Available online at www.sciencedirect.com
SciVerse ScienceDirect
Scripta Materialia 66 (2012) 849–853
Scripta MATERIALIA
www.elsevier.com/locate/scriptamat

Viewpoint Paper

Repeated frictional sliding properties of copper containing nanoscale twins

A. Singh,[a] N.R. Tao,[a,b] M. Dao[a,*] and S. Suresh[a]

[a]*Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139, USA*
[b]*Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, 72 Wenhua Road, Shenyang 110016, People's Republic of China*

Available online 17 February 2012

Abstract—Bulk dynamic plastic deformation (DPD) materials comprise a composite structure of nanoscale twin bundles and nanoscale grains. The tribological properties of DPD-processed pure nano-Cu have been investigated in this study and compared with conventional coarse-grained (CG) Cu under both monotonic and repeated frictional sliding. We demonstrate that DPD nano-Cu and CG Cu exhibit steady-state mechanical characteristics after repeated frictional sliding that are similar to those seen in nanotwinned (NT) Cu produced by pulsed electrodeposition.

Keywords: Contact sliding; Wear; Nanotwin; Nanocrystalline; Fatigue

1. Introduction

Grain refinement has been a traditional way to attain marked improvements in such properties as strength, wear and corrosion resistance, and diffusivity in metals [1–8]. Recent studies have also revealed that nanoscale twins introduced in the microstructure lead to improved mechanical properties not only in terms of higher strength, but also in retention of reasonable ductility, higher rate-sensitivity of deformation, and greater resistance to both fatigue crack initiation and growth [9–11]. These effects can be further enhanced through refining nanotwin (NT) spacing. However, high-quality pure nanotwinned specimens have thus far been produced successfully only in thin-film form [9–13]. This can impose severe constraints on potential applications of nanotwinned materials for bulk structural components.

Dynamic plastic deformation (DPD) offers a means to manufacture large specimens of nanostructured materials that comprise a mixture of nanotwins and nanograins [14,15]. While the basic mechanical properties of DPD Cu have been studied previously [14,15], the tribological response and the attendant microstructural stability of DPD nano-Cu under repeated frictional sliding have thus far not been investigated. Design against wear damage is a topic of considerable technological interest as it is one of the most common ways of material loss in most engineering applications [16]. Excessive material removal due to repeated frictional sliding and rubbing between contacting surfaces can lead to device dysfunction in micro electromechanical systems, failure of engineering components such as ball bearings and adverse immune response in the human body to metal-based biological implants. There have been only limited studies done on the wear response of nanograined (NG) and NT materials in general. In NG Ni, wear damage has been shown to decrease with decreasing grain size, but beyond the Hall–Petch breakdown point increased material loss was observed with grain refinement [6]. However, degraded wear response with strengthening has been observed in NG Ni–B alloy film produced by electrodeposition [17] and NG iron produced by rolling [18]. Grain coarsening under repeated sliding has been documented for NG Ni [8] as well as for NG Ni–W [19]. Moreover, a recent experimental study on repeated frictional sliding of NT Cu within ultrafine grains averaging 450 nm in size [11] confirmed that higher-density NT Cu exhibited greater resistance to both microstructure changes and surface damage after a frictional sliding pass; however, after many repeated sliding passes, copper samples with different NT densities were found to exhibit similar surface hardness and microstructure. This behavior bears a

* Corresponding author. E-mail: mingdao@mit.edu

1359-6462/$ - see front matter
doi:10.1016/j.scriptamat.2012.02.017

striking resemblance to the uniaxial strain-controlled fatigue behavior of medium-to-high stacking fault energy (SFE) metals, in which a uniform steady-state structure evolves after repeated mechanical loading as a result of rearrangements in defect structure facilitated by cyclic deformation.

Given the complexity of its microstructure, it is a challenging task to predict the tribological response of DPD nano-Cu only from its elasto-plastic properties. Moreover, the heterogeneous microstructure of DPD nano-Cu that comprises mixtures of nanotwin bundles and nano grains undergoes complex structural changes during frictional sliding because NG and NT materials influence structural evolution in different ways. It is therefore desirable to undertake experimental studies to ascertain the repeated frictional sliding response of DPD and contrast it with coarse-grained (CG) Cu under the same sliding test conditions, and to compare these results with NT Cu. Repeated frictional sliding tests provide controlled and quantitative measures that enable a fundamental understanding of the mechanisms underlying structural evolution during cyclic contact and the attendant wear damage processes.

The present work examines the tribological response of DPD nano-Cu and CG Cu by means of instrumented scratch tests under monotonic and repeated frictional sliding conditions. Friction coefficient and pile-up height are documented as functions of the sliding pass number. We have also studied the deformation-induced hardness evolution by indenting within the scratch tracks. These measurements enable the establishment of possible connections between tribological properties that develop in response to damage accumulation under repeated sliding and hardness/microstructure evolution. Repeated contact sliding (contact fatigue) is also seen to have an apparent similarity to strain-controlled uniaxial fatigue loading in that both types of loading produce plastic properties and the final corresponding microstructure that depend on the specific stress state, temperature and strain rate applied, but do not depend on the loading history for high SFE metals [20,21]. It is interesting to find that the eventual flow strength attained for CG Cu and DPD nano-Cu under similar experimental conditions was slightly lower than that obtained for NT Cu produced by pulsed electrodeposition (PED) [11]. This suggests that the processing method and the nature of twin boundaries (coherent twin boundaries in the case of PED vs. nanotwins generated from deformation twinning for DPD) may also be important factors that influence the final hardness and the corresponding microstructure attained in the deformation-affected zone.

2. Materials and experimental method

2.1. Materials

A copper cylinder with a diameter of 16 mm and a height of 26 mm was mechanically polished with SiC paper and subsequently annealed for 120 min in a vacuum (10^{-3} torr) at 700 °C to achieve a final average grain size value of approximately 250 μm. DPD treatment involved placing the copper cylinder on a lower anvil and compressing with an upper impact anvil at a strain rate of 10^2–10^3 s^{-1}. The specimen was totally immersed in a liquid nitrogen bath during each impact to prevent dynamic recovery and recrystallization. The deformation strain during the DPD process, defined as $\varepsilon = \ln(L_0/L_f)$, where L_0 and L_f are the initial and final thickness of the specimen, respectively, was 2.1. The resulting DPD nano-Cu specimen was essentially without any flaws or pores at the level of structural dimensions, and the final density and purity were found to be comparable to those of the original CG sample. The microstructure comprised ~67 vol.% of nano grains and ~33 vol.% of deformation twin bundles. The yield strength was measured to be 600 MPa and the ductility was 11% [14]. Essentially no strain hardening was observed. Further details of specimen preparation and microstructural and mechanical characterization can be found in [14]. In order to develop a basis for comparison for the foregoing DPD nano-Cu, a copper bar with an initial size of 8 mm × 8 mm × 12 mm was also mechanically polished with SiC paper and subsequently annealed for 120 min in a vacuum (10^{-3} torr) at 700 °C to achieve a final average grain size of approximately 250 μm. The bar, hereafter referred to as CG Cu, was directly used for companion experiments.

2.2. Experimental procedure

The frictional sliding experiments on DPD nano-Cu and CG Cu were performed using a NanoTest™ (Micromaterials, Wrexham, UK) instrumented indenter. A conical diamond tip (with a half tip angle of 70.3° and a tip radius of 2 μm) was used to make an array of scratches on the sample surface. This was accomplished by first making initial contact with the polished specimens (thoroughly cleaned in an ultrasonic ethanol bath), subsequent to which a normal load was applied on the specimen as the sample stage was moved laterally at a steady rate of 5 μm s^{-1}. The normal load was ramped up to its maximum value of 500 mN over the initial 50 μm of the sample-stage motion and allowed to stay constant for the remaining 450 μm length of the scratch. Thus, a 500 μm long scratch was introduced at the termination of one sliding pass. After that, the sample stage was first retracted by 15 μm away from the tip, and then returned to its original position

before the subsequent, new sliding pass. Multiple sliding over the same scratch path can be accomplished by repeating the process described above. The indenter was programmed to make 1, 17, 34, 50, 66, 82 and 98 sliding cycles, which left seven parallel tracks on DPD nano-Cu as well as CG Cu specimens. In order to avoid the interaction of strain-induced deformation zones between adjacent scratches, the grooved tracks were spaced amply apart from each other. Tangential loads were also acquired for the entire length of the scratch by force transducers mounted on either side of the indenter tip.

The same instrumented indenter was used to conduct all the indentations. First, 25 indentations were made on each specimen in order to ascertain the hardness. The same conical diamond indenter tip (with half angle of 70.3° and a tip radius of 2 μm) was used. The