Shear band formation and ductility in nanotwinned Cu

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The ductility and plastic flow behavior of highly aligned nanotwinned copper produced by interrupted magnetron sputtering is investigated. Tensile tests were performed at various strain rates at both room and liquid nitrogen (77 K) temperatures. Higher ductility and strength are reported for all samples tested at 77 K. The observed inhomogeneous deformation and shear band propagation are discussed as functions of the testing temperature, decreasing heat capacity at 77 K and low initial dislocation density, which leads to a yield peak.

Recent studies have shown the potential of highly nanotwinned Cu (nt-Cu), with improved ductility and high strength compared to nanocrystalline materials [1–3]. Lu et al. and Ma et al. have shown that electrodeposited copper with grain sizes of 400 nm to 1 μm having medium- to high-density nanotwins exhibit higher strength (600–900 MPa) than nanocrystalline Cu (~360 MPa) as well as higher ductility [4–6]. However, possible mechanisms that could explain the additional ductility have been limited, and the discussion has been focused on decreasing the lamella thickness (λ < 1.5 nm) [1–3], increased strain hardening [7–9] and the contribution of twins as nucleation sites for dislocations [10]. Enhanced ductility and strength at liquid nitrogen temperature (77 K) have been observed and reported in nt-Cu but related only to the presence of nanotwins [4].

In this study, we present a unique deformation mechanism for nanostructured nt-Cu, where the plastic deformation is caused solely by shear with an almost ideal plastic stress–strain response. Furthermore, we compare the deformation behavior during tensile tests at both room temperature (RT) and 77 K in order to elucidate the possible mechanisms that lead to the observed higher post-necking strain and overall ductility in highly nanotwinned metals at 77 K. We focus on the plastic flow behavior leading to shear band formation and expansion as a function of temperature. In comparison to other studies, the grains in this work are highly aligned. This can help in identifying single deformation mechanisms by eliminating random grain orientation.

High-purity (99.999%+) Cu foils (170 μm thick) were deposited onto (100) silicon wafers by interrupted DC magnetron sputtering, following the procedures described in previous publications [11,12]. The films were “freely” removed from the substrate and were handled as free-standing foils. Tensile tests (2–4 tests at a given strain rate) were performed at RT and 77 K using an Instron table-top universal testing machine. The gauge length of the dogbone-shaped samples is 6 mm (with a width of 3 mm). The initial Cu samples had grain widths of 500–800 nm, medium twin density (3.0 × 10⁶ m⁻²; Fig. 1 inset), high strength, low initial dislocation density and little to almost no strain hardening [11,12]. The samples tested at liquid nitrogen temperature were performed following a strict protocol to ensure that the temperature of 77 K was maintained throughout testing. The typical twin spacing is about 40 nm, as determined by focused ion beam (FIB; Multibeam JIB 4500 and FEI Nova 600 NanoLab) and a transmission electron microscope (Philips CM300-FEG) [11,13].

Tensile curves at various strain rates are presented in Figure 1 for tests performed at both RT and 77 K. The curves are not shown in true stress–strain since the deformation is highly non-homogeneous. It is noted that the results for the RT tensile tests are similar in both strength and total ductility (elongation to failure) when compared to studies on electrodeposited nt-Cu with similar twin spacing and density [3,5]. Further inspection of
Figure 1, together with Figures 2–4, highlights four distinctive features: the observation of a yield peak, which is due to the low dislocation density of the materials, as explained previously [11]; the lack of strain hardening in both types of testing environments; the deformation at both temperatures by shear band formation; and the samples tested at 77 K show both higher strength and post-necking ductility compared to the samples tested at RT. Of these, the latter two are the most puzzling [4,11,14]. Why do the samples tested at 77 K show both higher strength and higher ductility? Can this additional shear band formation really be labeled as ductility?

In order to address both questions, one must compare the failure mechanism for the samples tested at RT and 77 K. In Figure 2 we present a representative micrograph of the midsection of a tensile dogbone test deformed at RT at 10^{-4} s^{-1}. The sample clearly shows two deformation bands which, once they meet, become a failure site. This shear band type of deformation is typically found in bulk metallic glasses. It has also been reported in nanocrystalline materials, and highlights the typical lack of ductility of nanostructured materials [15–17]. If we compare the deformation micrograph to the stress–strain curves at RT, one can observe that one band forms and expands as the predominant deformation mode, while the second band is formed right after the yield peak. The shear band formation in respect to the yield peak is consistent with video observations by Carsley et al. in nanostructured Fe alloy [17,18]. This type of deformation was also observed by Wei et al. [19] in nanocrystalline (nc)-Fe, the behavior of which was described as “glasslike” due to the shear band formation and lack of strain hardening; the mode of deformation was believed to arise from dislocation-based mechanisms and not by adiabatic softening. In our manuscript, Figure 2a–c shows selected critical locations after deformation which are similar to the observations in nc-Fe [17,19]. Figure 2a is near the area of highest plastic deformation (several microns away from fracture edge), where some grain growth (grain width ~1.2 μm) is observed, while the twin spacing is still about the same size of the as prepared sample; in Figure 2b some grain rotation is observed (grain width ~650 nm), and in Figure 2c the grains and twin size do not change (grain width ~600 nm). In all three figures it is observed that the twins on either side of the boundary are out of register. This seems to point to a build-up of dislocations, as shown by Shute et al. on fatigued nanotwinned Cu samples [13].

In contrast, Figure 3 shows the sample tested at 77 K, which clearly exhibits necking (also seen in Fig. 4) as well as multiple deformation bands, which can also be observed in the shape of the stress–strain curve (Fig. 1), where a sequence of small yield peaks are associated with the sudden drops in the flow stress. The micrographs in Figure 3a–c are similar to those taken at RT, the main difference being that no out-of-register nanotwins were observed. It should be mentioned that in a recent study by Wang et al. [14], tensile tests performed at “cool” temperatures (meaning the sample was cooled to temperatures down to 120 K) deformed by detwinning. Additionally, Hong et al. [20] observed that the nucleation of shear bands in a twinned Cu–Al alloy subjected to dynamic plastic deformation started by bending and necking of the twin lamellae, followed...
by detwinning and subsequent formation of detwinned dislocation structures, which led to shear band formation. In our current study, we observed initial shear band formation without any detwinning (Figs. 2c and 3c). However, a more detailed analysis of detwinning mechanisms is currently underway and will be presented in a future publication.

The higher strength observed at liquid nitrogen temperature was expected, since dislocations move slower at 77 K and there is a general lack of mobile dislocations [21]. However, it is not clear why there is an increase in ductility at 77 K. It should be noted that the increase in total ductility comes mostly from post-necking elongation, in which the tests at 77 K show a 3–5% increase in post-necking strain. In both the RT and 77 K tests, the non-uniform plastic deformation occurred very shortly after the yield peak, and therefore there is no measurable change in the uniform plastic deformation (before necking).

In our study, there are several competing deformation phenomena which are unique to our system due to its high purity, low initial dislocation density and highly nanotwinned structure. First, we have more deformation bands at 77 K, each of which can carry large amounts of deformation compared to the RT tests. Here we propose that these additional deformation bands could be adiabatic, unlike the bands formed at RT. Even though one might typically think of adiabatic shear bands as being formed as part of ballistic deformation, they are also prevalent in low-temperature regimes where the heat capacity of a metal is low [22]. However, copper is not prone to forming adiabatic shear bands [23]. Therefore, the observation of adiabatic shear bands at 77 K is probably due to a combination of the effect of the nanotwins and the material’s heat capacity.

Basinski [24] observed a greater tendency for adiabatic shear band localization in metals when testing at low temperatures (4.2 K), and attributed this to a decrease in the material’s heat capacity as the temperature decreased. At very low temperatures, this propensity to form shear band can lead to a cup and cone type of fracture, as previously described by Chin and Backofen [25] and shown in Figures 3 and 4c. The temperature rise in plastic deformation is inversely proportional to the heat capacity (Cp); thus, the higher local temperature rise at 77 K should lead to a greater propensity to shear localization. For Cu, the heat capacity decreases by about half from RT to 77 K [26,27]. However, even taken into account the decrease in Cp, Cu is still not expected to form shear bands since it typically does not show a large change in flow stress at low temperatures [22]. Therefore, following the observations by Chin et al. [25], an additional event during testing, such as a yield peak, could help to nucleate the initial deformation needed to create favorable conditions for shear band formation. In our sample, the highly nanotwinned structure coupled with the low initial dislocation density leads to a yield peak at which an avalanche of dislocations is expected. A yield peak has been shown to be a contributing factor for adiabatic shear band localization in tests performed in Fe and other body-centered cubic (bcc) materials, which is highly linked to the fact that dislocation sources are strongly locked by interstitial solute atoms in the bcc structure [23,25,28], perhaps similar to the locking effect of the nanotwins.

Another critical factor in understanding the overall ductility is analyzing the strain hardening effects. In our study, all fracture surfaces for all tests at both temperatures showed ductile fracture, as seen in Figure 4b and d. It has been previously presented that on electrodeposited highly nanotwinned Cu samples there is an increase in strain hardening behavior linked to the increase in ductility [2,9]. In this study we observed an almost perfectly plastic behavior in the stress–strain curves, with a very slight increase in strain hardening at 77 K. Given that our material has a low initial dislocation density, it is difficult to observe any dislocation accumulation from our current figures. However, in our previous study of rolled nt-Cu (which leads to a high initial dislocation density), it was clearly shown that there is enhanced strain hardening at 77 K [11]. An increase in strain hardening as a function of temperature could be due to changes in the dislocation cell size as a function of temperature. Staker and Holt [29] showed that the dislocation cell size decreases for Cu as a function of temperature, and this can be directly correlated to the equation \( l_c = A \sigma b / \mu \) [30], where \( b \) is the Burgers vector (2.56 × 10^{-10} m), \( \mu \) is the shear modulus (4.21 × 10^{10} Pa), \( A_c \) is a constant, \( \sigma \) is the tensile stress and \( l_c \) is the dislocation cell size. From this equation, we can approximate that at RT the dislocation cell size is \(~500\) nm, close to the grain size (600 nm); therefore there is no dislocation accumulation in the sample since the existing dislocations are absorbed by the grain boundaries. At 77 K, the dislocation cell size is \(~300\) nm, half of that at RT, and therefore dislocations could accumulate inside the grain and some strain hardening can be achieved. However, as stated by Yu et al. [31] in their study of UFG Al, once the grain size has decreased below a critical value corresponding to the cell size at a given temperature, the mean free path of dislocations is no longer determined by the dislocation structure, but rather is limited by the grain boundaries. How exactly this statement relates to a highly nanotwinned fracture.
structure is still up for debate, including which factor (twins or grains) controls the strain hardening behavior.
In this manuscript we have proposed a combination of several mechanisms for the observed high “ductility” in our nt-Cu. These observations are based on a highly idealized material which contains aligned nanotwins and a low initial dislocation density.

We were able to show that the observed “enhanced ductility”, comprised mostly of post-necking strain, is due to the mutual effect of very low temperatures and the presence of a yield peak. However, it must be emphasized that the deformation mode for these materials was always by shear band formation and further research is still needed in order to understand the nt-materials deformation. The specific mechanisms discussed in this paper present a unique perspective for nt-materials and could enhance further understanding of non-homogeneous deformation in nanostructured materials.

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