

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 μm) 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 CuSO₄. 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 1×10^{-5} to $6 \times 10^{-2} \text{ s}^{-1}$ (for nt-Cu-coarse) and 1×10^{-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 1×10^{-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 1×10^{-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

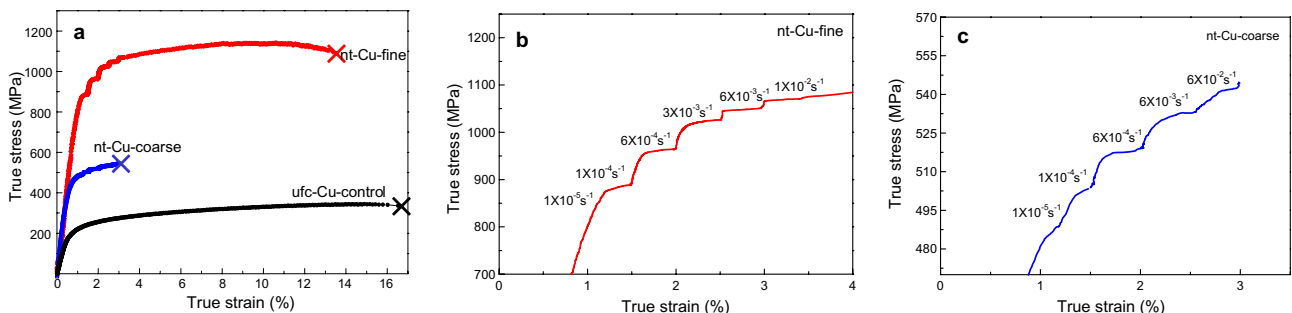


Figure 1. (a) Tensile stress–strain curves of the nt-Cu specimens. Magnified strain rate jump tensile curves for nt-Cu-fine and nt-Cu-coarse are shown in (b) and (c), respectively.

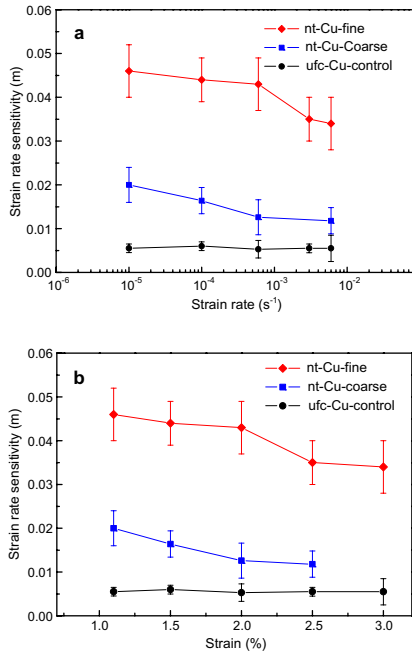


Figure 2. (a) The strain rate sensitivity, m , as a function of strain rate range for nt-Cu-fine and ufc Cu-control (1×10^{-5} to $1 \times 10^{-2} \text{ s}^{-1}$) and for nt-Cu-coarse (1×10^{-5} to $6 \times 10^{-2} \text{ s}^{-1}$). (b) SRS, m , as a function of the tensile strain for the Cu samples.

reported for ufc Ni from instrumented nanoindentation tests at different strain rates [4].

For each tensile jump test, the variation of m vs. the strain rates from the jump tests is shown in Figure 2(a). For this purpose, the strain rate sensitivity (SRS), m , is defined as [13]:

$$m = \left. \frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}} \right|_{\epsilon, T}, \quad (1)$$

where σ is the flow stress and $\dot{\epsilon}$ is the strain rate. Obviously, a strong SRS of flow stress in nt-Cu is captured in Figure 2 as a function of twin lamellar spacing. It is evident that m has a moderate reduction with increasing strain rate for nt-Cu whereas it is independent of strain rate for ufc Cu-control. The strain rate sensitivity for nt-Cu-fine is estimated to be about 0.043 ± 0.006 for a strain rate of $1 \times 10^{-5} \text{ s}^{-1}$, and 0.034 ± 0.005 for $1 \times 10^{-2} \text{ s}^{-1}$, which is significantly higher than that of nt-Cu-coarse ($m = 0.020 \pm 0.004$ at $\dot{\epsilon} = 1 \times 10^{-5} \text{ s}^{-1}$, and $m = 0.012 \pm 0.003$ at $\dot{\epsilon} = 6 \times 10^{-2} \text{ s}^{-1}$). The resulting m for the ufc Cu-control (about 0.005) is consistent with literature data for the conventional mc Cu, whose strength is generally rate-insensitive at RT [14]. The present results show very similar trends to m values determined from depth-sensing indentation and constant strain rate tests [9,15], and they match the trends of increasing rate sensitivity with increasing twin density. The effect of strain on m , Figure 2(b), also shows a similar variation as that of strain rate for the two twinned structures and the control condition.

The influence of tensile deformation on the microstructure of twinned Cu is shown in the TEM images. Figure 3(a) indicates that the as-deposited nt-Cu-fine sample has roughly equiaxed grains with clear large-

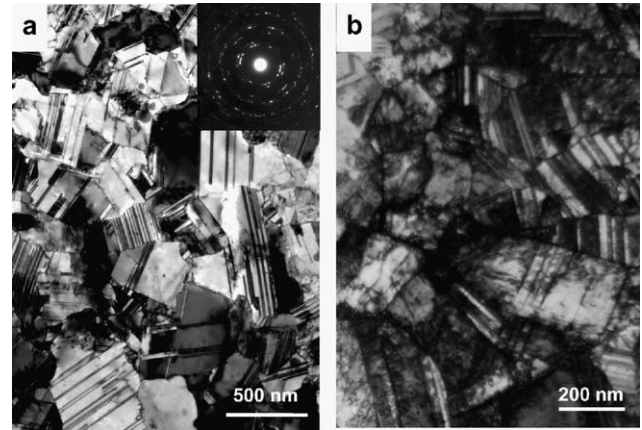


Figure 3. The microstructure of nt-Cu-fine (a) as-deposited state, and (b) after tensile-deformed state.

angle GBs and a high density of sharp, intra-crystalline TBs. Most growth twins are coherent $\Sigma 3$ boundaries. Obviously, the clear and straight coherent TBs separate the ultrafine-grained crystals into nm thick twin/matrix lamellar structures.

The post-tensile-failure microstructure of nt-Cu-fine is shown in Figure 3(b), where a high density of dislocations is evident in the strained sample. This is in distinct contrast to the as-deposited condition, Figure 3(a), where the sample is relatively thinly populated with dislocations. Stepped or even curved TBs are frequently seen in Figure 3(b), whereas only straight TBs were visible in Figure 3(a). In addition, numerous dislocation tangles are also seen inside the thick twin lamellae, Figure 3(b). Such tangles have been frequently observed in the deformed mc Cu. After deformation, the grain size of nt-Cu-fine did not change and the grains remained almost equiaxed with large-angle GBs.

In addition to the high concentration of dislocations blocked by either TBs or GBs, abundant dislocation debris is also observed in the vicinity of TBs/GBs in nt-Cu-fine sample, as shown in Figures 3(b) and 4. The significant strain contrast areas (in square 1 and 2, in Fig. 4) at TBs suggest the development of strong local stress concentration. The semicircular strain contrast regions (indicated by white arrows in Fig. 4) possibly suggest the emission of dislocation loops from TBs as also noted in the microscopy studies reported in Ref. [9]. The comparison of TEM observations before

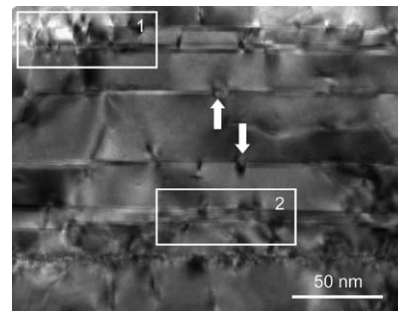


Figure 4. Typical TEM image of the structure close to the failure surface of nt-Cu-fine tension sample.

and after deformation suggests that most of the plastic strain would be taken by the dislocations piling up along the TBs, which are related to the shear strain accumulation on the TBs. For the nt-Cu sample with high density of nanoscale twins, GB diffusion as well as the GBs activities appear to be very limited, as also observed in ufc Cu-control (m is only 0.005). The enhanced strain rate sensitivity for nt-Cu samples, together with the observation of enhanced strengths from tensile jump tests, suggest that its rate-controlling mechanism is possibly different from that known for the mc and nc fcc metals, although it is noted that the trend of rate sensitivity variation with twin lamellar spacing is consistent with that of m vs grain size in fcc metals.

A key deformation mechanism for mc fcc metals is the cutting of forest dislocation which results in a low SRS. With grain size decreasing into ufc and nc regimes, the rate-limiting process is increasingly influenced by the interactions of GBs with dislocations, due to the abundance of GBs and/or sub-boundaries that serve as obstacles to dislocation motion [16,17]. For the nanotwinned Cu case, the main obstacle for dislocation motion should be the high concentration of nanoscale growth twins. We postulate that in nt-Cu specimens, in addition to the classical deformation by dislocation slip, the interactions between slip dislocations and coherent twin boundaries play an additional and critical role in the plastic deformation process, although the mechanistic origins are not known at this time.

Mechanistic models by Asaro and Suresh [18] pointed out that metals with nanoscale twins would nucleate and emit dislocations from twin boundaries, even deformation twins, during plastic deformation, and influence stress-strain response, very much like the GBs in nc fcc metals. It has indeed been found from computer simulations that defects at TBs assist dislocation generation [19]. Due to the highly concentrated dislocation activities in the vicinity of TBs, as shown in Figures 3(b) and 4, it is suggested that the dislocation interaction with TBs would take up most of the deformation and that the TB associated deformation mechanisms are likely to be much more rate-sensitive. This is consistent with the experimentally observed trends of the effect of twin density on strength and rate sensitivity of plastic flow, i.e., the stress as well as strain rate sensitivity of the nt-Cu specimen with respect to the concentration of the TBs and a relatively small m is observed in nt-Cu-coarse with a reduction in the concentration of TBs.

One phenomenon observed from the present tensile jump tests is that m decreases mildly and gradually as strain rate/strain increases for the nt-Cu specimens. However, for ufc Cu-control, there is no such dependence of rate sensitivity on either strain rate or strain, indicating that the presence of twin boundaries could influence how m is affected by strain rate or strain.

The decrease in m with increasing strain for nt-Cu can be understood by examining the change in microstructure due to the change in strain during tensile loading. From the TEM observations, the twin spacing appears to increase with plastic deformation although statistically significant large changes are not observed. Another observation is that the dislocation pile up along the TBs and between TBs increases with plastic defor-

mation. Dislocation-based plastic deformation processes would have a small or negligible dependence of strain and strain rate on SRS (m) [20,21] at small strains, i.e., less than 20%. If this is the case, then the plastic deformation-induced twin spacing increase is likely to be responsible for the mild decrease of m vs. strain, since the literature data [9,15] and the current results show that m decreases with an increase in twin spacing. While it is clear that the strain-dependent change in m is related to the twin spacing during plastic strain accumulation, it is not clear whether this change can account for the entire decrease in m with strain as observed in Figure 2(a) and (b). It is likely that both strain and strain rate contribute to the mild decrease of m during plastic flow, although the exact relative proportion is not known.

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