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Influence of gradient structure volume fraction on the mechanical properties of pure copper

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ABSTRACT

This paper reports the influence of gradient structure volume fraction on the tensile mechanical behaviors of pure copper processed by surface mechanical attrition treatment at cryogenic temperature. Superior combinations of tensile strength and ductility are observed in a certain volume fraction, in which strain hardening uprising after yielding is also observed. The gradient structure produces a synergetic strengthening and extra work hardening. These findings suggest the existence of an optimum volume fraction of gradient structure for the best mechanical properties.

1. Introduction

Making materials showing both ductility and hardness has been an important issue for materials science [1–3]. Severe plastic deformation (SPD) techniques have been extensively investigated to produce ultrafine-grained or nanocrystalline materials over several decades. Grain refinement in bulk metals can produce very high strength but disappointingly limited tensile ductility.

Recently, gradient structures (GS) have been introduced into some metals and produced excellent strength and ductility [4–7]. GS in materials exhibit a macroscopic gradual change in microstructure from surface to the core material. Gradient structures have been evolved over millions of years in nature and contributed to biological optimization against severe natural environments [8–10]. However, the investigation of the relationship between microstructures and mechanical properties, from the atomic to the macro-level and their interactions, has not garnered scientific attention until quite recently [11]. Further, there are limited studies analyzing the influence of the volume fraction of the GS layer on the tensile behaviors.

In this study, we produced GS layer at the top surfaces in commercial purity copper by surface mechanical attrition treatment (SMAT) at cryogenic temperature. The dynamic recovery and local recrystallization were greatly suppressed during cryogenic deformation [12]. In a certain volume fraction range, a synergetic strengthening effect and strain hardening uprising were observed.

2. Experimental

Copper samples were first prepared by rolling commercially-pure copper (99.995 wt%) and then annealing in vacuum at 873 K for 2 h to obtain homogeneous coarse grains. Four different sample thicknesses (2, 3, 4, and 5 mm) were chosen for this study to systematically vary the volume fraction of GS layers. These samples were polished to a mirror finish before SMAT treatment.

Details of the SMAT process have been described in a previous work [13]. Briefly, 180 stainless steel balls with 8 mm in diameter were placed at the bottom of a cylinder-shaped chamber and vibrated with a frequency of 50 Hz. Both sample sides were processed at cryogenic temperature.

After the SMAT process, dog-bone-shaped tensile specimens with a gauge length of 15 mm and width of 5 mm were cut using wire electric discharge machining. Tensile tests were performed on a Shimazu Universal Tester at room temperature under a strain rate of $5.0\times10^{-4}\ s^{-1}$.

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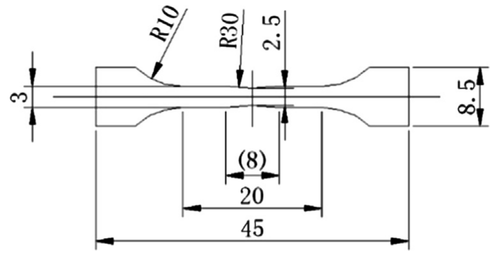

Fig. 1. Dog-bone sample geometry for in-situ SEM observation.

The hardness profiles of 3 mm samples along the depth were measured using a Vikers microhardness tester with a load of 10 g and duration of 15 s. Final results were determined by averaging the values of 8 indentation measurements.

The cross-sections of the SMAT samples treated were polished and etched for microstructure observation. The samples were etched in a solution containing $FeCl_3$ (5 g), HCl (50 mL) and H_2O (100 mL) for 30 s. The cross-sectional microstructure was analyzed using optical microscopy (OM, Olympus BX51M) and scanning electron microscopy (SEM, Hitachi S 3500N).

The samples prepared for in-situ tensile tests under SEM observation were prepared in a procedure similar to the preparation for dog-bone-shaped tensile specimens. In order to facilitate SEM observation, the middle of these samples was machined narrower with a radius of 30 mm to make strain concentrate in this area (Fig. 1).

3. Results

3.1. Microstructure

SEM observation revealed in depth grain size from the top surface down to the CG matrix (Fig. 2a). The average grain sizes are below 100 nm in the top 10-μm thick layer (NG layer), and increase to about 1 μm in the depth span of 10–100 μm (UFG layer). At depth greater than 100 μm are thought to be the deformed coarse grains layer (deformed CG layer), because of the higher hardness than annealed coarse grains (Fig. 3). For convenience, we define the whole gradient structure (GS) layer to include the NG layer, the UFG layer and the deformed CG layer. The thickness of whole GS layer is about 200 μm. Formation of this gradient structure from NG to CG can be understood by a strain and strain rate gradient during the SMAT processing at cryogenic temperature (CT-SMAT). The top surface layer was subjected to the largest strains and strain rates [14].

3.2. Vickers hardness

As shown in Fig. 3a, the hardness increases from about 0.65 GPa in the CG matrix to about 1.21 GPa at the top treated surface layer. After tensile testing, the hardness increases with increasing tensile strain. The strain hardening in the NG and UFG layers is limited. Fig. 3b shows the hardness increments (ΔH) profile along depth, indicating a ΔH maximum. ΔH of CG/GS interface is much higher than that of the CG matrix, and the maximum moves toward the CG region at higher tensile strain, which is consistent with previous observations in GS IF-steel [7]. If the GS layer and CG matrix deforms alone, the large grain sizes of CG matrix will have higher capability of accumulating dislocations to produce higher strain hardening than GS layer. The existence of the ΔH maximum near the GS/CG interface with higher strain hardening than the CG matrix provides direct evidence of extra strain hardening at the interfaces between the virtual necking fine-grained layer and the stable larger grained central layer [7]. The migration of the ΔH maximum indicates that the interfaces migrated dynamically.

3.3. Mechanical behaviors

Fig. 4a shows the engineering stress–strain curves of the CT-SMAT samples with different GS layer volume fractions and the annealed CG samples. Each curve is the average of three tensile tests. The CG samples exhibited yield strength (0.2% offset) of about 53 ± 5 MPa and a uniform elongation of 36 ± 1%. Meanwhile, the yield strength of the CT-SMAT samples are 160 ± 3 MPa, 190 ± 6 MPa, 220 ± 1 MPa and 235 ± 5 MPa and the uniform elongation are 29 ± 2%, 25 ± 2%, 14 ± 1% and 3 ± 0.3% for the GS volume fractions (VF_{GS}) of 0.08, 0.1, 0.13 and 0.2, respectively.

Fig. 4b shows that the tensile stress–strain curves systematically vary with VF_{GS}. In general, the curves obtained in this study can be categorized into the following four different characteristic types. Type 1 ($VF_{GS} < 0.08$): the curve shows continuous strain-hardening similar to that in pure CG copper. Type 2 ($0.08 < VF_{GS} < 0.1$): the curve exhibits a transition of tensile stress, in which tensile stress increases slowly first after yielding, then increases steeply for a small strain and finally follows the traditional strain-hardening behavior. Type 3 ($0.1 < VF_{GS} < 0.2$): the curve exhibits a yield peak followed by limited strain hardening. Type 4 ($VF_{GS} > 0.2$): the curve shows a clear yield peak followed by direct necking.

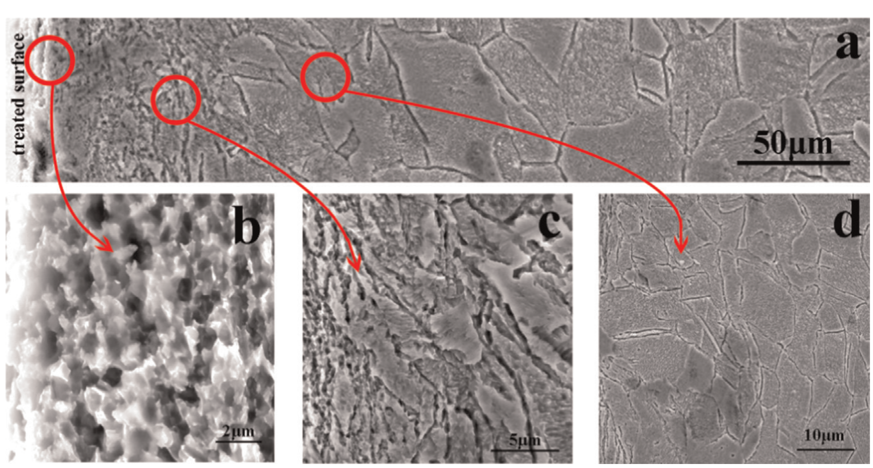

Fig. 2. (a) SEM cross-sectional image of a sample processed by SMAT at cryogenic temperature. (b–d) Higher-magnification SEM micrographs at different depths as indicated in (a).