

## Strain rate sensitivity of Cu with nanoscale twins

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The strain-rate sensitivity of ultrafine-crystalline Cu with different concentrations of nanoscale growth twins is investigated using tensile strain rate jump tests. Higher twin density leads to enhanced rate sensitivity, which decreases mildly with increasing strain rate and strain. Mechanisms underlying these effects are explored through post-deformation transmission electron microscopy. © 2006 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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In addition to their high strength and hardness arising from grain refinement into the nanometer regime, nanocrystalline (nc) metals and alloys (with both average and full range of grain sizes typically finer than 100 nm) are known to exhibit much higher sensitivity to the rate of straining than their microcrystalline (mc) counterparts (with grain size typically in excess of 1  $\mu\text{m}$ ) and ultrafine-crystalline (ufc) metals (with grain sizes in the range of 100 to several hundred nm). It is commonly found that nc face-centered cubic (fcc) metals exhibit lower tensile ductility and increased strain rate sensitivity of plastic flow [1–3].

Experimental evidence for enhanced rate sensitivity of electrodeposited Ni with fully nc grains has been demonstrated through instrumented, depth-sensing indentation at different rates of loading [4]. These experiments reveal that nc Ni, with an average grain size of about 40 nm, exhibits a much higher rate sensitivity than mc and ufc Ni [4]. These results are also consistent with the effects of loading rate on plastic deformation observed during simple uniaxial tensile tests [4].

The strain-rate jump test in tension provides direct and conclusive evidence for the rate-dependence of flow stress in metals. Such experiments are difficult to perform in truly nc metals because of their limited ductility, small dimensions and thin geometry of specimens, early

onset of necking, nonuniform plastic deformation and plastic instability [1,5]. Consequently, effects of grain size on rate sensitivity in Cu, for example, using tensile strain rate jump tests or compression strain rate cycling tests have been conducted primarily on ufc specimens (with an average grain size of 200–300 nm) produced by equal channel angular pressing (ECAP) with a high initial defect density [6,7].

Recent work by the present authors and their coworkers has shown that ufc Cu containing controlled concentrations of nanoscale twins, produced by pulsed electrodeposition (PED), also exhibits high tensile strength, hardness, and strain rate sensitivity [8–10]. These trends, observed from simple uniaxial tension tests [8,10] and instrumented indentation tests [9], are very similar to those seen in nc Cu without twins, but with grain sizes comparable to the twin spacing. However, unlike the nc Cu, the ufc Cu with nanoscale twins does not show reduced ductility as a consequence of decreasing twin spacing (or equivalently, increasing twin density) [8,10]. Pure ufc Cu with nanoscale twins thus provides a structure through which both enhanced strength and ductility can be achieved through appropriate process control.

In view of these considerations, nanotwinned Cu specimens, undergoing uniform plastic deformation until sufficiently large plastic strains, offer a means to explore the strain-rate-sensitivity of nanostructures through direct tensile strain rate jump tests over a wide range of strain rates. Our results from tensile strain rate jump tests, to be presented here, on ufc Cu with different

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concentrations of nanoscale twin densities demonstrate that the twinned structures have strongly increased strain rate sensitivities compared to ufc Cu of the same grain size without twins.

In this letter, high purity copper specimens (with in-plane dimensions of 20 mm × 10 mm and 100 μm in thickness) with nanoscale growth twin lamellae were synthesized by means of PED from an electrolyte of CuSO<sub>4</sub>. The as-deposited nanotwinned (nt) Cu sample has roughly equiaxed grains (with an average grain size of 400–500 nm), with a high concentration of coherent twin boundaries (TBs). A Cu sample with an average twin lamellar spacing of 15 ± 7 nm (hereafter referred to as nt-Cu-fine) and one with an average twin lamellar spacing of 100 ± 15 nm (nt-Cu-coarse) were selected. For comparison, ufc Cu sample of the same grain size, but essentially without twins (referred to as ufc Cu-control) was produced from the same electrolyte by means of direct current electrodeposition. Details of sample preparation procedure, purity, density as well as structural characteristics of the as-deposited nt-Cu can be found elsewhere [8–10].

To investigate the strength, ductility and strain rate sensitivity of nt-Cu, uniaxial tensile strain rate jump tests were performed on a Tytron 250 Microforce Testing System (MTS System Corporation, Eden Prairie, MN, USA). These tests were carried out by instantaneously changing the strain rate at a constant true strain in all specimens. Multiple flat, dog-bone shaped tensile specimens with a gauge length of 4 mm and a gauge width of 1.7 mm were prepared by electro-discharge machining from as-deposited Cu foils. The final thickness of tensile samples after electropolishing, about 20 ± 5 μm, was measured by a LEICA MPS 30 optical microscope.

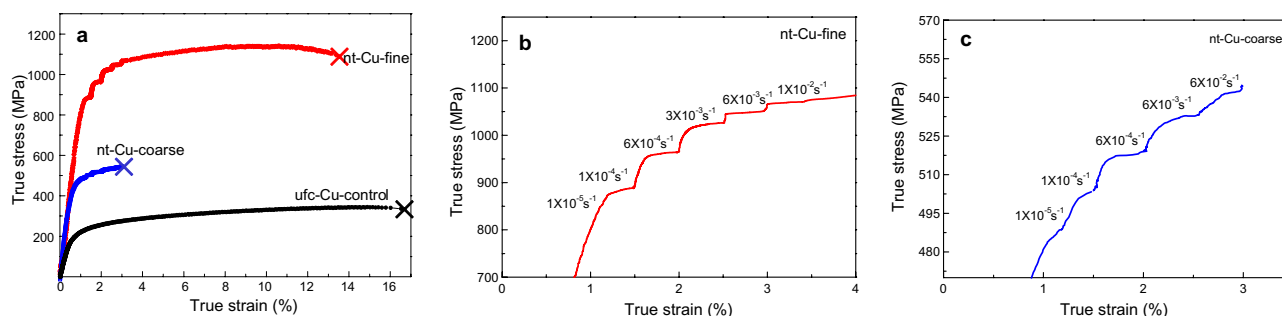
Microstructures of the Cu specimens in the as-deposited condition and after tensile deformation were characterized by means of a transmission electron microscope (TEM, JEM2000EXII). The deformed TEM Cu samples were cut from the area close to the fracture surface of the tensile specimens. Further details of TEM specimen preparation can be found elsewhere [9].

True stress–strain curves from the tensile strain rate up-jump tests on nt-Cu-fine, nt-Cu-coarse and ufc Cu-control are shown in Figure 1. The Cu samples with nanotwin structures show substantial tensile yield strengths. For the nt-Cu-coarse with twin lamellar spac-

ing of about 100 nm, the yield strength is close to 500 MPa. However, when the twin lamellar spacing decreases down to about 15 nm, the yield strength of nt-Cu-fine sample is close to 900 MPa, which is about 4.5 times higher than that of the ufc Cu-control sample with similar grain size. This strength is consistent with that of the nc Cu specimens with grain size of 20–30 nm with high angle grain boundaries (GBs) [11,12].

A noteworthy feature of Figure 1(a) is that the tensile ductility also increases considerably with decreasing twin lamellar spacing. For nt-Cu-coarse, tensile elongation-to-failure at a final strain rate of  $6 \times 10^{-2} \text{ s}^{-1}$  is only 3%. However, the elongation-to-failure is as high as about 14% for nt-Cu-fine at a final strain rate of  $1 \times 10^{-2} \text{ s}^{-1}$ . It is interesting to note that the gauge section of the tensile specimen deforms uniformly during a significant fraction of plastic straining prior to failure (i.e. more than 10% plastic strain for nt-Cu-fine). The extremely high yield strength and the large ductility detected for the nt-Cu-fine sample are unusual for metallic materials, in which strength and ductility usually exhibit opposite trends with refinement in the characteristic size scale of the structural dimension, such as the grain size. For most pure metals with nm-size grains, a comparable high strength could be obtained but only at a significant loss in ductility and in the extent of homogeneous plastic deformation [5].

Figure 2 also reveals the effects of variations in strain rate, spanning three orders of magnitude—from  $1 \times 10^{-5}$  to  $6 \times 10^{-2} \text{ s}^{-1}$  (for nt-Cu-coarse) and  $1 \times 10^{-5}$  to  $1 \times 10^{-2} \text{ s}^{-1}$  (for nt-Cu-fine)—of tensile stress–strain response. When the strain rate increases, the corresponding variations in plastic flow characteristics are observed to depend strongly on the twin density. With increasing strain rate, the strength increases monotonically. For example, the flow strength for nt-Cu-fine rises from about 900 MPa to 1070 MPa within the strain rate range of  $1 \times 10^{-5}$  to  $1 \times 10^{-2} \text{ s}^{-1}$ , as seen in Figure 1(b). The increment of flow stress for nt-Cu-fine is close to 300 MPa within the range of strain rates considered. However, a weak dependence of stress is found for nt-Cu-coarse with increasing strain rate. The increment of flow strength for nt-Cu-coarse is less than 50 MPa (from 492 MPa to 540 MPa) within the strain rate range of  $1 \times 10^{-5}$  to  $6 \times 10^{-2} \text{ s}^{-1}$ , as seen in Figure 1(c). As generally expected for fcc mc metals, a very small increase in strength is observed with increasing strain rate for ufc Cu-control sample. This is consistent with the trends



**Figure 1.** (a) Tensile stress–strain curves of the nt-Cu specimens. Magnified strain rate jump tensile curves for nt-Cu-fine and nt-Cu-coarse are shown in (b) and (c), respectively.

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