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Grain boundary excess volume and defect annealing of copper after high-pressure torsion

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Abstract

The release of excess volume upon recrystallization of ultrafine-grained Cu deformed by high-pressure torsion (HPT) was studied by means of the direct technique of high-precision difference dilatometry in combination with differential scanning calorimetry (DSC) and scanning electron microscopy. From the length change associated with the removal of grain boundaries in the wake of crystallite growth, a structural key quantity of grain boundaries, the grain boundary excess volume or expansion $e_{GB} = (0.46 \pm 0.11) \times 10^{-10}$ m was directly determined. The value is quite similar to that measured by dilatometry for grain boundaries in HPT-deformed Ni. Activation energies for crystallite growth of 0.99 ± 0.11 and 0.96 ± 0.06 eV are derived by Kissinger analysis from dilatometry and DSC data, respectively. In contrast to Ni, substantial length change proceeds in Cu at elevated temperatures beyond the regime of dominant crystallite growth. In the light of recent findings from tracer diffusion and permeation experiments, this is associated with the shrinkage of nanovoids at high temperatures.
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Keywords: Grain boundaries; Grain growth; Dilatometry

1. Introduction

Processing by severe plastic deformation (SPD) has been established as one of the most promising routes to produce bulk ultrafine or even nanocrystalline materials. These materials made by SPD exhibit exceptional mechanical properties (see e.g. Refs. [1–4]). A comprehensive understanding of these enhanced properties and of the process of grain refinement during severe plastic deformation is currently a major research topic in materials science. In particular, the roles of the various types of defects produced during deformation are widely studied. Furthermore, from a basic materials physics point of view, SPD metals offer the opportunity to study different types of deformation-induced defects and their mutual interaction. It has already been shown that athermally produced excess vacancies are present in high concentrations that are otherwise found only close to the melting temperature [5,6]. A complex defect annealing kinetics is suggested from volume and grain boundary diffusion studies in SPD-processed Cu and Ni [7,8]. During annealing, abundant and highly mobile vacancies may, for example, agglomerate or form a percolating porosity network in combination with triple

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junctions of grain boundaries. In addition, ultrafine-grained SPD materials give access to basic physical key parameters and processes such as thermally activated grain boundary relaxation prior to grain growth, as well as to the grain boundary excess volume, often also denoted as grain boundary expansion, of relaxed high-angle grain boundaries [9]. For this purpose, the direct experimental method of difference dilatometry is applied in the present work in combination with differential scanning calorimetry (DSC) and scanning electron microscopy (SEM). The subjects of the present study are high-purity samples of the face-centered cubic (fcc) metals Cu and Ni. Novel results obtained from difference dilatometry for copper are compared with results obtained by the other above-mentioned methods, as well as with results previously obtained for pure nickel.

2. Experimental

A Cu disk with a purity of 99.995 wt.% was deformed by high-pressure torsion (HPT) at room temperature, with six revolutions being applied at 2.2 GPa (for details see Refs. [10,11]). Samples were cut from this HPT-deformed disk (30 mm in diameter and 7 mm in height) at distances of at least 7.3 mm from the center. This corresponds to a von Mises equivalent strain of $\varepsilon > 23$, ensuring a regime, where the deformation is in saturation, i.e. where further deformation will not lead to further grain refinement.

For the dilatometric measurements, a total of nine prism-shaped specimens with the dimension of $3 \times 3 \times 7$ mm^3 were prepared. The direction of the length change measurement is defined with regard to the HPT deformation axis (see Fig. 1, axial, tangential and radial directions). Here, seven samples were prepared in the axial direction and one each in the tangential and radial directions.

Experiments were performed with a high-precision, vertical double-dilatometer (Linseis, L75VD500 LT), which allows the simultaneous measurement of two samples under an argon (5 N) gas flow. One of the two samples served as a reference and was made from the same Cu material, which was well annealed and coarse grained (grain size > 100 μm). The experimental data, plotted as a dilatometric length change curve $\Delta l/l_0$, represents the difference signal between the specimen and the reference (so-called difference dilatometry). The length change is directly related to the defect volume via the relation $3 \times \Delta l/l_0 = \Delta V/V_0$, assuming an isotropic distribution and annealing of defects [12]. The temperature of the maximum defect release rates can be determined from the minima of the derivative $d(\Delta l/l_0)/dT$ of the length change curve with respect to temperature. For the case of a constant linear heating rate, dT/dt, the temperature, T, is directly proportional to the time, t.

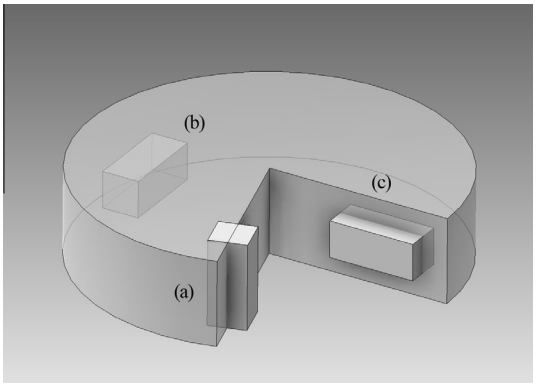

Fig. 1. Scheme of an HPT disk (diameter: 30 mm, height: 7 mm), with the dilatometric samples (dimensions: $3 \times 3 \times 7$ mm^3) denoted as (a) axial, (b) tangential and (c) radial with respect to the direction of the HPT axis.

For the DSC measurements, 14 samples, taken from three different positions of the HPT disk, were prepared. Six samples were cut from the disk at a radius of $r = 9$ mm at different heights. Four samples were prepared at radii of $r = 9.3$ and $r = 7.3$ mm. The measurements were performed with a Perkin Elmer DSC7 differential calorimeter, which determines the heat release for the annealing processes at different linear heating rates. A subsequent re-run served as the reference measurement and baseline for the analysis (for details see Ref. [13]).

Crystallite sizes were determined by a scanning electron microscope (LEO 1525 field emission scanning electron microscope, with a nominal resolution of 1.5 nm at 20 kV) equipped with a backscattering detector. For correlating the dilatometric length changes with modifications of microstructure, microscopy samples were prepared from the same part of the HPT disk as the dilatometric samples and subsequently annealed under identical conditions in the dilatometer up to predefined temperatures at a heating rate of 5 K/min, followed by rapid cooling to ambient temperature at a rate of about 30 K/min.

The crystallite size was determined from the SEM images by analyzing the area of the grains using the software program ImageJ [14]. A spherical grain shape was assumed for the analysis, and the arithmetic mean value of the diameters was calculated. The number of grains evaluated in each micrograph was about 380 except for the totally recrystallized sample annealed at 673 K, for which about 130 grains were considered. The error in the 1% range is due to uncertainties of assigning contrast features to grain boundaries.

3. Results

3.1. Correlation between microstructure and free volumes

Fig. 2 shows a typical dilatometric length contraction $\Delta l/l_0$, indicating the annealing out of defects associated with the release of free volume upon linear heating of the HPT-deformed Cu sample. Three substages, A, B and C, can clearly be discerned. In order to correlate these substages with annealing of specific types of lattice defects, a microstructural characterization by means of scanning electron microscopy was performed at the onset and at the end of each substage, as indicated by arrows in Fig. 2. In stage A, only a minor increase in the crystallite

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