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Strain hardening and large tensile elongation in ultrahigh-strength nano-twinned copper

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A high density of growth twins in pure Cu imparts high yield strength while preserving the capacity for efficient dislocation storage, leading to high strain hardening rates at high flow stresses, especially at 77 K. Uniform tensile deformation is stabilized to large plastic strains, resulting in an ultrahigh tensile strength of ~1 GPa together with an elongation to failure of ~30%. © *2004 American Institute of Physics*. [DOI: 10.1063/1.1814431]

Work hardening (cold work) and grain boundary (GB) hardening are the two conventional mechanisms used to strengthen a pure metal. In both cases the idea is to impede the movement of dislocations by increasing the density of obstacles—dislocations in the former case and GBs in the latter. In recent years, ultrahigh strengths have been achieved in elemental metals by employing heavy plastic deformation to produce dislocations and ultrafine-grained (UFG) microstructures, or by creating nanocrystalline (nc) grains.[1,2]

However, these ultrahigh-strength metals no longer have the high ductility typical of their conventional coarse-grained counterparts. Even though the UFG or nc materials may be intrinsically ductile (i.e., plastic deformation mechanisms remain operative), they tend to suffer from plastic instabilities upon deformation.[2,3] One example is the concentration of large deformation in shear bands.[4] In tension, the necking instability sets in early. The undesirable inhomogeneous deformation can lead to the onset of failure, severely limiting the useful ductility.[5] One major cause for the localized plastic deformation is the inability to sustain a sufficient strain hardening rate (Θ) to prevent the accumulation of large strains in local regions.[2,6] In the UFG and particularly nc metals, the dislocations that traverse the small grains to sustain the plastic flow tend to disappear into the opposing GBs, leaving little dislocation build-up[7] behind. Such dynamic recovery in the presence of abundant GBs takes away the most prolific conventional mechanism for strain hardening.

Therefore, it is a challenge to come up with a new micro-/nano-structure design strategy that can impart high yield strength to a metal, but without ruining its ability to sustain adequate Θ at high stress/strain levels. The idea discussed in the following uses growth twins with nanoscale lamellar spacings and coherent twin boundaries (TBs) in lieu of dislocations and general high-angle GBs, as strengthening agents. Such a micro-structure not only is highly effective in blocking the motion of dislocations,[8–10] but also retains the strain hardening capability, leading to an unusual combination of high tensile strength and ductility.

High-purity Cu (>99.99%) with growth twins was synthesized using pulsed electrodeposition from a Cu_2SO_4 bath. The twin density was controlled by adjusting the electrodeposition parameters, as explained in Ref. 9. The hydrogen and oxygen content in the as-deposited Cu was less than 20 ppm. X-ray diffraction patterns (not shown) suggest that the deposited foils have a strong {110} out-of-plane texture. For uniaxial tensile tests, the deposits were polished down to 25–40 μm and dogbone-shaped samples were cut with a gauge a length of 4 mm and a width of 1 to 2 mm. The tensile tests were performed at strain rates of 10^{-4}–10^{-1} s^{-1}, at room temperature (RT) or 77 K.

One example of the as-deposited Cu is shown in the transmission electron micrograph of Fig. 1(a). The average grain size (d) is of the order of 1 μm, but the grains contain a high density of growth twins with lamellar spacing ranging from a few to hundreds of nanometers. The length scales shown in Fig. 1(b) represent the distances between the boundaries (TBs and/or GBs) in the *perpendicular* direction. Note that for large global strains the deformation usually propagates throughout the entire sample, and the gliding dislocations would have to travel past these boundaries (barriers). On average, the barrier spacing (effective "d") is approximately 200 nm, i.e., in the UFG regime.

This microstructure is expected to lead to a strength much higher than would be expected from the micrometer-sized grains alone. Indeed, the 0.2% offset yield strength of the nano-twinned Cu is 620 MPa, as observed from the engineering stress–strain (σ)–(ε) curve at RT in Fig. 2(a). Its strength is higher than that of UFG Cu with d in the 200–300 nm range (~500 MPa),[11,12] suggesting that the low-energy TBs (special high-angle GBs), especially for lamellar with very small spacing, are highly effective in strengthening. Interestingly, the nano-twinned Cu also shows tensile elongation to failure of >10%, including significant uniform elongation (before the peak of the curve). This is to be compared with UFG or nc Cu that usually peak close to the beginning of the tensile plastic strain.[3,5,6] To illustrate this, a curve is included in Fig. 2(a) for an UFG Cu with d ~190 nm,[11,12] even though the 77 K test reduced dynamic recovery for enhanced Θ, the engineering σ–ϵ curve still peaks at small strains, showing limited uniform ductility.

The nano-twinned Cu, on the other hand, shows a tensile curve that peaks at >20% elongation when tested at the same temperature, Fig. 2(a). Its 77 K yield strength is 650 MPa, close to that of the 190 nm Cu. Because of the large *uniform* deformation, the engineering curve can be con-

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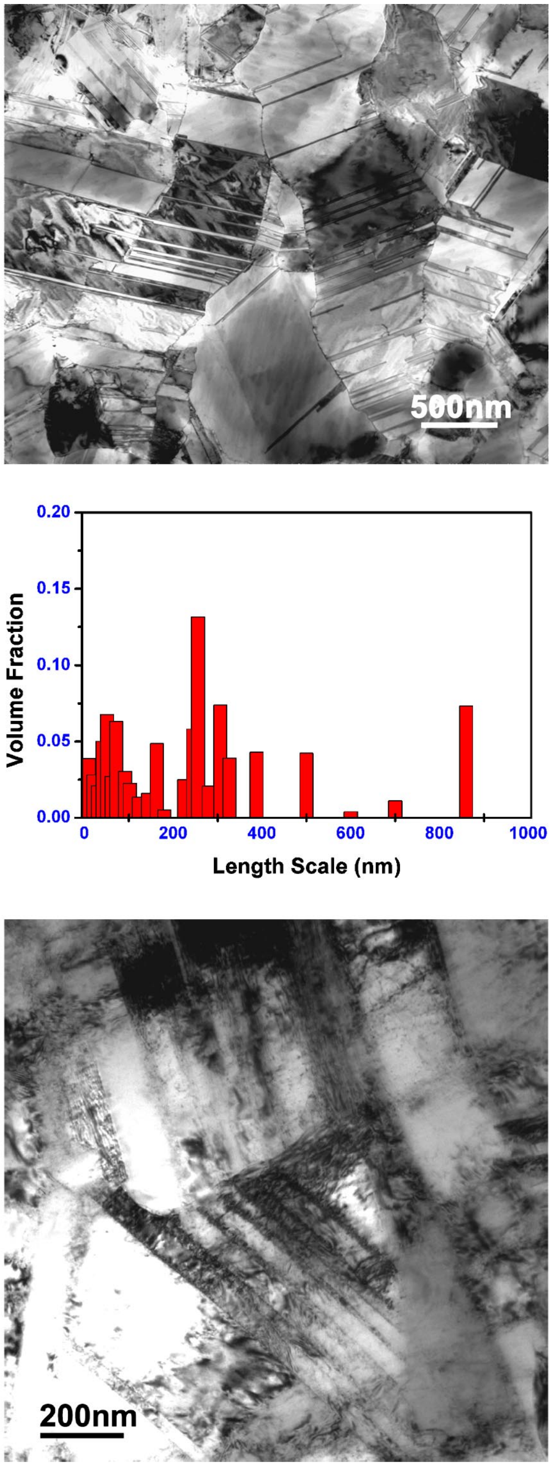

FIG. 1. (Color online) Electrodeposited Cu with high-density growth twins: (a) TEM micrograph of the as-deposited microstructure, (b) length scale (spacing) distribution determined from TEM micrographs for the direction perpendicular to the TBs, and (c) TEM micrograph after 77 K straining to ~30%, showing the accumulation of dislocations.

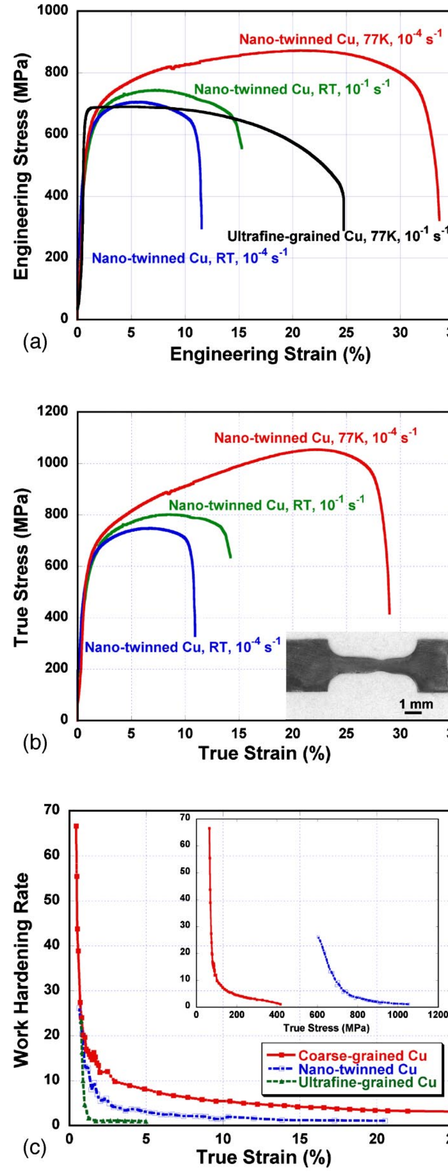

FIG. 2. (Color online) (a) Engineering tensile stress–strain curves of nano-twinned Cu tested at different strain rates and temperatures, in comparison with that of an UFG Cu tested at 77 K, (b) true σ–ε curves, (c) the normalized work hardening rate at 77 K, $\Theta=1/\sigma(\partial\sigma/\partial\varepsilon)_{\dot{\varepsilon}}$, where σ is true stress and ε true strain. At $\varepsilon=1\%$, $\Theta=20$ and 5 for nano-twinned and UFG Cu, respectively (and $\Theta=11$ and 1 at $\varepsilon=2\%$). Necking sets in when $\Theta\leqslant 1$, such that for UFG Cu the true strain and Θ cannot be evaluated after $\varepsilon=2\%$. Coarse-grained Cu is also included for comparison. The inset illustrates the sustained Θ at high stresses, in contrast with coarse-grained Cu.

verted to true σ-ϵ curve using standard formula assuming uniform cross-sectional area along the gauge length, as shown in Fig. 2(b). The strong strain hardening is obvious. As seen in Fig. 2(c), the normalized work hardening rate remains appreciable over a range of strains, whereas that of the UFG Cu diminishes very quickly. The inset of Fig. 2(c) compares the behavior between the two Cu materials (nano-twinned and coarse-grained) that do strain harden to large plastic strains at 77 K. The difference, obviously, is that the nano-twinned Cu offers pronounced Θ at high flow stresses. The stress range is in fact *beyond* what coarse-grained Cu can be work-hardened to reach (its Θ is too low to prevent plastic instability at such high stresses). As a result, the onset of necking is delayed (Considère criterion[2,6]), Fig. 2. At 77 K the nano-twinned Cu work hardens to an ultrahigh tensile strength of ~1 GPa and a uniform strain in excess of 20% in contrast to the few percent sustainable for nc metals.[2,13] The inset of Fig. 2(b) shows an optical micrograph of the sample that eventually necked and fractured. The neck developed, as well as the dimple features on fracture surfaces (not shown), are all characteristic of a ductile material.

The origin of the strong strain hardening at high strength levels is revealed in the TEM micrograph of Fig. 1(c). After deformation to 30% engineering strain at 77 K, the sample

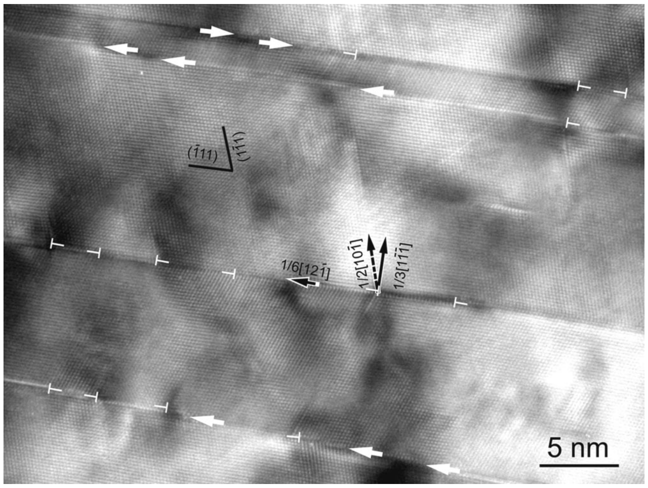

FIG. 3. TEM micrograph showing the deposition of Shockley (short arrows) and Frank (T) partial dislocations at twin boundaries, after RT deformation of nano-twinned Cu. Also included is a dissociation reaction of a unit dislocation into Frank and Shockley partials: $\frac{1}{2}[10\overline{1}]$ (black dashed arrow) $\rightarrow$ $\frac{1}{3}[1\overline{11}]+\frac{1}{6}[12\overline{1}]$.

stores high densities of dislocations. This is to be compared with the rather clean interior of the grains and twins before deformation, Fig. 1(a). It is clear that the twin-containing microstructure has ample room for the storage of additional defects (dislocations). The work hardening rate and uniform/total elongation are higher at an increased strain rate [the 10^{-1}/s curve at RT has been included in Fig. 2(a)], or a decreased deformation temperature (77 K), due to the reduced rates of dislocation annihilation in recovery.[11,12]

The large capacity for, and the fast rate of, dislocation storage originates from the following factors. First, the multimodal distribution of length scales [Fig. 1(b)], rather than a single *d* of 200 nm, naturally leaves the space in many twin lamellae for the retention of dislocations (easier in the wider twin lamellae). Second, the twin lamellar structure may be viewed as *inherently bimodal*,[6] because the length scale of the "2D grain" in the two dimensions parallel to the TBs is significantly larger than the ultrafine scale down to nanometers in the direction perpendicular to TBs (harder direction). Dislocations can thus accumulate, forming tangles to subdivide the twin lamellae [Fig. 1(c)]. Third, the TBs in large numbers also serve as the locations where a high density of dislocations can move and be built up starting from low levels. An example is seen in Fig. 3: during deformation nonglissile Frank partial dislocations $(1/3\ [111])$ of high densities are deposited on the TBs, due to a dissociation reaction (the glissile Shockley partials moving on the TBs[14] are also shown). The strain fields associated with the increasingly stepped and bent TBs, may add dragging forces on moving dislocations. After large deformation the TBs lose coherency and become dislocation sources.

In contrast, dislocation accumulation is not as efficient if GBs are used to provide the ultrahigh strength. For equi-axed small (e.g., nc) grains of uniform sizes, dislocations cannot be easily trapped inside[7] as they readily get re-incorporated into the GBs a short distance away in all directions. In other words, dislocations created by multiplication would escape to sinks before interacting with other dislocations. The "storage" at already-high-angle GBs is balanced out for the most part by reorganization and annihilation inside the boundaries and not expected to be effective in sustaining pronounced work hardening. Moreover, if *d* is even smaller than the average distance a dislocation glides before creating another, the multiplication process itself ceases altogether.[15] If dislocations are used for reaching a high yield strength and for producing an UFG structure,[16] e.g., by severe deformation (Fig. 2), the dislocation density is already too high for the material to be in any effective strain hardening stage. When the strength of Cu reaches as high as >600 MPa, the dislocations would be only very small distances apart. Dynamic annihilation would occur at any temperatures above zero to render a low Θ.[17] The necking instability would come in and dislocation storage is pronounced only in the necked region where huge strains are concentrated.[17]

In summary, this work illustrates again the importance of controlling length scales in the microstructure for sustained high rates of strain hardening.[6] We adopted the following nanostructuring strategy: the nanocrystalline grain boundaries are "replaced" with a high population of coherent growth-twin boundaries. Dislocations are "saved" for use in subsequent strain hardening at high flow stress levels. The nano-twinned structure allows room for the dislocation multiplication and storage to continue to large plastic strains. These effects are accentuated at 77 K, demonstrating an ultrahigh strength combined with impressive uniform elongation and tensile ductility. The nano-twinned microstructure is an addition to the repertoire of nanostructured metals that are being tailored for high strength and ductility.[6,13,16,18,19] For conductors used for ultrahigh field magnets, the 77 K properties required include a strength (ultimate and fatigue) in excess of 1 GPa, a ductility of $>10\%$, and a conductivity of 500% IACS.[20] Our work here demonstrates the potential to develop such a high-performance material based on pure Cu.

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