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Smaller is stronger: The effect of strain hardening

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Abstract

Single-crystal face-centered cubic metal pillars synthesized using a focused ion beam are reported to be stronger when compressed in smaller volumes. Using in situ Laue diffraction and crystal plasticity simulations it is shown that plastic deformation is initially controlled by the boundary constraints of the microcompression tests, followed by classical crystal plasticity for uniaxial compression. Taking the stress at which the change between the two modes occurs as strength of the pillar instead of the flow stress at a fixed amount of strain, the "smaller is stronger" trend is considerably reduced, if not eliminated, and what remains is a size dependence in strain hardening. The size-dependent increase in flow stress is a result of the early activation of multiple slip systems and thus the evolution of the microstructure during compression.

Keywords: Microcompression; Plastic deformation; Strain gradient; Single crystal; Strain hardening

1. Introduction

The development of a microcompression technique allowing compression of pillars with diameters below 10 μm has opened new routes for investigating the mechanical behavior of small volumes [1]. Such a technique is important for studying material properties for microtechnological applications [2–4], but the technique is also very promising for the investigation of small irradiated volumes or individual phases in complex advanced alloys. Microcompression revealed an increase in strength for single-crystal pillars when pillar diameters are reduced below 10 μm [1,5–8]. The "smaller is stronger" trend for single crystals has perplexed materials scientists, because it does not fit into classical crystal plasticity where the strength of a single crystal does not depend on its size but rather on the geometrically predicted dislocation slip system(s) for which the resolved shear stress is the highest [9]. The strength or resistance to permanent strain is expressed by the yield stress or the onset of percolative slip and is usually defined as the flow stress at 0.2% plastic strain [10]. When a single crystal is deformed to larger plastic strains, other mechanisms come into play such as dislocation interactions resulting in entanglements and self-organization mechanism forming crystallographic substructures. During this microstructural evolution, the metal hardens, i.e. the flow stress increases [11]. At very large strains a polycrystal composed of grains with different orientations is formed. The yield stress of a polycrystal is well known to increase with the inverse square root of the grain size which is ascribed to the piling up of dislocations at the interfaces between adjacent grains [12,13].

Because of the large stress–strain scatter observed in the initial stage of plastic flow in a microcompression test, the flow stress at a relative large amount of total strain (usually 5% or more) is used to demonstrate the "smaller is stronger" trend [1,5,6,14–16]. Since elastic strains in pure face-centered cubic (fcc) metals are well below 1% total strain

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[17], the origin of the remaining total strain has to be explored to justify the 5% criterion.

X-ray diffraction has been used for local probing of the microstructure and has been demonstrated to allow the spatial resolution of local strains within dislocation cells [18,19], the observation of grain rotation during plastic deformation [20] and the existence of dilatational strain gradients in Mo-alloy pillars of 550 nm diameter [21]. Here, polychromatic microfocused Laue diffraction was performed in situ during microcompression, as this technique elucidates the dynamics of the self-organization process of dislocations leading to the formation of subgrain structures [22]. Laue diffraction performed with a micron-sized beam on micropillars synthesized using a focused ion beam (FIB) has demonstrated the presence of strain gradients and defects prior to deformation [23–25]. Performed in situ, the technique has demonstrated the role a pre-existing strain gradient can play on the selection of the activated slip planes at large total strains [26], and also the formation of rotational gradients at high strains [27]. The present study focuses on lattice rotations captured in situ by tracking the path of the Laue spot at low total strains, with the goal of approaching as close as possible to the onset of percolative slip. The in situ Laue method is combined with crystal plasticity finite-element simulations, allowing us to study the influence of the boundary conditions on slip activation.

2. Materials and methods

2.1. Analysis of Laue latterns

A Laue diffraction pattern taken with a polychromatic X-ray beam is characterized by individual spots, each related to a different hkl-family. The position of the Laue spots depends on the crystal orientation and the shape of the unit cell. Therefore any crystal rotation and/or change in the shape of the unit cell will result in peak movements. For Laue analysis it is practical to decompose the strain tensor into a deviatoric and a hydrostatic (isotropic dilation) strain tensor [28,29]. Isotropic changes in the crystal unit cell do not change the position of the Laue reflections. Therefore, to determine the lattice parameter and the hydrostatic dilatation strain tensor, energy scans with a monochromatic X-ray beam are needed. On the other hand, deviatoric strains result in changes in the shape of the unit cell and therefore result in reflections that are slightly offset from their unstrained positions. Continuous streaking of Laue reflections obtained from polychromatic X-rays is therefore related to the presence of deviatoric strain gradients in the illuminated volume. Such gradients are often—but not necessarily—related to an excess dislocation content of one type. Indeed, an elastic tetragonal distortion of the unit cell will also cause deviatoric strains without the presence of dislocations. Discontinuous streaking points towards misorientations and is usually related to the presence of misorientations dislocation walls, forming geometrically necessary boundaries [30].

In this paper polychromatic Laue diffraction is applied and therefore the observed peak streaking is caused by deviatoric gradients. During in situ deformation, the movement of Laue peaks is interpreted as crystal rotation. In this way the path of the spots can be directly linked to a specific active slip system using the rotational Taylor model [31,32]. From the collective motion of all the Laue spots, the rotation of the compression axis can be quantified [33].

2.2. Investigated material

In situ tests were conducted on Au samples being oriented for single (⟨1 2 3⟩, ⟨3 4 6⟩) and double slip (⟨0 0 1⟩), and on single slip oriented Ni (⟨1 2 3⟩). Both the ⟨1 2 3⟩ - and ⟨0 0 1⟩-oriented Au, and the ⟨1 2 3⟩-oriented Ni pillars were synthesized from a bulk single crystal that was obtained from the melt by the Czochralski method and provided by Mateck. The ⟨3 4 6⟩-oriented samples were prepared from a well-annealed Au foil, of which more details can be found in the online material of Ref. [26]. It has previously been shown that the 3 μm Au⟨3 4 6⟩-pillar contained a twin [24]. All Au pillars were cut out from the bulk crystal using FIB, employing the annular milling procedure [6], providing a typical mean sidewall taper of ∼2.3° and aspect ratios ranging between 1.3 and 2.2 if the diameter is determined at half the pillar height. Au pillars with diameters between 2 and 10 μm were investigated. The 8.0 μm (A and B) and one 4.0 μm Ni pillars with an aspect ratio of 2.8 were prepared by the lathe FIB-milling technique [34] by M.D. Uchic and have no taper. In total 13 pillars were investigated, for which the diameter and initial compression axis orientation can be found in the first two columns of Table 2 below. Knowing the pillar axis orientation derived from the diffraction data enabled the Schmid factors (SFs) for each slip to be calculated. Table 1 provides the 12 SFs obtained for both the ⟨1 2 3⟩-oriented Au and Ni pillars. Small differences in SF between Au and Ni are due to small differences in alignment of the bulk crystals on the sample holder.

Table 1
Schmid factors (SF) for Au[1 2 3] and Ni[1 2 3].

Au			Ni		
Nr.	Slip system no.	SF	Nr.	Slip system no.	SF
1	(−1 1 1)[1 0 1]	0.472	1	(−1 1 1)[1 0 1]	0.475
2	(1 1 1)[−1 0 1]	0.365	2	(1 1 1)[−1 0 1]	0.374
3	(−1 1 1)[1 1 0]	0.340	3	(−1 1 1)[1 1 0]	0.349
4	(1 −1 1)[0 1 1]	0.302	4	(1 −1 1)[0 1 1]	0.281
5	(1 1 1)[−1 1 0]	0.194	5	(1 1 1)[−1 1 0]	0.195
6	(1 1 1)[0 −1 1]	0.174	6	(1 1 1)[0 −1 1]	0.179
7	(1 −1 1)[1 1 0]	0.171	7	(1 −1 1)[1 1 0]	0.160
8	(−1 1 1)[0 −1 1]	0.132	8	(−1 1 1)[0 −1 1]	0.126
9	(1 −1 1)[−1 0 1]	0.127	9	(1 −1 1)[−1 0 1]	0.121
10	(−1 −1 1)[0 1 1]	0.025	10	(−1 −1 1)[0 1 1]	0.024
11	(−1 −1 1)[1 0 1]	0.020	11	(−1 −1 1)[1 0 1]	0.019
12	(−1 −1 1)[−1 1 0]	0.005	12	(−1 −1 1)[−1 1 0]	0.005