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Characterization of dislocation microstructures in cyclically deformed [001] copper single crystals using high voltage scanning transmission electron microscopy

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ABSTRACT

Low-cycle fatigue tests of copper single crystals with the [001] stress axis were performed at room temperature under constant plastic shear strain amplitudes between $\gamma_{pl} = 3.5 \times 10^{-4}$ and 1.0×10^{-2}. Characteristic dislocation structures, which are highly dependent on a given plastic strain amplitude, were observed by high voltage scanning transmission electron microscopy. A vein-like dislocation structure, having a parallelepiped shape with two longitudinal (100) and (001) sides, was periodically formed along the [010] direction at around $\gamma_{pl} = 1.0 \times 10^{-3}$. Labyrinth structure with the (100) and (001) dislocation walls was then gradually developed with increasing plastic strain amplitude.

1. Introduction

It has been known that characteristic dislocation-wall structures, *i.e.*, "labyrinth" and "cell" structures, are developed when the contribution of secondary slip increases during the cyclic deformation of face centered cubic (fcc) crystals [1,2]. The geometrical features of the labyrinth structure on a mesoscopic scale have been elucidated, especially in [001] oriented single crystals [3–7]. However, the strain-amplitude dependence of development of labyrinth structure is still uncertain in the [001] copper single crystals, as there have been several different definitions of the observed microstructures. Jin and Winter found "uncondensed dislocation slabs" that are similar to, but are not identical with, the vein structure, while they simultaneously showing the existence of a typical labyrinth structure in the same specimen fatigued at a plastic shear strain amplitude of $\gamma_{pl} = 4 \times 10^{-3}$ [4]. Wang et al. also reported the formation of the "irregular labyrinth" structure in a fatigued specimen at a low strain amplitude of $\gamma_{pl} = 2.4 \times 10^{-4}$ [6]. Although Ackermann et al. observed a vein-like structure developed in a specimen fatigued at $\gamma_{pl} = 5 \times 10^{-3}$, they incorrectly interpreted the structure as a "well-saturated labyrinth" [3]. Furthermore, Gong et al. also observed a vein-like structure in a specimen fatigued at $\gamma_{pl} = 6 \times 10^{-4}$ by adopting the electron channeling contrast imaging technique, but they explained it as a regular labyrinth structure [7]. Thus, previous observations are rather fragmentary, and the evolution process and geometrical configuration of the labyrinth and related structures are still unclear. We will therefore focus on the strain-amplitude dependence of the evolution of characteristic dislocation microstructures in the cyclically deformed [001] copper single crystals, and we will reveal the crystallographic and geometrical features of the dislocation microstructures.

To observe the fine structures of defects in deformed specimens, there are clearly some advantages, such as reduced bend contours, thickness fringes, and dynamical contrast effects, in using the scanning transmission electron microscopy (STEM), instead of using the conventional TEM [8–10]. In addition, the use of high-voltage electron microscopy enables us to observe sufficiently thick samples (about 1 μm for Cu) for the detection of actual spatial configuration of dislocations in the bulk state. Therefore, in this study, the high voltage STEM (HV-STEM) was adopted for observation of dislocation microstructures in fatigued specimens.

2. Experimental Procedure

Single crystal plates of copper (purity: 99.9%) with the (100)

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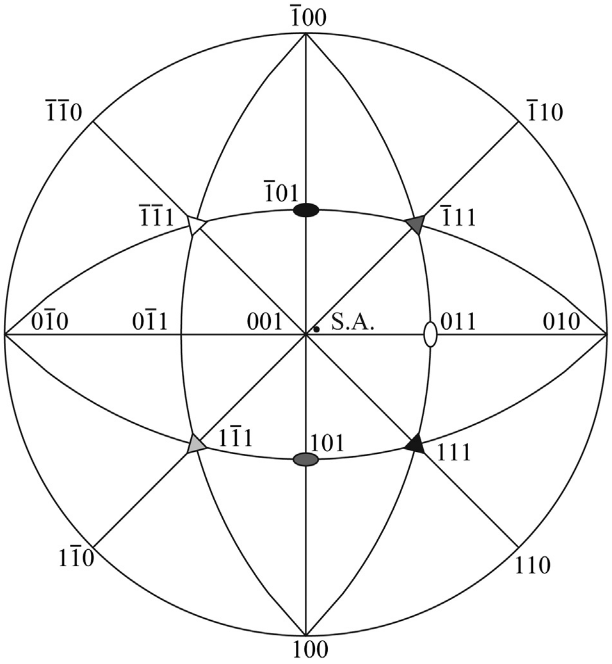

Fig. 1. A 001 stereographic projection of a cubic crystal. The stress axis (S.A.) is located within the basic stereographic triangle 001-011-$\bar{1}$11. $(111)[\bar{1}01]$, $(\bar{1}11)[101]$, $(\bar{1}\bar{1}1)[011]$, and $(1\bar{1}1)[\bar{1}01]$ are the primary, critical, conjugate, and cross slip systems, respectively.

surface were grown by the Bridgman method. The single crystals were cut into specimens for fatigue tests with gauge dimensions of 4 mm × 6 mm × 10 mm so that the stress axis becomes parallel to the [$\bar{1}$ 2 60] direction. The crystallographic orientation was measured using the back-reflection Laue technique with an accuracy of ± 1°, and the stress axis was inclined about 2° from the exact [001] direction. Under this condition, the primary $(111)[\bar{1}01]$ and critical $(\bar{1}11)[101]$ slip systems, for which the Schmid factor is 0.42, are expected to be simultaneously operative as shown in Fig. 1. As the Schmid factors of the conjugate $(\bar{1}\bar{1}1)[011]$ and cross $(1\bar{1}1)[\bar{1}01]$ slip systems are 0.41 and 0.39, respectively, there is a possibility to activate multiple slip. To confirm the active slip systems, therefore, slip lines were observed by a field emission scanning electron microscope (FE-SEM) (JEOL JEM-7001F) after fatigue tests.

Fully reversed tension-compression fatigue tests were performed at room temperature under the constant plastic shear strain amplitude γ_{pl} between 3.5×10^{-4} and 1.0×10^{-2} using a servo-hydraulic testing machine (Shimadzu EHF-LB2-10AL). Here, γ_{pl} was calculated with the highest Schmid factor of 0.42 for the stress axis orientation. The test frequency was controlled between 0.05 and 0.5 Hz by using a triangular command signal. Strain was measured with an extensometer mounted directly on the gauge section. The stress response was monitored and the hysteresis loops were recorded on a digital data logger. To observe how the dislocation microstructure develops, the fatigue tests were interrupted at a cumulative plastic shear strain γ_{cum} of 41.6, where γ_{cum} is defined as $4N\gamma_{pl}$, and N is the number of cycles.

The fatigued specimens were sliced into 3 mm discs parallel to (100), (010), and (001), respectively. These discs were ground down to a thickness of 0.2 mm with silicon-carbide paper. Thin foils were prepared by electrolytic polishing on a twin-jet polisher (Struers Tenupol-5) in a solution of 20% perchloric acid, 10% methanol, and 70% distilled water at 273 K. Microstructural observations were carried out in the bright field mode on a HV-STEM (JEOL JEM-1000 K RS) with an acceleration voltage of 1000 kV.

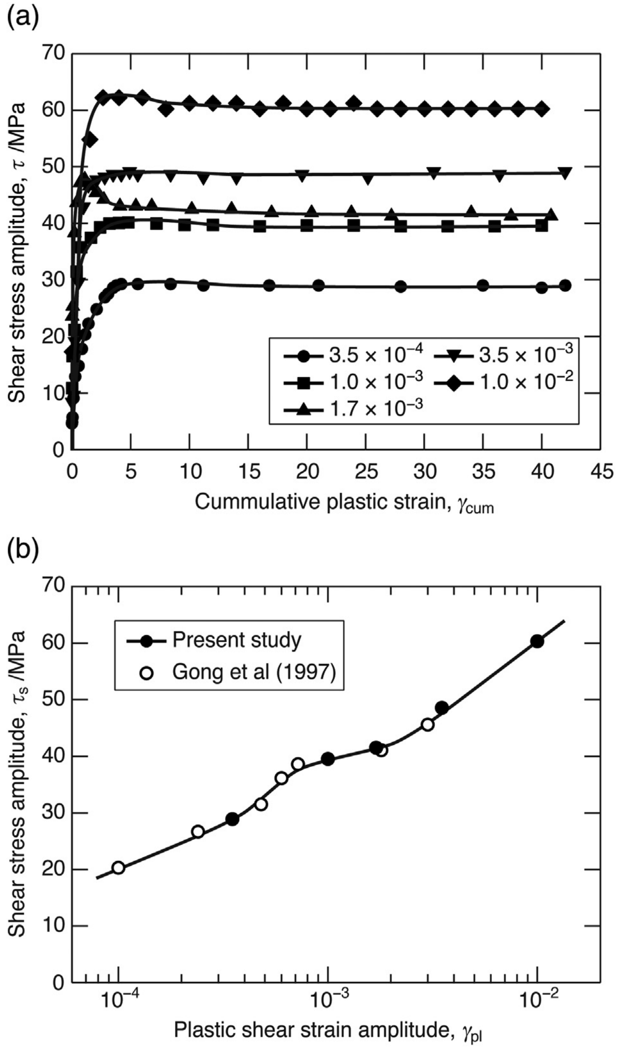

Fig. 2. (a) Cyclic hardening curves and (b) cyclic stress strain curve of the [001] oriented copper single crystals. The experimental data by Gong et al. [11] are also plotted for comparison.

3. Results and Discussion

3.1. Stress-Strain Response and Active Slip Systems

In all the fatigue tests conducted in this study, monotonic cyclic hardening to saturation was observed as shown in Fig. 2(a). Fig. 2(b) shows a cyclic stress strain curve (CSSC) obtained from the cyclic hardening curves by plotting the saturation shear stress amplitudes, τ_s, against controlled plastic shear strain amplitudes. The saturation stress amplitude increased monotonically with increasing given plastic strain amplitude, while a low hardening stage appeared at the intermediate plastic shear strain amplitude of approximately 1.0×10^{-3}. A similar low hardening stage was also found by Gong et al. [11] as shown in Fig. 2(b), though they did not mention it explicitly. As the saturation stress depends on the applied plastic strain amplitude, it is highly expected that the dislocation microstructures may also change depending on the plastic strain amplitude.

Slip lines in the (100) surfaces of the fatigued specimens were observed by FE-SEM as shown in Fig. 3. When the plastic shear strain amplitude is equal to or less than 3.5×10^{-3}, only the (111) and/or $(\bar{1}11)$ slip plane traces were visible, while the (111) and $(\bar{1}11)$ traces are not distinguished in the (100) surface observation. This means that the primary and critical slip systems were extremely active and, but the conjugate and cross slip systems were scarcely active. Although the