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Supporting Online Material for

Revealing Extraordinary Intrinsic Tensile Plasticity in Gradient Nano-Grained Copper

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Revealing extraordinary intrinsic tensile plasticity in gradient nano-grained copper

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Supporting Online Material

Materials and method details

- Sample preparation: surface mechanical grinding treatment (SMGT)
- Surface roughness measurements
- Microhardness tests
- Tensile tests
- Structure characterization (SEM/EBSD and TEM) experiments
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Sample preparation: Surface mechanical grinding treatment (SMGT)

Surface mechanical grinding treatment (SMGT) [S1] was used to synthesize a gradient nano-grained (GNG) surface layer on a CG Cu substrate. A commercial purity copper (99.97 wt.%) was used in the present study. Pure Cu rods were annealed at 723K for 1.5 h to obtain a coarse-grained polycrystalline structure. The average grain size of the as-annealed Cu is about 21 μm. The CG Cu rods were machined into dog-bone tensile bar samples, of which the geometry is indicated in Fig. S1. Then the tensile bars were processed by means of SMGT at liquid nitrogen temperature (~173 K). Only the gauge section and the transition sections were treated. 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}, the preset penetration depth of the tool tip into the sample a_p = 40 μm. A hemi-spherical 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 for achieving a thick and uniform GNG layer. 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.

Surface roughness measurements

Surface roughness of the tested samples was measured on a surface profilometer with a diamond stylus (Model 2205, Harbin Measuring & Cutting Tool Group Co. Ltd.). The roughness measurement accuracy is 0.001 μm with a load to the stylus of <0.7μN. The testing length is several millimeters along the axial direction in the gauge section of the tensile samples.

Microhardness tests

Microhardness measurements were carried out on a Mitutoyo MVK-H3 microhardness tester with a load of 50 g and loading time of 10 s. Hardness values were averaged from at least 20 indentations.

Tensile tests

For the GNG/CG and the CG bar samples, uniaxial quasi-static tensile tests were performed in an Instron 8801Testing System (MTS) at ambient temperature and a strain rate of $6x10^{-4}$ s^{-1}. For the thin foil GNG samples, uniaxial tensile tests were performed in a Tytron 250 Microforce Testing System (produced by MTS) at a strain rate of $6x10^{-4}s^{-1}$. A contactless MTS LX300 laser extensometer was used to calibrate and measure the strain of the sample upon loading.

Structure characterization (SEM/EBSD and TEM experiments)

Cross-sectional structural characterization of the processed samples was carried out on a FEI NanoSEM Nova 430 and a JEM-2010 transmission electron microscope (TEM) operated at a voltage of 200 kV. A layer of pure copper was electro-deposited onto the treated surface of the GNG/CG samples. Then cross-sectional samples was cut by using a low-speed diamond saw and polished for SEM observations. The electron backscatter diffraction (EBSD) tests were executed under a voltage of 20 kV and a current of 6.0 nA with a step size of 20 nm. Boundaries with misorientations higher than 15° were defined as high angle boundaries and

5° for low angle boundaries. The thin film specimens for TEM observations were prepared by using the conventional twin-jet electro-chemical polishing technique with an electrolyte consisting of 25% alcohol, 25% phosphorus acid and 50% de-ionized water (by volume) that was cooled in a dry ice bath.

References:

S1. W.L. Li, N.R. Tao, K. Lu, *Scr. Mater*. **59**, 546 (2008).

Figures

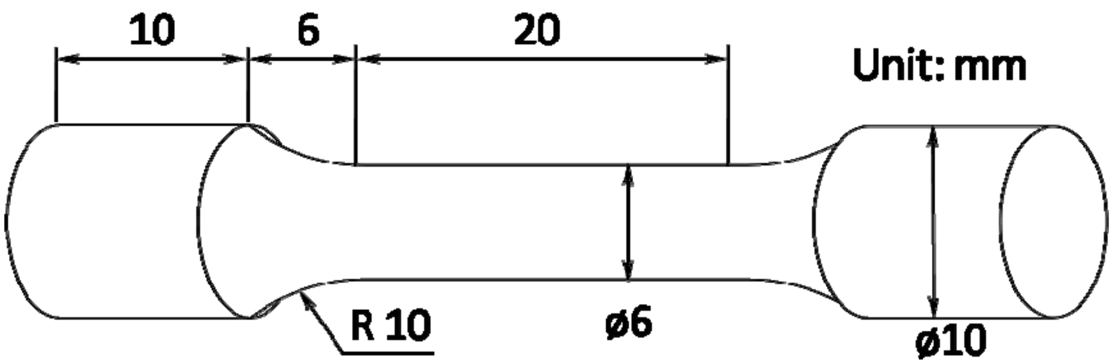

Figure S1. Illustration of the tensile bar sample geometry.

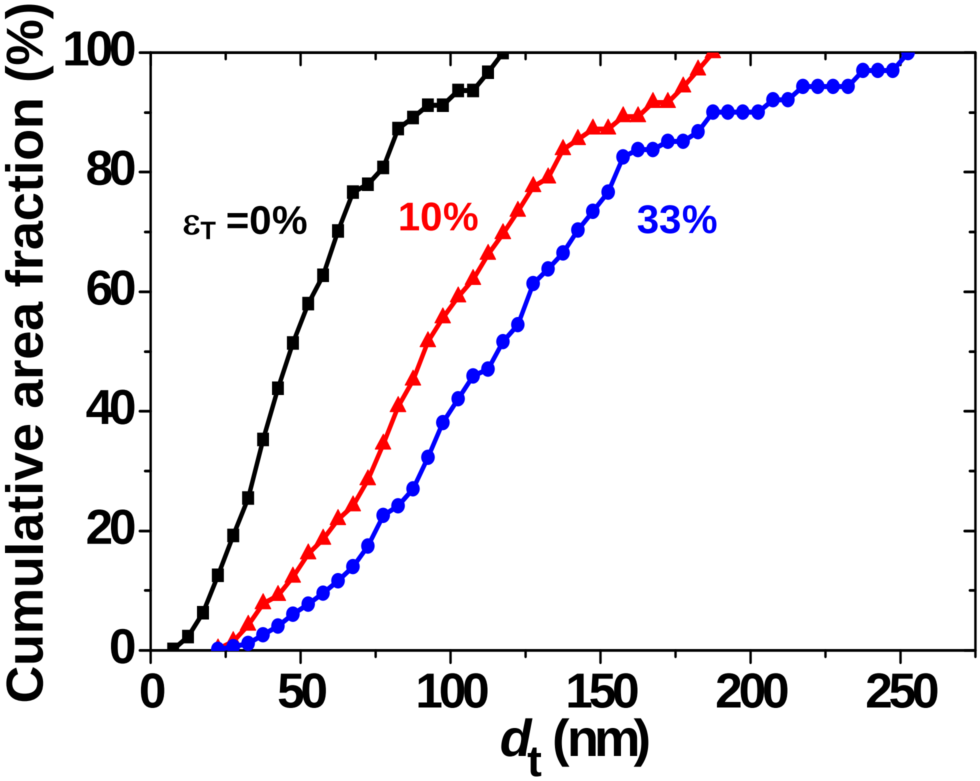

Figure S2. Area-weighted cumulative grain size distributions in the top GNG layer at tensile true strains of 0, 10%, and 33%, respectively, determined from TEM observations.