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Observations on persistent slip bands transferring through a grain boundary in a copper bicrystal by the electron channelling contrast in scanning electron microscopy technique

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ABSTRACT

To reveal the interaction of persistent slip bands (PSBs) with grain boundaries (GBs), a specially designed [134]/[18 2 7] copper bicrystal with a tilting GB and a common primary slip plane in its component crystals was prepared. The dislocation patterns within grains and near the GB in the copper bicrystal cyclically deformed at axial plastic strain amplitude of 0.9×10^{-3} were observed by the electron channelling contrast in scanning electron microscopy technique. The observation results show that the surface slip bands can transfer through the GB in the copper bicrystal continuously and do not induce any secondary slip near the GB. The ladder-like PSB structures can form in both [134] and [18 2 7] component crystals. On one surface, the ladder-like PSBs are still continuous beside the GB; however, the dislocation arrangements in those PSBs beside the GB become irregular and discontinuous on another surface. In addition, on the common primary slip (111) plane, a dislocation-affected zone with a width of 5–10 μm was observed on one side of the GB. Those observations reveal that the dislocation structures within the surface slip bands transferring through the GB in the copper bicrystal are not completely continuous during cyclic deformation even though the two grains have a common primary slip (111) plane.

§ 1. INTRODUCTION

The dislocation structures induced by cyclic deformation are generally observed by transmission electron microscopy (TEM). TEM investigations require thin-foil specimens and therefore the bulk specimens have to be destroyed. In addition, TEM requires tedious specimen preparation and permits only a relatively small specimen area to be investigated. Recently, the electron channelling contrast in scanning electron microscopy (SEM-ECC) technique has been applied to study the dislocation patterns in cyclically deformed metals such as nickel (Schwab *et al.* 1996, Bretschneider *et al.* 1997, Schwab *et al.* 1998), copper (Melisova *et al.* 1997, Gong *et al.* 1997) and stainless steel (Zauter *et al.* 1992). Compared with TEM, the SEM-ECC technique has many attractive features. This technique has been found to be extremely suitable for studying the dislocation arrangements over a large specimen area and at some special sites, for example near the GBs, within deformation bands (Melisova *et al.* 1997, Gong *et al.* 1997) and ahead of cracks. Consequently, it is

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more convenient to reveal the plastic strain localization in PSBs and the interactions of PSBs with GBs by the SEM-ECC technique. In our previous work, the cyclic deformation behaviour of copper bicrystals with perpendicular and parallel GB (Hu *et al.* 1996, Hu and Wang 1997, Zhang and Wang, 1998a,b) has been systematically investigated. It was observed that the PSBs cannot transfer through a large-angle GB and a grain-boundary-affected zone (GBAZ) formed owing to the plastic strain incompatibility (Hu *et al.* 1996, Zhang and Wang, 1998a). However, the PSBs and dislocation walls can pass through small-angle GB continuously and no GBAZ appeared (Zhang and Wang 1998b). In this work, a copper bicrystal with a tilting GB has been designed and prepared. In particular, the two component crystals are oriented for typical single slip and their primary slip planes are coplanar. The aim of the present work is to observe the interactions of PSBs with the GB during cyclic deformation.

§ 2. EXPERIMENTAL PROCEDURE

In the present study, a copper bicrystal with a large-angle GB was grown from oxygen-free high-conductivity copper of 99.999% purity by the Bridgman method in a horizontal furnace. A copper bicrystal specimen with a tilting large-angle GB was prepared from the as-grown bicrystal, as shown in figure 1 (*a*). By the Laue back-reflection technique, the orientations of the two component grains were determined as G1 $[\bar{1}34]$ and G2 $[18\,\bar{2}\,7]$ respectively. In particular, the primary slip planes of the

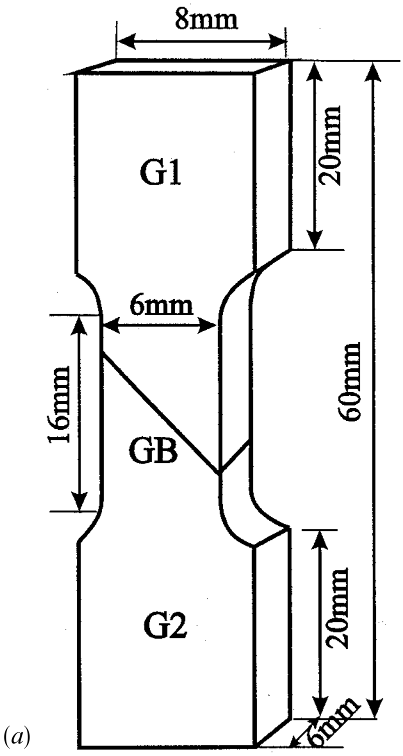
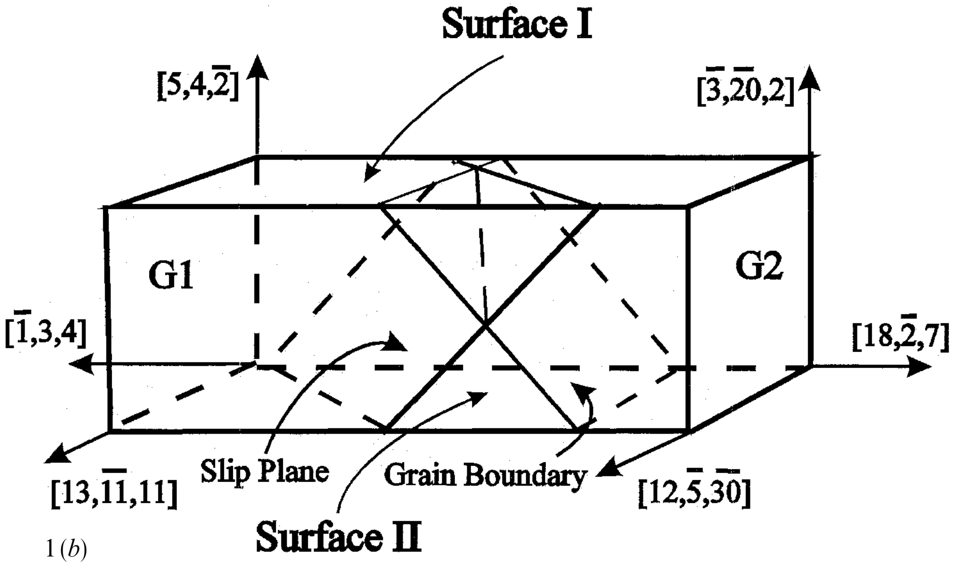


Figure 1. Fatigue specimen and the crystallographic relations of the copper bicrystal: (*a*) fatigue specimen; (*b*) crystallographic relations.



1 (*b*)

two crystals are coplanar, as shown in figure 1 (*b*). Before cyclic deformation, the specimen was carefully electropolished for surface observation. A symmetrical push–pull test was performed on a Shimadzu servohydraulic testing machine under a constant plastic strain amplitude of 0.9×10^{-3} at room temperature in air. A triangular wave with a frequency range 0.05–0.3 Hz was used. After cyclic saturation ($N = 10^4$ cycles), the surface slip morphology of the specimen was observed by scanning electron microscopy (SEM). Afterwards, the bicrystal specimen was polished to remove the surface slip traces and the dislocation patterns within grains and near the GB were observed by the SEM-ECC technique. The operating conditions can be found in a previous paper (Zhang and Wang 1998b).

§ 3. Results and discussion

3.1. *Surface slip morphology observations*

During cyclic deformation, the bicrystal exhibited rapid cyclic hardening and saturation behaviour with an axial saturation stress of 62.1 MPa. Surface observations by SEM showed the following features.

(1) Only the common primary slip system was activated in both grains G1 $[\bar{1}34]$ and grain G2 $[18\,2\,7]$.
(2) No secondary slip lines can be observed on the bicrystal surface including the vicinity of the GB.
(3) The common primary slip bands beside the GB were continuous, as shown in figure 2, which is similar to that observed near small-angle GBs (Zhang and Wang 1998b).

The slip bands transferring through a large-angle GB have never before been observed under cyclic loading. For the copper bicrystal without a common primary slip plane in two grains, all the slip bands were found to be terminated at the GB and unable to pass through the GB during cyclic deformation (Hu and Wang 1997, Zhang and Wang 1998a,b). The present result indicated that the stress and strain

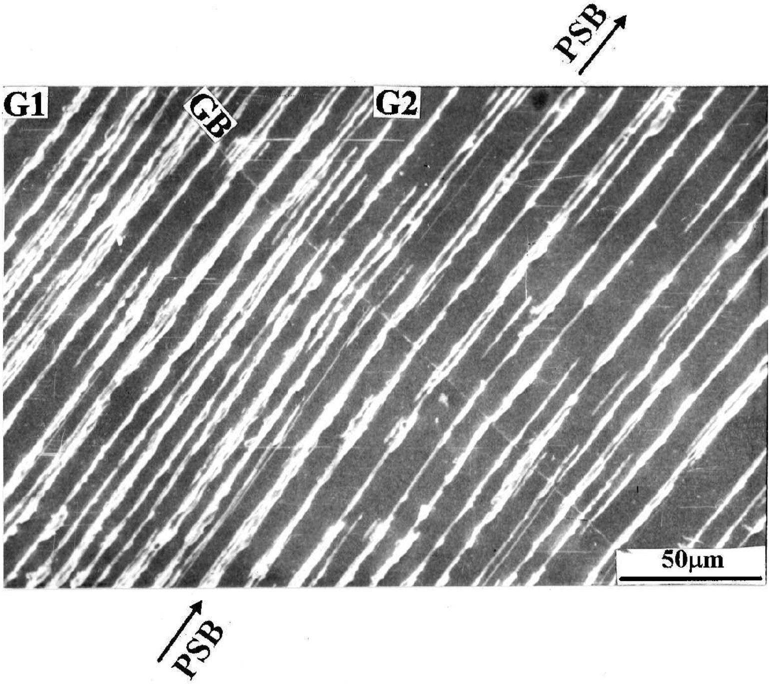


Figure 2. Primary slip bands passing through the GB on surface II in the bicrystal.

near the GB in the present bicrystal should be compatible during cyclic deformation. It is necessary to reveal the dislocation arrangements within grains and near the GB for better understanding of the effect of the GB. The SEM-ECC technique was adopted for this purpose.

3.2. *Dislocation patterns within grains and near the grain boundary*

The observations on the bicrystal surface by the SEM-ECC technique showed that the dislocation patterns in both G1 $[\bar{1}34]$ and G2 $[18\,\bar{2}\,7]$ grains consisted of typical two-phase structure of PSBs and dislocation veins, as shown in figure 3. These dislocation patterns are in good agreement with that observed in a copper single crystal (Winter 1974, Laird *et al.* 1986). By using the Schmid factors $(\Omega_{G1} = 0.47;$ $\Omega_{G2} = 0.49)$ of the grains $[\bar{1}34]$ and $[18\,\bar{2}\,7]$, the saturation resolved shear stress along the primary slip direction in the two grains can be calculated as follows:

$$\tau_{as}^{G1} = \sigma_{as}^{B}\Omega_{G1}, \tag{1}$$

$$\tau_{as}^{G2} = \sigma_{as}^{B}\Omega_{G2}, \tag{2}$$

where σ_{as}^{B} is the axial saturation stress of the bicrystal and equal to 62.1 MPa. τ_{as}^{G1} and τ_{as}^{G1} are the resolved shear stresses on the primary slip direction of the two grains, which were calculated to be 29.2 and 30.4 MPa respectively. It is clear that the surface slip bands in the two grains correspond to the ladder-like PSB structures and are in close agreement with its saturation stress of 62.1 MPa (Mughrabi 1978, Cheng and Laird 1981).

When the dislocation arrangements near the GB were observed at different surfaces of the specimen, two kinds of feature can be seen. On surface I in figure

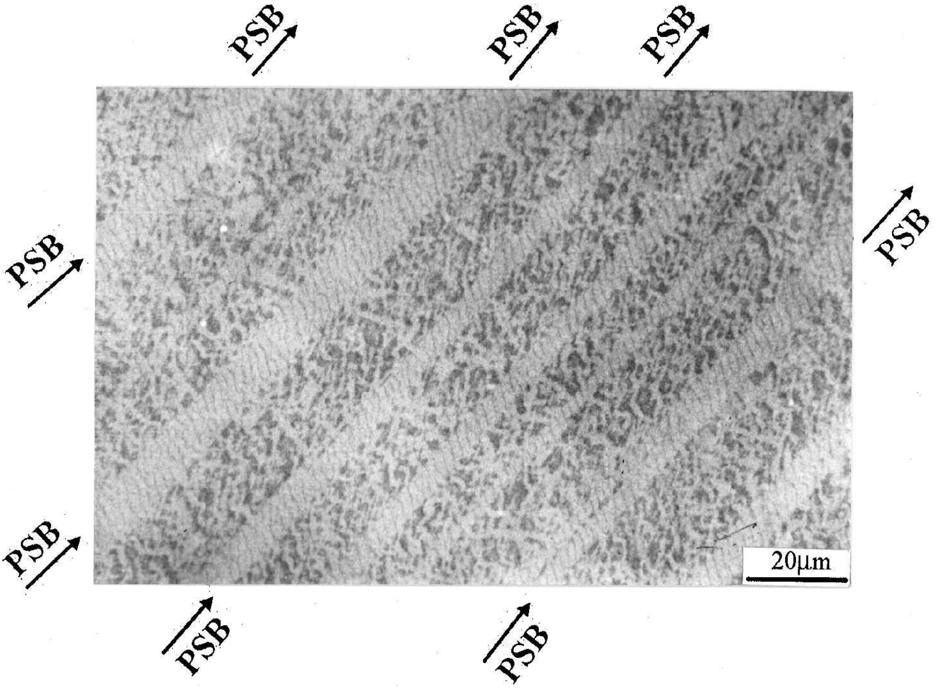


Figure 3. SEM-ECC image showing the two-phase structures of PSBs and dislocation veins in the $[18\,\bar{2}\,7]$ grain.

1 (*b*), the ladder-like PSB dislocation walls are also continuous and regular beside the GB, as shown in figure 4 (*a*). Those results indicate that the ladder-like PSBs can transfer through the large-angle GB continuously on this surface and are in good agreement with the surface slip bands near the GB, which is similar to that near the small-angle GBs (Zhang and Wang 1998b). When the surface in the bicrystal was observed, it seems that the ladder-like PSBs beside the GB also have one-by-one relation at low magnifications, as shown in figure 4 (*b*), which is in rough agreement with the surface slip bands (figure 2). If the dislocation patterns near the GB are magnified, it can be clearly seen that the ladder-like PSB structures within the $[\bar{1}34]$ grain (G1) can reach the GB, but the ladder-like PSB structures become rather irregular within the $[18\,\bar{2}\,7]$ grain (G2), as shown in figure 4 (*c*). In particular, the ladder-like PSBs in the $[\bar{1}34]$ grain (G1) seem to produce an affected zone near the GB within the $[18\,\bar{2}\,7]$ grain (G2), as indicated by arrows in figure 4 (*c*), which is not consistent with the surface slip bands in figure 2. Those observations indicate that the ladder-like PSBs cannot completely transfer through the GB on surface II even though the two grains have a common primary slip plane.

To reveal further the dislocation arrangements near the GB, the bicrystal was cut along the common primary slip plane and the dislocation patterns on (111) plane were also observed by the SEM-ECC technique. It is found that the dislocation arrangements on both $[\bar{1}34]$ and $[18\,\bar{2}\,7]$ grains are typical vein structures, as shown in figure 5 (*a*), which is in good agreement with that observed on the (111) plane in copper single crystal (Laird *et al.* 1986). When observing the dislocation arrangements near the GB, it is found that dislocation veins cannot reach the GB

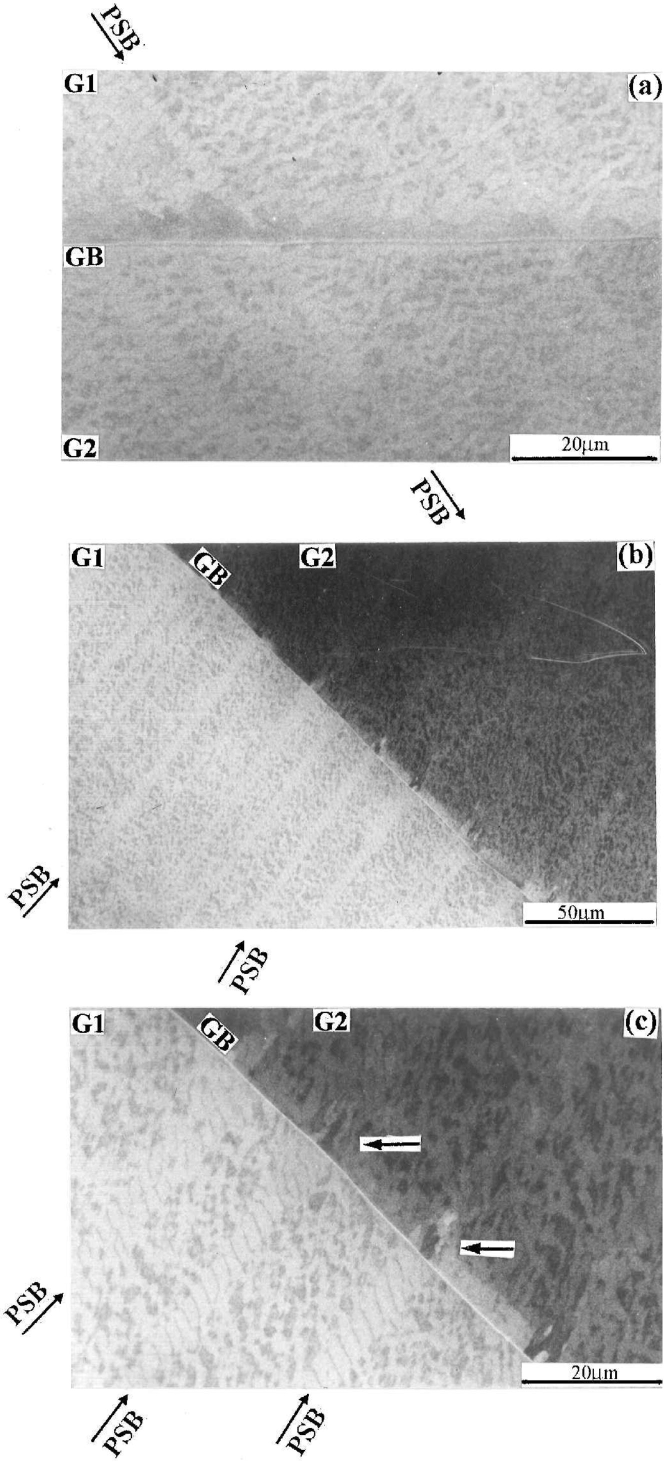


Figure 4. SEM-ECC images showing the dislocation patterns near the GB in the copper bicrystal (*a*) viewed from surface I; (*b*) viewed from surface II at a low magnification; (*c*) viewed from surface II at a high magnification.

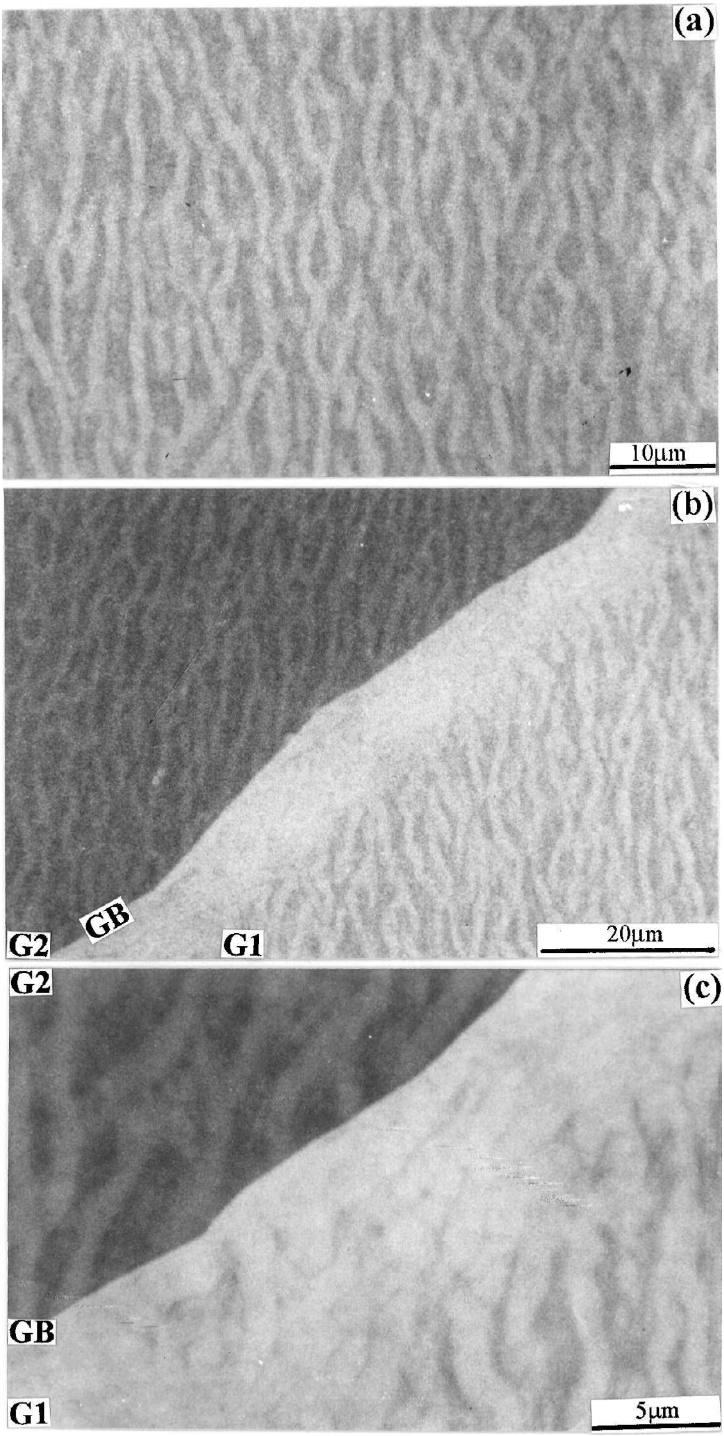


Figure 5. Dislocation patterns observed on the common primary slip plane in the copper bicrystal: (*a*) dislocation veins within $[\bar{1}34]$ grains; (*b*) the interaction of PSBs with the GB at a low magnification; (*c*) the interaction of PSBs with the GB at a high magnification.

and a dislocation-affected zone (DAZ) appeared, as shown in figure 5 (*b*). The DAZ is about 5–10 μm in width and appears only at one side of the GB. In appearance, it is very similar to the dislocation-free zone (DFZ) observed in copper polycrystals (Luoh and Chang 1996), but there is some difference between the DAZ and the DFZ. First, the width (5–10 μm) of the DAZ is about five times that (1–2 μm) of the DFZ; second, at high magnifications, it can be seen that there is still some dislocations in the DAZ, as shown in figure 5 (*c*). Obviously, the DAZ is not dislocation free but contains some dislocations. The DAZ may play an important role in intergranular fatigue damage during cyclic deformation and the formation mechanism need to be clarified by TEM in combination with the SEM-ECC technique.

§ 4. SUMMARY AND CONCLUSIONS

The dislocation patterns within grains near the GB were observed in a fatigued $[\bar{1}34]/[18\,\bar{2}\,7]$ copper bicrystal by the SEM-ECC technique. The following conclusions can be drawn. The surface slip bands can continuously transfer through the GB of the copper bicrystal and do not induce any secondary slip near the GB. The ladder-like PSB structures can form in both $[\bar{1}34]$ and $[18\,\bar{2}\,7]$ grains during cyclic deformation, which is in good agreement with its cyclic stress–strain response. On surface I, the ladder-like PSBs are still continuous beside the GB; however, the dislocation arrangements in those PSBs beside the GB become irregular and discontinuous on surface II. In addition, on the common primary slip plane, a DAZ with a width of 5–10 μm was observed on one side of the GB. Those observations indicate that the dislocation structures within the surface slip bands transferring through the GB in the copper bicrystal are not completely continuous during cyclic deformation even though the two grains have a common primary slip plane.

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