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Regular Article

A gradient nanostructure generated in pure copper by platen friction sliding deformation

S.Q. Deng [a], A. Godfrey [a,*], W. Liu [a], N. Hansen [b]

[a] Key Laboratory of Advanced Materials (MOE), School of Material Science and Engineering, Tsinghua University, Beijing 100084, PR China
[b] Danish-Chinese Center for Nanometals, Section for Material Science and Advanced Characterization, Department of Wind Energy, Technical University of Denmark, 4000 Roskilde, Denmark

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ABSTRACT

A modified friction sliding process with a large applied normal load has been used to develop a gradient nanostructure in Cu using only a short processing time. A quantitative characterization of the variation in microstructure and strength has been carried out by combined use of electron backscatter diffraction and hardness measurements, and the data used to estimate the effective strain profile resulting from the processing treatment. The affected deformation volume extends to a large depth of more than 1 mm, with a top surface hardness of 2.28 GPa, corresponding to a four-fold increase compared to the initial undeformed material.

A variety of techniques for generating gradient nanostructures in metals have been investigated, including surface mechanical attrition treatment [1,2], shot-peening [3,4], surface mechanical grinding treatment (SMGT) [5], burnishing [6–8] and sliding deformation [9–13]. In this study we investigate a variant of sliding where one planar surface is moved over another while under a compressive load. In contrast to similar previously reported techniques [9,10,13] our modified platen friction sliding deformation (PFSD) process [14] involves the use of a significantly larger compressive load during sliding and an enhanced surface roughness, with the dual objectives of increasing the strain gradient introduced during deformation (as well as the near-surface hardness), and increasing the depth to which hardening takes place below the surface.

A detailed knowledge of the resulting strain gradient is important as this provides the basis for both evaluation and further development of computer models of surface deformation processes [15]. The shear strain profile (representing the material flow) can in certain cases be estimated by the use of microstructural markers, as demonstrated recently by Moering et al. [16]. Here we use quantitative microstructural data to explore an alternative approach for estimation of the effective strain profile, representing the equivalent strain in bulk deformation resulting in the same microstructural scale and hardness. This is a more general approach and provides information on the combination of the shear strain gradient, together with the local strain rate gradient (and hence any heating) and the material chemistry.

For this purpose we use the electron backscatter diffraction (EBSD) technique for microstructural characterization. This has the advantage of allowing examination of large areas of material, and is therefore less sensitive to variations in the microstructure along the sliding direction at any given depth than transmission electron microscope-based observations. Additionally this technique allows a direct correlation of hardness measurements with the local microstructural characteristics.

As a model material, and to allow comparison with previous studies of friction sliding under different conditions [9,10,13,17–20] oxygen-free high conductivity Cu (99.9% purity) was used in this study. The initial material was in a fully recrystallized condition with an average grain size of 73 μm (determined ignoring annealing twins) and a nearly random texture. As illustrated schematically in Fig. 1a the PFSD system consists of two perpendicularly arranged loading frames. The sample is held with the outer surface in full contact with the friction platen (in this experiment a hardened steel bar) under a compressive load, applied using the horizontal loading frame. The compressive force is controlled using a load cell placed in line with the sample, on the opposite side of the friction platen. The sample is fully supported on its underside to prevent slippage during the friction sliding process. The friction platen is held by a separate vertical loading frame, which is used to push the platen down, resulting in deformation to the surface of the sample. The maximum sliding distance that can be achieved in one pass is determined by the space available for travel of the platen, which in our case was 60 mm.

In this study, Cu samples of size $11.5 \times 11.5 \times 9$ mm^3 were used, with the square face of the sample placed against the friction platen (of size $32 \times 32 \times 100$ mm^3). The PFSD process was carried out at room temperature using a normal (compressive) stress of 44 MPa, a platen-sliding speed of 6.7 mm s^{-1}, and a sliding distance of 60 mm. In order to highly refine the microstructure the PFSD process was repeated using

* Corresponding author.
E-mail address: awgodfrey@mail.tsinghua.edu.cn (A. Godfrey).

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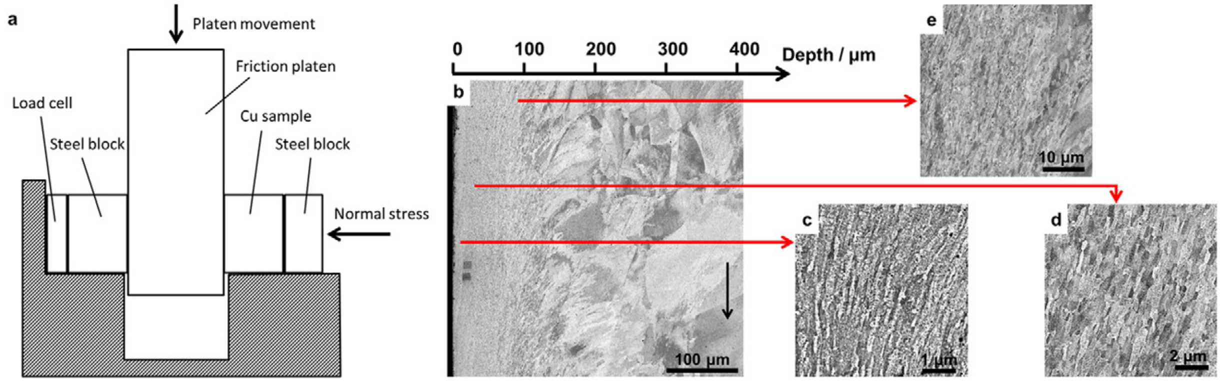

Fig. 1. (a) Schematic illustration of the PFSD set-up; (b–e) SEM micrographs of the Cu sample after PFSD, showing (b) the transition from highly refined surface to deformed grains in the top 400 μm of the sample; (c) nanoscale lamellae at 10 μm depth; (d) fine-grain structure at 30 μm depth; and (e) regular deformation lamellae at 90 μm depth. The arrow indicates the sliding direction of the friction platen. Direct evidence of shearing can be observed to at least a depth of about 160 μm, based on the curving of microstructural features such as twin boundaries and deformation bands towards the sliding direction.

four passes over the same area (resulting in a total sliding distance of ≈240 mm and a total deformation time of less than one minute). Moderately high values of initial surface roughness for both the Cu sample and the friction platen were chosen (measured as Ra = 5.1 μm and Ra = 3.2 μm, respectively). The effect of surface roughness on the PFSD process is described elsewhere [21]. After deformation all samples were held at −18 °C in a freezer to prevent recovery and recrystallization during storage.

The microstructure resulting from PFSD was examined on polished cross-sections (perpendicular to the sliding surface, and containing the sliding direction), and was characterized using a Tescan Mira 3 LMH thermal field emission scanning electron microscope (FE-SEM) equipped with an Oxford Instruments HKL Nordlys Max EBSD system. The strength was estimated from Vickers micro-hardness indentations, using a load of 10 g and a load duration of 10 s on the same cross-sections. These values resulted in indent sizes in the uppermost volumes in the range of 9–12 μm. Accordingly in the top 100 μm of the sample hardness indents were taken at 10 μm intervals, but offset from each other along the sliding direction to achieve a minimum spacing between indents of ≈30 μm. The indent in the cross section nearest the top surface was taken at a depth of 12 μm. To allow an estimation of the strain gradient introduced during PFSD, samples of the same initial material were also deformed by cold-rolling to a large von Mises strain of $\varepsilon_{vM} = 5.2$, and then examined using similar techniques.

Figs. 1(b–e) and 2 give an overview of the microstructural observations of the sample after PFSD, based on SEM and EBSD examination, respectively. As seen in Fig. 1b, a clear microstructural gradient is developed after PFSD. A large amount of plastic deformation is introduced in the top ≈100 μm, where the original grain boundaries can no longer be recognized. The microstructural morphology varies considerably, however, in a non-continuous manner, with depth from the friction surface. Near the top surface (Fig. 1c) the microstructure is composed of fine nanoscale lamellae lying nearly parallel to the sliding direction, with an average spacing of about 50–100 nm. Below this, the microstructure is composed of fine grains extended along the sliding direction (Fig. 1d). At greater depths the microstructure resembles a regular lamellar deformation microstructure, with elongated bands either parallel, or at a shallow angle, to the sliding direction (Fig. 1e). At still greater depths the initial grains can still be clearly identified, with some evidence of some plastic deformation inside the grains.

Based on both the FE-SEM and EBSD observations four depth regions, each with a typical microstructure, can be identified, as shown in Fig. 2.

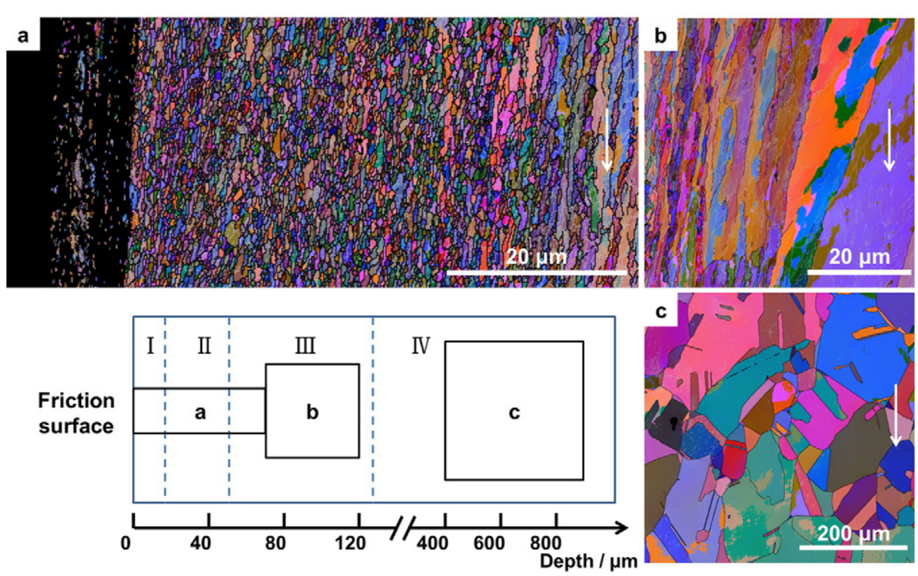

Fig. 2. Typical microstructural morphologies of Cu after PFSD as observed in cross-section by EBSD: (a) top 70 μm, showing the transition from nanoscale lamellae (region I, seen as the mostly non-indexed region), to fine grains (region II), to a regular lamellar deformation structure (region III); (b) the regular lamellar deformation structure (region III) at depth of 70–120 μm; (c) deformed grains (region IV) at a depth of 400–900 μm. The arrow indicates the sliding direction of the friction platen. Adjacent pixel misorientations of ≥1.5° and ≥15° are indicated by light grey and black lines, respectively.