBI and DBII.\textsuperscript{12} When $\gamma_{pl}$ continues increasing to $6.5 \times 10^{-3}$, which is far beyond the quasi-plateau region in the CSS curve, the majority of crystal surface has been covered with deformation bands of DBI and DBII, as shown in Fig. 5(a). Observations in some selected areas demonstrate that the region of intersection between DBI and DBII presents a more distinctive image contrast as compared to the other regions (see Fig. 5(b)), implying a concentrated plastic deformation. From Fig. 5(c) it was further found that the dislocation structures at the intersection region of DBI and DBII consist mainly of dislocation walls, which are the characteristic structure solely in DBII (see Fig. 4(c)). That is to say, at such
intersection region, the characteristic wall structure in DBII is basically retained, while the labyrinth structure in DBI tends to vanish. According to a previous work, DBI and DBII develop roughly along the primary slip plane \{111\} and the conventional kink plane \{101\}, respectively, and the plastic deformation concentration in DBII is more serious than that in DBI. Therefore, it is readily understood that the dislocation wall structure in DBII can overshadow the labyrinth structure in DBI at their intersection regions. Meanwhile, the interaction of these two types of DBs also leads to the formation of a number of dislocation cells near the wall structures, as seen in Fig. 5(d).

Based on the experimental results above, it can be concluded that the microstructures in two types of DBs, namely DBI and DBII, in fatigued [017] Cu single crystals are quite different. Combined with some existing findings, it is interesting to find that the crystallographic orientation seems to have a strong effect on the microstructures in DBI, but little effect on those in DBII in fatigued fcc pure metal single crystals, as schematically shown in Fig. 6. For example, the microstructures in DBI can consist of parallel PSB ladders (e.g. [135] single-slip crystals, see Fig. 6(a)), or labyrinths (e.g. the present [017] critical double-slip crystals and [001] multiple-slip crystals, see Fig. 6(b)), or dislocation walls and cells (e.g. [233] coplanar double-slip crystals, see Figs. 6(c) and (d)). In contrast, the microstructures in DBII seem to be just composed of dislocation walls in differently oriented single crystals (see Fig. 6(e)), in spite of the slight difference in the morphology of those dislocation walls, such as in [5 12 20] single-slip crystals, the present [017] critical double-slip crystals, [223] conjugate double-slip crystals, [155] coplanar double-slip crystals, and even in [011] multiple-slip single crystals of Ni or Ag etc. As is well known, for differently oriented fcc metal single crystals, modes and intensities of dislocation reactions among slip systems operated in cyclically deformed

![Fig. 5 Deformation bands and their dislocation structures in the [017] crystal cyclically deformed at $\gamma_{\text{pl}} = 6.5 \times 10^{-3}$. (a) low-magnification image of DBs, (b) magnified image of the region of interaction between DBI and DBII, (c) dislocation walls formed at the interaction region of DBI and DBII, and (d) an enlarged image of the lower-left portion of (c) showing dislocation cells near the wall structures.](image)

![Fig. 6 Schematics of the microstructures in DBs (DBI and DBII) in fatigued Cu single crystals. (a) PSB ladder structure in DBI, cf. Ref. 13); (b) labyrinth structure in DBI, cf. Ref. 14) and the present work, (c) dislocation wall structure in DBI, cf. Ref. 15), (d) dislocation cell structure in DBII, cf. Ref. 15), and (e) dislocation wall structure in DBII, cf. Refs. 16–20) and the present work.](image)
crystals may be different, and different characteristic dislocation structures would thus be formed to accommodate cyclic plastic strains. The DBI-type deformation band develops along the primary slip plane (111) and it carries the majority of plastic strains during cycling, so that the microstructures in DBI is generally determined by the orientation. However, the DBII-type deformation band is similar to the kink band, and its habit plane is close to the conventional kink plane {101}, so that the plastic deformation concentration in DBII is extremely serious. As the imposed cumulative strain carried by DBII reaches a certain capacity during cyclic deformation, the microstructures in DBII may easily transform from PSB ladders or other structures into a more stable structure, i.e., dislocation walls, which can accommodate a serious plastic strain concentration in DBII. Therefore, the microstructures in DBII are nearly independent of the orientation, and always composed of dislocation walls. A more general understanding of microstructures in different types of DBs formed in fatigued fcc single crystals still needs further in-depth experimental work.

4. Summary

By using the SEM-ECC technique, dislocation structures in fatigue critical double-slip-oriented Cu single crystals were observed, and they are found to be closely dependent upon the composed plastic strain amplitude, such as irregular labyrinths being formed at below the quasi-plateau region in the CSS curve, and a distinctive “two-phase” structure, i.e. PSB ladder-like structure and matrix labyrinth structure being formed at within the quasi-plateau region. As is beyond the quasi-plateau region in the CSS curve, two types of DBs denoted DBI and DBII dominate the cyclic plastic deformation. The dislocation microstructures in DBI and DBII consist mainly of regular labyrinth structures and dislocation walls, respectively, and the microstructures at the intersection region of DBI and DBII are comprised of dislocation walls together with a number of dislocation cells. The microstructures linked to DBI and DBII formed in fatigued fcc pure metal single crystals seem to exhibit a different orientation dependence.

Acknowledgments

This work was financially supported by the National Natural Science Foundation of China (NSFC) under Grant No. 50771029, and by the Fundamental Research Funds for the Central Universities of China under Grant No. N090505001, as well as by the Program for New Century Excellent Talents (NCET-07-0162) in University, Ministry of Education, P. R. China. Prof. X. W. Li is grateful for these supports.

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