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Influence of temperature on the strain rate sensitivity and deformation mechanisms of nanotwinned Cu

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ABSTRACT

The mechanical behavior of nanotwinned Cu was studied through indentation creep and constant strain rate indentation tests from 25 °C to 200 °C. The results showed an enhanced strain rate sensitivity of nanotwinned Cu with temperature, which was higher than that found in coarse-grained Cu. Transmission electron microscopy revealed the same deformation mechanism in the whole temperature range: confined dislocation slip between coherent twin boundaries and formation of dislocation pile-ups at the coherent twin boundaries. The mechanisms responsible for large increase in strain rate sensitivity of nanotwinned Cu with temperature were discussed to the light of experimental observations.

Metals with nanoscale twins have received increasing attention in recent years because of their unique properties [1–5]. In particular, nano-twinned (NT) metals (twin spacing <100 nm) achieve a strength and hardness similar to nanocrystalline (NC) metals (grain size <100 nm) [6], but are able to maintain substantial ductility [5,7]. In addition, NT metals present outstanding thermal stability, electrical conductivity and fatigue resistance [8]. These differences reflect the particular nature of coherent twin boundaries (CTB), as compared with standard grain boundaries, which lead to the activation of different deformation mechanisms. Obviously, further understanding of the dominant deformation processes under different conditions is necessary to design NT metals with improved properties. However, the current knowledge of the deformation of NT metals is still limited to ambient temperature despite that the plastic deformation of metals is a thermally activated process. This investigation was aimed at exploring the influence of temperature on the mechanical properties and deformation mechanisms of these materials.

Most of the work on NT metals has been focused in Cu. High density of NTs with modulated twin structure can be introduced in Cu by pulsed electrodeposition. NT-Cu processed following this route shows a tensile strength about 10 times higher than that of conventional course-grained (CG) Cu, and an electrical conductivity comparable to that of pure Cu [1]. This combination of properties comes about as a result of the large density of CTBs which block the dislocation slip while presenting extremely low electrical resistivity, that cannot be achieved by other types of grain boundaries. The mechanical behavior of NT-Cu at ambient temperature has been extensively studied by means of uniaxial tension [7,9,10], nanoindentation [9,11–13], micropillar compression tests [14,15] and theoretical models [16]. CTBs and grain boundaries (GBs) act as dislocation sources during deformation and plastic deformation is carried by the formation of dislocation pile-ups along the CTBs [9,17]. Recent studies have revealed that the strain rate sensitivity (m) of NT-Cu at ambient temperature and quasi-static loading (strain rate 10^{-4} s^{-1}–10^{-1} s^{-1}) was in the range 0.03–0.08. These values were much higher than those of its coarse-grained (CG) polycrystalline counterparts (0.004–0.007) [13] and contribute to delay necking and increase the ductility during tensile deformation.

In this investigation, the deformation mechanisms of NT-Cu were explored in the temperature range 25 °C to 200 °C by means of creep and constant strain rate indentation tests. The results of the mechanical tests (in terms of the strain rate sensitivity and activation volume) were completed with the help of transmission electron microscopy (TEM) observation to ascertain the dominant mechanisms of plastic deformation as a function of temperature.

High-purity Cu (99.99 wt%) sheets with nanoscale growth twins were synthesized by means of direct-current electro-deposition from an electrolyte of $CuSO_4$. More details about the deposition parameters can be found in [18]. All the deposition parameters (including temperature, pH, solution volume, current density, *etc.*) were kept almost

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constant during electrodeposition to ensure the homogeneity and consistency of the microstructure of the different sheets. The NT-Cu sheet was deposited on a Ni substrate. The final sheet thickness was >1.5 mm. A CG Cu with similar grain size prepared by the same electrolyte using DC electrodeposition was also studied for comparison [19]. The initial dislocation density of the CG-Cu was in the range 10^{10} to 10^{12} m^{-2}.

The textures of NT-Cu and CG-Cu sheets were analyzed by means of electron backscatter diffraction (EBSD), using a field-emission gun SEM (Helios Nanolab 600i, FEI) equipped with an HKL EBSD system. The average grain size was determined by the line intercept method from the EBSD micrographs. TEM was used to analyze the microstructure of twins and twin boundaries and their interaction with dislocations. A trenching-and-lifting-out focused ion beam technique was adopted to extract lamellae directly from the indentation imprints, which was subsequently thinned to approximately <100 nm for electron transparency. TEM observation was carried out in two-beam diffraction mode [20], employing different diffraction vectors, g, using the $g \cdot b = 0$ invisibility criterion, where b stands for the direction of the Burgers vector.

The mechanical properties of NT-Cu and CG-Cu samples were measured by means of indentation creep tests in a NanoTest™ platform III (Micro Materials, Wrexham, UK). This platform uses an independent tip and sample heating system that is designed to maintain the temperature within ±0.05 °C, which is the best strategy to achieve thermal equilibrium during indentation in order to minimize thermal drift. Load was applied with a diamond Berkovich indenter. It was initially increased up to 50 mN in 10 s in the nanoindentation creep tests and was maintained constant during 400 s followed by unloading at the rate of 5 mN/s. The evolution of the indentation depth, h, *vs.* time, t, was recorded during the holding time. Tests were carried out at 25 °C, 50 °C, 100 °C, 150 °C and 200 °C, respectively. At each temperature, thermal drift was carefully minimized (<0.1 nm/s) to equilibrate the temperatures of both the indenter and the sample. At least 5 tests were performed at each temperature and the results presented below for each temperature are the average value and the standard deviation of these tests.

The EBSD maps of the as-received CG-Cu and NT-Cu are shown in Fig. 1(a) and (b), respectively. The average grain size of the CG-Cu was 22 ± 4 μm with random texture and only a few twins were found within each grain. The average grain size of the NT-Cu was 14 ± 2 μm and the microstructure presented a strong (111) texture, which may result in smaller grain misorientation, and, therefore, reduces the driving force for grain growth at high temperatures [21,22]. High density of twins was found in most grains of the NT-Cu, leading to nm-thick twin/matrix lamellar structures, as shown in Fig. 1(c). The diffraction spots of the twins are clearly seen in the selected-area diffraction pattern of NT-Cu in the zone axes $\langle 01\bar{1} \rangle$ in Fig. 1(d). Only a few dislocations pinned at CTBs were observed in the as-received NT-Cu. The nanotwinned structure was retained after thermal exposure up to 200 °C. Fig. 1(e) plots the statistical distribution of twin lamella thickness in the as-received samples and after testing at 200 °C. The average twin thickness in the as-received sample was ≈25 nm and increased up to ≈40 nm after thermal exposure. It has been reported that the CTBs are more thermally stable than the grain boundaries due to their low coherent energy (24–39 mJ/m^2) [22,23] and,

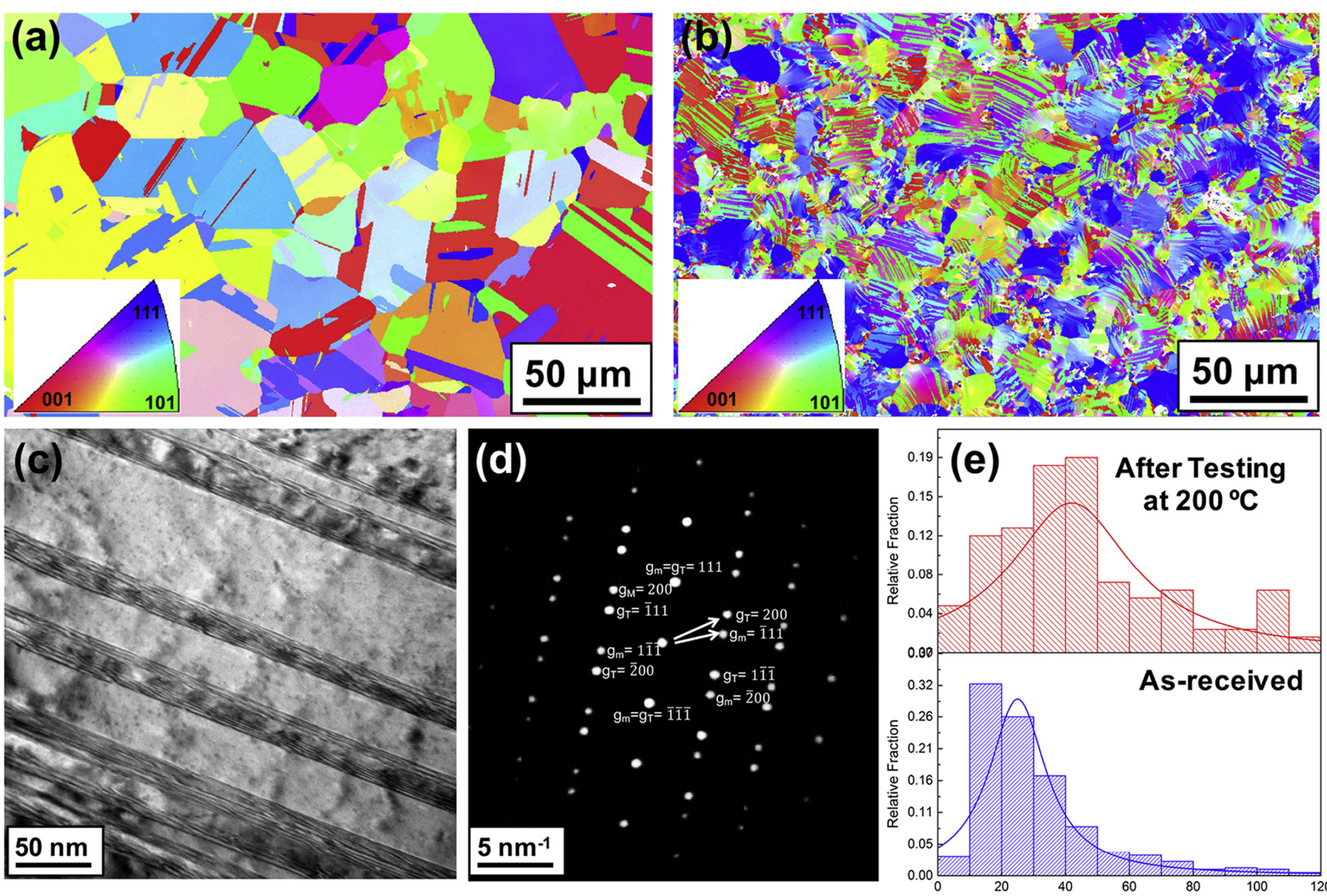

Fig. 1. (a) EBSD map of CG-Cu. (b) *Idem* of NT-Cu. (c) High resolution TEM of twin structure within a grain of NT-Cu. (d) Selected-area diffraction pattern of the NT-Cu in the zone axis $\langle 01\bar{1} \rangle$. (e) Histograms of twin thickness in NT-Cu in the as-received condition and after testing at 200 °C.