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Remarkable transitions of yield behavior and Lüders deformation in pure Cu by changing grain sizes

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Fully recrystallized ultrafine-grained (UFG) pure Cu specimens were fabricated by high-pressure torsion (HPT) and controlled annealing. The recrystallized UFG Cu with a minimum mean grain size of 0.51 μm showed high yield strength, good ductility, obvious yield drop and large Lüders strain during tensile test. The mechanical behavior of the Cu specimen became sensitive to the change in the grain size from 0.2 μm to 4.2 μm. The continuous transitions of yield behavior and Lüders deformation with grain sizes were discussed.

Pure Cu has been widely employed as a model material to investigate the fundamental deformation mechanisms and mechanical properties of face-centered cubic (FCC) metallic materials [1–3]. High strength can be obtained by refining the grain size, but the ductility also deteriorates with ultra grain refinement, which is known as the trade-off relationship [4,5]. Even though, superior balance between strength and ductility has been gained in Cu specimens with various microstructures, including nano-twinned structure [6], gradient structure [4], laminated structure [7], and bimodal structure [8]. In contrast to such specimens with well-designed microstructures, Cu specimens with homogeneous microstructures have been extensively studied in the coarse-grained regime (grain size larger than 3 μm, fully recrystallized) [9] and ultrafine-grained (UFG) regime (grain size smaller than 0.5 μm, plastically deformed) [10].

However, there are only few studies on UFG Cu with recrystallized structures since recrystallized UFG grains have been difficult to obtain by conventional plastic deformation and subsequent annealing treatment [11]. Recent research results in some metals and alloys indicate that the yield strength changes greatly when the grain size approaches 1 μm, and superior strength-ductility can be obtained when the recrystallized grain size is smaller than 1 μm [12,13]. Besides, some unconventional phenomena appear which can be related to the grain structure and dislocation density [14,15], i.e. clear yield drop and Lüders deformation in Al and Al alloys [5,16,17] and interstitial free (IF) steels [16]. Furthermore, these recrystallized UFG materials can even possess higher fatigue strength in contrast to the severely deformed counterparts [18,19], indicating that they can be potential candidates for industrial applications. It is thus meaningful to fabricate the recrystallized UFG Cu and reveal the corresponding deformation mechanisms.

In this study, pure Cu is processed by the high-pressure torsion (HPT) technique which can impose extremely high strain to refine the grain size [20,21]. Subsequent annealing treatment is carefully manipulated to fabricate Cu specimens with different grain sizes and dislocation densities. Tensile tests are conducted to reveal the mechanical responses of the Cu specimens by changing the grain structures.

Cu with a commercial purity of 99.9% was investigated in this study. The starting plate was 20 mm in thickness and had a mean grain size of 100 μm. HPT disks with diameter of 30 mm and thickness of 1.7 mm were processed by HPT at ambient temperature. All the disks were processed to 5 revolutions under an imposed pressure of 5 GPa. After HPT, the disks were then isochronally annealed for 5 min at 325 °C, 375 °C, 450 °C, 550 °C and 600 °C, respectively. All the specimens were annealed in a salt bath and then immediately quenched in water. After annealing, microstructural characterizations were undertaken by using a field-emission scanning electron microscopy (FE-SEM, LEO SUPRA 35) equipped with an electron backscattering diffraction (EBSD) system operated at an accelerating voltage of 20 kV. Microstructure

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characterization by EBSD was conducted on the transverse section of the HPT disks. The mean grain sizes of these specimens were measured by a line intercept method, and all the high-angle grain boundaries (HAGBs) including twin boundaries (TBs) were counted. Tensile specimens with gauge length of 4 mm, width of 1 mm and thickness of 1.5 mm were cut from the HPT disks. Tensile strain was accurately measured by using a digital image correlation (DIC) method, and the detail of this small-size tensile test was described elsewhere [22]. Tensile tests were conducted at an initial strain rate of 8.3×10^{-4} s^{-1} at ambient temperature, and two specimens were tested for each condition. The microstructures were studied at the positions of which distance from the center was larger than 5 mm to avoid the microstructural inhomogeneity in the near center regime. The center of the gauge part in the tensile specimens was also coincided to the position larger than 5 mm from the center of the disks.

Cu specimens having various mean grain sizes could be fabricated by the HPT and subsequent annealing process. Some specimens had deformation microstructures, but the other ones were fully recrystallized, as will be discussed later. Fig. 1 shows distributions of the grain size and misorientation angle for the Cu specimens processed by HPT and subsequent annealing treatments. The mean grain sizes were measured after HPT (0.20 μm) and after annealing at 325 °C (0.30 μm), 375 °C (0.51 μm), 450 °C (0.84 μm), 550 °C (2.43 μm) and 600 °C (4.20 μm). The values in the brackets were the mean grain sizes measured by counting HAGBs including TBs. With increasing annealing temperature, the grains grew steadily and the grain size distribution was shifted rightwards, as shown in Fig. 1a. In each case, the grain sizes distribute in a narrow range and roughly follow the normal distribution, indicating a process of homogeneous grain growth. Fig. 1b shows the misorientation angle distributions for the Cu specimens with different mean grain sizes. For the as-HPT specimen, the fraction of HAGB (f_{HAGB}) was 0.806 and the fraction of TB (f_{TB}) was only 0.048, because the as-HPT specimens involved many dislocations and low-angle dislocation boundaries and no annealing twins formed at this stage. When the HPT specimen was annealed at 325 °C for 5 min, f_{HAGB} increased to 0.875 and f_{TB} to 0.153, which could be explained by recovery, recrystallization, and formation of annealing twins. With increasing annealing temperature, f_{HAGB} reached a stable value of about 0.95; meanwhile, f_{TB} drastically increased to 0.373 at 375 °C and to 0.586 at 550 °C, indicating the fraction of annealing twins increased with grain growth process. The change of the TB fraction with annealing temperature is consistent with a Cu-6.8 wt%Al alloy processed by cold rolling and annealing treatments [12].

Tensile engineering stress-strain curves of the Cu specimens after HPT and subsequent annealing are shown in Fig. 2. For the as-HPT sample ($d = 0.2$ μm), the tensile stress-strain curve showed a high peak stress at a small tensile strain, and gradual decrease in the flow stress owing to necking, which was a typical feature of severely deformed materials and due to the high-density dislocations that limited strain-hardening capability. When the grain size was 0.3 μm, the tensile stress-strain curve exhibited a sharp drop of the stress and then gradual decrease in the flow stress due to necking of the specimen. The elongation to failure (18%) was even smaller than the as-HPT sample (25%), which could be attributed to the removal of mobile dislocations during annealing at 325 °C [23]. When the grain size increased to 0.51 μm, the tensile stress-strain curve showed an obvious yield drop and an unprecedentedly large Lüders strain (11%), which was also observed in the UFG pure Cu processed by spark plasma sintering (SPS) technique [11]. In addition, this specimen regained good strain-hardening capability, so that necking was significantly postponed. The upper yield strength approached to 348 MPa while the strain at the necking initiation point (marked by open square symbols in Fig. 2) was 0.184, indicating that a good balance of strength and ductility could be realized in this UFG specimen. When the grain size furthermore increased to 0.84 μm or 2.43 μm, the yield drop phenomenon still appeared but the Lüders strain decreased, indicating that the Lüders strain had an inverse dependence on the grain size [24]. The yield point phenomenon and Lüders deformation disappeared when the grain size became 4.2 μm, which was a typical stress-strain behavior in CG Cu.

Note that there was a sharp increase of tensile elongation when the mean grain size increased from 0.3 μm to 0.51 μm, which is supposed to be related to the change in microstructures by the annealing treatment. Fig. 3 shows the grain average misorientation (GAM) maps obtained by the EBSD analysis for the samples with different grain sizes of 0.2 μm, 0.3 μm and 0.51 μm. The higher GAM value corresponds to the higher

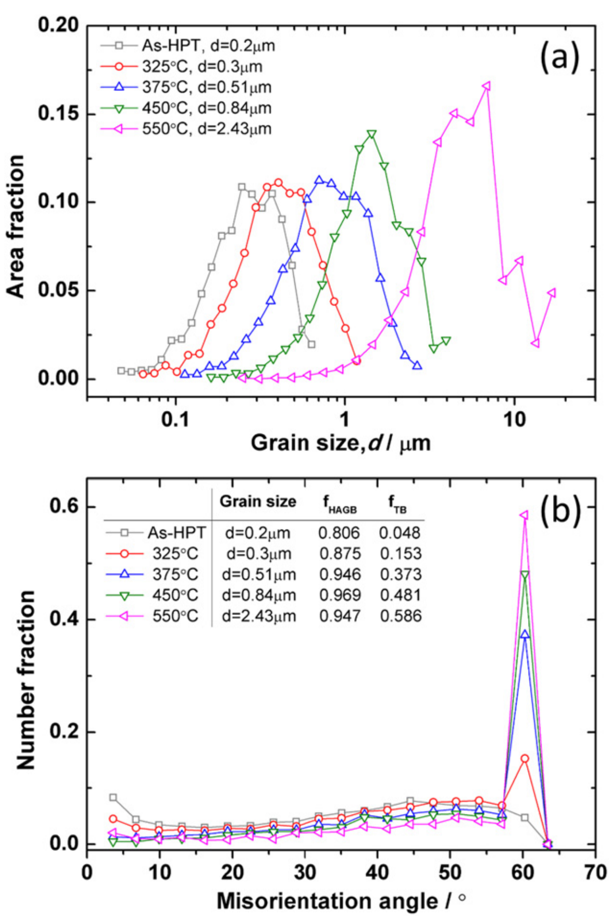

Fig. 1. Distributions of (a) grain size and (b) misorientation angle for the Cu specimens processed by HPT and subsequent annealing. f_{HAGB} and f_{TB} in (b) are related to the fraction of high-angle grain boundaries (HAGB) and twin boundaries (TB), respectively.

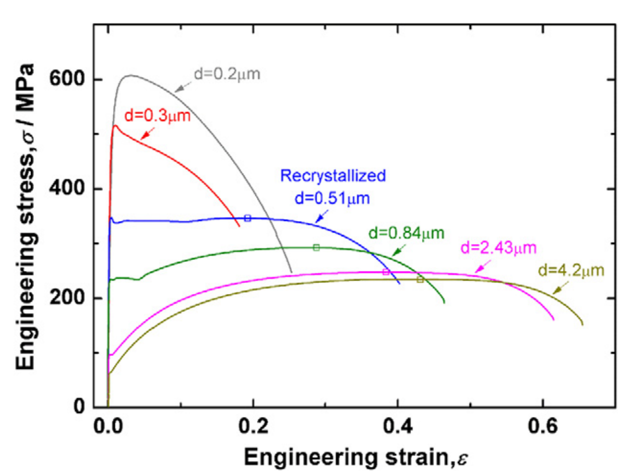

Fig. 2. Tensile engineering stress-strain curves of the Cu specimens with different grain sizes after processing by HPT and annealing. The necking initiation points were marked by open square symbols.