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Wear resistance and electroconductivity in copper processed by severe plastic deformation

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ABSTRACT

The wear properties and electroconductivity of three ultra-fine grained (UFG) commercially pure copper materials, subjected to combinations of high-pressure torsion (HPT) and equal-channel angular pressing (ECAP), were studied and compared with conventional coarse-grained (CG) copper. The results are discussed as a function of microstructure and microtexture. The UFG specimens demonstrate no significant decrease in electroconductivity by comparison with CG copper. The conductivity of an ECAP +HPT specimen showed a value of 99.3% of annealed copper and the same sample showed the lowest wear rate among the UFG specimens. However, all UFG specimens gave higher wear rates than CG copper and there was no evidence for the enhanced wear resistance reported for nanocrystalline copper obtained by surface mechanical attrition treatment (SMAT) and electrodeposition. This result is discussed in the context of two competing processes: shear by sliding and normal compression by impact load.

1. Introduction

The processing of ultrafine-grained (UFG) and nanostructured metallic materials using severe plastic deformation (SPD) [1] is a new and promising method of enhancing the properties of metals and alloys for advanced structural and functional applications [2]. Traditionally, there have been two main techniques for producing UFG materials using either equal-channel angular pressing [3] or high-pressure torsion [4,5] but other techniques are now available such as accumulative roll bonding (ARB) [6], multiaxial forging [7], twist extrusion [8], plain strain machining (PSM) [9] and others [10]. It is well established that metallic materials subjected to SPD can possess not only a UFG structure but also specific nanostructural features such as nonequilibrium grain boundaries (GBs) [1], nanotwins [11,12] and GB segregations [13]. Consequently, enhanced mechanical (high strength) and functional properties (electric, magnetic, corrosion, etc.) have been established for numerous different metals and alloys. In practice, SPD processing is attractive because the straining is practically unlimited due to the unchanging sample geometry and shape. However, there is a general tendency for a saturation in grain refinement for high melting metals [14] and recovery (and even recrystallization) for low melting materials [15] processed by continuing SPD methods. Nevertheless, a change in the deformation path or using a combination of different SPD techniques [16] may lead to further refinement of the microstructure. Recent investigations reported the successful cold consolidation of ball-milled powders [17], machining chips [18] and rapidly-quenched (RQ) ribbons [19]. This modification of the deformation path leads to greater refinement of the microstructures of metals and alloys and good consolidation properties for powders and chips.

The processing of UFG materials with advanced mechanical and functional properties are currently in transition from laboratory-scale research to industrial applications [2]. For example, there is considerable interest in using nanostructured titanium for biomedical applications [20,21] due to the excellent biocompability. The enhanced mechanical and functional properties achieved in nanostructured TiNi alloys by application of SPD techniques make this material very attractive for use as stents and other medical items [22,23]. Another area of possible application for UFG materials is in the hydrogen storage media because grain refinement can significantly improve the hydrogen storage capacity and adsorption/desorption kinetics of magnesium and magnesium alloys. Thus, improved hydrogen storage capacity and kinetics were achieved in bulk Mg-based alloys via ECAP [24] and HPT [25,26]. It is worth noting also the improved magnetic properties that can be achieved in UFG materials [27–29].

There is a considerable interest in creating UFG structure in pure copper and its alloys in order to get the optimum combination of low wear rate and high conductivity. However, pure copper has some distinct drawbacks, such as low strength and low

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thermostability, which tends to restrict its applications. There are two main strategies for strengthening of engineering metals: (i) through grain refinement and (ii) through alloying. Although the second approach is more efficient, alloying tends to significantly degrade the electrical conductivity. The residual electrical resistivity (RER), which is the reciprocal of the electrical conductivity, has been studied in pure copper processed by SPD [30,31] and it was revealed that, depending on the strain, the induced RER can increase by up to ~40 nΩ cm due to the accumulation of lattice defects during severe plastic deformation.

There are numerous reports on the application of SPD for grain refinement of pure copper by ECAP [32–37], HPT [38–40] and machining or plain strain machining [41] as well as surface mechanical attrition treatment (SMAT) [42]. The tribological properties have been studied in HPT copper [43], PSM copper chips [44], in SMAT copper specimens [45] and in copper processed by dynamic plastic deformation (DPD) [46]. Thus, HPT of copper led to significant grain refinement with a mean grain size estimated as ~200 nm and consequently there was an increased wear rate compared to the course-grained counterpart [43]. The tribological properties were enhanced with a nano-scale twin surface layer for SMAT copper [45] by comparison to the coarse-grained specimen for all loads and sliding rates, where this was assumed to originate from strengthening by twinning.

In all reports to date, significant wear resistance has been reported as a function of a single structural parameter, such as the mean grain size or the twin lamella distance. It was assumed for all UFG copper materials that they were textureless and there was no difference in any other structural parameters such as the fraction of high-angle boundaries. Large strain extrusion machining of copper provides some inhomogeneity in the microstructure, notably in the form of grain elongation, and it was reported [44] that the highest wear resistance was observed for nanostructured copper with a grain structure that was elongated in the extrusion direction. Thus, in these studies and many others the enhanced wear resistance is related to grain refinement and consequently to the increased microhardness of the samples. However, the practical use of copper and copper alloys is mostly as a contact wire which needs both good wear resistance and high electro-conductivity. These parameters are inversely related because for copper samples subjected to grain refinement an increase in the wear resistance with decreasing grain size will lead to a decrease in the electro-conductivity. Accordingly, this work was an attempt to evaluate concurrently the wear resistance and electro-conductivity in copper processed by a combination of HPT with PSM and ECAP in order to provide information on the relationship between the microhardness and grain size, the grain boundary statistics and the wear resistance and electro-conductivity.

2. Experimental material and procedures

Copper of 99.9% purity was annealed at 450 °C for 30 min to give an initial grain size of ~20 μm by electron back scatter diffraction (EBSD), hereafter designated the initial material, and then it was processed at room temperature by a combination of machining with HPT or using a more complicated routine including ECAP, machining and HPT. Full details of the sample processing are given elsewhere [19] but in brief some copper samples were machined into chips using a regular milling machine operating at a rate of 30 m/min with a shear strain imposed in the chips of $\gamma \approx 2$. The chips were consolidated using a constrained HPT facility [47] at room temperature under a pressure of $P=6$ GPa through a total of $N=5$ turns. The HPT discs prepared from the machined chips, henceforth denoted as M+HPT, were essentially fully-dense and there was no evidence of any visible cracking. Another type of specimen was prepared through a combination of ECAP and HPT. Thus, an ECAP copper rod, processed using route B_C through four passes at room temperature, was sliced into discs which were subjected to constrained HPT under the same conditions of $P=6$ GPa and $N=5$ turns at room temperature. These specimens are designated as ECAP+HPT. A third type of specimen was processed by ECAP, machined into chips and then the chips were also consolidated by HPT and they are denoted as ECAP+M+HPT. The wear experiments and microhardness measurements were conducted at the half-radius position on each HPT disc so that the radius is $r=2.5$ mm and the shear strain imposed by HPT is taken as $\gamma=2\pi rN/h$ where h is the final thickness of the disc. Thus, based on the additive rule for the imposed shear strains, γ, it is reasonable to estimate the imposed strain for these three types of specimens as $\gamma_{M+HPT} \approx 392$, $\gamma_{ECAP+HPT} \approx 394$ and $\gamma_{ECAP+M+HPT} \approx 396$. It follows that all values are practically identical although in practice the imposed shear strain is strain path and strain rate dependent so that it is preferable to characterize specimens by labeling as M+HPT, ECAP +HPT and ECAP+M+HPT. A schematic of the sample preparation methods is given in Fig. 1.

All consolidated discs were 10 mm in diameter and 0.2–0.3 mm in thickness. The discs were mechanically polished to a mirror-like finish for microhardness testing using a WOLPERT 420MVD facility with a load of 0.05 kg and a holding time of 10 s. Structural investigations were performed on sections perpendicular to the HPT axis using a Quanta 600 FEG scanning electron microscope (SEM) equipped with an electron back scatter diffraction analyzer incorporating an orientation imaging microscopy (OIM) system. The SEM specimens were cut from the discs at a radius of $r/2$ distance as shown in Fig. 2a and then mechanically polished on 1000 grit SiC paper and electropolished using a solution of 250 ml nitric acid, 750 ml methanol at 20 °C with a voltage of 10 V. The EBSD samples had a coordinate system as shown in Fig. 2b and the step size for EBSD scanning was 0.5 μm for coarse-grained (CG) copper and 20 nm for all UFG copper specimens. The mean grain size of all specimens was measured on the OIM images using the linear intercept method in the transverse and radial directions.

The overall electrical conductivity of all discs of identical geometry was measured using a four-point bridge technique or Wheatstone bridge circuit in which the unknown resistance is compared with a well-defined resistance. This method allows precise measurements for specimens having identical or very similar geometries.

Sliding wear tests of the UFG and CG Cu samples were conducted using a high-temperature tribometer (CSM Instruments) oscillating friction and wear tester with a ball-on-disc contact configuration. The Cu discs slide against Cr6 steel balls having a diameter of 6 mm with a hardness of $Hv \approx 1750$. The top surface layer of all tested samples was removed by polishing carefully to eliminate any surface roughness effect on the tribological behavior. The sliding wear tests were carried out at room temperature under a relatively low humidity without lubricant at an oscillating stroke of 1 mm, normal loads of 1.5 N, a sliding speed of 0.04 m/s and a fixed sliding distance of 100 m. The acquisition rate was 7 Hz. Wear tests of the annealed CG Cu sample with comparable roughness were also carried out under the same conditions for comparison purposes. After completing the wear tests, a precision contact profilometer SURTRONIC was used to acquire the profiles of the wear tracks at selected points on the specimens.

3. Experimental results

3.1. Microhardness, microstructure and microtexture of the copper specimens

The microhardness and other microstructural parameters are summarized in Table 1 and it is apparent that the microhardness has nearly doubled in all UFG samples compared with the CG sample: this is in accordance with a previous report [19]. The highest