High strength and high electrical conductivity in bulk nanograined Cu embedded with nanoscale twins

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A bulk nanograined Cu sample embedded with nanoscale twins is produced by means of dynamic plastic deformation at cryogenic temperatures. It exhibits a tensile yield strength of 610 MPa and an electrical conductivity of 95% IACS at room temperature. The unique combination of a high strength and a high conductivity is primarily attributed to the presence of a considerable amount of nanoscale twins which strengthen the material significantly while having a negligible influence on electrical conductivity. © 2007 American Institute of Physics. [DOI: 10.1063/1.2816126]

Mechanical strength and electrical conductivity are the most important properties of conducting materials. However, high strength and high electrical conductivity are mutually exclusive in materials strengthened via conventional approaches such as strain hardening, alloying, or grain refinement. Trade-off between strength and conductivity is always encountered in developing conducting materials. A recent investigation indicated that introducing a high density of nanoscale twins (NTs) in an ultrafine-grained (UFG) Cu may significantly strengthen Cu while keeping its high electrical conductivity. A high tensile strength (~1 GPa) combined with a high electrical conductivity [97% International Annealed Copper Standard (IACS)] was achieved when the average twin/matrix (T/M) lamellar thickness is as small as 15 nm in Cu thin foils produced by means of pulsed electrodeposition.

However, such a unique strength-conductivity combination could only be achieved in thin foil Cu samples, of which technological applications are limited. Synthesis of bulk Cu samples with NTs becomes a challenge. In this letter, we report our success in processing bulk nanograined Cu embedded with a considerable amount of NTs by means of dynamic plastic deformation (DPD) at cryogenic temperatures, which exhibits an excellent combination of a high strength and a high electrical conductivity.

A copper cylinder (18 mm in diameter and 25 mm in height) with a purity of 99.995% was processed by means of DPD at liquid nitrogen temperature (LNT). The setup and parameters of the DPD processing were described in detail in Ref. 3. The basic principle of the DPD process is that the sample is compressed at very high strain rates (~10^3 s^-1) and low temperatures, which ensure that the dislocation recovery is effectively suppressed. Under such circumstances, the critical stress for twinning is ready to be exceeded. Hence, deformation twinning (instead of dislocation activities) becomes the dominant plastic deformation mechanism. The Cu cylinder was deformed into a disk with a total true strain of 2.1, where the strain is defined as \( \varepsilon = \ln(L_f/L_0) \) (\( L_0 \) and \( L_f \) are the initial and the final thicknesses of the treated sample, respectively). The final sample dimension is about 50 mm in diameter and 3 mm in thickness. The deformation is fairly uniform and the as-processed sample is free of cracks. Measurements showed that the density and purity of the DPD Cu samples are identical to those of the original ones.

Figure 1(a) is a typical cross-sectional transmission electron microscopy (TEM) image of the bulk DPD Cu, which shows a mixed microstructure of nanograins and a considerable amount of twins. The T/M lamellar thickness is in the nanometer regime. Nanoscale T/M lamellae form bundles of several hundred nanometer to several micrometer thick and micrometer long, most of which are roughly aligned perpendicular to the loading direction. Detailed observations revealed that most twin boundaries (TBs) are stepped or curved, typical characteristics of boundaries for deformation twins at which a high density of dislocations exists, as shown in Fig. 1(b). The statistical measurements show that the volume fraction of twin bundles is about 35% and the average T/M lamellar thickness is about 44±2 nm [see Fig. 1(d)]. Most nanograins are elongated with an aspect ratio of ~2.5 and some grains are with diffuse grain boundaries (GBs) [as shown in Fig. 1(c)]. The histogram of the overall grain size distribution shows an average grain size (in short axis) of 66 nm [Fig. 1(e)].

Formation of a considerable amount of NTs in the DPD Cu sample is a direct consequence of the suppression of dislocation activities during the plastic deformation at high strain rates and low temperatures. The deformation twinning becomes a dominant mechanism for plastic deformation. In addition, formation of NTs is crucial for the formation of nanograins. As described in Ref. 3, most of the nanograins are derived from the fragmentation and shear banding of the nanoscale T/M lamellae, while a fraction of nanograins is evolved from dislocation cells which are associated with a high density of dislocations.

The tensile tests show that the bulk DPD Cu sample exhibits an impressive yield strength of 610±10 MPa, which is about one order of magnitude higher than that of coarse grained (CG) Cu. The elongation to failure is about 8%. It should be noted that the yield strength of the DPD Cu is significantly higher than those ever achieved for bulk Cu processed by means of severe plastic deformation (SPD) at room temperature (usually below 400 MPa). It is also higher than that of Cu processed by cold drawing at LNT, in which a small volume fraction of twin bundles were...
LNT can barely elevate the strength to about 460 MPa. Additional cold rolling (CR) of the SPD Cu at LNT can barely elevate the strength to about 460 MPa. Electrical conductivity of the DPD Cu was measured on strip specimens with a cross-sectional area of $0.5 \times 1 \text{ mm}^2$ and a length of 12 mm using the standard four-probe technique at ambient temperature. At least five specimens were measured to obtain an average value. The average conductivity value is $0.55 \pm 0.01 \Omega^{-1} \text{m}^{-1}$, which is about 95% (±2%) IACS. It is noteworthy that electrical conductivity of the DPD Cu is slightly lower than that of the CG Cu ($0.595 \times 10^8 \Omega^{-1} \text{m}^{-1}$) and is comparable to that of the UFG Cu processed via SPD (e.g., equal channel angular pressing or cold rolling), as plotted in Fig. 2. The conductivity of the DPD Cu is slightly lower than that of the UFG Cu thin foils with NTs ($0.571 \times 10^8 \Omega^{-1} \text{m}^{-1}$). However, the conductivity of DPD Cu is significantly higher than the reported values of pure Cu samples with nanoscale grain sizes. The nanograined Cu produced by ball milling exhibited a similar strength with our present sample, but a much lower electrical conductivity (51% IACS).

It is known that alloying Cu with other elements, such as Cr, Fe, Ni, etc., can elevate strength to a level of 500–700 MPa but accompanied with a significant loss in conductivity. The electrical conductivities of the corresponding Cu alloys range from 50% to 80% IACS. By plotting yield strength versus electrical conductivity of the SPD Cu and Cu alloys, as shown in Fig. 2, one may see that the data points fall in the shade regime at the lower-left side. Distinctly, the DPD Cu sample stands out from this regime, representing a superior strength-conductivity combination. Comparison with Cu–Cr–X alloys (widely used as conductors), the DPD Cu exhibits a similar strength but a much higher conductivity.

The high strength of the DPD Cu is in part a consequence of the extremely refined nanograins. Previous investigations indicated that the classical Hall-Petch relation is valid for nanograined Cu with grain size as small as 10 nm. In terms of this, a yield strength of about 455 MPa is expected for an average grain size of 66 nm. TBs are also effective in blocking dislocation motions, hence strengthening the materials like conventional GBs. A yield strength of about 831 MPa was estimated for the deformation twins with an average $T/M$ lamellar thickness of 44 nm. Following the rule of mixture, a total yield strength of the DPD Cu sample is estimated to be about 590 MPa, which agrees reasonably with our measurement strength (610 MPa). Obviously, the strengthening effect of NTs is predominant in the present sample relative to the grain size effect, although the volume fraction of NTs is lower than that of the nanograins.

The slight drop in electrical conductivity of the DPD Cu relative to the CG Cu originates from the presence of plenty of interfaces including GBs and TBs, as indicated in the nanocrystalline Cu and the Cu samples deformed at cryogenic temperatures. The intrinsic GB resistivity of Cu is closely related to the GB structures and energy state and can vary from $0.5 \times 10^{-7}$ to $2.5 \times 10^{-7} \text{n}\Omega\text{m}^{-2}$ with GB misorientations increasing from low to high angles. The electrical resistivity of coherent TB is usually taken as half of the intrinsic resistivity of the materials like conventional GBs.
specific stacking fault resistivity, i.e., $1.5 \times 10^{-8}$ nΩ m² in Cu, which is much lower than those of conventional GBs. Recent systematical investigations showed that the numerous dislocations accumulated at the TBs have a negligible influence on the specific TB resistivity. Consequently, the contribution of deformation TBs to the overall electrical resistivity should be approximately in the order of 0.1 nΩ m in the present sample.

In the present sample, characteristics of the GBs are complicated. First, GB misorientations in the present sample vary from low to high angles, the volume fraction of which is difficult to be determined due to the nanoscale grain size. Second, parts of the nanograins are derived from the fragmentation of $T/M$ lamellae, of which misorientations slightly deviate from the exact $\Sigma 3$ relationship by a few degrees. However, these parts of GBs are expected to still possess a lower resistivity compared with the conventional GBs. For simplicity, it is supposed that the specific GB resistivity varies in the same range as the above mentioned, then the total GB contribution to the electrical resistivity should be between 0.89 and 4.4 nΩ m. In comparison with GBs, the contribution of TBs to the electrical resistivity is negligibly small, though the TB area per unit volume is comparable to that of the GBs. In other words, the decrease of electrical conductivity in the DPD Cu compared with the bulk CG Cu primarily resulted from the presence of a large number of GBs. In terms of the above analysis, the conductivity drop in the DPD Cu owing to GBs is estimated to be about 2%–18%. Considering the measurement uncertainties in grain sizes and the GB characteristics, the predicted value is in an acceptable agreement with the experimental result.

In order to verify the contribution of NTs and nanograins to strength and electrical conductivity, the DPD Cu sample was subjected to an additional CR with a rolling strain of 50% at ambient temperature (the rolling strain is defined as $e = (S_f - S_i)/S_i \times 100\%$, in which $S_i$ and $S_f$ are the cross-sectional areas of the sample before and after CR), and details about the CR are in Ref. 11. After CR, the volume fraction of NTs decreases to about 24% primarily due to the fragmentation of NTs into nanograins. Meanwhile, the volume fraction of nanograins becomes larger, and the average grain size increases slightly to about 90 nm. In addition, GB relaxation occurred during rolling and some GBs exhibited sharper contrasts. Tensile tests showed that the DPD+CR Cu exhibits a yield strength of 545 MPa, which is lower than that of the DPD Cu. The electrical conductivity at room temperature is about 97% IACS. The yield strength decrease is directly resulted from the reduction of the volume fraction of NTs. In terms of the rule of mixture, the predicted strength reduction is about 55 MPa, which is consistent with the measured data. The majority of the strength drop originates from the decreasing volume fraction of twin bundles while the slight increase in grain size plays a minor role.

Upon CR, the volume fraction of nanograins increases with a slight increase in grain size while the volume fraction of NTs decreases. Hence, the slight increase in electrical conductivity after CR can be reasonably attributed to GB relaxation. These results suggest that the electrical conductivity in the DPD Cu is determined by the amounts and characteristics of GBs while the TBs play a minor role.

In summary, a bulk nanograined Cu sample with embedded NTs was produced using the DPD processing at LNT, which exhibits a unique combination of high strength and high electrical conductivity. The high strength originates from the extremely refined nanostructure (NTs and nanograins), in which the NTs play a predominant role in strengthening. The high electrical conductivity is mainly attributed to the high density of TBs with extremely high specific electrical conductivity. The present work demonstrates clearly that introducing a high density of NTs into bulk Cu is an effective approach to elevate strength without sacrificing its high electrical conductivity.

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