Stress-Amplitude-Dependent Deformation Characteristics and Microstructures of Cyclically Stressed Ultrafine-Grained Copper

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Various mechanical behaviors of ultrafine-grained (UFG) materials produced by equal channel angular pressing (ECAP) generally exhibit distinctive characteristics from those of conventional grained materials, so that the relevant studies have received sustained attention in recent years.[1–16] Apparently, the understanding of the fatigue properties (among various mechanical properties) of such UFG materials is of particular importance for their practical engineering applications, many investigators have thus been attempting to explore the fatigue deformation mechanisms of UFG materials, especially of UFG copper, with different emphases.[17–34] It is now commonly recognized that the low-cycle fatigue damage in UFG materials primarily results from the formation of shear bands (SBs), which can extend over far larger scales than the initial grain size,[18–26] and that the cyclic softening phenomenon as well as the deterioration in fatigue life under strain-amplitude-controlled tests in the low-cycle fatigue (LCF) regime is attributed to grain coarsening due to instabilities of the ECAPed structures during cycling.[21,22,27,32,33] On the contrary, the fatigue life of UFG materials obtained under stress-controlled tests in the high-cycle fatigue (HCF) regime is normally enhanced greatly as compared to their conventional counterparts.[21,27] However, the details about cyclic deformation microstructures and shear band features in LCF and HCF regimes, respectively, are less understood so far. To further understand the possibly different fatigue deformation mechanisms of UFG copper in LCF and HCF regimes, the present contribution is to report the effect of the applied stress amplitude on the cyclic stress response behavior, surface damage features as well as relevant microstructural changes of such UFG copper materials. The applied stress amplitudes ranging from 100 MPa to 200 MPa are adopted to ensure that fatigue tests are carried out within both LCF and HCF regimes.

Experimental Procedures

The rod of UFG copper (99.98 %) of 20 mm in diameter was produced by the ECAP process. Ten passages of extrusion were performed to introduce an equivalent shear strain of about 11.5. All pressings were carried out by rotating each sample about the longitudinal axis by 90° in the same direction between consecutive passes (i.e. so-called route Bc). Fatigue specimens with a gauge section of 2 × 2 × 4 mm3 were carefully spark-machined from the as-prepared rod. Before the fatigue tests, the specimens were electro-polished to produce a strain-free and mirror-like surface for microscopic observations. Stress-controlled (R = –1) fatigue tests were performed in vacuum at room temperature with a frequency of 5 Hz using a servo-hydraulic testing machine (Shimadzu SEM Servopulsor). Three different applied stress amplitudes Δσ/2 of 100 MPa, 150 MPa and 200 MPa were adopted. The plastic strain was measured from each stress-strain hysteresis loop. Surface deformation characteristics and fracture surface morphologies were examined care-
fully by using an optical microscope and a scanning electron microscope (SEM). Fatigue microstructures of UFG copper were observed by transmission electron microscope (TEM) using JEOL 3010 electron microscope operated at 300 kV. TEM thin foils were first sliced from the gauge part of the fatigued specimens by spark cutting perpendicular to the loading axis, then mechanically thinned to dozens of microns thick and, finally, polished by a conventional twin-jet method.

Results and Discussion

As-produced Microstructure

The bright-field TEM image of as-produced microstructure was given in Figure 1, where the corresponding selected area electron diffraction (SAED) was insetted. It is clear that the microstructure in the as-produced condition consist mainly of relatively equiaxed grains with large- and small-angle boundaries and an average size of about 250 nm; however, typical fine lamellar structures were also found in some local areas (for details see Ref.[29]).

Cyclic Deformation Response Curves

Figure 2 presents cyclic deformation curves of the UFG copper at different stress amplitudes. Obviously, cyclic softening occurred at each stress amplitude and this phenomenon took place even earlier and more notably with increasing stress amplitude. The fatigue lives at stress amplitudes of 100 MPa, 150 MPa and 200 MPa are $7.72 \times 10^5$, $3.11 \times 10^5$ and $3.27 \times 10^4$, respectively. Clearly, the fatigue life at the low stress amplitude of 100 MPa is close to $10^7$ and it can thus be considered to fall in the HCF regime, but the life at the high stress amplitude of 200 MPa basically falls in the LCF regime.

Surface Deformation Features

Figure 3 shows the essential surface deformation features of UFG copper at different stress amplitudes. It can be seen that the commonly prominent feature is, without exception, the occurrence of shear bands on the surface at all stress amplitudes. That is to say, the plastic deformation of UFG copper under cyclic stressing is governed by the formation of SBs. However, the SBs formed at different $\Delta \sigma/2$ exhibited distinctive features. For example, the SBs seem to form separately and locally at a low $\Delta \sigma/2$ of 100 MPa, and single shear band can be clearly seen as marked by the arrows in Figure 3(a). These single shear bands extended over about 0.1 mm, which is much larger than the initial grain size of 250 nm, constituting in groups macroscopic SBs in local areas, meanwhile, some local areas remained undeformed as indicated by “U” in Figure 3(a). With increasing of $\Delta \sigma/2$ to 150 MPa, single shear band can hardly been detected and coarse SBs (indicated by the arrow in Fig. 3(b)) consisting of many single shear bands formed locally with some areas being still undeformed as marked by “U” as well in Figure 3(b). According to the feature and distribution of these coarse SBs, we can call them SB clusters. At the highest $\Delta \sigma/2$ of 200 MPa, two sets of large-scale SBs, which are roughly perpendicular to each other, were observed to appear on the maximum resolved shear stress plane making approximately 45° with the loading direction. These SBs almost extended across the entire surface of the fatigue specimen, leading to a serious surface damage of the deformed specimen.

From the above-observed surface features of such SBs it is reminiscent of the deformation bands (DBs) recently found in fatigued copper single crystals, which disrupt an initially smooth surface and affect the fatigue crack nucleation and subsequent propagation. Analogously, the formation of SBs in the present fatigued UFG copper is also a decisive factor affecting crack initiation and premature failure. For example, SEM examinations of surface damage morphologies demonstrated that crack initiation did take place along SBs in all samples, as shown in Figure 4. At the lowest $\Delta \sigma/2$ of 100 MPa, the cracks formed separately along many long and straight single shear bands (see Fig. 4(a)); this morphology is fairly similar to the crack nucleation along persistent slip bands (PSBs) in fatigued copper single crystals. When $\Delta \sigma/2 = 150$ MPa, many micro-cracks formed together within
SB clusters, as indicated by the arrows in Figure 4(b). At the highest $\Delta \sigma/2$ of 200 MPa, serious extrusions were found on the surface and cracks or voids nucleated along the large-scale SBs (see Fig. 4(c)).

Several investigators have given some explanations on the formation of SBs in deformed UFG copper,\textsuperscript{[21,22,25,26,30,40–46]} and some disputable opinions have arisen. For examples, Mughrabi and Höppel\textsuperscript{[21]} suggested that the formation of SBs might be related with local grain/subgrain coarsening during cycling, whereas Wu et al.\textsuperscript{[25,26]} reported that no detectable grain coarsening was observed in the vicinity of SBs, and they held that the formation of SBs may be attributed to both oriented distribution of defects along shear plane of last pressing and local reversible and irreversible deformation.

Another often-mentioned explanation is based on grain boundary sliding. Vinogradov and Hashimoto\textsuperscript{[22]} have given an evidence for it and they thought the formation of SBs as being the result of a sum of mesoscopic and microscopic shear events along grain boundaries. This explanation seems to be also applied to the present case of SBs formed at different $\Delta \sigma/2$. However, the characteristics of SBs are strongly dependent upon the applied $\Delta \sigma/2$, as shown schematically in Figure 5. At the low stress amplitude, i.e. within HCF regime (see Fig. 5(a)), macroscopic SBs consist clearly of many short single shear bands, with local areas being undeformed. With the increase in stress amplitude, the density of SBs increases and single shear band become unseen, and the surface is occupied by some dense SB clusters, as shown in Figure 5(b). When fatigue tests are performed in LCF regime, i.e. at the high stress amplitude, large-scale DBs extending across the whole specimen surface form and disrupt the surface (see Fig. 5(c)). In a word, the plastic deformation of UFG copper, in any case, is governed mainly by the formation of SBs, regardless of their features. At low stress amplitudes (HCF regime), the elastic component of the induced total strain amplitude is dominant during cycling, so that the formation of SBs comprising single shear bands are sufficient to accommodate the plastic strain. In contrast, provided the applied stress amplitude is getting high enough (i.e. in the LCF regime), the plastic component would become much larger, and in this case, the plastic strain has to be accommodated by the formation of large-scale SBs with a high density.

Fracture Surface Features

The fracture surface features at different $\Delta \sigma/2$ are shown in Figure 6. It is apparent that the applied stress amplitude has an obvious effect on the fracture surface morphology. The fracture surface at the lowest $\Delta \sigma/2$ is mainly featured by cleavage planes (see Fig. 6(a)), indicating an obvious brittle fracture mode. As $\Delta \sigma/2$ rose, some clear cleavage steps can be found, as marked by the arrows in Figure 6(b). Micro-plasticity exited at the cleavage steps, which is analogous to the
feature of tear ridges often observed in quasi-cleavage fractures. The stress amplitude continued increasing to 200 MPa (i.e. in the LCF regime), the fracture surface is a mixed-mode fracture featuring dimples and some cleavage planes (see Fig. 6(c)), showing an enhanced plastic deformation. It should be noted that the dimple size is much larger than the initial grain size, which should be ascribed to the obvious grain coarsening. Undoubtedly, from Figures 6(a) to (c), one can see a clear change in fracture mode from brittle to more ductile as the applied stress amplitude increases, or as cycling is going from HCF regime to LCF regime.

**Microstructures**

The microstructures observed by TEM in UFG copper fatigued at different stress amplitudes were given in Figures 7–9. Observations demonstrated that after cyclic stressing at the lowest $\Delta \sigma/2$ of 100 MPa the UFG copper specimen underwent substantial structural changes, i.e., grain coarsening, as shown in Figure 7(a). Under this circumstance, except for some single dislocations or dislocation tangles (indicated by the arrows in Fig. 7(b)), no typical dislocation arrangements are observable in these coarsened grains. As the stress amplitude increased to 150 MPa, an obvious grain coarsening took place as well, but in this case, except for single dislocations (Fig. 8(a)), some typical dislocation arrangements, such as dislocation walls (Fig. 8(b) and loose cell structures (Fig. 8(c)), are discernible in a few of coarsened grains. It should be
noted that such dislocation arrangements are not well-developed. For example, the vestige of cell structure feature seems to be indicated by the arrow in Figure 8(a). With raising the stress amplitude to 200 MPa, it was found that some typical dislocation structures, such as dislocation walls (Figs. 9(a) and (b)), dislocation cells (Fig. 9(b)–(d)) and PSB ladder-like walls (Fig. 9(d)), have formed in many coarsened grains, showing an enhanced cyclic softening and plastic deformation. These typical dislocation structures shown in Figure 9 were frequently observed in fatigued copper polycrystals with a conventional grain size\textsuperscript{[48,49]} and copper single crystals\textsuperscript{[50–53]}. It should be pointed out that there are still more or less unchanged ultrafine grains, in all cases (Fig. 10), which should correspond to the undeformed areas as shown in Figures 3 and 5. Such a stress-amplitude dependence of microstructures accounts for the corresponding cyclic softening response behavior shown in Figure 2.

Summary

Fatigue and fracture behavior of UFG copper produced by ECAP were investigated at different stress amplitudes ranging from 100 MPa to 200 MPa. According to the magnitude of applied stress amplitudes, the fatigue tests were carried out, in fact, in three regimes, i.e. HCF regime, LCF regime and in-between regime. It was found that cyclic softening occurred in all cases and it took place even earlier and more notably with increasing stress amplitude. On cyclic stressing in these three regimes, the plastic component of the total strain amplitude would, to different extents, contribute to the fatigue deformation, so the main conclusions can be catalogued, according to these three regimes, as follows.

1) In the HCF regime, the surface deformation feature was manifested by the macroscopic SBs consisting clearly of many short single shear bands, and cracks formed separately along the single shear bands. The UFG copper specimen finally fractured in the form of a cleavage brittle mode. The corresponding microstructural changes were embodied by grain coarsening, and no typical dislocation arrangements were visible in coarsened grains, except for some single dislocations or dislocation tangles.
In the in-between regime, the surface deformation morphology was featured by locally-formed dense SB clusters, and many micro-cracks formed together within SB clusters. The fracture mode was basically cleavage brittle, but microplasticity coincided at the cleavage steps. Remarkable grain coarsening took place as well, and some developing dislocation arrangements, such as thin walls and loose cell structures, formed in a few of coarsened grains.

In the LCF regime, the surface deformation morphology was mainly characterized by the large-scale SBs extending over the whole specimen surface, and cracks or voids formed along these large-scale SBs. A more ductile mixed-mode fracture surface featuring dimples and some cleavage planes was found. Different dislocation structures, such as dislocation walls, dislocation cells, and PSB ladder-like walls can be found to form in many coarsened grains, depending upon the orientation of those coarsened grains.

Fig. 10. Schematics of stress-amplitude-dependent microstructures in fatigued UFG copper. (a) low stress amplitude (i.e. HCF regime), (b) intermediate stress amplitude (regime between HCF and LCF), and (c) high stress amplitude (i.e. LCF regime).


