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Specimen size and grain size effects on tensile strength of Ag microwires

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The coupled effects of grain size and specimen size on strength in miniaturized specimens remain largely unknown. Here, the tensile strength of Ag wires of thickness (t) 20–50 μm and grain size (d) 3.5–40.6 μm was found to depend on both t and d. The data also revealed the existence of a strengthening effect depending on shape as the ratio t/d decreases from ~3, in addition to the effects of the absolute specimen and grain size.

Keywords: Plastic deformation; Tensile test; Whiskers; Polycrystalline; Silver

The strength of polycrystalline metals is well known to depend on grain size, via the Hall–Petch effect [1,2], as well as its inverse effect in the nanocrystalline regime [3]. More recently, advances in understanding have also been made concerning the effect of specimen size on strength in the miniaturized size regime spanning from sub-millimeter to sub-micron. First, in the size regime of tens to hundreds of microns, Fleck et al. [4] reported that polycrystalline copper wires exhibited significant size dependence of strength only in torsion but not in tension, and such observations led them to propose that a strong gradient in plastic strain exists in torsion which is accommodated by a high density of geometrically necessary dislocations which would then give rise to strengthening. Other situations in which plastic strain gradients are significant include bending [5] and indentation [6], and strength indeed depends on size in these situations. In uniaxial tensile tests, strong, overall strain gradients are not expected, but unlike Fleck et al. [4], who reported an insignificant size effect on tensile strength on microwires, other researchers who performed tensile testing on polycrystalline metal foils with sub-millimeter thicknesses indeed observed significant dependence of strength and fracture strain on the specimen size [7–11]. In the sub-micron-to-micron regime, dislocation sourcing appears to be an important factor leading to a strong size effect, as evidenced by the observation of strength data approaching the theoretical limit in early tensile tests on single-crystal metal whiskers of micron thicknesses [12,13], as well as in more recent compression or tensile tests on micron-sized single-crystal metal specimens [14–20]. In such micron-sized single crystals, dislocations can escape easily, resulting in a sustained low accumulation state [17,21,22], and their size-dependent [14–22], jerky and stochastic [23] nature of deformation is thought to be due to the need to trigger new dislocation sources along the strain path [24–29]. However, dislocations can be made to remain inside a micron-sized volume by applying an external coating or internal filling of a foreign material [30], or by incorporating a grain boundary within the specimen to form a bicrystal configuration [31]. These micropillars exhibit significant dislocation storage and hence much smoother deformation, yet a significant size effect on strength still exists [30,31]. Also, in the body-centered cubic structure, dislocations cross-slip frequently and may not escape as easily from a small crystal volume as in the face-centered cubic structure [32], but experimentally strong size effects on strength were still seen [19,33].

While the effects of specimen size on deformation have been intensively researched in recent years, as summarized above, in either situation with and without strong overall plastic strain gradients, the coupled effects of grain size and specimen size are much less known. In the polycrystalline situation, the grain size serves as an internal microstructural length scale which is known to affect strength via the well-known Hall–Petch effect, whereas the external specimen size may serve as another length scale affecting strength. Only a few studies have

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been devoted to this topic [9–11,34,35], and a consistent picture has not emerged. As mentioned above, Fleck et al.'s observation of insignificant size effect on tensile tests of polycrystalline microwires [4] does not agree with others who observed significant size effect in uniaxial tests in similar size ranges [21–24], or in the regime of sub-micron specimen sizes and nanometric grain sizes [34,35]. In this letter, we investigate the tensile deformation of Ag microwires with varying grain size and wire diameter, in an attempt to understand some of the effects of these two length scales, in a uniaxial testing situation.

Ag wires with an initial diameter of ~52 μm were annealed in a vacuum furnace at a vacuum level of 10^{-5} torr for 0.5 h under different temperatures from 170 to 750 °C to obtain different grain sizes. In order to determine the grain size of the as-received and the annealed specimens, longitudinal sections along their length were milled out by a focused-ion beam (FIB) in an FEI Quanta 200 Dual Beam FIB system. After milling, the specimens were properly tilted for ion channeling contrast images of the grains, as shown in Figure 1.

The average grain sizes of each condition of wires, shown in Table 1, were determined using the linear interception (LI) method [36]. It is important to note that the wires annealed at 550 and 750 °C contained only a few, sometimes one or two, grains across the wire thickness, so a precise definition of the grain size becomes problematic here. Nevertheless, a rough measure is still needed for discussion purposes, so, in addition to the LI method, the equivalent circle diameter method [36] was also used to calculate their grain sizes. The results show that the difference in the values from both methods is no larger than 1 μm. To obtain different wire diameters while keeping the average grain size constant for each annealing condition, the wires were etched in 65% nitric acid for different times. The resultant wire diameters were ~20, ~30, ~40 and ~50 μm respectively, with a typical variation of ±2 μm.

A nanometric-resolution tensile tester (Agilent Technology T150 UTM) was used for the tensile tests. Each wire sample was glued by an epoxy resin onto a rectangular paper template with a diamond-shaped window cut in it, so that the two ends of the wire were glued onto the paper while its central portion, which was the gauge length, stood freely within the diamond-shaped window. The wire template assembly was then fixed into the grips of the nanotensile tester, and the paper template was then carefully cut into two pieces at the two sides of the diamond-shaped window, freeing out the Ag wire. All the tensile tests are performed with a 20 mm gauge length at a nominal strain rate of $5 \times 10^{-4}\ s^{-1}$ at room temperature. For each condition of Ag wires with a given average grain size and wire diameter, 6–12 specimens were tested. In some of the tests the wire broke at or near the epoxy resin glue, and the corresponding data were excluded from the subsequent analysis, since the stress state near a fixed end is likely to be more complex than within the gauge length. The number of valid tests for each condition is shown in Table 1. Figure 2a and b shows the typical engineering stress (σ)–engineering strain (ε) curves of the Ag wires with various diameters and grain sizes. As usual, the engineering stress/strain was calculated as the applied load/tensile displacement divided by the initial cross-section area/initial length of the tested wire. Figure 2a reveals that, at a given wire diameter of ~40 μm, as the grain size increases within the experimental range, both the tensile elongation and the ultimate tensile strength decrease. Figure 2b shows that, at a constant grain size, the elongation of the wires decreases as their diameters decrease. Moreover, the yield strengths of the as-received wires with different diameters are higher than those of the 750 °C-annealed ones, which is the same as what Figure 2a indicates. For the as-received wires with grain size d = 3.5 μm, the dependence of the yield strength on wire diameter

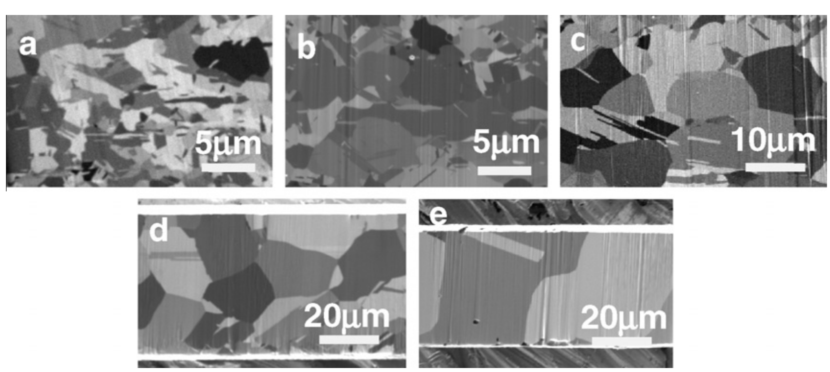

Figure 1. Ion channeling contrast images obtained by ion-optics in the FIB showing the polycrystalline structures in (a) as-received Ag microwires and Ag microwires annealed at (b) 170 °C, (c) 450 °C, (d) 550 °C and (e) 750 °C.

Table 1. Grain sizes of as-received and annealed Ag wires, and number of valid tests for each condition.

Annealing temperature (°C)	As-received	170	450	550	750
Grain size (μm)	3.5 ± 0.5	5.1 ± 0.1	10.6 ± 1.0	21.0 ± 2.6	40.6 ± 6.8
Wire thickness	Number of valid tests				
20 μm	8	6	8	8	7
30 μm	9	10	8	8	10
40 μm	10	7	11	7	10
50 μm	5	Nil	Nil	6	10