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The criteria for microstructure evolution of Cu and Cu-Al alloys induced by cyclic loading

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ABSTRACT

The microstructure evolution behaviors and corresponding quantitative rules of polycrystalline pure Cu and Cu-Al alloys under cyclic push-pull loading with rather large strain amplitudes ($\Delta\varepsilon/2 = \pm 2\% \sim \pm 9.5\%$) were systematically investigated. Besides common dislocation structures, deformation twins (DTs), shear bands (SBs) and ultra-fine grains (UFGs) were observed for the first time in fatigued specimens with certain composition and strain amplitude. Based on careful observation and brief classification, a distribution diagram of above microstructures at various Al contents and strain amplitudes was summarized. Then, three corresponding criteria were proposed by developing theoretical models of microstructure evolution, to provide critical distributions of dislocation structures, DTs and UFGs. The calculated results display good agreements with the experimental observations. Both the experimental results and the theoretical analyses indicate the existence of general rules that dominate the microstructure evolution behaviors under cyclic loading. The investigation of these rules is of great significance for the further achievements on both the fatigue mechanism and the life prediction.

1. Introduction

For metallic materials, microstructure evolution during cyclic deformation is an important issue for the investigation of fatigue behaviors. It significantly influences almost all the fatigue properties, including the procedure of cyclic hardening, the mechanism of fatigue damage and fracture, as well as the fatigue life [1]. Researches on the microstructure evolution under cyclic loading began in the 1900s [2] and became a hot research topic since the 1960s [3]. Among those researches, the most systematic achievement is obtained in single crystals with face-centered cubic (FCC) structure [4–6], which indicates that the stacking fault energy (SFE) can significantly influence the microstructure evolution.

For FCC metals with high SFE, cell structure is the most common dislocation configuration formed under cyclic loading [7–9]. Take pure Al as an example, the microstructures changed from veins to walls/labyrinth and finally to cells with the increase of cyclic stress amplitude [10–13]. In contrast, in materials with medium SFE such as Cu [5,14–16], Ni [17–20] and Ag [21,22], the persistent slip bands (PSBs) [14,15,23–25] emerge as a typical dislocation configuration in a range of stress/strain amplitudes between the region of veins/patches [26] and labyrinth/cells [27–30]. By further decreasing the SFE, the above regular dislocation configurations gradually disappear. Instead, multi-polar dislocation arrays and persistent Lüders bands (PLBs) dominate the microstructure evolution process [16,31,32], as observed in Cu-Al [31,33,34], Cu-Zn [35,36] and high-manganese austenitic steels [37,38].

Researches of these microstructure evolution processes have been conducted not only in the single crystals with various orientations [32,39], but also in bicrystals [40,41] as well as polycrystals including coarse grains [42–46] and ultra-fine grains (UFGs) [47,48]. Besides the special structures formed in grain boundary affected zone, microstructures formed under cyclic loading in coarse grains are similar to that in single crystals with double-slip or multiple-slip orientations [43]. Vein, labyrinth, as well as cells can be frequently observed, while PSBs become less in polycrystals [44]. Relevant investigations on UFGs usually focus on the behaviors of grain coarsening and shear banding [47], which can be reflected by the cyclic softening behavior [48].

According to the above introductions, the microstructure evolution behaviors change with chemical components, loading conditions and original microstructures of materials, and then lead to different fatigue properties. Therefore, it is significant to study the microstructure evolution under cyclic loading systematically and comprehensively. So far, there remain some problems need to be solved to complete this investigation. One of them is about the microstructure evolution of materials under extremely-low-cycle fatigue (ELCF) [49]. As another extreme condition just like the ultra-high-cyclic fatigue (UHCF), the ELCF has its own theoretical

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bases and application prospects [50–53]. However, its microstructure evolution behaviors have rarely been studied: all the achievements mentioned above are obtained under strain amplitudes on the order of magnitude of 10^{-3}. What will happen if the strain amplitude continues to increase to very high levels? Another issue is about the corresponding theory development. Even though there are many laws and regulations summarized from the large amount of experimental results (including some we mentioned above), a uniform rule with clear physical meaning is still a challenging mission. Can we go a step further?

The above two problems can be briefly summarized as: (i) *the conditions of microstructure evolution under larger strain amplitudes*; and (ii) *the uniform rules of microstructure evolution under cyclic loadings*. With such purposes, the microstructure evolution behaviors of pure Cu and single-phase Cu-Al alloys with different Al content were comprehensively investigated in this study, based on our former research on the ELCF properties [54]. Cyclic push-pull loading tests with extremely high strain amplitudes (up to $\pm 9.5\%$) were conducted, followed by careful microstructure representation. Then a summary diagram of typical microstructures at different chemical contents and strain amplitudes was presented based on detailed investigation and brief classification. Finally, three qualitative criteria corresponding to the dividing lines of different microstructures in the diagram were proposed, to give physical explanations of the microstructure evolution behaviors of Cu and Cu-Al alloys under cyclic loading.

2. Experimental procedures

In this study, pure Cu of 99.97% purity and Cu-Al alloys of three different Al contents (Cu-5 at%Al, Cu-8 at%Al, Cu-16 at%Al) were selected. The pure Cu and Cu-Al alloys were cold-rolled and then annealed at 800 °C for 2 h to obtain homogeneous microstructures with an average grain size of $\sim 150\ \mu m$. Push-pull strain-controlled cyclic deformation were then carried out on an Instron 8850–250 kN testing machine with a strain ratio of -1 and a strain rate of $1 \times 10^{-2}\ s^{-1}$ in ambient air at room temperature. As total strain amplitudes were relatively large ($\Delta\varepsilon/2 = \pm 2\%, \pm 4\%, \pm 6\%, \pm 8\%, \pm 9.5\%$), the specimen were machined into podgy round bars (gauge dimension: $\Phi 10\ mm \times 12\ mm$) to ensure the stability during cyclic deformation [54]. The deformed microstructures were characterized by transmission electron microscopy (TEM) with an FEI Tecnai F20 microscope, operated at 200 kV. Thin foils for TEM observations were firstly cut from the fatigue specimens parallel to the loading axis by a wire cutting machine, with an original thickness of $300\ \mu m$, then were mechanically reduced to about $50\ \mu m$, following with a twin-jet polishing in a solution of $H_3PO_4:C_2H_5OH:H_2O = 1:1:2$ (vol.) with a voltage of 8–10 V at -6 °C.

3. Experimental results

Due to the large strain amplitudes, microstructures such as deformation twins (DTs), shear bands (SBs) and UFGs, which usually form in tensile tests or even under the severe plastic deformation (SPD) methods can be observed in our fatigued specimens. In this section, these special microstructures, as well as typical dislocation structures, will be introduced in detail.

3.1. Typical dislocation structures

Despite other microstructures, dislocation structures are still the most common and basic microstructures in the post-fatigue samples for both pure Cu and Cu-Al alloys. However, unlike the well-developed dislocation configurations formed after common fatigue tests, these dislocations cannot be fully evolved to stable structures due to the extremely short fatigue lives(less than 1000 cycles) [54]. These primitive dislocation structures can be roughly classified into walls, cells, bands and planar arrays according to their morphologies, which vary with the Al contents and loading conditions.

Dislocation walls (usually the labyrinth structures) were observed in some grains of pure Cu and Cu-5 at%Al (Fig. 1(a)-(b)). The walls are thinner and more regular in pure Cu than in Cu-5 at%Al, and become thicker and less regular when increasing the strain amplitude. Dislocation cells (Fig. 1(c)-(d))are the most common structure for pure Cu and Cu-5 at%Al, and they also exist in Cu-8 at%Al under rather large strain amplitudes ($\Delta\varepsilon/2 = \pm 6\%$, $\pm 8\%$). Just like the way the dislocation walls change, the boundaries of cells turn thinner with the decrease of Al content or strain amplitude. Besides, dislocation bands (Fig. 1(e)-(f)) are the typical dislocation structure of Cu-8 at%Al, which can also be found in some grains of Cu-5 at%Al. These structures look very similar to the band-shaped microstructure formed by one-pass ECAP [55], with sub-grain boundaries as edges of the bands. In contrast, dislocations in Cu-16 at%Al are generally scatter in grains and roughly distribute in planar arrays (Fig. 1(h)). This kind of arrays also exist in Cu-8 at%Al with relatively low strain amplitudes ($\Delta\varepsilon/2 = \pm 2\%$, $\pm 4\%$). Moreover, dislocation lines become straighter with the increase of Al content. In some regions of Cu-16 at%Al, dislocations can even expand and form narrow stacking faults (Fig. 1(g)).

According to above experimental results, the dislocation structures vary with both the Al content and the strain amplitude, and the former plays a relatively more crucial role. With increasing the Al content, the typical dislocation structures transform from walls and cells to bands and planar arrays, and finally to narrow SFs. The strain amplitude has similar but weaker influence on dislocation structure evolution as alloying component. Taking the Cu-8 at%Al as an example, the typical dislocation structures change gradually from bands to cells with the increase of strain amplitude. Both of the factors are relevant to the change of dislocation slip mode, which can be considered as the intrinsic mechanism of all the above phenomena. Further discussions will be conducted in Section 4.1.

3.2. Typical twinning structures

For FCC metals and alloys, deformation twinning can rarely happen during common high- or low-cycle fatigue processes, probably for their small stress/strain amplitude. In our tests, DTs have been observed in Cu-5 at%Al specimens at strain amplitudes larger than $\pm 4\%$ and all the Cu-8 at%Al and Cu-16 at%Al specimens (the smallest strain amplitude is $\pm 2\%$). In comparison with the DTs formed under tensile loading, DTs formed in fatigue tests share the similar rules in the way the Al content and the loading condition influence the amount and morphology of DTs [56]. For example, as displayed in Fig. 2(a)-(c), for specimens deformed under the same strain amplitude ($\pm 8\%$ for instance), DTs can only be observed in several small regions in Cu-5 at% Al alloy but become widespread in Cu-16 at%Al alloy.

Despite above similarities, DTs formed during cyclic loading display some unique features. One is the popularity of secondary twins. Under cyclic loading conditions, secondary twinning occurs more frequently than under tensile tests, which may be owing to the large accumulative strain and cyclic hardening effect. Based on a simple laminated structure (see Fig. 2(d)), the appearance of secondary twins introduces lamellas in another direction, initiated from (Fig. 2(e)) or cross through (Fig. 2(f)) the initial structures. Another notable feature is the emergence of de-twinning behavior.