Cyclic Deformation Behavior of Ultra-Fine Grained Copper Produced by Equal Channel Angular Pressing

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Cyclic deformation behavior of ultra-fine grained (UFG) Cu of 99.99% purity processed by equal-channel angular pressing was investigated. In tension-compression fatigue tests under strain control, UFG Cu showed cyclic softening. Shear bands were formed along the direction inclined by about 45° from the loading axis. Observations using an electron backscattering diffraction technique and transmission electron microscopy revealed that local grain growth took place in the shear bands and overall grains elongated along the shear direction. Cyclic softening can be understood as a result of dynamic grain coarsening occurred intensively in the strain localized shear bands.

Keywords: ultra-fine grained copper, equal channel angular pressing, cyclic deformation, cyclic softening, shear bands, grain coarsening

1. Introduction

In recent years, much attention has been given to the mechanical properties of ultra-fine grained (UFG) materials produced by severe plastic deformation (SPD) techniques such as equal channel angular pressing (ECAP),1-4 accumulative roll-bonding (ARB)5,6 and high-pressure torsion (HPT).7,8 In considering practical applications, the cyclic deformation and fatigue properties of UFG materials have been studied extensively. In particular, the following characteristic features in cyclic deformation of UFG Cu processed by ECAP have been sporadically reported: (1) cyclic softening is frequently detected as a stress-strain response,9-13 (2) macroscopic shear bands (SBs) are formed inhomogeneously,9,10,13-18 (3) coarse grains are locally developed.9,12,14,15,19-21 For example, Wu et al.14,15 observed no grain coarsening, while they found apparent SBs by scanning electron microscopy (SEM) observation. Therefore, they concluded that there is no relationship between the SB formation and the grain coarsening. Contrary to this result, Mughrabi and Höppel19 suggested that the grain coarsening is closely related to the formation of the SBs and proposed two possible SB formation mechanisms. As far as the authors know, there has been no consistent explanation to understand all the above features of the cyclic deformation of UFG Cu. To obtain a thorough understanding of the microstructural changes during cyclic deformation of UFG materials, it is essential to conduct precise observations with the combined use of SEM and transmission electron microscopy (TEM).

In this study, plastic-strain-controlled cyclic deformation tests were performed using high purity UFG Cu produced by ECAP. Based on the results of SEM and TEM observations, the relationship among cyclic softening, SB formation and local grain coarsening was discussed.

2. Experimental Procedure

UFG copper (99.99% purity) billets of 10 mm in diameter and 60 mm in length were processed by the ECAP technique. Each billet was subjected to pressing for 8 passes in such a way that after each pressing the billet was rotated by 90° in longitudinal direction, usually denoted as route Bc.3,4

Specimens for fatigue tests with the gauge dimensions of 4 mm x 6 mm x 10 mm were taken from the central part of the ECAP billets by spark erosion in the direction parallel to the rod axis. The specimens were electrolytically polished to avoid any influence of mechanical pre-treatment. Fully reversed tension-compression low-cycle fatigue tests were carried out at room temperature (RT) under the constant plastic-strain amplitudes ranging from $\varepsilon_{pl} = 2 \times 10^{-4}$ to $\varepsilon_{pl} = 2 \times 10^{-3}$ using an electro-hydraulic testing machine (Shimadzu Servopiet). Strain was measured with an extensometer mounted directly on the gauge section and a constant strain rate of $1 \times 10^{-3}$ s$^{-1}$ was employed using a triangular command signal. The stress response was monitored on a digital oscilloscope. For microstructural observation, the fatigue tests were interrupted when the stress amplitude decreased and reached 85% of the maximum stress amplitude.

Surface observations were performed on both as-pressed (as-ECAPed) and fatigued specimens by using a Hitachi S-4300 scanning electron microscope equipped an electron backscattering diffraction (EBSD) system (Oxford INCA crystal). Grain maps were constructed with an individual step size of 40 nm and a lower cut-off misorientation angle of 5°.

The fatigued specimens were sliced into 3 mm discs and ground down to 0.2 mm using silicon-carbide paper. Final thin foils were prepared by electrolytic polishing, and then TEM observations were carried out on a JEOL 2011 microscope at an accelerating voltage of 200 kV.

3. Results and Discussion

3.1 Cyclic stress-strain response

Cyclic hardening/softening curves of UFG Cu specimens
To discuss the strain-amplitude dependence all plastic-strain amplitudes as previously reported in the literature. To discuss the strain-amplitude dependence all plastic-strain amplitudes as previously reported in the literature. To discuss the strain-amplitude dependence all plastic-strain amplitudes as previously reported in the literature. To discuss the strain-amplitude dependence all plastic-strain amplitudes as previously reported in the literature. To discuss the strain-amplitude dependence all plastic-strain amplitudes as previously reported in the literature. To discuss the strain-amplitude dependence all plastic-strain amplitudes as previously reported in the literature. To discuss the strain-amplitude dependence all plastic-strain amplitudes as previously reported in the literature. To discuss the strain-amplitude dependence all plastic-strain amplitudes as previously reported in the literature. To discuss the strain-amplitude dependence all plastic-strain amplitudes as previously reported in the literature.

The results obtained from strain-controlled tests on UFG Cu by Höppel et al. and those on conventional coarse-grained polycrystalline Cu by Mughrabi and Wan are also shown in Fig. 3. It is found that there is a good agreement between the present result and that obtained by Höppel et al.. For the cyclic stress-strain response of polycrystalline cubic metals, the power-law relationship

\[ \sigma_n = k\varepsilon_p^n \]  

has been established, where \( k \) is the cyclic strength coefficient and \( n \) is the cyclic strain hardening exponent. Applying eq. (1) to the experimental results of the UFG Cu, the values of \( k \) were received. Similarly, the values of \( k \) were received.

### 3.2 Microstructural characterization

Figure 4 shows a set of SEM images taken from specimens cyclically deformed at various plastic-strain amplitudes. On the surfaces of all deformed specimens, so-called SBs were developed. The length of the SBs varied widely from 1 \( \mu \)m to 100 \( \mu \)m in each specimen. The morphology of the SBs was quite similar to the extrusions frequently formed in persistent slip bands of fatigued Cu single crystals. The SBs were also inclined by about 45° from the stress axis. This indicates that the SBs are formed parallel to the planes of maximum resolved shear stress. Since micro-crack initiation along the SBs was detected, strain localization indeed occurs in the SBs during cyclic deformation. These structural features of the SBs characterized by the present study are consistent with those reported in the literature.

Changes in grain size and grain morphology of the fatigued specimens were observed using the EBSD method. Grain maps obtained from an as-ECAPed material and a specimen...
fatigued at $\varepsilon_{pl} = 2 \times 10^{-4}$ are shown in Figs. 5(a) and 5(b), respectively. It can be seen from Fig. 5(a) that fine grains with an average size of approximately 500 nm were formed after 8 passes of ECAP. As shown in Fig. 5(b), coarse grains with sizes of about 10 $\mu$m long were locally developed in the cyclically deformed specimen. These grains were banded along the direction almost parallel to the trace of SBs (see Fig. 4(a)). Here, it is needed to pay attention to the actual sizes of the coarsened grains since small-angle boundaries with misorientations of up to $5^\circ$ are indistinguishable in our SEM observation. The results of TEM observations to elucidate the grain interior in the fatigued specimens will be shown later in this paper.

It also turns out from Fig. 5(b) that the other major grains slightly grow up to 1–2 $\mu$m and elongate into the same direction as that observed in the coarse grains. Although Wu et al.,14) Kunz et al.,13) Lukáš et al.,17,27) and Xu et al.18) conducted SEM observations in fatigued commercial-purity UFG Cu (99.8%–99.9%) produced by ECAP, they all concluded that no detectable grain coarsening took place during cyclic deformation. This result is obviously in disagreement with the present result. On the other hand, Höppel et al.12,20,28) have reported that shear banding and pronounced grain coarsening take place during cyclic deformation of high-purity UFG Cu (99.99%), while they have not observed the grain elongation as represented in the present study. Therefore, the results of our study accord qualitatively with those obtained by Höppel et al.12,20,28)

From all these findings, it is evident that the stability of UFG structure during cyclic deformation strongly depends on the purity of materials.

The relationship between the SB formation and the local grain coarsening is one of the most interesting problems in cyclic deformation of UFG Cu. In the previous studies mentioned above,13,14,17,18,27) it has been concluded that there is no relationship between the SB formation and the grain coarsening since as-ECAPed grain structures are kept in fatigued specimens with clear SBs. On the contrary, Mughrabi and Höppel,19) and Höppel et al.20,28) have insisted that the grain coarsening plays a dominant role in the formation of the SBs. From Fig. 4(a) and Fig. 5(b), it is reasonable to conclude in the present study that the grain coarsening is closely related to the SB formation. The following experimental results of TEM observations will give strong evidence for this conclusion.
The grain morphology in a specimen after ECAP through 8 passes is shown in Fig. 6. Dense dislocations are found to be tangled in the grain interiors. In contrast, it is obvious from Fig. 7 that local grain growth occurred characteristically in a specimen cyclically deformed at $\epsilon_{pl} = 2 \times 10^{-4}$. It is noteworthy that the coarse grains having a low dislocation density are aligned along the direction approximately parallel to the SBs as represented in Fig. 5(b). The average size of the coarse grains was measured as about 2 µm, and this value is much smaller than that obtained from the grain map shown in Fig. 5(b). It can be readily understood from Figs. 5(b) and 7 that each elongated grain about 10 µm in length actually consists of subgrains whose size is in the range of 1–3 µm. From our multi-scale observations of the SBs by SEM and TEM, we can justify the conclusion that the plastic strain localization during cyclic deformation of UFG Cu induces the dynamic grain/subgrain growth and causes the development of the SBs.

**Fig. 6** TEM photograph taken from an as-ECAPed specimen.

**Fig. 7** TEM photograph taken from a specimen cyclically deformed to $\epsilon_{cum} = 3.3$ at $\epsilon_{pl} = 2 \times 10^{-4}$. Stress axis is in the vertical direction. Subgrain structure was developed heterogeneously in the specimen.
3.3 Correlation among cyclic softening, shear banding and local grain coarsening

In the present study, isolated coarse grains with sizes of 1–3 μm were also observed frequently in specimens cyclically deformed at various strain amplitudes. A typical result is shown in Fig. 8. Since the dislocation wall structure is well developed in this isolated coarse grain as originally reported by Agnew and Weertman, it may be expected that the strain localization takes place even in the isolated grain. This is consistent with the fact that the length of the SBs varied widely from 1 μm to 100 μm. To explain the formation mechanism of the SBs, Mughrabi and Höppel have proposed two possible scenarios. One is that grain/subgrain coarsening is locally initiated and leads to SBs in a large scale. The other is that a catastrophic extended SB firstly happens and subsequently triggers the formation of a new coarsened microstructure within the SB. A reasonable implication of our observations is that the former scenario is more likely to occur as the SB formation mechanism.

As shown in Fig. 1, the cyclic softening was observed at all plastic-strain amplitudes. The SB formation and the grain growth were also detected by the SEM and TEM observations. These findings inevitably raise the question of whether the development of the SBs is related to the cyclic softening. Xue et al. and Kunz et al. have concluded that there is not a one-to-one correlation between the SB formation and the cyclic softening since they both have found distinct SBs even in the case of cyclic hardening. Moreover, they could provide no microstructural evidence of the cyclic softening. On the other hand, the present study gives strong evidence for the direct relationship between the SB development and the local grain coarsening. Therefore, it is quite reasonable to suppose that the cyclic softening is closely related to the strain localization and the resultant shear-banding phenomenon, while the purity of materials strongly affects the stability of the UFG structure.

4. Conclusions

Plastic-strain-controlled cyclic deformation of high purity UFG Cu (99.99%) produced by ECAP was performed at RT. The stress-strain response and the microstructural change were investigated. The conclusions can be summarized as follows.

(1) UFG materials show cyclic softening at all plastic-strain amplitudes.

(2) SBs are developed along the planes of maximum resolved shear stress. Grain coarsening occurs intensively in the SBs and gives rise to subgrain formation.

(3) Cyclic softening can be understood as a strain localization phenomenon and is closely related to the SB formation and the dynamic grain coarsening.

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