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Size effect on tensile creep behavior of micrometer-sized single-crystal gold

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ABSTRACT

In order to clarify the size effect on the tensile creep properties of micrometer-sized single-crystal Au at room temperature, we conducted long-term (up to 14 h) creep experiments for two sets of specimens of approximately 0.5 and 1.5 μm in size, and they were observed with *in situ* field-emission scanning electron microscopy (FESEM). We directly measured the specimen elongation from the FESEM images to eliminate the measurement error in the displacement sensor owing to thermal drift, which ensured accurate creep strain evaluation. On one hand, at high stress range, where the stress σ was close to the yield stress σ_Y (i.e., $\sigma/\sigma_Y \approx 1$), the creep strain continuously increased, and the creep curve consisted of typical transient and steady-state creep regions for both specimens of sizes ~0.5 and ~1.5 μm. On the other hand, at low stress range ($\sigma/\sigma_Y \approx 0.8$), the creep was mainly induced by intermittent strain bursts, which were not observed in bulk metal. Thus, the creep behavior transitioned from continuous to discrete as the applied stress decreased. The smaller specimens required higher stress to reach a strain rate on the order of 10^{-7}–10^{-6} s^{-1}, indicating that the resistance to creep deformation increased with the decrease of specimen size. The "smaller is stronger" trend presented in the long-term creep deformation in the size range of ~0.5–1.5 μm. This finding implies that such small crystals can sustain high stress over a long period and can be used as elements of micro devices.

1. Introduction

The mechanical properties of sub-micrometer and micrometer materials have attracted significant attention because they are different from those of bulk counterparts. The size effects on the plasticity of micrometer-sized metal samples [1–13] have been of particular interest because compression and tensile experiments for metallic crystals revealed that the resistance to plastic deformation increases with the decrease of sample size [2,11,13]. This "smaller is stronger" trend is widely accepted as a universal tendency [1,12]. One of the mechanisms of this size effect in nano- and sub-micrometer range is explained by the dislocation starvation model [2,13]. The mobile dislocations inside a small crystal have a greater probability of becoming annihilated at a free surface than of interacting with one another, which decreases the overall dislocation density. Such processes would lead to a dislocation-starved state requiring very high stresses to nucleate new dislocations from surface sources. For micrometer-sized samples with non-zero dislocation density, the source truncation model [3–10,14] is generally accepted. A small sample mainly has single-arm dislocation sources (SAS), where dislocation arms are pinned at the point inside the crystal and at the point of their termination on the surface. In such a case, shorter sources can exist in smaller samples, and therefore, higher stress is required to operate the dislocation sources. In the micrometer range, thus, smaller crystals generally have higher plastic resistance.

However, the time-dependent deformation or creep of such small crystals is more important than the time-independent plasticity for the long-term use and reliability of micro devices. It is unclear whether small crystals can sustain high stress over a long period. Studies are very limited due to the difficulty in conducting long-term experiments for a small sample, so the creep properties and size effects of such small crystals have not yet been clarified. Ng and Ngan [15] conducted compressive creep experiments for aluminum (Al) single-crystal pillars of diameters in the range of 4–6.3 μm at room temperature, and they reported that the creep rate of micrometer-sized samples was smaller than that of their bulk counterpart. They attributed this to the depletion of dislocations in the small sample. In addition, the samples showed intermittent strain bursts under creep, which were not observed in bulk samples.

Unlike time-independent plastic deformation, however, the "smaller is stronger" trend is not common for creep properties. Typical creep mechanisms include diffusion creep and dislocation creep (power-law creep). Diffusion creep is further classified into lattice diffusion creep (Nabarro–Herring creep [16,17]) and grain-boundary diffusion creep (Coble creep [18]). On one hand, in the diffusion creep mechanism, as grain boundaries are the fast diffusion paths, creep rate increases as the grain size decreases. Choi et al. [19] carried out creep experiments for polycrystalline nickel pillars of diameters 0.6–2.0 μm with a grain size of 30 nm at room temperature. The results indicated much higher creep strain rates in smaller pillars, demonstrating the "smaller is weaker"

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Table 1
Sizes of tensile specimens.

Specimen code	Thickness B, μm	Width W, μm	$D=\sqrt{BW}$, μm	Length L, μm
T05-1	0.51	0.47	0.49	3.01
T05-2	0.65	0.47	0.55	3.04
T05-3	0.44	0.45	0.44	3.01
T05-4	0.35	0.49	0.41	3.05
T10-1	1.22	0.95	1.08	3.01
T15-1	1.64	1.50	1.57	3.54
T15-2	1.73	1.57	1.65	6.00
T15-3	1.43	1.43	1.43	6.06
T15-4	1.29	1.50	1.39	6.12

trend, which was attributed to diffusion-controlled mechanisms such as free-surface-assisted grain-boundary diffusion and grain-boundary sliding. For single-crystal micro samples [20,21], similar trends were observed, due to active surface diffusion. On the other hand, in the dislocation creep mechanism, the "smaller is stronger" trend is expected. However, due to the difficulty in conducting long-term experiments, the size effects on creep properties have not been elucidated in detail. All the aforementioned creep experiments [15,19–22] were conducted for very short periods up to several hundred seconds; creep experiments for hours or more have rarely been conducted for such micrometer-sized crystals. Moreover, almost all the creep experiments were carried out under compression and therefore, the samples had taper and small aspect ratios to avoid buckling instability. In such specimens, stress gradient inevitably occurs and a complex stress field due to the loading probe contact affects the results. Thus, long-term tensile experiments under uniform stress are favorable for the investigation of creep properties and mechanisms.

This study aimed at clarifying the uniaxial tensile creep properties and their size effect in micrometer-sized gold (Au) single crystals at room temperature. The use of single crystals eliminates the grain-boundary-mediated mechanisms. Two sets of tensile specimens approximately 0.5 and 1.5 μm in size were fabricated from a bulk Au single crystal using focused ion beam (FIB). Long-term creep experiments were conducted for up to 14 h under *in situ* field-emission scanning electron microscopy (FESEM) observation.

2. Materials and methods

An Au single crystal of purity 99.999% grown by the Czochralski process [23] was used as a sample. After cutting a cube of side 3 mm with {100} surfaces via wire-electrical discharge machining, dumbbell-type tensile specimens shown in Fig. 1(a) were fabricated using FIB (FEI Company, present Thermo Fisher Scientific Inc. Versa 3D) at an accelerating voltage of 30.0 kV. The surface of the specimen was (001) and the tensile direction was [100]. The length of parallel section L, specimen width W, and thickness B are defined as shown in Fig. 1(a). The dimensions W and B of specimens used in tensile experiments were approximately 0.5, 1.0, and 1.5 μm, designated as Specimens T05, T10, and T15, respectively, and those of specimens used in creep experiments were approximately 0.5 and 1.5 μm, designated as Specimens C05 and C15, respectively. To apply tensile force to the specimen with a gripper as described later, a head section of size 2.0×5.0 μm or 2.5×6.0 μm was prepared at one end of the parallel section. The aspect ratio L/W was set at ~6 for Specimen C05 and ~4 for Specimen C15. Filleted shoulders were made at both ends to avoid stress concentration. Finish processing was conducted at a small beam current of 10 pA to minimize possible FIB-induced damage. Considering the inclination of the FIB-milled surface owing to a beam flare, the beam direction was adjusted so that the thickness became uniform and the contact surface of the head section to the gripper became perpendicular to the loading direction. The sizes of all the specimens are listed in Tables 1 and 2. The side faces of the parallel section were inclined at ~2° due to FIB, and the average width of the upper and lower bases of trapezoidal cross-section was defined as the width W.

A mechanical loading system (Hysitron, Inc. Picoindenter PI-85, rated force: 10 mN, rated displacement: 5 μm), in which the force F is electrostatically applied and the displacement δ is measured using a capacitance sensor, was used for the tensile and creep experiments. A gripper for applying tensile force to the head section of the specimen was fabricated using FIB from a diamond blank probe. A schematic of the loading method is shown in Fig. 1(b).

All the experiments were conducted under *in situ* observation in the vacuum chamber of FESEM (JEOL Ltd. JSM-7001F or Thermo Fisher Scientific Inc. Versa 3D). The pressure was below 9.6×10^{-5} Pa in JSM-7001F and below 1.5×10^{-4} Pa in Versa 3D. The room temperature was 294–297 K, and its variation during the experiment was less than 2 K. The nominal stress σ was defined as F divided by the cross-sectional area of the parallel section BW, and the nominal strain ε was defined as δ divided by the length of the parallel section L.

The tensile experiments were conducted under a closed-loop displacement control, and the strain ε was increased at a rate of 3.3×10^{-3} /s. The creep experiments were performed using a closed-loop load control, and the stress σ was increased at a rate of 30 MPa/s to a target stress value and thereafter maintained constant for the testing duration (6 or 7 h). Subsequently, the specimen was unloaded at the same rate. For some experiments, a second creep experiment was conducted under the same conditions immediately after the first creep experiment. The applied stress and testing time of all the creep experiments are listed in Table 3.

The creep experiments were started at least 12 h after the loading apparatus was powered on in the FESEM chamber and the stability of the apparatus was verified using a method described in the following sentences to avoid the thermal drift of the system. In this method, a stiff column that did not exhibit creep deformation was fabricated near the specimens. The same force as that used in the subsequent creep experiment was applied to the column using the gripper and maintained constant for 1800 s. If the variation of displacement δ was less than 10 nm (equivalent to the displacement rate of 5.6×10^{-3} nm/s and thus the strain rate of 1.9×10^{-6} 1/s for specimens with $L=3$ μm), we considered that the thermal drift was small and started the subsequent creep experiment. After the creep experiment, a similar drift check was performed again to confirm the stability of the system.

The thermal displacement drift in the creep experiments was, however, sometimes not negligible even after the above stabilization. This loading apparatus (PI-85) measures the displacement using a capacitance sensor, which is far away from the gripper [24]. The measured displacement hence includes the thermal drift of machine frame, if present. Therefore, in order to measure the displacement more precisely, the elongation of the gauge section of the specimen was evaluated using *in situ* FESEM images. A location at which displacement was measured is schematically indicated in Fig. 1(b). FESEM images were captured every 1 ks, and the specimen length L_1 was measured at six places and averaged. A fast scan rate (5 frames per second and averaging 16 frames) was used for accurate measurement. The specimen elongation (displacement) was evaluated as $\delta=L_1-L_0$ where L_0 is the initial specimen length