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Sample size and orientation effects of single crystal aluminum

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ABSTRACT

The mechanical behavior of single crystal Al sample with different sizes in mini-, micro- and nano-scales and different loading directions along [111] and [110] have been systematically investigated. The sample size and orientation effects are examined. The results are compared with previously reported data on other face-centered cubic Ni, Cu, Au and Ag, establishing an interesting trend for sample size effect with respect to stacking fault energy. With higher stacking fault energy and thus higher dislocation mobility such as Al, the sample size dependence would be lower.

1. Introduction

In recent years, the fast development of nano-technology adopts numerous materials in small scale for new devices, including sensors, actuators, and microelectronic devices. Such device sizes have been reduced down to micro- or nano-scales. The understanding of mechanical properties in micro- or nano-scales and the significance of size effects have become an essential task. The size effect, particularly, the idea of "smaller is stronger" has been explored for some nanocrystalline materials [1] and single crystals [2–5]. Deformation of miniaturized single crystals with physical dimensions in the micro to nano range typically exhibit higher flow stresses. The size effect was purposed to be a result of the reduced sample size smaller than the characteristic length for dislocation multiplication, resulting in dislocation starvation [6,7]. An increase in flow stress for reduced micropillar samples under compression was first reported by Uchic et al. [2], using the focused ion beam (FIB) technique and the nano-indenter equipped with a flat diamond tip. By using this method, the micro/nano-scaled measurements for size effects have been researched on various materials such as Au [3,4,6,8], Cu [9–11], Al [7,12], and Ni [2,13–15].

For typical face-centered cubic (FCC) Ag, Au, Cu, Ni and Al with various stacking fault energy values (∼22, ∼45, ∼78, ∼128 and ∼166 mJ/m^2, respectively) [16], it was found that when the sample size was reduced to nano-scale, the mechanical properties such as yield strength and plasticity would both increase [2,5,6,17,18,19]. This phenomenon is related to restriction of dislocation motion. There is no space to induce abundant dislocations. The extended elongation originated from lattice constant extension and nano-twinning are activated. Among all the previous reports, after normalized by shear modulus and Burger's vector [20], the stress increment with decreasing sample size does not seem to exhibit strong dependence with respect to the stacking fault energy. It is also rare to conduct size effect from the same single crystal from bulk down to nano scales, as well as the size effect for different sample orientations. Thus, in this study, the single crystal Al samples, along the [111] and [110] directions, are selected to be loaded in mini-, micro- and nano-scales. The extracted results are discussed and compared with previous findings.

2. Experiment method

Single crystal Al with the rod crystal parallel to [111], measuring 40 mm in height and 10 mm in diameter, was provided by Ames Laboratory, Iowa, USA. The purity is 99.95%. The growth orientation of this Al single crystal was investigated by X-ray diffraction (XRD, SIEMENS D5000) with Cu-K$_\alpha$ radiation. After careful mechanical grinding, polishing and electropolishing to result in lustrous and smooth surfaces, the exact crystalline orientations were determined by electron back scattering diffraction (EBSD) attached on scanning electron microscope (SEM, JSM-6330). We select two orientations, [111] and [110], for our study for the ease of sample extraction. Samples were accordingly sliced from the single crystal and, subsequently, tested in both tension and compression.

Three dimensions of the mechanical testing specimens were

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prepared, namely, the dog-bone shape samples for mini-tension test (in mini scale), cylindrical pillar shape samples for micro-compression (in micro scale), and specially designed dual-gage samples for nano-tension test (in nano scale), as described in details below. Multiple tests were conducted and the representative and average data are presented. Because of the difficulty (and the cost) in preparing micro-scaled tensile samples using our FIB method, we only carry out micropillar compression for the micro-scaled tests. There might be doubts if we can compare the tension results in the mini- and nano-scales with the compression results in micro-scale. The tension/compression asymmetry needs to be explored further. Yu et al. [21] have examined the yield stress σ_y asymmetry in AA1050 commercially pure Al samples with various grain sizes. For fine-grained Al samples with the average grain size of 0.35–0.75 μm, the σ_y asymmetry can be at most 20%, with the higher compression stresses. When the grain size is increased to 2.5 μm, the tension/compression asymmetry is no longer observed. In addition, the tension/compression asymmetry in Al single crystals was found to be very minor [22–24]. We thus consider the minor σ_y asymmetry (even for 0-20%) of the current Al samples for our wide-scaled comparison (for 300–1000% σ_y difference) on the size and orientation effect is logical and acceptable.

The mini-scaled samples, with tensile gauge length of 1.5 mm, width of 0.5 mm and thickness of 0.5 mm, were machined with the tensile direction parallel [111] or [110]. The mini tensile tests were conducted using the MTS Tytron-250 Microforce Testing System (minitester). The strain rate was set to be $2 \times 10^{-3}\,s^{-1}$, within the quasi static state.

The micro-scaled samples were prepared by the dual focused ion beam (FIB, Seiko, SEIKO SMI3050 SE), following the Uchic method [2]. A Ga^+ ion beam operated at 30 keV and 7–12 nA was initially directed perpendicular to the surface of the Al single crystal to mill a crater with a much larger size island located in the center. Then, the much lower currents of 0.7–0.09 nA, in avoidance from appreciable Ga^+ damage [25], were selected to refine the preserved island in the center to get a desired diameter and height of the pillar. The pillar samples have a minor taper angle less than 2°, with 4.5 ± 0.5 μm in height and ~2.0 ± 0.2 μm in diameter. The samples were loaded in uniaxial compression by nano-indentation system MTS Nano-Indenter XP equipped with a flat-end Berkovich indenter under the displacement control mode. The strain rate was set to $7 \times 10^{-3}\,s^{-1}$, also within the quasi static state. The maximum displacement was preset to be about 320 nm.

The nano-scaled samples were also prepared by the FIB process, and also loaded by the MTS Nano-Indenter XP. The configuration of this newly designed nano-tension samples has been reported in our earlier paper [26], as shown in Fig. 1. This sample contains one central beam under compression receiving the indentation load, and two parallel gage sections under tension when the central pillar is pressed downward. The tensile gage section of this sample has a dimension of about 400 ± 100 nm in length, 200 ± 50 nm in width, and 150 ± 50 nm in thickness, maintaining a typical of gage length to width (or thickness) ratio of about 2:1. The central compressive beam is essentially a much larger and stronger pillar, which has a dimensional width over five times of the tensile gage section to ensure effective tension deformation within the tensile gage section with minimum deformation from the central beam. The nano-scaled specimens were loaded at a constant displacement rate mode 0.4 nm/s, or a strain rate about $1 \times 10^{-3}\,s^{-1}$. The maximum displacement was set to be 400 nm.

3. Results and discussion

The Al single crystal sample was characterized by XRD along the cross-sectional plane of the rod and the result is shown in Fig. 2(a). Its single peak clearly reveals the growth direction of the crystal parallel is [111]. This orientation is further confirmed by the EBSD inverse pole figures, as shown in Fig. 2(b).

Results obtained from mini-scaled tension tests are demonstrated in Fig. 3 for the [111] samples. The representative stress-strain curve is shown in Fig. 3(a), from which the Young's modulus of 70 ± 2 GPa and 0.2% offset yield strength of ~60 ± 3 MPa are measured. It is well known that pure Al is among the most isotropic metals, with Poisson ratio of 0.362, very close to the ideal isotropic value of 0.333, plus E_{111}/E_{iso} or E_{110}/E_{iso} above 0.95 (where E_{111}, E_{110} and E_{iso} represent the modulus along the [111], [110], and theoretically calculated isotropic value based on elastic constants) [27]. The tensile elastic strain is only ~0.1% and the tensile failure elongation reaches 8 ± 1%. All the related mini-scaled data are compiled in Table 1. Parallel tests were also performed on the [110] mini samples, and the tensile yield strength is ~40 ± 3 MPa. From the SEM images of the deformed sample, the slip trace can be seen on the gauge section surface, as shown in Fig. 3(b). No twinning was observed from careful surface trace examination. The 35° marked in the figure is the angle between the tensile loading direction, [111], and the slip direction, one of the ⟨110⟩ family such as [110].

Results from the micro-scaled tests are presented in Fig. 4. The representative stress-stain curve for the [111] samples is shown in Fig. 4(a). Since the initial loading would be affected by the surface flatness of the pillar top, the elastic modulus is usually extracted from the slope of the unloading curve, which value gives about calculated by the unloading part slope of the stress-stain curve is about 72 ± 4 GPa. The micro-scaled yield strength is

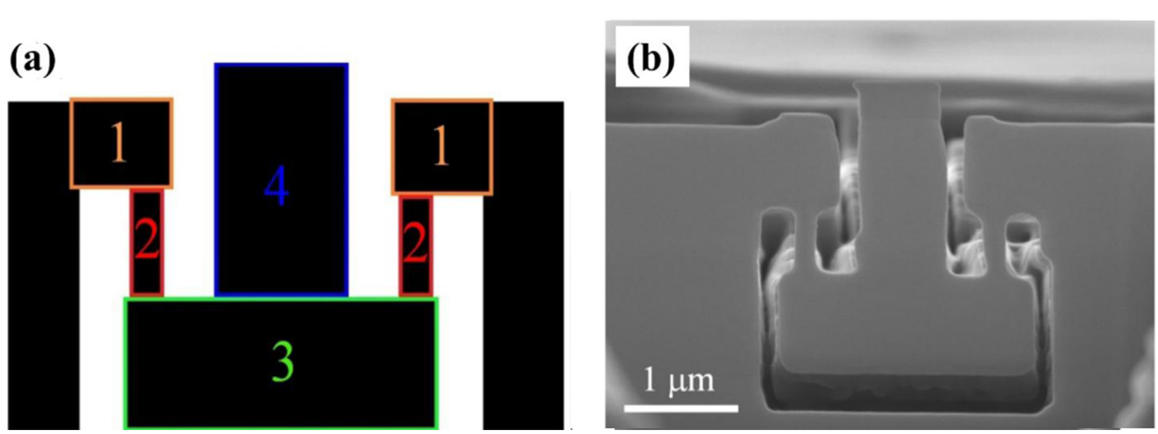

Fig. 1. Schematic drawing (a) and SEM micrograph (b) of the self-designed nano-tension specimen.