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Effect of strain rate on microstructure of polycrystalline oxygen-free high conductivity copper severely deformed at liquid nitrogen temperature

B. Zhang, V.P.W. Shim *

Impact Mechanics Laboratory, Department of Mechanical Engineering, National University of Singapore, 9 Engineering Drive 1, Singapore 117576, Republic of Singapore

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Abstract

The microstructure of polycrystalline oxygen-free high conductivity copper subjected to severe uniaxial single compression at liquid nitrogen temperature and strain rates ranging from 10^{-2} to 10^{5} s^{-1} is characterized using transmission electron microscopy, X-ray diffraction and differential scanning calorimetry. A difference in strain rate leads to a change in the density, character and arrangement of dislocations, as well as the size and configuration of dislocations cells/(sub)grains in the deformed sample. A threshold strain rate of 10^{3} s^{-1} is identified for the formation of localized deformation bands, which characterizes heterogeneity of deformation at high strain rates. These bands are composed of grains that are significantly smaller than those outside them, as well as those obtained at strain rates lower than 10^{3} s^{-1}. Under particular conditions, grains as small as several nanometers can be generated in the vicinity of these bands, through the activation of rotational dynamic recrystallization. Amorphization is identified as a deformation mechanism in structures consisting of grains smaller than ~13 nm, and this offers an explanation for the "inverse Hall–Petch effect". A model that illustrates the initiation and propagation of an amorphous phase during deformation is proposed. Deformed samples exhibit the tendency of an increase in strength with the value of the Zener–Hollomon parameter, which captures strain rate and temperature rise during deformation. This study suggests that a strain rate in the order of 10^{2} s^{-1} should be adopted in severe plastic deformation techniques to produce nanometer-sized grains.

Keywords: Strain rate effects; Dislocations; Nanometer grains; Amorphization

1. Introduction

Over the past two decades, much research interest from metallurgists has been focused on metals/alloys subjected to large deformation. Their investigations can be classified into two categories according to the objectives: (i) to elucidate the underlying mechanism that accounts for different work hardening behaviors corresponding to stages III, IV and V, respectively, which encompass the large deformation strain range [1–5]; (ii) to refine an originally coarse-grained structure to the ultrafine/nanometer regime in order to achieve an improvement in mechanical properties of the processed material [6–8]. Investigations related to work hardening behavior have revealed different rates of generation and annihilation of dislocations in stages III, IV and V, occurring either in dislocation cell walls or in the cell interior [1,9], or associated with specific types of dislocations (i.e., screw or edge dislocations) interacting during each stage [10,11]. The rate of dislocation generation and annihilation determines the rate of work hardening. Studies focusing on grain refinement demonstrate gradual subdivision of coarse grains, leading to the formation of a structure with refined grains and high-angle grain boundaries [6,12]. Although these investigations have different objectives, both show the crucial role of dislocation activity in transforming the microstructure of materials undergoing plastic deformation. An example is the

* Corresponding author. Tel.: +65 6516 2228; fax: +65 6779 1459.
E-mail address: mpespwv@nus.edu.sg (V.P.W. Shim).

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rearrangement of dislocations in cell walls occurring from stages III to V, leading to the transformation of polarized dipolar dislocation walls into polarized tilt walls [13–15]. Also, the gradual accumulation of glide dislocations at the walls of ultrafine/nano-sized dislocation cells, which results in the transformation of cell boundaries into subgrains or even high-angle grain boundaries [6,16–19]. However, the investigations carried out have been done mainly at room or higher temperatures and exhibit characteristics typical of relatively low-strain-rate deformation (usually $<10\ s^{-1}$), cf., Refs. [20–30]. Although the microstructural characteristics of metals/alloys deformed either at temperatures lower than room temperature (RT) or strain rates as high as 10^5–$10^6\ s^{-1}$ have been elicited, cf., Refs. [31–35] and [36–41], respectively, there seem to be few investigations that characterize the microstructure of samples deformed to large strains at both low temperature and high strain rate. It is noted that, by employing multiple high-strain-rate ($\sim 10^3\ s^{-1}$) deformation processes at liquid nitrogen temperature (LNT), Li et al. recently [42] managed to reduce the size of grains from an initial order of a few hundred microns down to that of tens of nanometers, overcoming the limitation associated with grain size saturation which is encountered in conventional severe plastic deformation (SPD) techniques [43]. Systematic investigation on how strain rate affects the resulting microstructure of samples deformed to large strains at low temperatures appears to be lacking. The present effort examines the effect of strain rate on the microstructure of oxygen-free high conductivity (OFHC) copper severely deformed at LNT (77 K).

Characterization of material microstructure is often performed using transmission electron microscopy (TEM) or X-ray diffraction (XRD). Although TEM enables direct observation of the microstructure, only a miniscule volume of the specimen is examined. In contrast, XRD scans a much larger volume of the specimen, leading to more representative statistics of the microstructure, and is thus used in this study in conjunction with TEM to observe the microstructure more comprehensively. In XRD, microstructural characteristics such as dislocation density and crystallite size can be extracted from peaks in the XRD scan outputs by applying evaluation techniques such as the Warren–Averbach analysis (WAA) [44], Williamson–Hall plot (WHP) [45] and the variance method [46]. Recently, the WAA and WHP have been upgraded by the introduction of a parameter called the "contrast factor of dislocations" and are referred to as the modified WAA (MWAA) and the modified WHP (MWHP) techniques, respectively [47]. The MWAA and MWHP have been verified as being reliable and precise in evaluating dislocation density, grain size, etc. [48–50], and hence are adopted in the current analysis.

Polycrystalline OFHC copper is chosen as the material of study because it is easily available, possesses good ductility and there is abundant existing data for comparison of the results obtained. Uniaxial single compression is selected as the deformation mode by which strain rates encompassing different orders of magnitude can be imposed using the available facilities.

2. Experimental procedure

2.1. Deformation

Cuboid samples are fabricated by wire-cutting them from a rod of OFHC copper which has been annealed and possesses a microstructure characterized by grains measuring $\sim$100 μm in size, as well as annealing twins (Fig. 1). Samples are immersed in a liquid nitrogen bath for durations sufficient to cool them down to LNT, and then compressed in an atmosphere of liquid nitrogen. Compressive deformation at various strain rates is performed using a Shimadzu Universal Testing Machine (SUTM), a drop tester, a split Hopkinson pressure bar (SHPB) and gas gun, and the respective true strains and strain rates induced are presented in Table 1, whereby ε and $\dot{\varepsilon}$ are defined, respectively, as

$$\varepsilon = \ln(L_0/L)$$
$$\dot{\varepsilon} = \varepsilon/t \quad (1)$$

L_0 and L represent the initial and final length, respectively, of a sample, and t is the duration of compression. The deformed samples are stored in a refrigerator prior to subsequent analysis.

2.2. Microstructure characterization

2.2.1. TEM

A JEM-2010F TEM microscope operating at 200 kV was used for microstructural examination of sample surfaces perpendicular to the compression axis. The samples

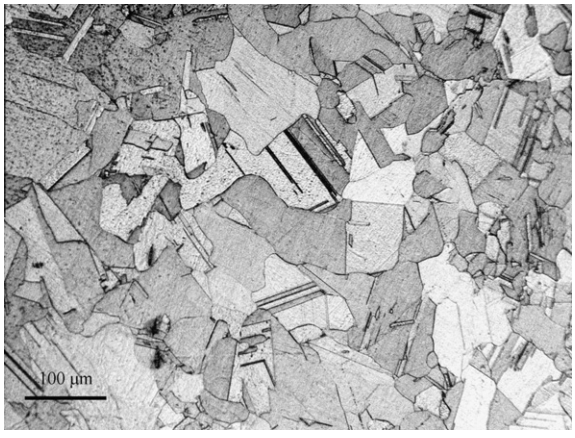

Fig. 1. Optical micrograph showing microstructure of annealed samples.

Table 1
Parameters for uniaxial single compression tests.

Equipment	Temperature	True strain, ε	True strain rate (s^{-1}), $\dot{\varepsilon}$
SUTM			9.4×10^{-2}
SUTM			1.9
Drop tester	LNT (77 K)	2.3	4.8×10^2
SHPB			5.7×10^3
Gas gun			6.9×10^4