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Cyclic softening behaviors of ultra-fine grained Cu-Zn alloys

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ABSTRACT

Low-cycle fatigue tests were carried out on ultra-fine grained (UFG) Cu and Cu-Zn alloys to reveal the mechanisms of cyclic softening and the effects of dislocation slip mode. Based on careful examinations of the grain coarsening (GC), shear band (SB) evolutions and surface hardness change during cyclic deformation, the microscopic mechanisms of the cyclic softening process and the correlations between GC and SBs were deeply revealed. Besides, a general and coincident relationship was found between the softening velocities and the fatigue lives for UFG Cu and Cu alloys. Finally, it is approved that through alloying to increase the slip planarity, the cyclic softening caused by GC and SBs can be largely restrained such that the fatigue life may be improved effectively.

1. Introduction

Ultrafine-grained (UFG) materials with grain sizes of several hundred nano meters have been extensively explored for their extremely enhanced strength relative to their coarse grained (CG) counterparts [1,2]. Nevertheless, the sharp reduction in the elongation because of the exhausted work-hardening capacity is commonly accepted as the major drawback for these materials [1–4]. Therefore, many researchers have focused on improving the overall tensile properties through tuning the microstructures, which have made some progresses [1–5]. However, from the prospective of the engineering application, besides the static mechanical properties such as tensile strength and plasticity, the cyclic deformation behavior is also significant, which has not been studied sufficiently in the past for the UFG materials [1,3]. Therefore, it is of great importance from both scientific and engineering views to evaluate the fatigue properties of these materials, especially for the low-cycle fatigue (LCF) behavior, which correlates closely with the tensile plasticity [4,6].

Previous studies on the fatigue behaviors of UFG materials mainly contain Cu and Al and their alloys, Mg alloys, Ti, invar alloy and low-carbon steel [7–19]. The main experimental results can be summarized as follows. The stress controlled high-cycle fatigue (HCF) life of the UFG materials is generally longer than that of their CG counterparts, and the improvement amount increases with the stress amplitude [7–14]. However, in the case of the strain-controlled LCF, the fatigue life of the UFG metals is often shortened under a Manson-Coffin plot [6,12,15]. The deteriorating LCF performance of the UFG materials can be mainly ascribed to their cyclic softening behavior, accompanied microscopically by grain coarsening (GC) and the formation of shear bands (SBs), which are considered to be the two basic damage mechanisms for the UFG materials under cyclic loading [13,16,17]. Therefore, it is of great significance to reveal the mechanism of SBs and GC for better understanding the fatigue behavior of the UFG metals.

At present, the previous researches yield great controversies in the details of the SBs formation and in whether there exist some correlations between SBs and GC. Wong et al. [20] found that SBs resulted from dislocation tangling and the width of SBs was less than grain size. However, more studies [21–25] verified that SBs are thicker than the grain diameter and several grains were embodied in a single SB. Some researchers proposed that SBs originated from the grain boundary (GB) sliding along the shear plane of the last pass of equal-channel angular pressing (ECAP) [21], which was later denied as the observation of several sets of intersecting SBs [26,27]. Mughrabi et al. [12] and Malekjani et al. [28] found grain growth nearby SBs using the scanning electron microscope (SEM) based electron channeling contrast (ECC) technique and Mishra et al. [29] also found dynamic recrystallization along SBs for UFG Cu by using electron back scattering diffraction (EBSD). These studies indicate that a tight connection exists between SBs and GC for the UFG

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materials. However, the essence of this connection and the respective role that SBs and GC play in the cyclic softening process need to be further revealed, which is a primary object for the present study.

Besides, as the low LCF life of the UFG materials can be traced down to the appearance of GC and SBs, to hinder the formation of GC and SBs should contribute to improving the LCF properties [30]. It is well established that the dynamic recovery and recrystallization are thermally activated, which are facilitated by the cross-slip of dislocations [16,17,31]. Accordingly, the LCF life may be increased through adjusting the dislocation slip mode to inhibit the dislocation cross-slip. For example, researchers found that the UFG metals with planar slip mode (e.g. UFG Ti, Mg alloys and Cu-Zn or Cu-Al alloys with high solute contents) show both stable microstructure and relatively high fatigue lives [26,27,32–34]. As to the transition of the dislocation slip mode, researchers found that several factors might be the causes, such as short range order (SRO), stacking fault energy (SFE) and yield stress, among which the SRO is confirmed to play the major role [35,36]. For Cu-Zn alloys used in this study (and also Cu-Al alloys cited in this study [26]), with the increase of Zn or Al contents, the degree of SRO increases and meanwhile the SFE decreases, both of which facilitate the transition of slip mode [35–38]. Therefore, it is significant to figure out whether adjusting the dislocation slip mode through alloying is valid in improving the microstructural stability and LCF life for UFG materials. This is another target of the present study.

Therefore, according to the above introductions, we employed UFG Cu and Cu-Zn alloys to carry out LCF tests in the present investigation, aiming at clarifying two issues: 1) whether there exist correlations between GC and the formation of SBs and what roles do they play in the cyclic softening process; and 2) whether the microstructure stability and LCF properties can be improved through adjusting the dislocation slip mode.

2. Experimental procedures

Three materials, Cu of 99.9% purity, Cu-5at.% Zn and Cu-11 at.% Zn alloys were selected to obtain various SFE values (Cu: 45 mJ/m^2; Cu-5at.% Zn: 38 mJ/m^2; Cu-11 at.% Zn: 33 mJ/m^2) and SRO degrees [35–40]. The two Cu-Zn alloys were first cast into ingots and then hot rolled into rods of 20 mm in diameter, and Cu was supplied by bars of 20 mm in diameter with cold-drawn state. Before pressing, all the bars were cut into rods of 105 mm in length and annealed at 800 °C for 2 h to achieve homogeneous microstructure. Then, all the rods were processed by ECAP for 4 passes using route Bc at room temperature (RT), with a pressing velocity of 20 mm/min.

The tensile and fatigue specimens were cut into shapes of dog-bone by electronic spark-cutting technique with gauge dimensions of 12 mm × 4 mm × 3 mm and 14 mm × 4 mm × 5 mm, respectively. Tensile tests were performed with an Instron 8862 testing machine operating at a strain rate of 10^{-3} s^{-1} at RT, and symmetric tension-compression LCF tests were also carried out on the Instron 8862 testing machine at a frequency of 0.5 Hz controlled by the total strain amplitude. After fatigue tests, the sample surfaces were observed with a LEO Supra 35 field emission SEM, operated at 20 kV. The microstructures of the samples after fatigue tests were characterized by transmission electron microscopy (TEM) with an FEI Tecnai F20 microscope, operated at 200 kV. The thin films for TEM observations were cut parallel to the tensile direction of the samples. To characterize the cyclic softening behavior of the SBs, an AMH-43 Vickers microhardness tester was employed to measure the hardness variation of the fatigued sample surfaces, with a loading of 100 g for 13 s.

3. Experimental results

3.1. Microstructures and tensile properties

The microstructure characteristics of the three materials have been reported in the previous study [41], which will be simply described in the present study. Elongated and equiaxed grains coexist comparably in amounts for Cu and Cu-5at.% Zn, while for Cu-11 at.% Zn, the microstructure was nearly homogenized to the equiaxed grains after ECAP for 4 passes. For Cu-11 at.% Zn, some deformation twin (DT) bundles can be observed frequently because of the relatively low SFE [42,43]. The morphologies of the equiaxed grains of the three materials are shown in Fig. 1a–c. The average grain sizes of the equiaxed grains are ~230 nm–160 nm and ~100 nm for Cu, Cu-5at.% Zn and Cu-11 at.% Zn, respectively.

The tensile engineering stress-strain curves for the three materials are shown in Fig. 1d. It is clear that with increasing the Zn content, the UTS increases obviously and the total elongation remains nearly the same, in accordance with the previous studies on the Cu-Al and Cu-Zn alloys [41–44]. The increased UTS with increasing the Zn content is owing to the more refined grains. This can be explained by the effect of the dislocation slip mode on the grain refinement during severe plastic deformation: with increasing the dislocation slip planarity, cross slip is refined so that the dynamic recovery and recrystallization were also hindered, therefore, the saturated grain size was reduced [41–43].

3.2. Cyclic softening behaviors and Coffin-Manson plots

Fig. 2 shows the relationship between the stress amplitude and the cyclic numbers for the three materials under log-log coordinates (2a-2c) and at three different total strain amplitudes under linear-linear coordinates (2d-2f). Overall, three general trends can be abstracted from Fig. 1) Cyclic softening happened for nearly all the samples of the three materials and it became more serious with the increase of strain amplitude, as also reported by the previous studies [8,26,30]. 2) With the increase of Zn content, the cyclic softening degree is reduced for all the strain amplitudes, as clearly reflected by the slope of the linear segments of the curves in Fig. 2d, e and f. This indicates that increasing the slip planarity facilitates the improvement of the microstructural stability during LCF. 3) Under linear-linear coordinates, the cyclic softening curves show very similar shape, a laid down "S", for all the three UFG materials at all strain amplitudes. This reveals that the softening processes of the UFG materials under LCF follow some general rules associating with similar microscopic damage mechanisms, which will be fully discussed in the next section.

Fig. 3 illustrates the dependence of the reversal numbers ($2N_f$) on the total strain amplitude ($\Delta\varepsilon_t/2$) for the three materials. Although the total strain amplitude rather than the plastic one was used as the vertical axis, it also shows fairly good linear relationships for the three metals under log-log coordinates, indicating that the fatigue damage caused by plastic strain at the low strain amplitude part is comparable to that caused by elastic strain at the high stress amplitude part [6,12]. From Fig. 3, it is obvious that with increasing the Zn content, the fatigue life is overall improved for all strain amplitudes, in accordance with the effect of slip planarity on the cyclic softening behaviors indicated by Fig. 2. However, on closer inspection, the improvement degree of the LCF life is rather different for the high and low strain amplitude parts: the higher strain amplitude, the less improving effect. This may be owing to the nondistinctive tensile elongations of the three metals, as illustrated by Fig. 1d. For further understanding the above LCF behaviors, the surface damage morphologies and microstructure evolution after fatigue require to be further investigated.