Materials Science & Engineering A 609 (2014) 273–282

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Texture and fatigue behavior of ultrafine grained copper produced by ECAP

Oscar Fabián Higuera-Cobos [a,b,*], Jonathan Antonio Berríos-Ortiz [c], José María Cabrera [a,d]

[a] Departamento de Ciencia de los Materiales e Ingeniería Metalúrgica, ETSEIB – Universidad Politécnica de Cataluña, Av. Diagonal 647, 08028 Barcelona, Spain
[b] Facultad de Ingeniería Mecánica, Universidad Tecnológica de Pereira, Vereda La Julita, Pereira, Colombia
[c] Facultad de Ingeniería y Arquitectura, Escuela de Ingeniería Mecánica, Universidad de El Salvador, El Salvador
[d] Fundació CTM Centre Tecnológic, Plaça de la Ciència 2, 08243 Manresa, Spain

ARTICLE INFO

Article history:
Received 19 March 2014
Received in revised form
5 May 2014
Accepted 6 May 2014
Available online 14 May 2014

Keywords:
Equal channel angular pressing (ECAP)
Inverse Pole Figures (IPF)
Copper
Texture
Orientation Distribution Function (ODF)
High-cycle fatigue

ABSTRACT

Electrolytic Tough Pitch (ETP) and Fire Refined High Conductivity (FRHC) copper samples were severely deformed by equal channel angular pressing (ECAP) and the effect of plastic deformation on the microstructure, texture and fatigue was investigated. The microstructural behavior was determined by analysis of the final texture through Inverse Pole Figures (IPF) and Orientation Distribution Function (ODF) maps, which revealed a marked decrease in the normal grain size of the ECAPed coppers and also the presence of recrystallization processes. The effect of the initial texture on the evolution of the texture after 8 ECAP passes for the two coppers was also analyzed. The results showed that the annealed materials presented a marked anisotropy, with a texture controlled by the ⟨110⟩ fiber. Additionally, the fatigue behavior of ultrafine grained coppers produced by ECAP was discussed briefly with respect to the macroscopic shear banding and fatigue lives. Experimental results also suggest that the fatigue behavior of ultrafine grained coppers processed by ECAP is quite different from conventional grained coppers, as the deformed materials presented a significant increase in the fatigue limit.

1. Introduction

Presently, due to the rapid development of the electrical and electronic industries, there is a high demand for materials that combine both high strength and good electrical conductivity, which has led to the development of pure Cu or Al with ultrafine or nanometric grain sizes. Such materials can be developed through severe plastic deformation (SPD) techniques [1]. Among the SPD methods, the equal channel-angular pressing (ECAP) technique is considered the most effective in terms of the amount of material used for the production of bulk metals with ultrafine grains [2]. Through this technique a large amount of shear strain is introduced in the processed samples, with no change in the shape of the workpiece, modifying the microstructure of the processed material and resulting in improvements in both the physical and mechanical properties (tensile/compression strength, yield stress, elongation and fatigue life).

Besides the improvements in the physical and mechanical properties, as a consequence of the ECAP deformation, the materials develop a crystallographic texture, due to crystal reorientation, which is related to the dominant slip systems given by the crystallographic structure. Also, additional texture components may be developed, depending on the processing conditions and on the occurrence of the recrystallization processes or mechanical twinning. The evolution of the deformation texture with strain has been subjected to various investigations [3–5].

Recently, research focused on fatigue of ultrafine grained materials has increased noticeably, due to the key influence of fatigue properties on the use of these materials in practical applications. Fatigue can be associated with cumulative damage processes in materials, resulting in the fracture of a given material under the application of cyclical stress levels below the static tensile strength. Total fatigue life has conventionally been divided into two regions corresponding to the time required: first for crack nucleation and then for crack propagation. In order to delay the initiation of cracks, the materials should exhibit high mechanical resistance, while large ductility allows greater tolerance to the propagation of the crack. Ultrafine grained materials obtained by SPD, which can be associated with a combination of high strength and good ductility, suggest the possibility of a significant improvement in fatigue life. A correlation

* Corresponding author at: Departamento de Ciencia de los Materiales e Ingeniería Metalúrgica, ETSEIB – Universidad Politécnica de Cataluña, Av. Diagonal 647, 08028 Barcelona, Spain. Tel.: +34 57 63137124x111; fax: +34 57 63137362.
E-mail addresses: osfahico@gmail.com (O.F. Higuera-Cobos), jose.maria.cabrera@upc.es (J.M. Cabrera).

http://dx.doi.org/10.1016/j.msea.2014.05.011
0921-5093/

between the cyclic deformation and the fatigue properties of ultrafine grained (UFG) materials showed that the fatigue behavior depends strongly on the parameters of the ECAP procedure, the purity of material and the type of fatigue loading [6]. As the low-cycle fatigue (LCF) behavior of ultrafine grained materials is generally inferior to that of their coarse grained counterparts, and the high cycle fatigue (HCF) resistance is generally significantly enhanced [7], in this investigation both Electrolytic Tough Pitch (ETP) and Fire Refined High Conductivity (FRHC) coppers, severely deformed by ECAP, were analyzed in the HCF domain. Also, a correlation between texture and fatigue behavior was investigated.

2. Experimental procedure

Cylindrical specimens of 10 mm diameter and 60 mm length from ETP and FRHC coppers with a 99.97% and 99.81% purity, respectively, were initially subjected to annealing at 600 °C in inert atmosphere (Argon) for 30 min. Subsequently, the materials were subjected to severe plastic deformation by the ECAP technique at room temperature to a maximum equivalent strain of 8 (i.e. 8 passes), following route Bc (which implied a sample rotation of 90° along its longitudinal axis in the same direction after each pass [8]). For the ECAP process, the die used (Fig. 1) was manufactured with a tool steel insert where two channels intersect at an internal angle of $\Phi=90°$ and an external angle of $\Psi=37°$, resulting in an approximated deformation per pass of 1 ($\varepsilon \sim 1$) [9]. The extrusion rate was 0.02 m/s, and molybdenum disulfide was used as a lubricant. The microstructures and texture were characterized by electron backscatter diffraction (EBSD). The samples were cut from the center of the ECAP billets and mechanically polished using 0.02 μm colloidal silica solution following standard metallographic procedures. EBSD measurements were performed using a scanning electron microscope (SEM) with a Field Emission Gun JEOL JSM-7001F (at a voltage of 20 kV) operating with the Oxford Instruments

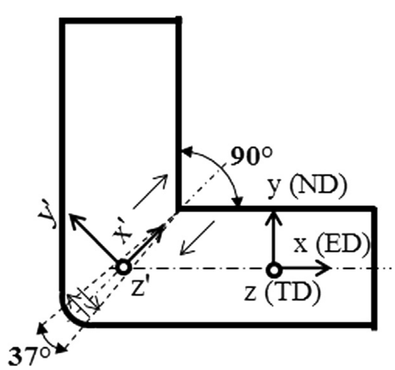

Fig. 1. Die configuration and the reference system used in this study.

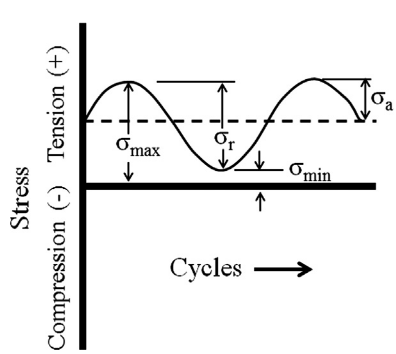

Fig. 2. Repeated stress cycle of the fatigue behavior applied in this study [11].

HKL Channel 5 software. The texture was represented by the orientation distribution function (ODF), because it provides a better representation of the texture components than pole figures and presents the small changes in the location of the texture components [10]. Different step sizes were used, as 0.4 μm for the annealed coppers and 0.05 μm for the samples with 8 ECAP passes.

Prior to fatigue tests the copper samples were subjected to microtensile tests in the longitudinal direction at room temperature. Tensile specimens were machined with standard gauge dimensions of 4 × 1 × 1 mm and the tests were performed in a Microtest DEBEN machine at a cross-head speed of 3.3×10^{-3} mm/s. As for the study of the fatigue behavior of the ECAPed samples and their comparison with the annealed state, fluctuating stresses were applied, i.e. the stress ratio between minimum and maximum was $R=\sigma_{min}/\sigma_{max}=0.1$. It should be noted that both the minimum and maximum stresses applied were tensile stresses, as shown in Fig. 2. All the experiments were performed at room temperature.

The stress range was deduced as the algebraic difference between the maximum and minimum stresses of each cycle [11], as can be seen in Fig. 2.

$$\sigma_r = \sigma_{max} - \sigma_{min} \tag{1}$$

The stress amplitude was defined as being equal to half of the interval

$$\sigma_a = \sigma_r/2 \tag{2}$$

The dimensions of the fatigue specimens were machined according to the ASTM E466 standard. Machining was carried out in a numerically controlled lathe, using low cutting depths in order to minimize the introduction of residual stresses (see Fig. 3). Afterward, the samples were longitudinally sanded with SiC 2500 sandpaper in order to eliminate the circumferential notches produced during machining and then circumferentially polished using abrasive disk plush and abrasive paste. Fatigue tests were performed on a RUMUL 654 machine with axial loading, having a maximum load of 10 kN and controlled by mechanical resonance at a high operating frequency of 150 Hz.

3. Results and discussion

3.1. Microstructural characterization

Fig. 4 summarizes the microstructural behavior of the two studied materials, both in the annealed state and deformed after 8 ECAP passes. The two materials in annealed state exhibit a heterogeneous microstructure composed of different grain sizes with equiaxed grains among which elongated grains can be found. These elongated grains are associated with special grain boundaries of the twin type, resulting from the annealing process to which they were subjected (see Fig. 4a and b). As for the materials after 8 ECAP passes (see Fig. 4c and d), the heterogeneous microstructure was maintained and the presence of large twinned grains can also be observed.

After the ECAP process, the coarse grains were refined presenting an average size of 2.3 μm for the ETP copper and 1.54 μm for the FRHC copper. Regarding the ultrafine matrix, the two materials exhibit very close grain sizes. This behavior is similar to the one identified by Etter et al. [12] on a commercially pure copper and by Huang et al. [13] on an oxygen free high conductivity copper (OFHC), where their microstructures after 8 passes presented large grains free of dislocations and mostly twinned. Wang et al. [14] associated the presence of large grains with the onset of the dynamic discontinuous recrystallization process, even at room temperature, as the driving force for the grain boundaries' migration is provided due to the large amount of dislocations generated