Materials Characterization 138 (2018) 263–273

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

Ultrafine gradient microstructure induced by severe plastic deformation under sliding contact conditions in copper

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ARTICLE INFO

Keywords:
Sliding contact
Copper
Severe plastic deformation
Grain refinement
Recrystallisation
Nanohardness

ABSTRACT

Sliding contact induces severe plastic deformation (SPD) at the surface of ductile materials and induces a microstructural gradient associated to a significant increase of hardness toward the surface. This gradient allows observing all stages of grain refinement in SPD, as illustrated here by the analysis of polycrystalline electrolytic copper tested in a coaxial tribometer. Materials tested in the cold-rolled state and after annealing were characterised by high-resolution electron backscattering diffraction and nano-indentation. The incremental plastic strain produces an ultrafine microstructure in the top layers, which gradually changes to the original size in unaffected material. In cold-rolled material, an intermediate recrystallised layer is observed. The separation of the Misorientation Angle Distribution (MAD) in a low-angle portion and a high angle portion allows characterising the accumulation of strain induced misorientation, while the Kernel Average Misorientation (KAM) provides information on the evolution of substructure at the finest levels. The results point toward a process where strain-induced effects compete continuously with recrystallisation, except for the surface layer in the cold-rolled material, where dynamic recrystallisation is dominant. Combining the information from KAM and subgrain size distribution, the measured hardness can be explained as a combination of grain size and dislocation hardening.

1. Introduction

When ductile materials are subject to sliding wear conditions, strong microstructural modifications are observed close to the surface. One of these modifications is the formation of a tribolayer with a thickness between several hundreds of nanometres to tens of microns [1], depending on the contact conditions and the initial microstructure [2]. Rigney [3–5] explained the formation of the tribolayer as the combination of severe plastic deformation (SPD), mechanical mixing (MM) and material transfer. Kapoor [6–8] described the corresponding SPD-process as the accumulation of small plastic strains due to cyclic loading, unlike most common SPD process in which the total strain is achieved in a few cycles of very large plastic strains [9–11]. It has been suggested that tribolayers are responsible for a decrease in wear rate and friction coefficient in sliding contact of metals [12–14]. The technological importance of this process has been illustrated by studies on bearings [15], railway tracks [16–19], brake disks [20,21], journal bearings [22] and ceramic materials [23–25].

The effect of SPD on tribological behaviour has been reported for low carbon steel, titanium and copper [26–29]. This improvement is often attributed to the proposal by Archard [30], in which wear resistance is proportional to the material hardness. Nevertheless, this empirical relation is overly simple, suggesting the need for a combination of ductility and strength. Such a combination may be obtained by introducing a gradient microstructure by superficial SPD methods like surface mechanical grinding treatment (SMGT) [31,32], surface mechanical attrition treatment (SMAT) [33] or platen friction sliding deformation (PFSD) [34].

Since grain size is a key microstructural factor affecting the mechanical behaviour of polycrystalline metals, the interest for developing new processing techniques, which allow modifying grain size without changing composition has increased. An attractive method for

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https://doi.org/10.1016/j.matchar.2018.02.017
Received 13 October 2017; Received in revised form 7 February 2018; Accepted 13 February 2018
Available online 15 February 2018

producing ultrafine-grained and nanostructured materials is SPD, due to the possibility to enhance the strength up to a factor of eight in pure metals and by 30–50% for alloys [35,36]. Using a combination of high hydrostatic pressure and shear deformation, the straining is practically unlimited while the shape of the part is almost constant [37,38].

The grain refinement by SPD in high or medium stacking fault energy (SFE) materials like Fe, Al or Cu is due to the formation of a dislocation-based substructure. This refinement is more pronounced at low or medium strains, while in later straining stages, grain boundary misorientation increases [39–42], leading to the formation of predominantly high angle grain boundaries (HAGB) in materials with accumulated strains above 6 to 8 [11,43]. The evolution from a homogeneous dislocation forest to elongated dislocation cells and subsequently sub-grains with increasing plastic strain has been described extensively in several review papers [44–46]. A quantitative analysis of the increment of subgrain boundary misorientation, and hence their evolution from LAGB to HAGB has been provided by Pantleon & Hansen [47] and Pantleon [48].

An alternative viewpoint is that ultrafine grained structures obtained by SPD at relatively low temperatures are produced by continuous dynamic recrystallization (CDRX) which is characterised by a slower kinetics than conventional dynamic recrystallization (DRX) because of the requirement of large strains ($\varepsilon > 3$) [49]. In CDRX new grains form during deformation as a result of the continuous accumulation of dislocations increasing the sub-boundary misorientation, producing a gradual transformation from low angle grain boundaries (LAGB) into HAGB [49–51]. The grain refinement in ultrafine or nanostructured metals increases the mechanical strength, combined with the effect of work hardening by increasing dislocation density [43]. Although a significant decrease in ductility is expected and often observed, it has been reported that UFG materials produced by SPD sometimes show a good combination of strength and toughness [43,52–54].

Although in most known SPD techniques like High Pressure Torsion (HPT) or Equal Channel Angular Pressing (ECAP) the material flow is more complex than in a classical torsion test, it is assumed that the deformation mode is simple shear [55,56]. The crystallographic texture obtained in torsion tests on face centred cubic (FCC) metals has been extensively studied [57–60]. Two fibres and one component are observed: the A-fibre {111}⟨uvw⟩, B-fibre {hkl}⟨110⟩ and the C-component {100}⟨110⟩ [34]. Ideal orientations associated to the mentioned fibres have been defined based on simulation and experimental data; these orientations are shown in Table 1, in which {hkl}⟨uvw⟩ refers to the set of planes and directions parallel to the shear plane and shear direction respectively.

In previous research, the superficial modification in Cu, Al-Sn, and Cu-Mg-Sn systems was analysed, showing the influence of annealing in the tribological behaviour and the formation of tribolayers in two-phase alloys [29,63]. In the present work, a coaxial tribometer developed by the authors [64] was used to modify the surface of pure copper, inducing a microstructural gradient which allows for a systematic study of the microstructure, texture evolution and their interrelation with hardness and grain size. The microstructure and texture were analysed by means of detailed EBSD scans along the cross section to the worn surface, while nano-hardness measurements were performed in the previously scanned zones.

Table 1
Ideal texture components observed in FCC materials subject to simple shear [58,61,62].

Component	{*hkl*}⟨*uvw*⟩	φ_1	Φ	Φ_2
A	$\{1\bar{1}\bar{1}\}\langle 110\rangle$	0	35.26	45
$\overline{A}$	$\{\bar{1}11\}\langle\bar{1}\bar{1}0\rangle$	180	35.26	45
A^*	$\{\bar{1}\bar{1}1\}\langle 112\rangle$	35.37	45	0
		125.37	90	45
$\overline{A}^*$	$\{11\bar{1}\}\langle 112\rangle$	144.74	45	0
		54.74	90	45
B	$\{\bar{1}12\}\langle 110\rangle$	0	54.74	45
		120	54.74	45
$\overline{B}$	$\{1\bar{1}\bar{2}\}\langle\bar{1}\bar{1}0\rangle$	60	54.74	45
		180	54.74	45
C	{100}⟨110⟩	90	45	0
		0	90	45
A fiber {111}⟨*uvw*⟩				
B fiber {*hkl*}⟨110⟩				

2. Materials and Methods

Commercial slabs of electrolytic tough pitch copper with purity of 99.9% were cold rolled to a final reduction of 88% equivalent to a Von Mises strain of $\varepsilon_{VM} = 2.5$ and subsequently annealed at 873 K for 45 min. The material was tested under two different conditions: cold rolled (CuCR) and recrystallized (CuRX). The surface modification was conducted at room temperature using a coaxial tribometer [64]. The experiment consists in the application of a constant normal load of 100 N by means of a cylindrical pin of AISI9840 steel with a spherical cap of radius equal to 200 mm in contact with the surface of a Cu-coupon of 20 mm × 20 mm. The pin rotates on its own axis for 300 s at a constant angular speed of 60 rpm. Before the tribological test, Cu surfaces were prepared by conventional mechanical polishing to a mean square roughness of 0.62 μm and 0.64 for CuCR and CuRX respectively. Based on the mechanical work dissipated in system and considering the aluminium sample holder as an infinite heat sink, it can be calculated that the temperature increase in the Cu-coupon is < 1 °C.

The sample reference system follows the traditional rolling convention, representing the rolling direction as RD, transversal direction as TD and normal direction as ND. The worn surface is taken perpendicular to ND. Samples for microstructure analysis were taken perpendicular to TD, which for this observation plane is also the sliding direction. Transverse sections trough the centre of the wear track were prepared by conventional mechanical polishing and analysed unetched on a FEI Quanta 450-FEGSEM operating at 20 kV with spot size of 5, corresponding to a beam current of 2.4 nA. TSL-OIM Data collection version 6.2 was used to acquire EBSD patterns, the working distance was 20 mm with a tilt angle of 70°. Step size used for coarse-grained and cold-rolled zones was 0.1 μm, while for the layer closest to the worn surfaces it was 55 nm, in hexagonal grid scan mode.

EBSD scans were performed to analyse the microstructural gradient from top of the wear-affected zone to a zone where the original microstructure was evident. To ensure the statistical validity of data, the size of the areas analysed was varied according to the grain size. The orientation data were post-processed with MTEX [65] quantitative texture software analysis version 4.0.23. A clean-up procedure to eliminate pixels with neighbour confidence index correlation < 0.1 was performed before the microstructural and textural analysis. To define grains, a cut-off angle of 5° was considered for the minimum boundary misorientation in LAGB and 15° for HAGB.

KAM was calculated for the six nearest neighbours of each pixel (excluding grain boundaries) with threshold of 3°. This parameter quantifies small local differences in orientation which are not identified as LAGB under the threshold mentioned before. KAM therefore indicates the presence of local lattice bending due to dislocation structures which are not otherwise resolved. The grain boundary misorientation angle distribution (MAD) was obtained from the experimental data. A frequency distribution was obtained by fitting an 8th degree polynomial to the empirical distribution function of measured grain boundary misorientation and calculating the derivative. The polynomial was forced to be equal to 0 and 1 at the lowest and highest misorientation in the dataset, respectively, with derivative equal to 0 in both points. Twin boundaries were excluded from the calculation A theoretical MAD was calculated from the measured texture, rather than using the classical Mackenzie distribution which is valid for random textures. This calculated “random” MAD represents a