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Fatigue behavior and damage characteristic of ultra-fine grain low-purity copper processed by equal-channel angular pressing (ECAP)

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Abstract

The S–N and Coffin–Manson plot, cyclic stress–strain response, changes of microstructure, and the surface morphology of ultra-fine grain (UFG) low-purity copper processed by ECAP were tested and observed in present study. And the formation mechanism of shear bands was discussed in detail. The results show that the UFG Cu represents longer lifetime under stress-controlled fatigue, but lower fatigue resistance under strain-controlled fatigue when compared with the coarse grain counterpart. Cyclic stress–strain responses of UFG Cu under stress-controlled fatigue alter from cyclic softening to cyclic hardening as stress amplitude decreases. But the responses always show cyclic softening under strain-controlled fatigue in present testing. By electron back scattering diffraction and transmission electron microscope technique, the shear bands were discovered on the surface of all cycled samples and no grain coarsening was discovered near the shear bands, which indicated that there was no inevitable relationship between formation of SBs and cyclic softening/grain coarsening. The discovery should be related to impurities in copper. The oriented distribution of defects along the shear plane in the last ECAP processing is one of the major mechanisms of SBs formation.

Keywords: Copper; Ultra-fine grain structure; ECAP; Fatigue behavior; Shear bands

1. Introduction

With promising physical and mechanical properties and without contamination or porosity, Ultra-fine grain (UFG) materials prepared by equal-channel angular pressing (ECAP) have drawn considerable interests from researchers during the last two decades [1–4]. The ECAP is a cold/warm working technology, which realizes grain refinement and significant hardening with extremely large imposed strains repeatedly and without fracture in bulk. In the working process of ECAP, extremely fine-grained structure formed by simple shear [1]. ECAP provides new opportunities for comparative study of the properties between bulk UFG materials, which herein are free of porosity, and conventional coarse grain (CG) materials. By ECAP technology, grain was refined to 0.1–1 μm scale along with dislocation hardening, which results in considerable enhancement of material strength and hardness, while its ductility remains sufficient.

Up to now, much work has been carried out mainly on microstructures evolvement, mechanical properties and strengthening mechanism of UFG materials prepared by ECAP [5–11]. In consideration of engineering applications, more and more attentions have been paid to the study of their fatigue properties in recent years [12–31]. Materials tested mainly included copper, aluminium and some of its alloys, magnesium alloys, α-brass, titanium, invar alloy and low-carbon steel. The main experimental results can be summarized as follows. The stress-controlled fatigue life of ECAP materials is longer than that of their CG counterparts; on the other hand, their fatigue life is most often shortened in the case of strain-controlled cycle, in some cases there is no measurable effect [1,2,20]. The shortening of fatigue life under strain-controlled tests is believed to be closely related to the instability of the UFG structure produced by ECAP. Formation of shear bands (SBs) and their cracking are considered to be the most important damage mechanism [18], but the details of SBs formation still remain unclear. Mughrabi

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and Höppel [21] concluded that the formation of persistent-slip-bands-like SBs would not be expected to occur in materials with extremely fine grain size, unless, their microstructure coarsened during cyclic loading in UFG Cu of 99.99% purity. Wu et al. [17–19] studied related changes of SBs microstructure by means of SEM-ECC in UFG Cu of 99.98% purity; no relationship was found between coarsening of microstructure and SBs. Kunz et al. [25] studied the fatigue behavior of UFG Cu of 99.9% purity through EBSD and TEM, the results did not reveal any substantial change of the microstructure caused by fatigue. The results from available literature are ambiguous and full of contradictions. So, it is still necessary to carry out further studies of the fatigue cumulative processes.

As for materials, the purity of Cu used in previous works was very high. For example, the purity of Cu tested by Wu et al. [17–19] was 99.98%, by Mughrabi and Höppel [21] was 99.99% and by Vinogradov et al. [24] was 99.98%. As well known, stability of the microstructure is determined by the details of the dislocation structure, impurities and precipitates. Grain boundaries can be pinned by impurities and precipitates, which is a very common way to stabilize the structure. All these effects can influence the grain/cell boundary mobility and the fatigue lifetime. From this viewpoint, impurities or alloying elements might be of great significance to fatigue resistance. In present work, the commercial copper was of substantially lower purity than those tested in Refs. [17–19,21–29]. It would be more close to engineering applications.

We employed copper of 99.8% purity and processed it by ECAP to study its cyclic deformation behavior under stress-controlled and strain-controlled conditions, respectively. The attention was mainly paid to cyclic stress–strain response, changes of microstructure and surface morphologies after fatigue, in order to shed some light on the mechanisms of cyclic deformation behavior of UFG materials.

2. Experimental material and procedures

The commercial copper of 99.8% purity was used in this study. The chemical composition of the copper is given in Table 1. The billets of 14.5 mm × 14.5 mm × 90 mm were pressed through intersecting at 90° square channel, the pressing was performed with the press velocity of 2 mm/s at room temperature via the so-called “route C”, when the sample was rotated through 180° about working axis between subsequent passes. The pressing strain ε is about 1 for each ECAP pass. The samples for testing were shaped by spark erosion to prepare specimens with the gauge length of 10 mm and the square cross-section of 4 mm × 4 mm. As shown in Fig. 1, the axis of the specimen was aligned with the direction of extrusion. The copper prior to ECAP had nearly equiaxial grain of ~30 μm in average size, which was used in the course of experiments for comparison. The gauge length of specimens was polished carefully to remove the damaged layer from the surface. The fatigue experiments were carried out on a hydraulic servo-electric load frame at loading frequency of 0.5 and 30 Hz for strain-controlled and stress-controlled fatigue, respectively. Tests were performed by strain control under fully reversed tension/compression at constant plastic strain amplitudes, $\varepsilon_{\text{pl}} = \Delta\varepsilon_{\text{pl}}/2$, which were ranged from 2×10^{-4} to 10^{-2} at room temperature in air. The tensile tests were conducted on the same testing machine at the constant strain rate of 1 mm/min.

Table 1
Chemical composition of copper (wt.%)

Element	Bi	Sb	As	Fe	Ni	Pb	Sn	S	Cu
Content	0.02	0.04	0.01	0.03	0.02	0.005	0.04	0.005	Balance

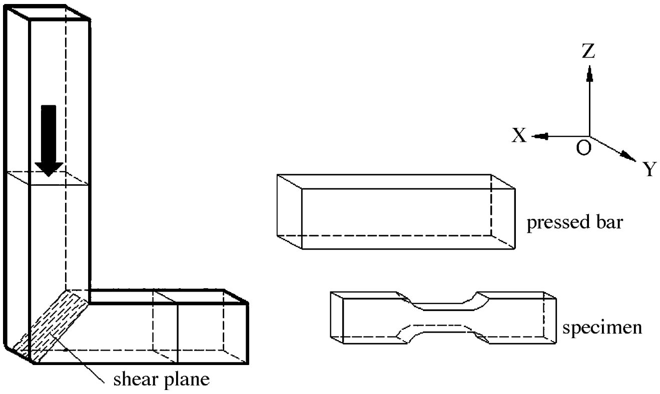

Fig. 1. Illustration of the orientation of specimen to the pressed bar and shear plane of the last ECAP pressing.

A transmission electron microscope (TEM, JEOL 200CX) was used in this experiment at 160 kV for microstructure characterization. Thin foils for TEM observation were prepared by a twin-jet polishing technique using a mixture of 33 vol% nitric acid and 67 vol% methanol. A scanning electron microscope Jeol JSM 6460 with electron back scattering diffraction (EBSD) system INCA Crystal by Oxford Instruments Analytical was applied to measure the grain map. The EBSD procedure is capable of revealing the microstructure in terms of grain orientation. The grain maps were constructed as regions with orientation range within 5°. Another scanning electron microscopy (SEM, FEI Quanta 200) was employed for observation of surface morphologies after fatigue.

3. Results and discussion

As shown in Fig. 2, a fine and uniform structure of the copper, with the average grain size of 305 ± 15 nm, was obtained after six passes of ECAP. The basic mechanical properties of copper are listed in Table 2. The improvement of tensile strength of the 6-pass ECAPed samples is evident when compared with that of the CG counterpart. However, the ductility δ became considerably lower and necking started at very low strains as soon as the ultimate tensile strength was achieved. The increases of the tensile strength of UFG copper might be due to the following reasons. Firstly, the grain size fined with the ECAP passes. The effect of grain size on strength follows the classical Hall–Petch type equation in general, i.e. the yield strength increases with grain size decreasing [32,33]. Secondly, the accumulated plastic strain increased with increasing passes of ECAP for materi-