Investigation of the role of grain boundary on the mechanical properties of metals

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Abstract. Compression testing of micropillars was used to investigate the gain boundary effect on the strength of metals which is especially interesting in ultra fine grained and nanocrystalline metals. Single and bicrystal micropillars of different sizes and crystallographic orientations were fabricated using a focused ion beam system and the compression test was performed with a nanoindenter. A reduction of the pillar size as well as the introduction of a grain boundary results in an increase in the yield strength. The results show that the size and the orientation of different adjoining crystals in bicrystalline pillars have an obvious effect on dislocation nucleation and multiplication.

1. Introduction
The mechanical properties investigation of metals by microindentation shows that the hardness increases by decreasing the grain sizes. However, the trend of this change for grain sizes between 20 nm and 1 µm is altering [1]. This clearly reveals the local interaction between dislocations and grain boundaries in small grain materials. In this range strain rate sensitivity is also observed [2]. Yang et al [3] used nanoindentation to examine the role of dislocations-grain boundary interaction in this grain size range. They indented within only one grain in order to investigate the interaction between grain boundary and dislocations. Their results provided the interaction of dislocations with all grain boundaries surrounding the indented grain. In this paper we introduce a novel method based on micropillar compression testing [4], to investigate the interaction between dislocations and a specifically chosen single grain boundary.

2. Experiments
Micropillars were cut in a coarse grained pure nickel (99.99%) sample which was prepared according to the procedure described in [5]. The Orientation Imaging Map (OIM) shown in figure 1a used to choose the arbitrary orientations, in which single crystalline (figure 1c) and bicrystalline (figure 1e) pillars were cut using a Strata™ dual beam focused ion beam (FIB) system manufactured by FEI. These pillars, with different diameters, were compressed in a Hysitron® TriboIndenter™ nanoindentation system equipped with a diamond flat punch tip. The compression tests were performed in two ways: load control and displacement control, where a constant loading rate or strain rate was applied, respectively.
3. Results and discussion

3.1. Single crystalline micropillars (SCM)

The stress-strain curves of \( \approx [153] \) oriented micropillars with diameters \( \approx 7 \) and \( \approx 3 \) \( \mu \)m are shown in figures 2a and 2b. The curves show following differences: 1) the larger pillar has a continuously elastic to plastic deformation. The smaller pillar, however, shows an elastic deformation followed by several strain bursts (pop-in) at constant loads. 2) The larger sample has a lower strength in comparison to the smaller micropillar which is in good agreement with previous observations [4, 6, 7].

Figure 1: a) OIM of the coarse grained Ni sample used for producing micropillars. The orientations of grains are marked on the inverse pole figure. b,d) SEM images of grains (A and B) and the grain boundary, where the micro SC and BC pillars are produced. c,e) SEM images of SC and BC pillars. The SC Pillar is cut inside one grain (e.g. grain A) and BC pillar on the grain boundary between grains A and B.

A new approach in this work was the stepwise compression test. This means that the stress-strain curves are not resulted from one single compression test but they are sum of consecutively performed compression tests on a pillar with increasing maximum load in the load function. Between each step the sample was observed in SEM, as shown in figure 2b, which provided more detailed information from intermediate steps of pillar deformation. Additionally by means of stepwise loading it was possible to observe the plastic deformation initiation in micropillars.

Figure 2: The stress-strain curves of SCMs of different sizes a)\( \approx 7 \) \( \mu \)m and b)\( \approx 3 \) \( \mu \)m with the SEM micrograph taken in intermediate steps of compression test.

3.2. Birystalline micropillars (BCM)

As mentioned the aim of this work was the investigation of the grain boundary effect on the plastic deformation of fine crystalline metals. Therefore the compression tests were performed on BCM’s with different diameters in the range of 1 to 7 \( \mu \)m. The stress-strain curves for
two different BCM’s with \( \approx 1 \) and \( \approx 3 \) \( \mu \)m diameters are shown in figure 3. The observations show, as in the case of the SCM, by decreasing the size of the BCM the strength increases.

Figure 3: a) Stress-strain curves of BCM’s with different sizes. b) Post-compression SEM image of a \( \approx 3 \) \( \mu \)m BCM, a dislocation loop is shown schematically. One side of the loop reaches the free surface, which results in formation of steps. The other side of the loop reaches the grain boundary, which results in the pile-up of dislocations and bowing out of the grain boundary. c) Post-compression SEM image of a \( \approx 1 \) \( \mu \)m BCM. The pillar deforms as a SCM and shear bands cross the grain boundary.

Figure 4: Yield stress vs. micropillar diameter of SCMs and BCMs. The curves show two parameters affecting the yield stress: 1) the pillar size and 2) the grain boundary. It shows that there are two important parameters affecting the yield stress of pillars: 1) the pillar diameter and 2) the grain boundary. An explanation to the first effect can be found in [8]. A decrease of the pillar’s diameter leads to a lower dislocation density due to the loss of dislocations through
the free surface image stresses. The grain boundary effect is more complicated compared to the size effect. First of all the BCM’s can be considered as two single crystals adjoining each other. In the first order approximation we can consider the pillars elastic isotropic and write the relation 1;

\[ \epsilon_{BCM} = \epsilon_1 = \epsilon_2 \Rightarrow \sigma_{BCM} = \sigma_1 = \sigma_2 \]  

where are the strains and are the stresses in BCM, grain 1 and grain 2, respectively. It means that by ignoring the effect of the grain boundary a BCM can be equivalated to two SCM with the following relation

\[ d_{BCM} = d_{SCM} \sqrt{2} \]  

where and are the diameters of BCM and equivalent SCM’s, respectively. Since the Schmid factors of two grains are different, the one with a higher Schmid factor will tend to yield earlier. Therefore, the measured yield stress can be associated with the grain with the higher Schmid factor i.e. equivalent SCM containing this grain. Therefore, we can expect a shift to the left for BCM’s yield stress curve in figure 4 by considering the BCM’s as two single crystals adjoining each other. This is shown in figure 4 by the dash line ”equivalent SCM for BCM”. In spite of this shift, the curve dose not overlap with the curve belonging to the single crystals. It shows the strengthening effect of grain boundary for small sizes. The other interesting observation during the compression tests on BCM’s was the difference in their plastic deformation behaviors as their sizes changed. As shown in post-compression SEM image of \( \approx 3 \ \mu m \) BCM, the grain boundary was deformed under the loading. One can imagine that, the plastic deformation initiates in the grain with the higher Schmid factor (left side grain of BCM in figure 3b by the activation of a Frank-Read source. The activated dislocation loop starts to grow by further increasing of the load. One side of the dislocation loop reaches the free surface and results in the formation of steps, while the other side of the loop reaches the grain boundary and results in the pile-up of dislocations behind the grain boundary. Thus by increasing the load the pile up force on the grain boundary increases and results in the bowing of the grain boundary as shown in figure 3b. In the \( \approx 1 \ \mu m \) BCM shown in figure 3c, however, the grain boundary seems to be absent and the whole pillar deforms like a single crystalline pillar and shear bands crosses the micropillar. This effect is still not fully understood and further experiments are required to clarify this size effect in small BCM’s.

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