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Structural instabilities during cyclic loading of ultrafine-grained copper studied with micro bending experiments

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ABSTRACT

The cyclic mechanical properties and microstructural stability of severe plastically deformed copper were investigated by means of micro bending experiments. The ultrafine-grained structure of OFHC copper was synthesized utilizing the high pressure torsion (HPT) technique. Micron sized cantilevers were focused-ion-beam milled and subsequently tested within a scanning electron microscope in the low cycle fatigue regime at strain amplitudes in the range of $1.1 - 3.2 * 10^{-3}$. It was found that HPT processed ultra-fine grained copper is prone to cyclic softening, which is a consequence of grain coarsening in the absence of shear banding in the micro samples. Novel insights into the grain coarsening mechanism were revealed by quasi *in-situ* EBSD scans, showing i) continuous migration of high angle grain boundaries, ii) preferential growth of larger grains at the expense of adjacent smaller ones, iii) a reduction of misorientation gradients within larger grains if the grain structure in the neighborhood is altered and iv) no evidence that a favorable crystallographic orientation drives grain growth during homogeneous coarsening at moderate accumulated strains, tested here.

1. Introduction

Grain size reduction by severe plastic deformation (SPD) is well established to synthesize bulk ultrafine-grained (UFG) or nanocrystalline (NC) samples. Similar to the static strength, the performance in the high cycle fatigue (HCF) regime has been proven to be enhanced significantly compared to the coarse grained (CG) counterparts for UFG [1–4] and NC materials [5–8]. The reason for enhanced fatigue limits or HCF performance can be explained by a shift of the onset of microplasticity, necessary to induce fatigue damage, to higher stress levels than in the CG condition. Unfortunately, the performance of such materials in the low cycle fatigue (LCF) regime, where higher plastic strain amplitudes are present, is deteriorated and cyclic softening is promoted. For UFG materials, which will be in the focus of the current work, cyclic softening has been found to occur not only under strain controlled conditions (decreasing stress amplitude) for purity levels above 99.9% [9–12], but also in stress controlled experiments above a certain stress amplitude, where the softening is reflected in a continuously increasing strain amplitude [11,12]. However, these studies have conflicting viewpoints about the impact of the strain amplitude. Although it is frequently reported that cyclic softening is more pronounced at higher strain amplitudes [1,13,14], other experiments show that low strain amplitudes and the concomitant enhanced lifetime enable time dependent thermally activated processes to occur and promote cyclic softening [11]. Also, material parameters can be decisive for the occurrence or magnitude of cyclic softening, for instance, a high purity level [14] or the grain shape [1]. Although it is well known that different SPD procedures generate materials of different grain boundary structures, the grain boundary misorientation has been disregarded in the context of the cyclic mechanical response for a long time. Although cyclic softening was revealed for structures consisting of major fractions of low angle grain boundaries (LAGB) [1] or high angle grain boundaries (HAGB) [11], their direct influence has not been investigated systematically.

For UFG materials, three mechanisms have been found to contribute to the observed cyclic softening, which are i) shear band formation, ii) coarsening of the fine scaled grain structure [15,16] as well as iii) a reduction of the defect density, in especially dislocation density [3,9], or a combination of them. These mechanisms can lead

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to early strain localization, fatigue damage and failure of the samples. The small grain size and the resulting large grain boundary fraction was suspected to be responsible for these microstructural instabilities as it offers large driving forces for grain growth.

Despite the extensive work carried out on this topic, detailed knowledge about the initiation and evolution of these structural instabilities is still at its infancy. As an example, the nature of the grain growth process in these fine-scaled structures is currently not completely understood. A thorough description, whether grain growth proceeds in discrete events or in a rather continuous manner, if a certain incubation time is needed, or which grains will start to grow, is still unclear. Further issues include whether a certain crystallographic orientation is favored to grow or shrink, as well as the importance of the grain boundary type (LAGB, HAGB).

The ambiguities about the processes inducing structural instabilities prevent an understanding of how cyclically stable materials need to be designed. A sound identification of the driving forces for the underlying processes and how they evolve are of utmost importance to control the microstructural stability in a successful way.

To gain a thorough understanding about the mechanisms occurring during cyclic loading of UFG structures, a suitable experimental setup was looked for. Cyclic micro bending experiments, conducted inside a scanning electron microscope (SEM), allow for a unique correlation between changes in the microstructure and the local stress-strain state. Further, tracing the same sample area throughout the entire experiment enables the detection of crucial changes in the surface morphology, for instance shear bands or fatigue cracks which in turn may affect the softening process. Special emphasis was placed on the identification of possible correlations between boundary misorientation and crystallographic orientations with structural instabilities.

2. Experimental

Quasi constrained high pressure torsion (HPT) [17] was used to synthesis an oxygen free high conductivity (OFHC) copper (99.95%) with ultrafine grains. The HPT disk diameter d and its thickness t were 15 mm and 7.5 mm, respectively. Deformation was conducted at a pressure of 3.5 GPa for 15 revolutions n, which resulted in an equivalent v. Mises strain ε_{eq} of 87 at an HPT radius r of 12 mm, from which the sample was extracted, according to Eq. (1)

$$\varepsilon_{eq} = \frac{2\pi nr}{t\sqrt{3}}. \tag{1}$$

Such strains are sufficient to obtain constant mechanical and structural properties throughout the entire HPT disk, except the very center (r < 1 mm). In this steady state region the grain size was measured by electron back scatter diffraction (EBSD) to be 530 nm (area weighted).

For the cyclic micro bending experiments, a platelet was extracted from regions of saturated microstructure, which was subsequently cut into rods of 1 mm × 1 mm in cross-section. The top of the rods were electrochemically etched to form tips, where the final bending beam was focused-ion-beam (FIB) milled with a Zeiss LEO 1540 XB dual beam FIB workstation. For the bending beams, thickness to length ratios between 1:1 and 1:2 were used. A schematic drawing of a bending beam is shown in Fig. 1. The actual size of all samples tested can be found in Table 1.

To illustrate the mechanical and the structural changes, two samples have been selected for this study, denoted sample A and B. The cyclic micro bending experiments were conducted inside a SEM (Zeiss LEO982) using an ASMEC UNAT (sample A) and a Hysitron PI85 (sample B) microindentation system to impose the strain amplitude. A FIB milled tungsten double blade gripper was used to impose the cyclic load onto the samples. The bending beams were loaded under displacement control, resulting in plastic strain amplitudes $\varepsilon_{a,pl}$ of $1.1 - 3.2 \times 10^{-3}$ at the outer fiber, with a stress ratio $R = -1$ at a strain rate $\dot{\varepsilon}$ in the range of $2.6 - 3.8 \times 10^{-3} s^{-1}$ (for details see Table 1). For sample B EBSD scans were conducted after 100, 200, 300 and 400 cycles to track the coarsening process.

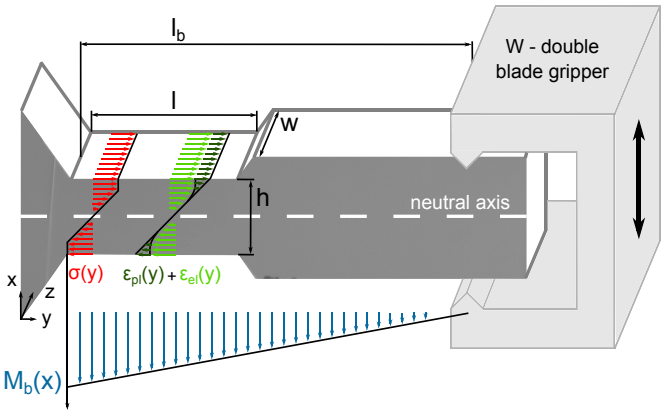

Fig. 1. Schematic drawing of a miniaturized bending beam with dimension labels of the width w, height h, length l and bending length l_b, as well as the distribution of the normalized bending moment $M_b(x)$ along the x-axis. Strain $\varepsilon(y)$ and stress $\sigma(y)$ values along the y-axis are indicated.

The elastic contributions of the measured displacement stemming from the needle, SEM-stage and the specimen holder were taken into account by correcting the measured compliance using a method according to Wurster et al. [18]. The outer fiber stress σ_s was calculated from the force-displacement data, based on elastic bending beam theory according to Eq. (2):

$$\sigma_s = \frac{6Fl_b}{wh^2} \tag{2}$$

Although this is a good approximation for small strain amplitudes, it overestimates the stress at larger strain amplitudes. The outer fiber strain ε_s according to Eq. (3):

$$\varepsilon_s = \frac{uh}{2ll_b} \tag{3}$$

is simply derived from the applied displacement and assumed to be constant in the gauge length. The measured force is F, the bending length l_b, gauge section width w, gauge section height h, the beam deflection u and the gauge section length l.

3. Results

3.1. Cyclic hysteresis loops

The cyclic hysteresis loops of sample A are shown in Fig. 2 for two different plastic strain amplitudes, $\varepsilon_{a,pl} = 1.1*10^{-3}$ (a) and $1.9 * 10^{-3}$ (b-d), respectively. For both strain amplitudes only certain cycle numbers (90, 990, 2990, 4990, 5790 for $\varepsilon_{a,pl} = 1.1*10^{-3}$ and 90, 790, 990, 1190 after the increase to $\varepsilon_{a,pl} = 1.9*10^{-3}$) are plotted to ensure a better visibility. The maximum tensile and compressive surface stresses at the lower strain amplitude $\varepsilon_{a,pl} = 1.1*10^{-3}$ in Fig. 2a stay nearly constant up to 5790 cycles, corresponding to an accumulated plastic strain $\varepsilon_{acc,pl}$ of 25.5 according to Eq. (4):