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Mechanically-induced grain coarsening in gradient nano-grained copper

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Gradient nano-grained Cu subjected to tensile tests yields at a stress almost twice of that of the conventional coarse-grained Cu at or below the room temperature. Beyond the yield stress, a uniform plastic strain of larger than 30% can be achieved, accompanied by homogeneous grain coarsening in nano-grained surface layer. The observed grain coarsening may induce certain degree of "strain softening". The measured grain coarsening rates strongly depend on temperature, stress and strain rate, suggesting that the grain coarsening is presumably limited by thermally activated dislocation activities at defective grain boundaries, rather than via diffusional process on atom-by-atom basis. A non-diffusional (source-limited) Mott-Turnbull rate equation has been proposed to interpret the observed grain coarsening phenomenon.

1. Introduction

According to the well-known Hall-Petch relation [1,2], the strength of polycrystalline materials depends on grain size [3,4]. Nano-grained (NG) metals with grain size less than 100 nm usually exhibit high strength. However, with elevating temperature or by applying stress or strain, nano-grains tend to grow and the material strength tends to decrease. Softening due to grain coarsening or grain growth seems spontaneous and inevitable in many NG metals when the specimen is held at or even below the room temperature (RT) [5,6]. In particular, such "unusual" grain coarsening may occur more readily under mechanical deformation (termed as mechanically-induced grain coarsening), as observed in the indentation [7–9], rolling [10], compression [11] and tensile loading [12,13] tests. For example, it has been revealed that a gradient nano-grained (GNG) surface layer on a bulk coarse-grained (CG) pure Cu rod may sustain a tensile true strain exceeding 100% at RT accompanied by significant grain coarsening approaching to submicrometer levels [14].

Proper control of grain coarsening is essential to maintain the strengthening role of grain boundaries (GBs). Both thermal and mechanical events at GBs and within grains triggering grain boundary migration (GBM) must be taken into account in order to address the physical mechanisms underlying mechanically-induced grain coarsening. Among possible GB-based mechanisms, the shear-coupled GBM has been evidenced as a dominant one by both experimental observations [15–19] and a number of molecular dynamics simulations [20–25]: the normal motion of a GB is coupled to tangential displacement of adjacent grains results from an applied shear stress [26]. So far, only the role of stress on the GBM processes has been emphasized, whereas the elementary mechanisms occurring during the shear-coupled GBM are not yet well understood [27], in which temperature and strain-rate effects are still need to be addressed.

To understand the mechanism and general kinetics of the mechanically-induced grain coarsening, we have examined the mechanical behaviors of a GNG Cu layer on the surface of a bulk CG pure Cu rod (hereafter referred to as the GNG/CG Cu) deformed at different temperatures and strain rates. With these samples, significant grain coarsening can be observed in the top NG layer under uniaxial tensile deformation and the rate of grain coarsening can be measured over a broad range of grain sizes at various strains or loading durations. Besides, the softening due to grain coarsening

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can also be monitored at various temperatures and strain rates.

2. Experimental

2.1. Sample preparation

Commercial-purity Cu rods (99.97 wt%) consisting of well-annealed equiaxed coarse grains (25 μm) were machined into dog-bone-shaped tensile samples with a gauge length of 20 mm and a gauge diameter of 6 mm. Both the gauge sections and the arc transitions of the samples were subjected to surface mechanical grinding treatment (SMGT) following the procedure well described in Ref. [28]. The SMGT processing was repeated six times to obtain the top GNG layer of our samples.

2.2. Tensile tests

Uniaxial tensile tests of SMGT-processed Cu samples were performed on an Instron 5982 testing machine (Instron, UK) with a load capacity of 100 kN. An Instron 2620-601 clip-on extensometer was used to measure strain at RT (300 K), while an Epsilon model 3542 clip-on extensometer was used to measure strain at cryogenic temperature (123 K). To obtain each data set, at least three repeated tensile tests were carried out. The samples before and after tensile deformation were refrigerated at 258 K (−15 °C). For comparison, CG Cu samples were also tested under the same conditions.

In order to estimate the strength of the top NG layer, the surface 20 μm thick NG layers of the GNG/CG Cu were removed by electrolytic polishing. The samples without NG layers, hereafter referred to as the gradient ultra-fine grained (GFG) Cu, were tensioned under the same conditions for that of GNG/CG Cu. Based on the rule-of-mixture, i.e.,

$$\sigma_{GNG/CG} = V_{NG}\sigma_{NG} + (1 - V_{NG})\sigma_{GFG/CG} \quad (1)$$

where V_{NG} is the volume fraction of the NG layer in the GNG/CG samples, $\sigma_{GNG/CG}$, $\sigma_{GFG/CG}$ and σ_{NG} are the strength of the GNG/CG Cu, GFG/CG Cu and NG Cu layer, respectively, the strength of the top surface NG layer as a function of plastic strain can be estimated by accurately measuring $\sigma_{GNG/CG}$ and $\sigma_{GFG/CG}$ from the tensile tests.

2.2.1. Temperature

Uniaxial tensile tests at 300 K and 123 K were carried out at a strain rate of 1×10^{-2} s^{-1}. GNG/CG samples were deformed to $\varepsilon_T = 5\%$ and 25%, then unloaded for further microstructural examination. To be consistent, all strains referred in this study are true strains. The tensile tests at 123 K were carried out in an Instron environmental chamber, which was cooled by pouring liquid nitrogen inside the chamber in a continuous flow. The temperature was controlled automatically by a *T*-type thermocouple temperature controller.

2.2.2. Strain rate

Four different strain rates, i.e. 1×10^{-2} s^{-1}, 1×10^{-3} s^{-1}, 1×10^{-4} s^{-1} and 1×10^{-5} s^{-1}, have been used in our tensile tests. The microstructural evolutions of the GNG layer deformed at 1×10^{-2} s^{-1} and 1×10^{-5} s^{-1} were investigated further by scrutinizing their microstructure at strains of $\varepsilon_T = 5\%$, 15% and 25%, respectively.

2.3. Microstructural characterization

The longitudinal sections of the SMGT Cu were examined by a FEI NanoSEM Nova 430 field emission gun scanning electron microscope (FEG-SEM). The microstructures of the top GNG surface layer before and after tensile deformation were examined by a JEOL-2010 transmission electron microscope (TEM) operated at a voltage of 200 kV. The grain size distributions of the top GNG Cu layer were measured using bright-field TEM images. Over 800 grains from about 60 TEM images were measured to determine the grain size distribution profile for each condition.

3. Results

3.1. Microstructural characterization

As-prepared SMGT Cu samples show shining surfaces with minor roughness ($R_a \approx 0.3$ μm) and are crack-free. As shown in Fig. 1a, the specimen is characterized by a gradient structure composed of a GNG layer, a deformed CG layer and an undeformed CG core. The thickness of the mechanically treated surface layer in the radial direction is about 700 μm. TEM observations (Fig. 1b) indicate that

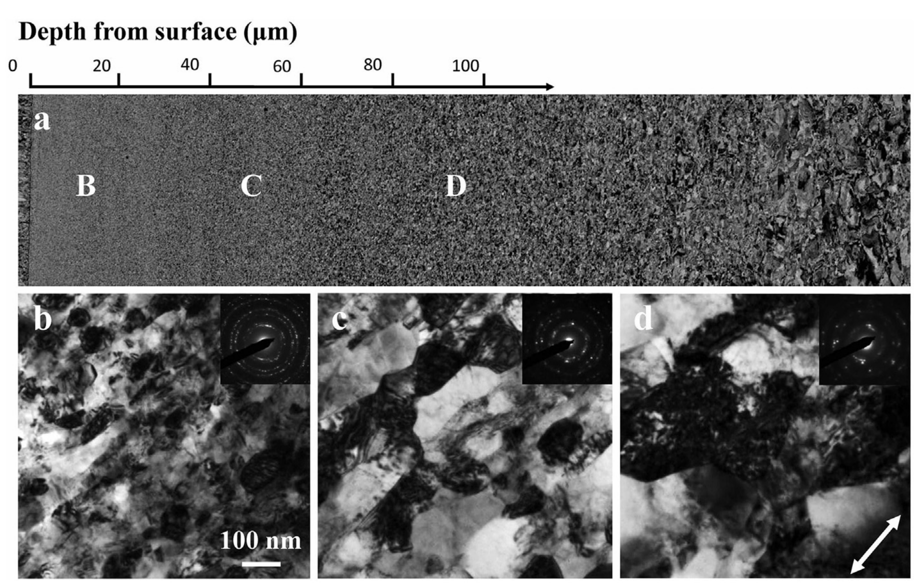

Fig. 1. (a) Typical longitudinal section SEM image of the SMGT Cu sample. (b, c, d) Bright-field TEM images at positions B, C and D indicated in (a), respectively. The double-headed arrow in (d) represents the tensile loading direction (the same as in the following Figures).