Available online at www.sciencedirect.com

ScienceDirect

Scripta Materialia 77 (2014) 17–20

Scripta MATERIALIA

www.elsevier.com/locate/scriptamat

Tension-induced softening and hardening in gradient nanograined surface layer in copper

T.H. Fang,[a] N.R. Tao[a] and K. Lu[a,b,*]

[a]*Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China*

[b]*Herbert Gleiter Institute of Nanoscience, Nanjing University of Science & Technology, Nanjing 210094, China*

Received 31 October 2013; revised 7 January 2014; accepted 7 January 2014
Available online 13 January 2014

With surface mechanical grinding treatment, a gradient nanograined (GNG) surface layer is produced on a bulk coarse-grained (CG) pure Cu, where the grain size increases gradually from 20 nm (topmost surface) to micrometer scale. Microhardness measurements of the GNG/CG sample after tension revealed that tension induces softening for grains smaller than 165 nm and induces hardening above this size. This critical size agrees with the strain-induced saturation grain size of Cu subjected to severe plastic deformation.

Keywords: Hardness test; Grain growth; Grain refinement; Gradient nanograined (GNG) structure; Copper

Large-strain plastic deformation of metals induces substantial grain refinement. Coarse-grained structures are progressively refined via complicated dislocation and/or twinning activities into ultrafine-grained (UFG) structures, accompanied by remarkable hardening. As grains become smaller, the strain-induced grain refinement process slows down and eventually ceases, typically in the submicrometer scale for pure metals [1–3]. Many UFG metals and alloys have been produced by means of various plastic deformation techniques [4–6].

Recent experimental observations have revealed that plastic deformation may induce obvious grain coarsening in nanograined (NG) metals and alloys, accompanied by obvious softening [7–13]. Nanosized grains may grow into submicrosized ones when the NG metals are strained at ambient temperature (or even liquid nitrogen temperature) under indentation [7,8], compression [9,10] or tensile loading [11–13]. The coarsening phenomenon of NG structures has been interpreted as a mechanically driven grain boundary (GB) migration process [11–13], which becomes more pronounced as the grain size becomes extremely small. The coarsening tendency is weakened as grains become larger.

Thus, under a given deformation condition, a critical grain size in a material is anticipated that corresponds to the transition from deformation-induced grain refinement to deformation-induced grain coarsening. Below this critical size, deformation induces coarsening and softening, while above it deformation induces refinement and hardening.

The present study is aimed at identifying this softening–hardening transitional grain size in pure Cu by using a unique gradient nanograined (GNG) structure [14,15]. By means of surface mechanical grinding treatment (SMGT) of a bulk coarse-grained metal, a GNG surface layer can be produced in which spatial variation of grain sizes is graded, covering a wide grain size spectrum from nanoscale in the topmost surface to microscale away from the surface. With a GNG surface layer on a bulk coarse-grained substrate of the same material, very large tensile strains can be achieved, as large as the coarse-grained (CG) state [15]. Therefore, this architecture provides a unique way to explore the existence of the transitional grain size.

Commercial-purity copper (99.97 wt.%) rods with a CG structure (average grain size of 21 μm) were machined into dog-bone-shaped tensile bar samples with a gauge diameter of 6 mm and a length of 20 mm. The

* Corresponding author at: Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China; e-mail: lu@imr.ac.cn

1359-6462/$ - see front matter
http://dx.doi.org/10.1016/j.scriptamat.2014.01.006

gauge and the transition sections of the bar samples were subjected to SMGT at cryogenic temperature (~173 K; for details see Ref. [14]). In brief, the sample was rotated at a high speed and its surface was ground by a lubricated hemispheric WC/Co tip. The surface layer of the sample was deformed plastically at a high strain and strain rates (>10^3 s^{-1}). The SMGT processing parameters are as follows: rotating velocity of the sample $v_1 = 600$ rpm, sliding velocity of the tool tip $v_2 = 3$ mm s^{-1}, preset penetration depth of the tool tip into the sample $a_p = 40$ μm. A hemispherical WC/Co tool tip (with a radius of $r = 3$ mm) was used. For each sample, the SMGT process was repeated six times with the same processing parameters, to achieve a thick and uniform GNG layer. No material was removed during the SMGT. Plastic deformation is rather uniform in the surface layer, with a small surface roughness ($R_a \approx 0.3$ μm). No crack was identified in the surface of the SMGT-processed samples.

Tensile testing was performed on an Instron 8801 machine at room temperature with a strain rate of 6×10^{-4} s^{-1}. A contactless MTS LX300 laser extensometer was used to calibrate and measure the strain of the sample upon loading. Structural characterization of the processed samples was carried out on an FEI NanoSEM Nova 430 scanning electron microscope (SEM) and a JEOL-2010 transmission electron microscope (TEM) operated at a voltage of 200 kV. The thin film specimens for TEM observations were prepared by using the conventional twin-jet electrochemical polishing technique with an electrolyte consisting of 25% alcohol, 25% phosphorus acid and 50% deionized water (by volume) that was cooled in a dry ice bath. Microhardness measurements were carried out on a Mitutoyo MVK-H3 microhardness tester with a load of 50 g and a loading time of 10 s. Hardness values were averaged from at least 10 indents.

SMGT induced a substantial grain refinement in the surface layer of the bulk Cu specimen (Fig. 1) [15]. In the top layer, which was ~60 μm thick, nanosized elongated grains (with an aspect ratio of ~1.7) with random crystallographic orientations are formed. The average transverse grain size increases gradually from about 20 nm in the top surface to about 100 nm at 60 μm deep. Grain sizes increase to about 300 nm in a depth span of 60–150 μm. Deeper than 150 μm, typical deformation structures form in coarse grains, characterized by dislocation tangles or cells with sizes ranging from several hundred nanometers to about 20 micrometers. The thickness of the overall deformed surface layer is ~600 μm. The gradient variation of grain sizes with depth from the treated surface as determined from TEM and SEM observations is shown in Figure 1b.

Quasi-static tensile tests of the SMGT samples with a GNG/CG architecture showed a yield strength (0.2% offset) of 129 ± 17 MPa (twice that of the CG sample) and an elongation-to-failure of 60% (a uniform elongation of 31 ± 2%) [15]. During tension the GNG surface layer deforms coherently with the CG core without any surface cracking or delaminating, even in the neck region, where the true strain exceeds 100%. After failure, half of the tensile sample was sectioned longitudinally after depositing a layer of pure copper to protect the surfaces. The microhardness in the GNG surface layer was measured at different positions in the gauge section, i.e. with different tensile true strains, which are estimated from the area reduction: $\varepsilon_T = \ln(D_0^2/D^2)$, where D is the sample diameter at a specific position and D_0 is the original diameter before tension.

As shown in Figure 2, in the GNG surface layer of the as-SMGT sample, hardness decreases gradually from 1.66 ± 0.12 GPa in the topmost surface to 0.75 ± 0.02 GPa in the undeformed substrate. After tension, the surface hardness drops obviously, accompanied by an increase in the hardness of the substrate, resulting in a smaller hardness gradient. At a true strain of 56%, the surface hardness drops to 1.2 ± 0.05 GPa while that in the CG substrate increases to 0.97 ± 0.07 GPa. As the true strain exceeds 100%, the hardness values of the surface and the substrate become much closer, with a very small hardness gradient (from 1.15 ± 0.07 GPa in the surface to 1.05 ± 0.07 GPa in the substrate). From the hardness contour maps measured in a large area with various strains (Fig. 3), one can see a similar phenomenon of softening in the top surface layer and hardening in the deep layer and the CG substrate.

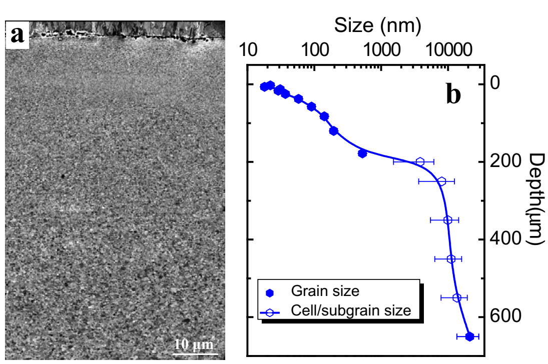

Figure 1. (a) A cross-sectional SEM image of the GNG surface layer in the as-SMGT Cu sample. (b) Measured grain/cell sizes as a function of depth in the as-SMGT sample.

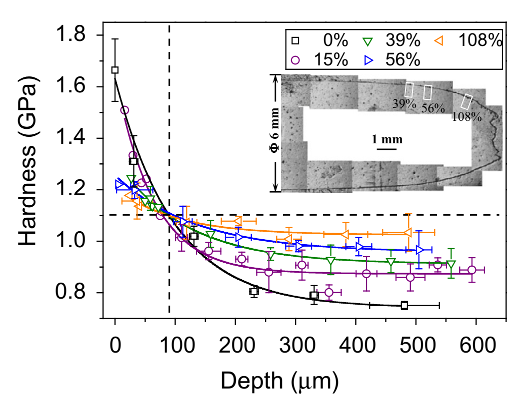

Figure 2. Variations of measured microhardness with depth in the GNG surface layer of the tensile samples with different true strains, as indicated in the SEM image of the longitudinal section of the tensile sample after failure (inset). Each datum point is averaged from more than 10 indents.