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FIB-induced dislocations in Al submicron pillars: Annihilation by thermal annealing and effects on deformation behavior



Subin Lee ^a, Jiwon Jeong ^{a, b}, Youbin Kim ^c, Seung Min Han ^c, Daniel Kiener ^d, Sang Ho Oh ^{e, a, *}

^a Department of Materials Science and Engineering, Pohang University of Science and Technology (POSTECH), Pohang 37673, Republic of Korea

^b IBS Center for Integrated Nanostructure Physics (CINAP), Institute for Basic Science, Suwon 16419, Republic of Korea

^c Graduate School of Energy Environment Water and Sustainability, Korea Advanced Institute of Science & Technology (KAIST), Daejeon 34141, Republic of Korea

^d Department Materials Physics, Montanuniversität Leoben, Jahnstraße 12, 8700 Leoben, Austria

^e Department of Energy Science, Sungkyunkwan University, Suwon 16419, Republic of Korea

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ABSTRACT

In-situ transmission electron microscopy (TEM) annealing of submicron Al pillars (~300–450 nm in diameter) fabricated by focused ion beam (FIB) shows that the dislocation loops formed by the high energy Ga⁺ ion beam impact near the material surface can be removed by annealing at around half of the melting point (T_m). Quantitative analysis of real-time TEM data reveals that the dislocation loops first show a ripening behavior at around $0.4T_m$, i.e. larger loops coarsen at the expense of smaller ones. Subsequently, the ripened loops start to shrink and are eventually annihilated at $\sim 0.5T_m$ by the diffusion of thermally activated vacancies. Microcompression tests on the as-fabricated and annealed pillars suggest that the FIB-induced defects, particularly dislocation loops, distinctively affect the deformation behavior of submicron Al pillars; while the yield and flow stresses appear unaffected by the annealing, strain bursts are larger and more frequent in the annealed pillars compared to those of as-fabricated samples. In-situ TEM compression revealed that the initial plastic deformation and subsequent plastic flow of Al pillars are significantly altered by the presence of FIB-induced dislocation loops, as they actively respond to the applied stress. We observe that the initial dislocation activity in most FIB-prepared pillars was the glide of FIB-induced dislocation loops. With further straining, the dislocation loops escaped the pillar, leaving slip steps at the pillar surface and/or dislocation debris within the pillar volume via the interaction with other mobile dislocations. The subsequent dislocation slip is mostly localized at these locations, thereby forming large slip steps. Contrarily, in the case of annealed pillars, the deformation was rather homogeneous with the formation of multiple fine slip steps. The present direct TEM observations contribute to the understanding of the nature of FIB-induced defects and reveal their distinct roles in the deformation behaviors of submicron pillars.

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1. Introduction

Size effects in mechanical properties of face-centered cubic (fcc) single crystals have attracted tremendous interest since Uchic et al. reported that the yield strength of single crystal Ni pillars increases as the pillar diameter decreases to micron or submicron sizes [1–4]. This phenomenon is referred to as “smaller is stronger”, and is now understood as a common feature of single crystal metallic

materials, valid not only for fcc but also for body-centered cubic [5–7] and hexagonal close-packed metals [8,9]. Several models have been suggested to explain the mechanical size effects. One is the source truncation model, which is based on an increase of the average stress for the activation of dislocation sources with decreasing sample size [10,11]. The other model is based on the exhaustion of mobile dislocations due to the limited glide distance from a source to the pillar surface and thus the lack of mutual interaction with other dislocations, which limits possible multiplication processes [12,13].

Most of the experimental work on mechanical size effects has been conducted through microcompression testing of single crystal

* Corresponding author. Department of Energy Science, Sungkyunkwan University, Suwon 16419, Republic of Korea.

E-mail address: sanghooh@skku.edu (S.H. Oh).

pillars fabricated by using a focused ion beam (FIB). The high energy Ga^+ ion beam used for FIB milling, however, is known to induce various structural defects near the pillar surface, such as vacancies, interstitials, and also their aggregates forming stacking fault tetrahedra or dislocation loops [14]. The damaged region containing such defects extends up to several tens of nanometers from the surface, of which the exact thickness varies depending on the milling condition and target material [15,16]. These FIB-induced defects, particularly dislocation loops, may critically affect the deformation behavior and/or the mechanical properties of FIB-prepared pillars. For instance, the effects of FIB milling were investigated by dislocation dynamics simulations, showing that the flow stress of submicron fcc pillars with a diameter in the range of 0.5–1.0 μm increases by 10–20% due to dislocation pile up at the damage layer [17]. A FIB-induced surface hardening effect was also observed experimentally for single crystal Mo alloys when increasing the acceleration voltage of the Ga^+ ion beam during FIB milling [18,19]. Kiener et al. reported that the flow stress of FIB-fabricated Cu pillars can increase due to the solid solution hardening effect of Ga in Cu [20]. All these studies suggest that FIB milling alters not only the strength, but also the plastic flow characteristics of metals in small dimensions.

Removal of FIB-induced defects is therefore crucial for exploring the true mechanical size effects and inherent deformation behavior of metal pillars. Shan et al. showed that FIB-induced dislocation loops in Ni pillars can be removed by applying a small amount of (plastic) strain, which was coined as “mechanical annealing” [21]. Another practically more acceptable way to remove FIB-induced defects would be a thermal annealing. Unwanted formation of a surface oxide or contamination layer can be suppressed by conducting the annealing in high vacuum. Recently, Lowry et al. reported that the yield stresses of FIB-prepared Mo pillars are recovered close to the theoretical strength after thermal annealing [22]. Similar results were also obtained by Kiener et al. for Cu pillars [23]. Although several previous studies have shown that the FIB-induced defects affect the mechanical properties of metal pillars and this can also be modified by thermal annealing, the basic thermal processes leading to defect annihilation, as well as the detailed role of these defects in the stochastic plastic deformation of small volumes, are still not understood well.

In the present study, to elucidate the underlying mechanisms controlling the annihilation of FIB-induced dislocation loops and their influences on the mechanical properties and the deformation behavior of metal pillars, we carried out in-situ transmission electron microscopy (TEM) annealing, as well as in-situ TEM compression experiments of as-prepared and annealed Al submicron pillars. We chose Al as a model fcc metal because a moderate annealing below 200 °C is quite effective to remove most of the FIB-induced defects due to its low melting point (T_m). When a FIB-prepared Al pillar is annealed in TEM, the dislocation loops first exhibit ripening behavior at $0.4T_m$, followed by their annihilation, which is facilitated by the diffusion of thermally activated vacancies as the temperature approaches $0.5T_m$. The in-situ TEM compression directly shows that in the FIB-prepared pillars the first dislocation activity was the expansion and glide of the FIB-induced dislocation loops, resulting in the formation of slip steps on the surface and/or immobilized dislocations through interactions with other dislocations. Upon further deformation dislocation slip localized at these locations, thus forming large slip steps. Contrarily, in the case of annealed pillars, slip steps are distributed more uniformly along the pillar axis. One of the most pronounced differences in the deformation behavior of the two types of pillars is the strain burst distribution; the average strain and the load drop per strain burst event were much larger in the annealed pillar compared to those in the as-prepared one. This clearly demonstrates that the FIB-

induced defect influence the nanoscale deformation of Al pillars by affecting dislocation escape processes.

2. Experimental procedures

2.1. Fabrication of Al pillars using FIB

A (111)-orientated Al single crystal was polished and subsequently etched with a diluted hydrofluoric acid etchant to remove the native surface oxide. Submicron pillars were fabricated onto this polished surface using a FIB (Quanta 3D FEG, FEI, Hillsboro, OR). First, a thin lamella was lifted out from the Al single crystal and then attached onto a supporting Cu grid by using Pt deposition. Then, coarse micropillars with a radius of 10 μm were fabricated using the Ga^+ ion beam with a beam current of 15 nA at 30 kV in an annular milling mode. During the consecutive fine FIB milling of the pillars, the ion beam current was gradually decreased to reduce ion beam damage, and 10 pA were used for the final polishing step. The prepared pillars had a diameter in the range of ~300–450 nm at mid sample height and a length of around 1 μm , giving them an aspect ratio of ~3:1, well suited for microcompression experiments [24]. The aspect ratio of the pillars prepared for in-situ TEM compression, however, was higher than 3:1, specifically around 5:1 in average. This aspect ratio was chosen to secure a sufficient field-of-view of the pillar when viewed from the side in TEM while minimizing the bending or buckling [25]. A scanning electron microscopy (SEM) image of a representative FIB-prepared pillar is shown in Fig. 1a. A slight taper of ~3° is unavoidable using this top down annular milling approach [26,27]. The insert in Fig. 1a depicts the orientation of the Thompson tetrahedron. Since the loading direction is aligned closely parallel to the [111] direction, there are six slip systems with a Schmid factor of ~0.27 on the three inclined slip planes, (a), (b) and (c) according to the Thompson tetrahedron notation.

In order to estimate the thickness of the FIB-induced surface damage layer, the trajectory of Ga^+ ions was simulated using the SRIM software package [28], see Fig. 1b. The parameters employed for the final stage of FIB milling were used for the simulation, which are: an acceleration voltage of 30 kV, a beam current of 10 pA, and an incidence angle of 89.0° [27]. Note that the SRIM code does not take into account any channeling effect of the incoming Ga^+ ions which might arise for a highly symmetric low-index crystal orientation as in the present case.

2.2. Transmission electron microscopy: in-situ annealing, in-situ compression, and X-ray elemental mapping

A TEM (JEM-2100F, JEOL Ltd., Tokyo, Japan) operated at 200 kV was used for in-situ TEM experiments. Using a double tilt heating holder (Gatan 654, Gatan Inc., Pleasanton, CA) the heating experiments were carried out following two different routes. In the first annealing treatment, the FIB-prepared pillars were heated to 120 °C within 10 min, and then the temperature was increased to 200 °C in 10 °C steps after stabilization for 10 min at each temperature. This was intended for the clear observation of microstructural changes with minimizing a sample drift. In the second heating route, an isochronal annealing was conducted at a constant ramping rate of 5 °C/min for a quantitative assessment of the activation energy of the dislocation annihilation process. To document the temporal evolution of FIB-induced dislocation loops, real-time TEM movies were recorded using a charge coupled device camera (ORIU200D, Gatan Inc., Pleasanton, CA) at 25 frames per second. The size of the dislocation loops was determined by manual measurement of the projected area of the loops on the snapshot TEM images with minimizing the errors by applying a Canny edge detection filter to the selected regions of uniform contrast.

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