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Microstructure development and lateral distribution of defects in ultra-fine grained copper prepared by high-pressure torsion

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Abstract

A defect study of ultra-fine grained (UFG) Cu prepared by high-pressure torsion (HPT) will be reported. Conventional positron annihilation spectroscopy (PAS) including positron lifetime (PLT) and Doppler broadening (DB) techniques was employed as the main experimental tool. PAS was combined with transmission electron microscopy, X-ray diffraction and Vickers microhardness (HV) measurements. First, lattice defects introduced by HPT were characterized. A very high concentration of defects created during HPT deformation was observed and the two kinds of defects could be identified: dislocations and small vacancy clusters (microvoids). Further investigations were focused on (i) the radial distributions of defects and (ii) the evolution of microstructure during HPT processing. The results of the present study are consistent with an increase of shear strain from the sample centre toward its periphery. Extended lateral mapping of microstructure was performed using HV and DB techniques. The latter one reveals a significant non-uniformity of defect distribution which was less pronounced in the HV measurements.

Keywords: Ultra-fine grained copper; high-pressure torsion; lateral distribution of defects; dislocations; microvoids; positron annihilation spectroscopy.

1. Introduction

Materials with mean grain size reduced down to several hundreds of nanometers are referred to as the ultra-fine grained (UFG) materials. Grain refinement of ordinary polycrystalline materials often brings an improvement of

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their mechanical properties, for example, a high strength combined with a reasonable ductility [1]. Grain boundaries (GB's) become to play a substantial role in UFG materials and obviously assist e.g. to an increase of diffusion activity of atoms [2] and ductility [3]. A remarkable grain refinement can be attained by the techniques based on severe plastic deformation [1], e.g. by the high-pressure torsion (HPT). The HPT deformation is performed by placing a material between two anvils and straining it by the rotating anvil under a uniaxial pressure of several GPa. In many metals or metallic alloys, grain refinement to a typical grain size of ≈ 100 nm is achieved by means of HPT treatment [1]. Among such materials, UFG copper is a good model system for comparative studies of different preparation techniques because pure copper is easily accessible and various characterization methods are applicable.

HPT treatment results in disk-shaped bulk specimens having typically a diameter of 10 – 15 mm and a thickness of ≈ 0.3 mm. A huge amount of lattice defects (vacancies, dislocations) is created during HPT deformation. It can be anticipated, that the HPT imposed shear strain is raised proportionally to the radial distance from the disk centre and to the number of torsion turns [1]. Consequently, the microstructure at the disk centre can expected to differ from that at the disk edge. Such a microstructure pattern was supported by a lower hardness at the disk centre compared to its periphery measured on HPT deformed Ni [4]. On the other hand, the microstructure differences between the sample periphery and its centre were indicated to become smaller under a higher pressure applied during HPT deformation and with an increasing number of turns [5]. Hence, a detailed knowledge of the microstructure variations in dependence on preparation conditions, in particular the type and spatial distribution of defects induced during HPT processing, is obviously a key issue in attempts to understand unusual properties of HPT deformed materials and to optimize material preparation procedure.

In the present Contribution, a detailed defect study of UFG Cu prepared by HPT was performed. The Contribution is focused on (i) the characterization of defects introduced by HPT, (ii) the lateral distribution of defects and (iii) the microstructure evolution during HPT processing. Conventional positron annihilation spectroscopy (PAS) combined with X-ray diffraction (XRD), transmission electron microscopy (TEM) and Vickers microhardness (HV) measurements were involved [6,7].

2. Experimental

Copper of 99.95 % purity was HPT deformed at room temperature. A series of samples subjected to 1, 3, 5 10, 15 and 25 revolutions were investigated in order to follow the evolution of microstructure during HPT treatment. A possible effect of compressive pressure was also studied using samples deformed under 2 and 4 GPa. HPT processing resulted in disk-shaped specimens having a diameter of ≈ 9 mm and a thickness of ≈ 0.3 mm.

HV measurements were performed using a STRUERS Duramin 300 hardness tester with a load of 100 g applied for 10 s. HPT deformed Cu samples were polished to a mirror-like quality for HV investigations. The homogeneity of microstructure was characterized by HV measured on a rectangular $x - y$ grid with the incremental spacing of 0.5 mm. Colour coded maps of HV results were constructed to provide pictorial displays about the homogeneity of microstructure across the sample.

Two positron sources, made of carrier-free ^{22}Na carbonate salt (iThembaLABS) deposited and sealed between two 2 μm thick mylar C foils (Dupont), were used: (I) a stronger source, having ≈ 1 MBq strength and an activity spot of ≈ 3 mm diameter, and (II) a weaker source (≈ 0.5 MBq strength, < 1 mm spot size). Positron source was sandwiched between two identical disks of the material studied.

Positron lifetime (PLT) measurements were performed using the source (I) and a fast-fast PLT spectrometer similar to that of Ref. [8]. The spectrometer exhibited a time resolution of 150 ps (FWHM) and a coincidence count rate of ≈ 100 s^{-1} for the above positron source. Positron annihilations in the source and its cover foils contributed to measured PLT spectra with the two weak components exhibiting lifetimes (intensities) of 0.368 ns (8 %) and 1.5 ns (1 %). These components were extracted based on PLT measurements with the well-annealed Cu reference material. Radial variations of microstructure were investigated by making PLT measurements with the positron source positioned at the sample centre (i.e. at a radial distance of $r \approx 0$) and at a periphery position ($r \approx 3$ mm).

Doppler broadening (DB) experiments were carried out using the weaker source (II) and an ordinary HPGe γ-ray spectrometer having an energy resolution of 1.3 keV (FWHM) at the 511 keV γ-ray energy. Doppler profiles were described in terms of the ordinary sharpness and wing parameters, S and W, respectively, normalised to the shape parameters measured at the centre of the sample after one HPT revolution (S_0 and W_0 , respectively). The lateral variations of microstructure were mapped in detail using DB measurements performed for different source positions