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Direct evidence for grain boundary motion as the dominant restoration mechanism in the steady-state regime of extremely cold-rolled copper

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Abstract

Ultra-fine-grained high-purity copper (99.99%) deformed by means of high-pressure torsion into the steady-state regime was subjected to additional rolling deformation. The microstructural changes as a function of the applied strain were analysed by means of orientation imaging microscopy. It was found that after a distinctive rolling strain a steady state with respect to microstructural features such as grain size, misorientation distribution and texture evolves again. A special spilt specimen technique was used to perform quasi in situ observations of the microstructure between additional strain increments. Profound insights into the local deformation and restoration processes within the steady-state regime were gained. The observations lead to the conclusion that grain boundary migration perpendicular to the rolling direction leads to the disappearance of certain grains, enabling the occurrence of a steady state.

Keywords: Severe plastic deformation (SPD); Cold-rolling; Copper; Grain boundary migration; Steady state

1. Introduction

In the last decades, severe plastic deformation (SPD) methods have made it possible to easily apply ultra-high strains to metals at low homologous temperatures. The applied strain leads to enormous grain refinement, resulting in ultra-fine-grained (UFG) or nanocrystalline (nc) materials. However, grain refinement is not indefinite and terminates after a certain amount of strain and the processed materials reach a steady state in deformation regarding microstructural features such as grain size, grain shape or misorientation distribution [1–9]. Additionally, defect densities such as vacancies or dislocations also remain constant. This steady-state grain size represents a lower limit for grain fragmentation as additional straining does not lead to further refinement. The occurrence of a distinct minimum grain size for a specific deformation technique at a given temperature and strain rate is confirmed by deforming a material with a starting grain size smaller than the steady-state grain size obtained by a certain SPD process. By doing this, the metal coarsens towards the steady-state size obtained by deforming a coarse-grained starting material [10]. In contrast to the starting grain size, deformation temperature, amount of impurities and strain rate influence the resulting steady-state grain size [1,2].

In the steady-state regime, restoration mechanisms have to take place at the same time in order to keep grain features constant. Estimating the minimum grain size after a certain SPD process is truly a challenging task; however, several attempts with contradicting assumptions have been recently made to predict or model the steady-state grain

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size [11–15]. In Ref. [11] it was suggested that the steady state is a consequence of the equilibrium between grain growth due to adiabatic heating and the refinement caused by straining. Divinski et al. [12] proposed that cracks initiate and propagate during SPD and limit the minimum grain size achievable. If crack initiation can be avoided during deformation, the steady-state grain size will be larger than the one calculated by their model. Edalati and Horita developed a model for predicting the steady-state hardness of pure metals after SPD as a function of their atomic bond energy and related parameters [13]. Another model proposed that the minimum grain size is a consequence of recovery and generation of dislocations, with the stacking fault energy being one of the most important parameters for the obtainable steady-state grain size [14,15]. In Ref. [1] it was suggested that the mobility of the grain boundaries should be the crucial parameter for the occurrence of a minimum grain size during SPD and larger grains become subdivided again by the formation of low-angle grain boundaries (LAGBs).

Despite the vast amount of scientific research in the field of SPD in the last decades and the above-mentioned recently published models for predicting the minimum grain size, the actual deformation and restoration mechanisms within the steady state have not been investigated directly in an experiment due to the obvious experimental difficulties one would have to face. In this contribution these difficulties were overcome by using an innovative experimental technique. The model material, UFG copper, was pre-deformed via high-pressure torsion (HPT) into the saturation regime and deformed further through rolling. The characteristics of rolling allowed for the use of a split specimen and to study quasi in situ the processes during the steady state, applying for SPD standards very moderate strain increments. The information gained from these experiments will give valuable input for further modelling activities.

2. Experimental

UFG copper with an area-weighted grain size of 530 nm was produced by high-pressure torsion. For that, discs of pure copper (99.99%) with a diameter of 30 mm and a height of 6.5 mm were processed with an applied pressure of 3.5 GPa for 15 revolutions, with a rotational speed of 0.07 rotations min^{-1}. This results in a strain of $\varepsilon = 92.1$ at a radius of $r = 11$ mm according to Eq. (1) with the equivalent plastic strain ε, n the number of revolutions, r the radius and t the specimen thickness:

$$\varepsilon = \frac{2\pi n r}{t\sqrt{3}} \tag{1}$$

Details concerning the setup of the HPT tool used in this study can be found elsewhere [16]. Microhardness measurements along the radius of the copper disc were carried out to ensure that mechanically homogeneous properties were found across the entire disc, except for the very centre, $r < 0.5$ mm. The UFG copper discs were subsequently cold-rolled to different thickness reductions in a conventional rolling mill. The strain rate was kept fairly low. As a consequence, only small incremental thickness reductions and low rolling speeds were allowed in order to minimize self-heating of the copper sheet. The strain rate was estimated from Eq. (2) [17] to be $\dot{\varepsilon} = 10^{-1}\ \mathrm{s}^{-1}$, where Δt is the difference in thickness before and after a rolling pass, R is the radius of the rolls, ω is the angular speed of the rolls and ϕ is the logarithmic thickness reduction:

$$\dot{\varepsilon} = \frac{2}{\sqrt{3}} \frac{\omega\phi}{\sqrt{\frac{\Delta t}{R}}} \tag{2}$$

The applied rolling strain ε (equivalent strain) was calculated according to Eq. (3), with t_1 the thickness of the copper sheet after cold-rolling and, t_0 the thickness of the as-processed HPT disc (6.5 mm):

$$\varepsilon = \frac{2}{\sqrt{3}} \ln\left(\frac{t_1}{t_0}\right) = \frac{2}{\sqrt{3}}\phi \tag{3}$$

Microstructures after HPT processing and after different amounts of additional cold-rolling were captured from electron backscatter diffraction (EBSD) data obtained with a Zeiss LEO 1525 field emission gun scanning electron microscope (SEM). Grain size was calculated from these data using an orientation imaging microscopy (OIM) analysis software package. About 2000 grains were analysed for each condition. Crystallographic texture of the samples was characterized using a Rigaku SmartLab five-axis diffractometer equipped with Cu Kα radiation, a parabolic multilayer mirror in the primary beam and a secondary graphite monochromator. The measurements were performed using a Schultz reflection technique. The collected pole figure data were evaluated using the software package Labotex™ in order to quantify volume fraction of individual texture components.

Throughout this paper, the cold-rolled material will be described using rolling direction (RD), transverse direction (TD) and normal direction (ND), see Fig. 1. In addition to the microstructural and texture investigations, accompanying microhardness measurements with a load of 300 gf were carried out. An overview of the single deformation experiments specifying the geometrical changes and degrees of deformation is given in Table 1.

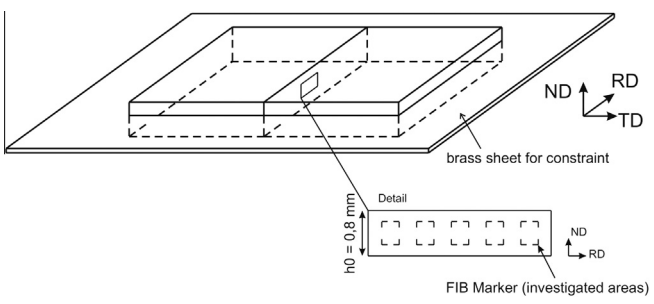

Fig. 1. Schematic of the experimental setup used for the quasi in situ EBSD experiments (inset image not to scale).