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Dislocation density of pure copper processed by accumulative roll bonding and equal-channel angular pressing

Yoji Miyajima [a,*], Satoshi Okubo [a], Hiroki Abe [a,1], Hiroki Okumura [a,2], Toshiyuki Fujii [b], Susumu Onaka [a], Masaharu Kato [a]

[a] Department of Materials Science and Engineering, Graduate School of Science and Engineering, Tokyo Institute of Technology, 4259-J2-63, Nagatsuta-cho, Midori-ku, Yokohama 226-8502, Japan
[b] Department of Metallurgy and Ceramics Science, Graduate School of Engineering, Tokyo Institute of Technology, 2-12-1-S8-7, Ookayama, Meguro-ku, Tokyo 152-8552, Japan

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ABSTRACT

The dislocation density of pure copper fabricated by two severe plastic deformation (SPD) processes, i.e., accumulative roll bonding and equal-channel angular pressing, was evaluated using scanning transmission electron microscopy/transmission electron microscopy observations. The dislocation density drastically increased from ~10^{13} m^{-2} to about 5×10^{14} m^{-2}, and then saturated, for both SPD processes.

1. Introduction

Ultrafine grained (UFG) metals fabricated by severe plastic deformation (SPD) processes having the grain size d less than 1 μm have been widely investigated because of their high strength [1]. Such high strength can be understood qualitatively by both the high density of grain boundaries and high dislocation density ρ via grain refinement strengthening and dislocation strengthening, respectively. Although many papers report grain size d, the number of papers reporting the dislocation density ρ is limited [2–5]. This is because the experimental procedure for evaluating ρ is relatively complicated compared with that for evaluating d.

Among several methods to evaluate ρ, such as, X-ray diffraction line profile analysis (XLPA) [6,7], differential scanning calorimetry (DSC) [6], electrical resistivity measurements [3], hardness measurements [8], and transmission electron microscopy/scanning electron transmission electron microscopy (TEM/STEM) [2,4], the TEM/STEM method is the only one by which dislocations are observed directly. The dislocation density obtained by the TEM/STEM method is often said to be smaller than the real density: some dislocations may disappear from the surface of the thin foil specimen due to the image force, or, some dislocations satisfy the invisible condition for the diffracted electron beam. Nevertheless, we believe that the advantage of the direct observation compensates for the possibility of underestimation of ρ.

As far as the authors know, there is only one systematic TEM/STEM study on the change in ρ in SPD processed aluminium, i.e., 2N–Al (purity of 99%) [4], whereas, no systematic investigation has been reported for copper apart from a few reports of relatively-highly deformed copper by the SPD process [2,9,10]. Therefore, the aim of this study is to systematically evaluate ρ of SPD processed pure copper using TEM/STEM.

2. Materials and methods

Pure Cu sheets and Cu rods having the purity of 99.99% (4N–Cu) annealed at 873 K for 2 h (7.2 ks) were subjected to an accumulative roll bonding (ARB) and an equal-channel angular pressing (ECAP) processes, respectively [1,11,12]. Both SPD processes were performed at room temperature. The initial grain size of 4N–Cu is about 35 μm.

The ARB process consists of four steps; cutting a metal sheet into two pieces, cleaning with acetone and subsequent wire brushing on one side of each sheet, stacking the two pieces of sheet as the two brushed surfaces stack, and roll bonding with the rolling reduction of 50%. Fig. 1(a) shows the schematic diagram of ARB and its sample

* Corresponding author.
E-mail address: miyajima.y.ab@m.titech.ac.jp (Y. Miyajima).
[1] Current affiliation: Denso Corporation, Kariya 448-8661, Japan.
[2] Current affiliation: Mitsubishi Materials Corporation, Tokyo 100-8117, Japan.

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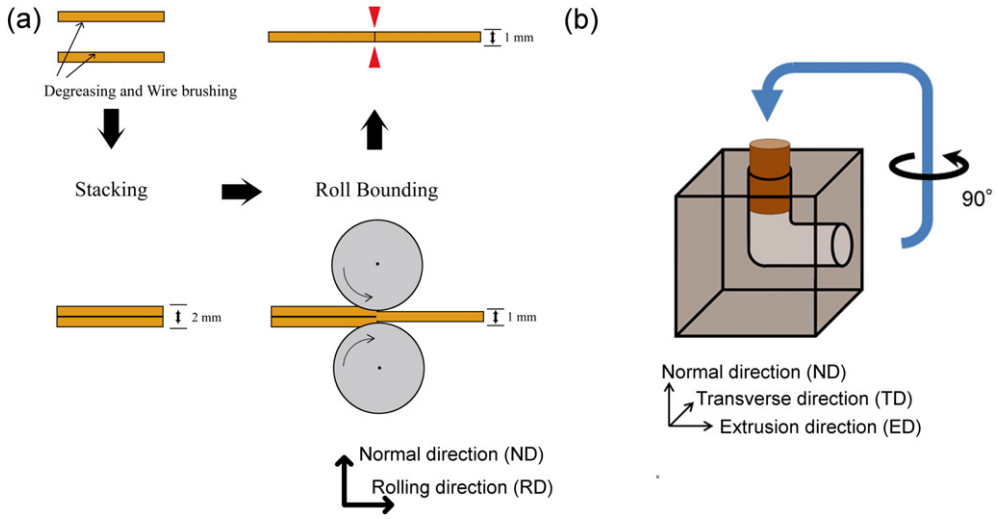

Fig. 1. Schematic illustration of (a) accumulative roll bonding process and (b) equal-channel angular pressing process. The sample coordinates were also displayed.

coordinates. Rolling, transverse and normal directions are denoted as RD, TD and ND, respectively. Here, TD is perpendicular to both ND and RD. In this study, the rolled sheet was immediately water quenched in order to avoid the effect of process heat during the roll bonding. The surface of the rolls used for the ARB process was lubricated by machine oil, and the ARB process was applied up to eight cycles. Hereafter, the ARB *N* cycle sample is denoted as ARB *N*c, and *N* is the ARB cycle number. The detail of the ARB can be found elsewhere [2–4,11,12]. The initial sizes of the sheets were 1 mm, 90 mm, and 400 mm for the thickness, the width, and the length, respectively.

The ECAP process was performed using a metal die having a channel path bent 90°. The cross sectional shape of the path is circular, and extrusion direction (ED), TD and ND are defined as shown in Fig. 1(b). The sizes of the cylindrical samples were 10 mm and 60 mm for the diameter and the length, respectively. The cylindrical samples were inserted into the path via route B_c, in other words, the cylindrical samples were rotated by 90° around the height direction at each pass. The channel of the metal die and the cylindrical samples was lubricated by molybdenum disulphide. The ECAP process was applied up to 12 passes. Hereafter, ECAP *n* passes sample is denoted as ECAP *n*p.

Both the ARB and the ECAP processed 4N–Cu were observed by STEM and TEM including high-voltage electron microscopy (HVEM). JOEL JEM-2100F (STEM mode) with the acceleration voltage of 200 kV with Gatan bright field (BF) detector was used for STEM, whereas, Hitachi H1250 with the acceleration voltage of 1000 kV and JOEL JEM-2100F (TEM mode) with the acceleration voltage of 200 kV were used for TEM.

All the TEM and STEM specimens were cut from both the ARB and the ECAP processed samples using an electrical wire discharge machine, Brother HS-300, as the observations were performed from TD. The surface of the specimens was mechanically polished using SiC paper until the thickness of the specimen becomes about 150 μm. The specimens were also electrolytically polished using Struers TenuPole 3 with the applied voltage at 7 V in the mixture of distilled water, phosphoric acid, and ethanol for 7:2:1 in volume at 273 K until the thickness becomes around 20 μm. Subsequently, the specimens were electrolytically polished with the applied voltage at 7 V in the mixture of nitric acid and methanol for 1:3 in volume at 223 K for perforation.

Apart from annealed 4N–Cu and ECAP 1p, seven grains satisfying diffraction conditions for visible dislocations in the grains were observed by STEM in order to evaluate dislocation density ρ of the specimen with Ham's intersection method [13]. Whereas, ρ of the annealed 4N–Cu and ECAP 1p were measured from only one grain since the grain size was too large to measure several grains. The procedure for Ham's intersection method is schematically shown in Fig. 2. First, a mesh is drawn on a TEM image of a grain satisfying the diffraction condition, and the total length L of the mesh is measured. Secondly, the number of intersections m between the mesh and dislocations are counted. Finally, Eq. (1) is used to evaluate ρ.

$$\rho = \frac{2m}{Lt} \tag{1}$$

Here, the thickness of the specimen t was evaluated from thickness fringes which appear at a high-angle grain boundary (HAGB) under two-beam condition or systematic reflection [4]. It is known that the thickness fringes appear periodically with discrete depth, the so-called extinction distance ξ_g [14], and therefore, the specimen thickness can be evaluated once the number of thickness fringes at HAGB is counted.

Table 1 shows some relationships between the reflection and ξ_g for Cu, which is copied from Table A.4.2 in reference [14]. It is pointed out that the values of ξ_g in the literature are for 100 kV electrons, but, the acceleration voltage of TEM used in this study is 200 kV. Thus, the values of

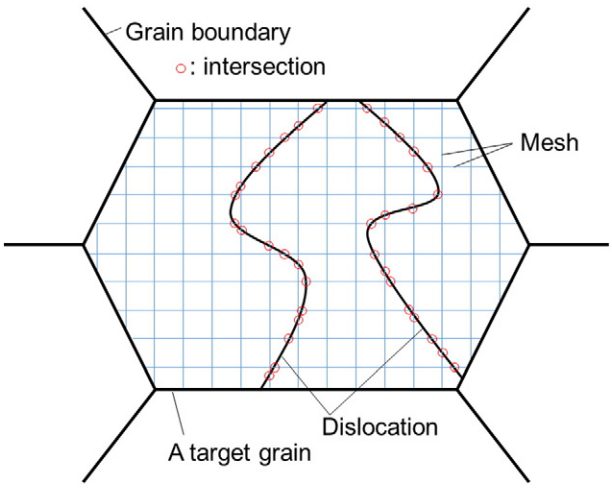

Fig. 2. Schematic illustration of Ham's method. The thick lines represent grain boundary, mesh consists of thin lines, small circles indicates the intersections, and bent lines are dislocations.