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Surface nanocrystallization of Cu and Ta by sliding friction

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ABSTRACT

Optimization of the surface structure and properties is of great concern since the failures of engineering materials such as wear, erosion and fatigue usually occur on the surface of materials. Plastic deformation from the sliding friction process has been utilized to realize surface nanocrystallization in commercial pure copper and tantalum plates in this work. The optical microscopy, transmission electron microscopy, X-ray photoelectron spectra, and uniaxial tensile testing results suggested that clean nanocrystalline surface layers of pure copper and tantalum were obtained, significantly strengthening the materials after the treatment.

1. Introduction

A new family of severe plastic deformation processes has attracted considerable scientific interests in order to generate nanocrystalline surface layer for bulk materials [1]. Various surface deformation techniques have been developed by transforming the initial coarse-grained (CG) structure of a bulk material into refined-grain structure, such as surface mechanical attrition treatment (SMAT) [1], air blast shot peening [2], wire-brushing [3], and surface mechanical grinding treatment (SMGT) [4]. These advances enhance several types of mechanical properties while keeping the overall chemical composition of the target material unchanged.

Friction is a dissipative process encountered in daily life and industry manufacture. Many experimental results have illustrated that microstructure in the near-surface layer might be changed accordingly during friction and wear process under different contact conditions. Surface microstructure observations showed that plastic deformation could result in the formation of grains with a size falling into the nanometer regime. Grains in the surface layer of pure copper with a size less than 60 nm have been found in an earlier study on subsurface structure of pure copper abraded against SiC paper [5]. After sliding against the zirconia disc, the grain size in the surface of the 316 stainless steel could be refined to 8–23 nm [6]. According to Hughes's experimental results, the average geometrically necessary boundaries were only 10 nm, with nanocrystalline surface layer depth in excess of 4 μm [7]. During friction process, only one-tenth of the energy was undertaken by wear, while most of the energy was consumed in deformation, heat and noise etc. [8]. Thus it is supposed that in case a suitable process is selected, wear loss can be controlled and more energy can be consumed in plastic deformation. A thick nanocrystalline surface layer in excess of 100 μm underneath the worn surface of pure copper induced by sliding against a WC-Co ball by utilizing a generic friction and wear tester has been reported elsewhere by the current authors [9]. These results further experimentally verified the sliding friction as a potential approach to create nanocrystalline structures on the surface layer of metallic materials.

As such, we have developed a surface nanocrystallization technology by means of sliding friction treatment (SFT) with a specified device with enlarged sliding amplitude and improved controllable contact condition to scale up the sample size. This work described the generation of nanostructured surface layer on pure copper and tantalum by the sliding friction process and reported subsequent changes in the mechanical properties of the treated surfaces.

2. Experimental procedures

99.95 wt% of pure copper and tantalum plates with a size of $200 \times 200 \times 3$ mm^3 and a roughness of 0.4 μm were annealed at

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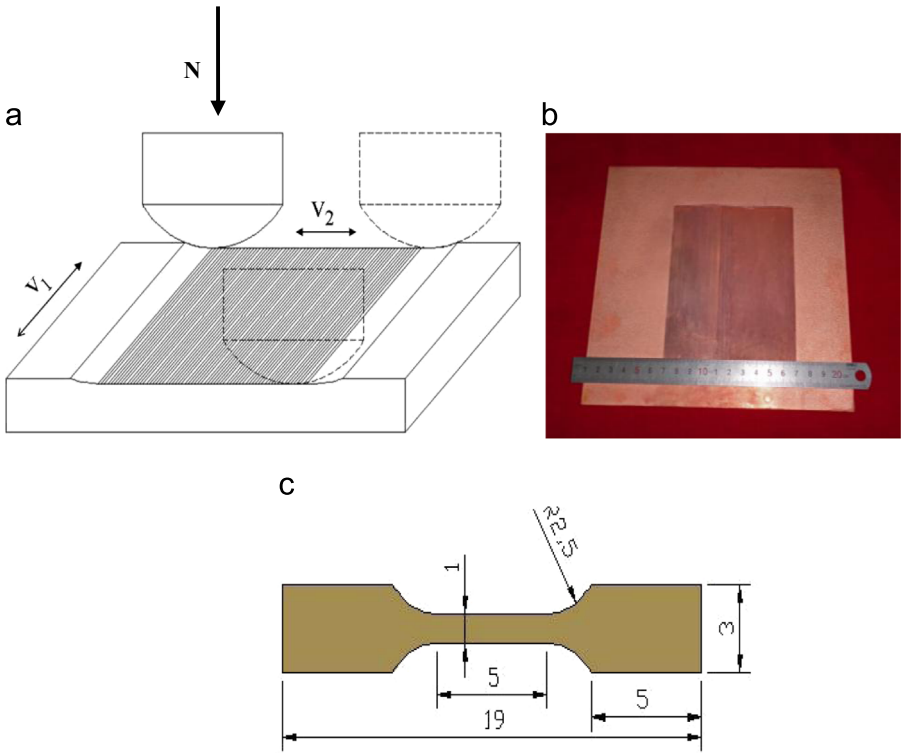

Fig. 1. (a) Schematic illustrations of the sliding friction treatment set-up, (b) a photograph of a Cu sheet after sliding friction treatment and (c) the geometry of the tensile specimen.

973 K and 1273 K, respectively, for 60 min, in order to eliminate the effect of mechanical processing on the surface and to homogenize the microstructure. The experiments were conducted on a specially designed device in a ball-on-disc contact configuration as shown in Fig. 1(a), in which a spherical WC-Co ball of 10 mm in diameter bearing a normal load was static and the specimen (copper or tantalum) subjected to SFT was firmly installed in a table and pressed together with the WC-Co ball under a preset normal force of 100–500 N. The table moved independently along the x and y axes driven by two motors. The sliding of the specimen with an amplitude of 50 mm (d_1) at a speed of 0.2 m/s (v_1) with respect to the WC-Co ball along the x axis was executed first, then the table with the specimen was shifted along the y axis for a step of 100 μm (d_2), and the sliding process continued until the area on the specimen surface was sliding treated. The sliding was carried out under chlorcosane as a lubricant for the tantalum while the copper was conducted under a dry condition in an ambient environment. An image of a Cu sheet after SFT with a surface roughness of 0.7 μm and treated surface area of 100 mm × 150 mm is shown in Fig. 1(b).

The microstructure of the surface layer of the treated samples was characterized by using an Olympus PMG 3 optical microscopy (OM) and a JEOL JEM-2100 transmission electron microscope (TEM) operated at a voltage of 200 kV. Thin foil samples for TEM observations were cut from the treated surface layer and thinned by ion thinning at low temperatures.

X-ray photoelectron spectroscopy (XPS) was carried out on a Thermo Fisher ESCACAB 250XI spectrometer, by using monochromatic Al Ka radiation and detection pass energy ranging between 30 and 100 eV. Argon sputtering was applied at a pressure of 1×10^{-7} Pa under a 3 kV beam accelerating voltage.

The as-treated plates were mechanically polished on the top surface to remove 1–2 μm in depth to eliminate the roughness effect on tensile properties. Down-sized tensile specimens shown in Fig. 1(c) were cut with the testing direction along the sliding direction by using electro-discharging machining. Afterwards, mechanical polishing was performed from back side of the treated sample to a mirror finish with a designed thickness. Tensile tests were performed on a Tytron 250 Microforce Testing System (MTS System Corporation, with a precision of force measurement of 10 mN) at a strain rate of $5 \times 10^{-3}\,s^{-1}$ at room temperature. A contactless MTS LX300 Laser extensometer was used to calibrate and measure the strain of the tested sample during loading.

3. Results and discussion

Fig. 2 shows the microstructure from longitudinal cross-section of the Cu sample before and after SFT. Plastic deformation and traces of plastic flow are evident in the treated surface layer, instead of the original CG structure. After SFT at 100 N, the topmost deformed subsurface layer features are discontinuous in a wave pattern. This is similar to the vortex structure that appeared in some local zones of the uppermost worn subsurface, which has been usually found [10–12] and checked in detail by Yao [13] to be composed of severely refined grains. It is apparent that the plastic flow lines extended to a greater depth as the load increases to 200 N, and the deformed subsurface layer tends to be continuous, with grain boundaries bent towards one direction with decreasing depth toward the surface.

As revealed by TEM morphologies and the corresponding selected area electron diffraction (SAED) pattern in Fig. 2(d) and (e), elongated grains with random crystallographic orientations are formed in the top surface layer of the sample. The average transverse axis grain size (d_t) and longitudinal axis grain size (d_l) are 60 and 110 nm, respectively, although a certain number of grains approaching 200 nm are also present. The grain size is slightly larger than that in the surface layer of the Cu sample subjected to SMGT (22 nm and 45 nm in transverse axis and