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Size effect in bi-crystalline micropillars with a penetrable high angle grain boundary

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ABSTRACT

The implications of various size effects on the deformation behavior of and near grain boundaries is not yet fully understood. In this manuscript, slip transfer mechanisms through a general high angle grain boundary (HAGB) allowing for easy transfer are investigated in order to understand the size dependence of the dislocation-grain-boundary interaction. Complementary *in situ* micro compression tests on copper single and bi-crystals in the scanning electron microscope and with x-ray Laue microdiffraction were used to correlate the mechanical response with the evolving microstructure. It is shown that no dislocation pile-up is formed at the boundary. The lack of pile-up stresses results in a deformation process which is dominated by the initial dislocation source statistics. This is evidenced by similar size scaling of the single and bi-crystalline samples with the grain size being the characteristic length scale.

1. Introduction

Grain boundaries (GB) strongly influence the mechanical properties of materials, as independently shown by Hall [1] and Petch [2]. This is mainly due to the fact that dislocations cannot simply transmit through a GB, as the Burger's vector of one grain is generally not a lattice vector in the adjacent grain. The inability of dislocations to transmit easily through a GB had been known ever since the concept of dislocations was introduced, however, until today a thorough quantitative understanding of dislocation slip transfer is still lacking. One reason for a poor quantitative understanding of slip-transfer mechanisms is, that even for one particular boundary several different dislocation reactions might take place simultaneously in bulk materials. This often hinders a direct observation of a specific deformation mechanism. Nevertheless, already by analyzing the slip traces at the surface of bi-crystalline samples first insights into slip transfer mechanisms were revealed [3,4]. In the 1970's and 80's, transmission electron microscopy (TEM) investigations were applied to study dislocation-GB reactions. Following mechanism were observed: dislocation absorption at the GB [5,6], dislocation reflection [7], dislocation nucleation from the GB [8] and, under specific conditions, dislocation transmission through the GB [5,9].

Today, two transmission criteria which predict the transmitting slip systems are well-established. The first criteria (further denoted as the T-criteria, described in eq. (1) [4]) accounts for the amount of shear transfer from one slip system to the other. Hence, $T = 1$ results into full shear transfer, which would not give rise to grain-size hardening [10]. The second criteria is based on the θ angle defining M criteria (see eq. (2)). θ is defined as the angle of the two intersections of the slip planes and the GB plane [5,9]. This is particularly important as the dislocation line needs to reorient in the GB plane, which often involves thermally activated processes [11]. Hence, slip systems with high M criteria have small θ angles and are predetermined to show slip transfer.

$$T = \frac{\overrightarrow{b_1} \cdot \overrightarrow{b_2}}{\left|\overrightarrow{b_1}\right| \cdot \left|\overrightarrow{b_2}\right|} \cdot \left(\overrightarrow{n_1} \cdot \overrightarrow{n_2}\right) + \left(\frac{\overrightarrow{b_1}}{\left|\overrightarrow{b_1}\right|} \cdot \overrightarrow{n_2}\right) \cdot \left(\overrightarrow{n_1} \cdot \frac{\overrightarrow{b_2}}{\left|\overrightarrow{b_2}\right|}\right) \quad (1)$$

$$M = \overrightarrow{L_1} \cdot \overrightarrow{L_2} \quad (2)$$

For both slip criteria the pure slip geometry defined by the Burger's vectors $\vec{b}$, the unit normal to the slip plane $\vec{n}$ and the

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intersections between the GB and the slip planes $\vec{L}$ are considered.

As previously mentioned local dislocation-GB interactions in bulk materials are greatly influenced by simultaneous plasticity in adjacent grains. Therefore, all aforementioned dislocation-GB interactions can simultaneously take place in the material but, eventually, the local dislocation type and GB structure [9] determine the dominating mechanism. To be able to identify the impact of one discrete interphase on the mechanical properties, a micro fabricating technique of single- and bi-crystalline samples firstly introduced by Uchic is nowadays widely used [12]. Until today, numerous micromechanical experiments on single crystalline samples were conducted revealing size dependent plasticity of micron and submicron sized samples [13–16]. This is manifested by (i) an increased yield strength for smaller samples [17] and (ii) a stochastic mechanical response (e.g. Ref. [18]) which both are caused by source size distribution in single crystals [19–21].

Experiments on micro bi-crystals with one GB have been conducted to investigate the influence of both, the GB and size effects on the dislocation-GB interaction. First work on 6 μm sized, focused ion beam (FIB) milled aluminum bi-crystals demonstrate smaller strain bursts and a higher strength compared to their single crystalline counterbodies, which was mainly explained by dislocation storage in distinct grains [22]. Imrich et al. showed the formation of smaller slip steps and pronounced hardening for micron sized copper bi-crystals containing a general high angle grain boundary (HAGB) compared to equally sized single crystals [23]. This was attributed to source truncation hardening in individual grains. Both studies represent the case when the GB acts as an obstacle for dislocation motion. In contrast to this, Kunz et al. showed an absence of dislocation storage in aluminum bi-crystals and suggested that the GB is a dislocation sink [24]. Surprisingly, they revealed an identical behavior of single- and bi-crystals independently on the samples size. On the contrary, Kheradmand et al. [25] showed dislocation transmission through the GB for large nickel bi-crystals. Arguing that the dislocation-dislocation interactions define the deformation process in larger samples [26], Kheradmand et al. show less transmission events when decreasing the sample size down to 1 μm. That was explained by increased transmission stresses [25] and more frequent dislocation-GB reactions in this size regime compared to larger samples.

Though many attempts have been undertaken to resolve the impact of different GBies on the deformation behavior, further studies are necessary to achieve a thorough and quantitative understanding of size-dependent dislocation-GB interaction. Therefore, state-of-the-art *in situ* characterization methods like TEM or synchrotron based Laue microdiffraction (μLaue) are required to interlink the type and density of dislocations to the mechanical response.

Here, we apply *in situ* μLaue diffraction compression tests. μLaue diffraction allows to assess the crystallographic phase, the deviatoric strain tensor [27–30] and – in an ideal case – the density and type of geometrically necessary dislocations (GNDs) [31]. Combined with small scale deformation, e.g. in terms of micro pillar compression, unprecedented insights into the deformation behavior of crystalline materials had been achieved during the last decade. For instance, many studies were conducted to unravel origins of the sample size effect [32], the influence of FIB milling on the mechanical properties of small scale structures [32,33] as well as the activation of non-favored slip system in compression [32–34] and tension [35]. This makes *in situ* μLaue diffraction a method well-suited to investigate the dislocation-GB interaction at micron scale.

This work is aiming for a quantitative understanding of the size dependent slip transfer through a nominally penetrable general HAGB, which complements former work on twin boundary containing pillars (being no obstacle for dislocation slip [23,36,37] and bridges to current work on impenetrable boundaries [23,38]). As model material, copper bi-crystals grown by the Bridgman technique are used, and subsequently analyzed by *in situ* micro compression in a scanning electron microscope and at BM32 of the European Synchrotron Radiation facility (ESRF).

2. Experimental details

A bulk bi-crystal was grown using two seed crystals by the Bridgman method from 99,88 at % purity copper. The bi-crystal contains a single, HAGB with tilt and twist components with misorientation angle of 30°around [1 2 10] and compression directions close to <225> and <479> for grain A and grain B respectively.

Two types of experiments were conducted:

(i) *in situ* μLaue measurements, where freestanding rectangular shaped micro samples (Laue samples) are used (Fig. 1d–f), and
(ii) *in situ* scanning electron microscopy (SEM) compression tests where cylindrical shaped samples (annular samples) of different sizes are investigated (Fig. 1a–c).

In both cases, small slices of $5 \times 5 \times 0.5$ mm were cut from the bi-crystalline bulk material (i) parallel to the growing direction for μLaue samples (see Fig. 1d) and (ii) perpendicular to the growing direction for annular samples (see Fig. 1a) so that the compression direction stays identical irrespective of the sample geometry. To obtain a smooth and defect free surface the slices were first mechanically and then electrochemically polished in phosphoric acid using voltage from 15 V to 2 V for coarse and fine etching.

The annular micro samples were then directly milled on the polished slices. The μLaue samples requires further thinning of the slices to a wedge form with a rounding diameter to 1 μm at the top where the subsequent micro samples are placed following the approach of Moser et al. [39]. Multiple annular micro samples can be prepared on one macro specimen, whereas only one bi- and several single crystals can be prepared on one wedge which therefore requires the preparation of multiple wedges for numerous compression tests of μLaue bi-crystals. In both cases, a Zeiss Auriga® operated with Ga^+ ions at 30 keV was used for micro sample preparation gradually reducing currents from 16 nA for coarse to 600 pA for fine milling. After compression tests, SEM images were taken and further analysis with regard to activated slip system, slip steps and GB behavior was conducted.

2.1. In situ μLaue compression tests of rectangular pillars

All micro specimens were shaped by focused ion beam milling to the size of $5 \times 5 \times 15\ \mu m^3$ with sides being parallel to the compression direction to ensure a constant cross-section throughout the pillar height (see Fig. 1e–f). Due to the fact, that samples with cross-sections smaller than 3 μm in copper do not allow for a separation of elastic bending and GND caused curvature [40], as well as the diffraction signal might greatly be influenced by the tales of the primary beam [41], only one sample size of 5 μm was tested. To establish a top sample surface being parallel to the flat punch indenter, an overtilt of 1.5° was used at the final milling stage. The GB is placed in the center of the micro sample parallel to both, compression direction and sample sides so that, ideally, no shear stress component acts on the grain boundary during compression. The flat punch indenter was etched out of 300 μm tungsten wire and subsequently thinned by FIB milling to the final shape. The compression experiments were performed at the