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ULTRA-LARGE GRAIN PURE COPPER MICROSTRUCTURE UNDER LOW CYCLE FATIGUE

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Key words: ultra-grain, dislocation, fatigue, re-evolution.

ABSTRACT

The dislocation structure evolution in polycrystalline copper at constant strain amplitude during low cycle fatigue is well understood. Single crystal, ultra-large grain polycrystalline copper dislocation development has received little attention. Ultra-grain polycrystalline copper with 600 μm average grain size was used in this study to investigate the dislocation development at different fatigue strain amplitudes. The results show that; (1) the stress curve vs. number of cycles (S-N curve) produces hardening in the first stage followed by softening regardless of the strain amplitude. At the same time, no plateau is found in the S-N curves. (2) The fatigue saturation stress increases consistently with increased grain size. (3) The special dislocation morphology of ultra-grain copper during fatigue displays a loop patch structure or veined structures embedded in a long band area in parallel dislocation. This is because the larger grain has larger saturation stress, producing a large area with the same slip system band that regulates the high saturation stress.

I. INTRODUCTION

It is well known that fatigue fracture occurs due to dislocation interaction. The evolution of dislocation structure under the fatigue process is among the stacking fault energies in the material structure, such as face-center cube (FCC) (Laird et al., 1986; Ma and Laird, 1988; Chen et al., 2003; Toribio and Kharin, 2006), Therefore, the optimum strength can be o body-center cube (BCC) (Mughrabi et al., 1976; Mughrabi et al., 1981; Buchinger et al., 1986; Sommer et al., 1988) and hexagonal close packing (HCP) (Steveson and Breedis, 1975; Gu et al., 1994). The dislocation structures can be cataloged into two modes. The first is wavy form, which is observed in high stacking fault energy materials, and subsequently develops a loop patched structure, veined structure, persistent slip bands (PSBs), walled structure, cell structure and miss-orientation cell structure (Winter et al., 1981; Ackermann et al., 1984; Laird et al., 1986; Ma and Laird, 1988; Chen et al., 2003; Toribio and Kharin, 2006). The second form is a planar material that exhibits low stacking fault energy with a persistent Lüder band dislocation structure, regardless of the fatigue cycle progression (Buchinger et al., 1986; Inui et al., 1990). The dislocation fatigue structures differ in the space between the Lüder bands. This means that the distance between the persistent Lüder bands decreases with increased plasticity strain accumulation during fatigue.

The wavy form of dislocation structure has been widely researched in the literature. Pure copper FCC materials have been extensively investigated, such as polycrystalline copper under variable strain amplitude (Ma and Laird, 1988; Huang, 2003), low strain amplitude (Buchinger et al., 1984; Laird et al., 1986), frequency effect (Yan and Laird, 1986), temperature effect (Basinski et al., 1980; Sommer et al., 1988; Basinski and Basinski, 1989), load type effect on dislocation structures (Ma et al., 1990; Llanes and Laird, 1993), grain size effect (Lianes et al., 1993; Morrson, 1994) and single crystal copper for fatigue dislocation evolution (Buchinger et al., 1984; Holwarth and Eßmann, 1993). The results from the above mentioned reports reveal that the developed dislocations are similar regardless of the load condition, temperature, strain amplitude, frequency, grain size and polycrystalline type. The difference is the variation in dislocation fatigue acceleration or retardation evolution, high or low saturation stress or the space between the walls. However, the evolution of extra-large grain structural dislocation (about several hundred micro-meters) has seldom been reported. The microstructural evolution of polycrystalline copper with extra-large grain size is therefore studied in this research.

II. EXPERIMENTAL

A polycrystalline copper rod with oxygen free high purity

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Table 1. The fatigue test data at constant strain amplitudes.

Sample	Strain Amplitude	Fatigue cycles	Fracture	TEM
A	0.3%	3000	No	Yes
B	0.3%	19246	Yes	Yes
C	0.2%	6000	No	Yes
D	0.2%	39851	Yes	Yes
E	0.1%	119376	Yes	Yes

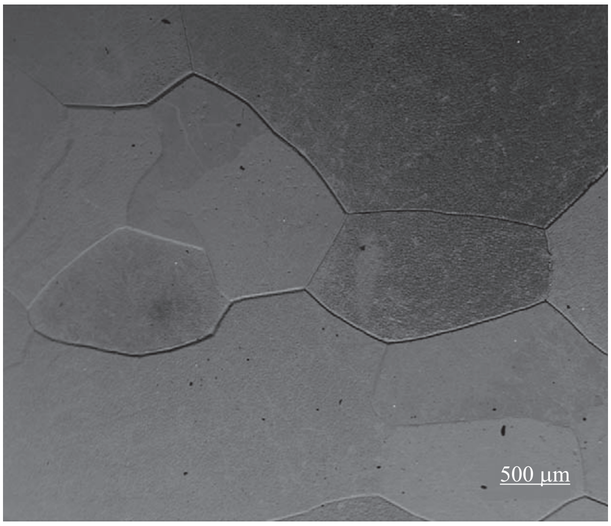

Fig. 1. The grain size of OFHC pure copper annealed at 10^{-5} Torr. vacuum for 4 hours. The average grains size is about 600-800 µm.

(OFHC, 99.99%) was used for this study. The specimens were annealed at 800°C for 4 hours in a vacuum at 10^{-5} Torr. Samples were then cooled in the furnace. The specimen grain sizes were 650-700 µm, as shown in (Fig. 1). The specimen preparation followed the ASTM E647 instructions for hour-glass. The specimen configuration is shown in (Fig. 2).

Before fatigue, the glass specimens were polished using 240, 400, 600, 1000, 1200 mesh abrasive papers. The polished specimen was processed using Al_2O_3 powder (0.3 micron). Low cycle fatigue tests were performed on a computerized Instron 8801 hydraulic testing machine at strain ratio R ($R = \varepsilon_{min}/\varepsilon_{min} = -1$) and a frequency of 1 Hz. The fatigue condition is shown in Table 1.

Specimens prepared through the low cycle fatigue process were cut into 0.6 mm thick slices along the cross section to observe the dislocation structures. The slices were ground to a thickness of 0.1-0.15 mm using abrasive paper and then punched into disks 3 mm in diameter. The 3 mm disks were twin-jet polished using Struer D_2 solution at 10 V and −10°C. A Philip 200 CM transmission electron microscope (TEM) was employed to investigate microstructures of the low cycle fatigue specimens.

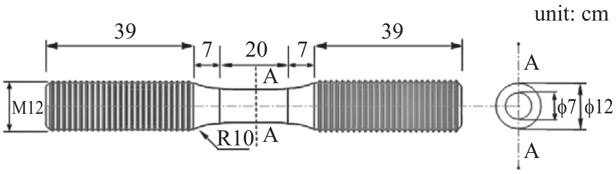

Fig. 2. Specimen dimensions.

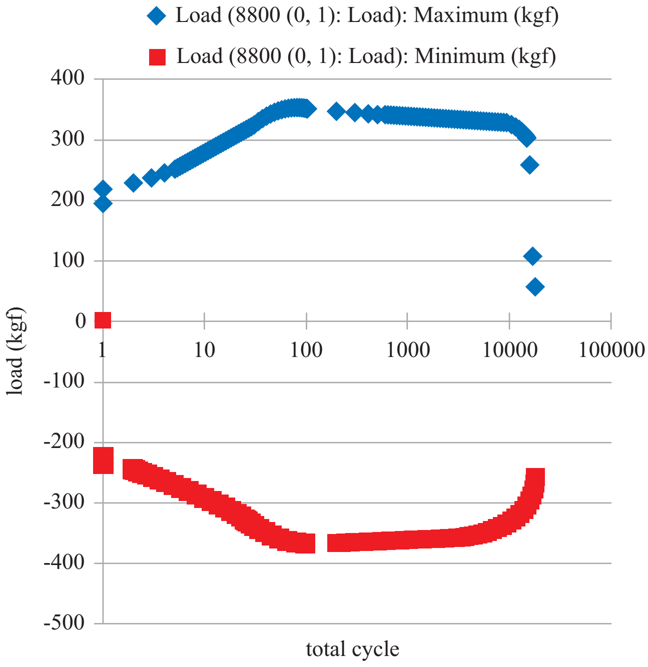

Fig. 3. The stress vs. number of fatigue cycles diagrams at 0.3% strain amplitudes.

III. RESULTS AND DISCUSSION

After low cycle fatigue under 0.3%, 0.2% and 0.1% strain amplitude, the stress vs. fatigue number of cycles for 0.3% strain amplitude specimens is shown in (Fig. 3). The S-N curves for 0.2% and 0.1% strain amplitude are similar to the 0.3% strain amplitude samples. This result shows that regardless of the strain amplitude, the S-N curves show hardening initiated and then softening until fatigue fracture. At the same time no plateau area is shown in any S-N curve. These results vary from single crystal specimens (Buchinger et al., 1984). The saturation stress (11.04 kgf/cm^2) for ultra-large grain is larger than that for the large grain (Lianes et al., 1993; Morrson, 1994). However, the S-N curve in this study showed no secondary hardening effect. This result is different from that for small and large grain size specimens under the same strain amplitude (Wang and Mughrabi, 1984; Laird et al., 1989; Wang et al., 1989). The microstructures were observed using a Philip 200 CM transmission electron microscope, shown in Figs. 4-8. Fig. 4 shows the dislocation structure at 3000 cycles under 0.3% strain amplitude. It reveals a similar loop patch structure or veined structures embedded in a long band clipped in parallel dislocation walls. Vein structures, cells and miss-orientation cell structures were also observed. The dislocation structures in the

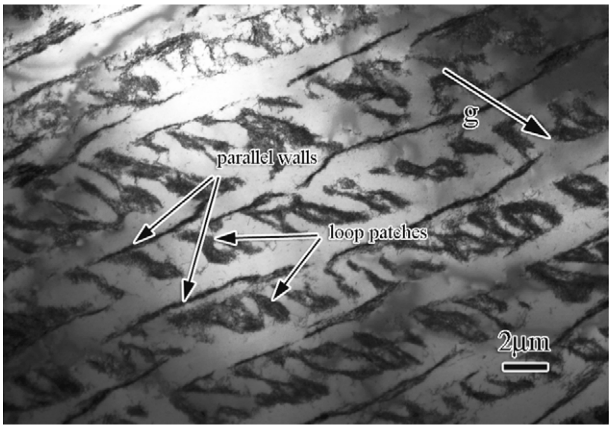

Fig. 4. **The dislocation structure in specimen A (0.3% strain amplitude fatigue to 3000 cycles) reveals loop patches and veins structure embedded in two parallel walls.**

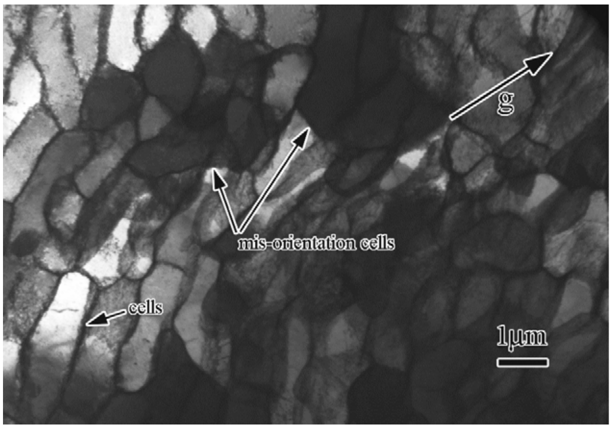

Fig. 5. **The dislocation structure in specimen B (0.3% strain amplitude fatigue to fracture) shows cells and mis-orientation cells.**

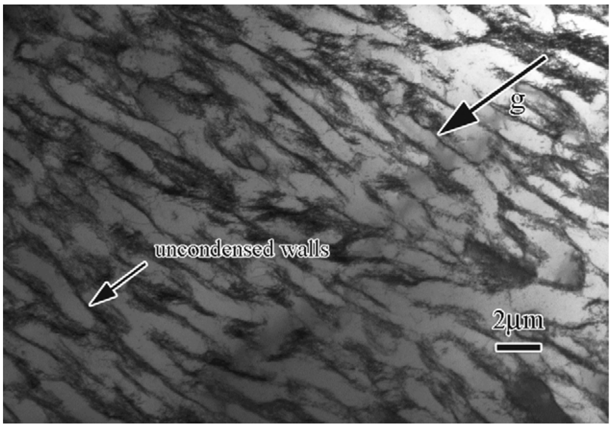

Fig. 6. **The dislocation structure in specimen C (0.2% strain amplitude fatigue to 6000 cycles) shows uncondensed cells.**

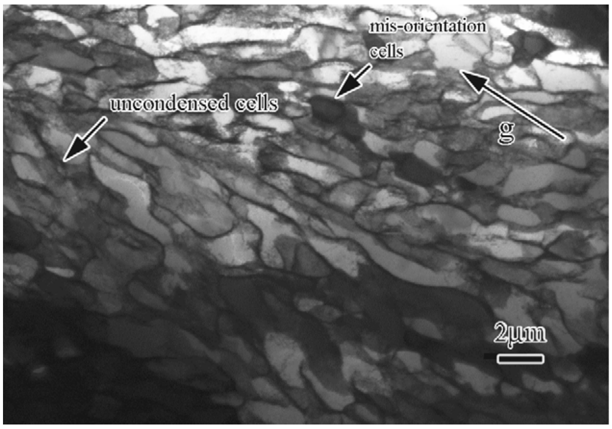

Fig. 7. **The dislocation structure in specimen D (0.2% strain amplitude fatigue to fracture) reveals uncondensed cells, cells and mis-orientation cells.**

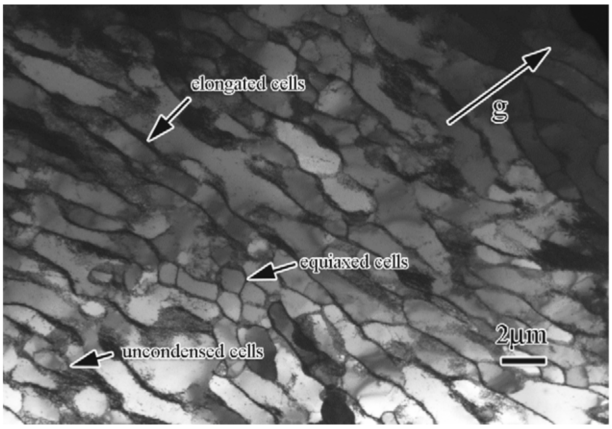

Fig. 8. **The dislocation structure in specimen E (0.1% strain amplitude fatigue to fracture) revealed loop patch elongated cells and equiaxed cells.**

fracture at 0.3% strain amplitude are revealed in (Fig. 5). This result is similar to that reported for copper materials (Laird et al., 1986). Fig. 6 shows the dislocation structure at 6000 cycles under 0.2% controlled strain amplitude. A similar loop patch or veined structure is shown in the uncondensed dislocation wall structure. The dislocation fracture structures at 0.2% strain amplitude present a cell structure (Fig. 7). Similarly, the dislocation structure at 0.1% fatigue strain amplitude fatigue also presents a cell structure (Fig. 8). The difference between Figs. 5, 7 and 8 is the cell size and cell shape. The cell size (0.5~0.7 μm, average 0.6 μm) is smaller when the strain amplitude is increased. This is consistent with that reported in the literature (Laird et al., 1986). The dislocation cell shape at 0.1% strain amplitude varies compared to 0.2% and 0.3% strain amplitude, the strain amplitude change dislocation cell morphology shown in Table 2. This is because the strain amplitude is too low to create a loose multiple slip density system.

Based on the results above the dislocation structures in the ultra-large grain size are similar to those in the small or large grain size. The differences among these specimens are shown in (Fig. 5). The S-N curve reveals softening after the initial hardening stage with no secondary hardening effect. According

Table 2. The cell morphology at change strain amplitudes.

Strain Amplitude	Non-Saturation (3000 cycles)	Saturation
0.1%	The dislocation structures dominated by elongated cells and equiaxed cells. The equiaxed cells are majority.	equiaxed cells
0.2%	The dislocation structures dominated by elongated cells and equiaxed cells. The elongated cells are majority.	equiaxed cells
0.3%	The elongated cells take the most part of the dislocation structures. The equiaxed cells are rarely.	elongated cells

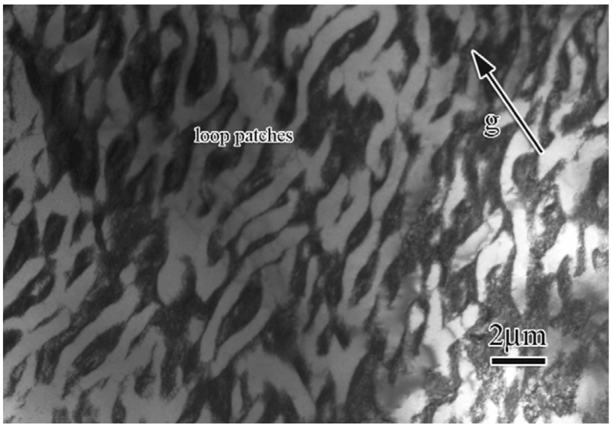

Fig. 9. The dislocation structure in specimen A (0.3% strain amplitude fatigue to 3000 cycles) reveals loop patches with high density dislocation.

to the literature reports, the loop patch and veined structure occur in the hardening phase and the persistent slip band occurs in the softening phase (Winter et al., 1981). Because the larger grain has higher saturation stress (Lianes et al., 1993; Morrson, 1994) and higher dislocation density (Fig. 9), there is a larger slip system with the same direction upon the second slip system created when the fatigue reaches saturation stress. This result induces softening in the S-N curve. At the same time, the plasticity strain accumulation is smaller than that in the previous step due to the softening effect. Therefore, the same directional slip systems that induce the slip band remain within a narrow band and continue to regulate the plastic strain accumulation. Based on above two factors, the S-N curve exhibits continued softening with no secondary hardening observed. Unless the plastic strain accumulation becomes large enough to result in multiple slip systems, the second slip system continues operate. Therefore, the dislocation morphologies in Fig. 4 were formed. In other words, the dislocation structure shown in Fig.4 is the ultra-grain result with high saturation stress and the strain amplitude is insufficiently large to create a multiple slip system or extend the slip band in the same direction to form a cell structure.

IV. CONCLUSIONS

This study revealed that dislocation obeys a wavy form type development. This means that the dislocation evolves from a loop patch, veins, PSBs, walls, cells and then mis-orientation at the plastic strain accumulation during fatigue. Special dislocation morphology was observed in ultra-large polycrystalline copper grain. This phenomenon is due to the larger grain with larger saturation stress to regulate. The high saturation stress induces a larger slip system band. At the same time the S-N curves indicate softening. The strain amplitude is based on the average grain size in fatigue specimens regardless of the load conditions.

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