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Hall–Petch effect and strain gradient effect in the torsion of thin gold wires

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Gold wires with diameters of 20 and 50 μm were annealed in the temperature range 330–750 °C to obtain different grain sizes. Torsion and tension experiments on these wires confirm that the Hall–Petch effect is stronger than the strain gradient effect in the small grain size region (below 5 μm). With the grain size becomes larger, the strain gradient effect is evident. We find that these two mechanisms are synergistic, and this interaction strongly depends upon the grain size.

Keywords: Torsion test; Hall–Petch effect; Strain gradient; Gold wires; Size effect

The strengthening of crystalline materials is one of the key issues concerned in both engineering application and scientific research. There are many ways by which the flow strength of crystalline materials is increased by restricting dislocation motion, such as boundary strengthening, solid-solution strengthening, particle hardening, strain gradient strengthening, etc. Various types of hardening mechanisms, either alone or in combination, can be present in a crystalline material to produce size effects [1–3]. Among those, the effect of grain size in metals (Hall–Petch effect) [4,5] and strain gradient strengthening [6–10] have attracted considerable attention. As early as in 1950's, Hall [4] and Petch [5] had concluded that the yield strength of metals can be expressed as $\sigma_y \propto d^{-1/2}$, where d is the grain size of metal, as recently reviewed by Armstrong [11]. It is argued that grain boundaries hinder dislocation movement and hence a greater stress is required to yield metals. However, recent studies on nanocrystalline materials show that as the grain size is smaller than 20 nm, the strength of metal would exhibit an inverse Hall–Petch effect, manifesting through softening with decreased grain size due to the activation of grain boundary-assisted deformation mechanism [12,13]. For example, Haque and Saif [14] studied the mechanical behavior of 30–50 nm thick aluminum films by a uniaxial tension device and found that the yield stress decreases with grain size.

In the past two decades, many experiments including nano/micro-indentation [15–17], torsion of micron-dimensioned wires [6,18–21], and bending of micron-dimensioned thin films [22,23], all show that the strength of metallic components undergoing inhomogeneous plastic deformation is inherently size dependent when the characteristic length scale is on the order of microns. Typically, Fleck et al. [6] performed torsion tests on copper wires with different micrometer diameters and found that the normalized torque increases by a factor of three as the wire diameter decreases from 170 to 12 μm, while no size effect was observed in simple tension. This unexpected phenomenon was further confirmed by many other experiments [18–23]. These observations cannot be captured by conventional theories of plasticity, because such theories do not contain intrinsic material length-scales. This type of size effect is generally attributed to geometrically necessary dislocations [6,9] associated with plastic strain gradients, which has motivated the development of various continuum theories of strain gradient plasticity [6–8,10].

Both Hall–Petch effect and strain gradient effect play key roles in the strengthening of metals, and there does exist an interaction between these two effects [6,22]. For example, in the wire torsion tests performed by Fleck et al. [6], the larger diameter wires have the larger grain

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sizes. However, only a few works have been dedicated to study this coupling effect [17,23]. In this paper, we develop a combined method consisting of micro-torsion and micro-tension tests that can be used to test pure gold wires over a range of diameter and grain size. These results allow us to unravel the contribution that each phenomenon adds to the strength, and to investigate the relationship between these two effects.

The polycrystalline gold wires with diameters of 20 and 50 μm were supplied by MKE (Kunshan, China), with 99.99 wt.% purity. They were firstly annealed in a furnace at 330, 360, 390, 420, 450 and 500 °C for 4 h to obtain different grain sizes. The quasi-single crystal wires were obtained by annealing samples at 750 °C for 12 h, as shown in Figure 1 in Supplementary Material. As for the quasi-single crystal wires, the strengthening due to Hall–Petch effect is negligible, and therefore the individual strain gradient effect can be found. In order to determine the grain size, the wire specimens were cut through cross-section by using Focused Ion Beam (FIB) in a Quanta 3D Dual Beam system. Typical images of grains are shown in Figure 1, and the grain size were determined by linear interception method [24].

Torsion experiments of the annealed samples were conducted using a self-made torsion tester. Details of the experiments are given elsewhere [19–21]. In order to decrease the random error, at least three specimens were tested for each Au wire with a certain diameter and grain size. Each stress–strain curve was obtained by averaging the experimental data of these specimens. Due to the difference of radius and length of the sample, the strain rate of each sample is not the same, but all below 0.3×10^{-3}/s. All the tests were carried out at room temperature.

The torsional responses of all samples at different annealing temperatures are shown in Figure 2(a)–(c). Following Fleck et al. [6], the torsion data are displayed in the form of normalized torque Q/a^3 versus surface shear strain κa. Here, Q is the torque, a the wire radius and κ the twist per unit length. The results are presented in a manner that highlights the separate dependences on grain size and wire diameter. Those summarized in Figure 2(a) and (b) are for wires with the same diameter ($2a = 20$ and 50 μm, respectively) but differing grain size (see Table 1), revealing the increase in flow stress with decrease in grain size at a fixed wire diameter. That is, the grain size has a significant impact on the strength of polycrystalline gold wires. Thereafter, the wires with diameters of 20 and 50 μm were annealed at 750 °C for about 12 h to achieve a quasi-single crystal state, as shown in Figure 1 in Supplementary Material. The torsional results in Figure 2(c) are for wires with the quasi-single crystal but two different diameters ($2a = 20$ and 50 μm). The increase in flow stress with decrease in wire diameter is evident. In this case, it is believed that the Hall–Petch effect is negligible in the different diameter wires. Therefore, the elevation of the strengths of quasi-single crystal wires is attributed to strain gradients.

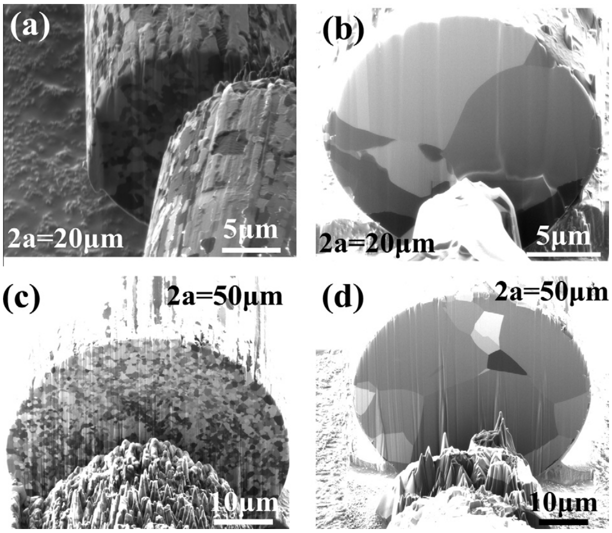

Figure 1. Typical FIB images of the cross-section of polycrystalline gold wires: (a) $2a = 20$ μm, 330 °C; (b) $2a = 20$ μm, 500 °C; (c) $2a = 50$ μm, 330 °C; and (d) $2a = 50$ μm, 550 °C.

In order to figure out the coupling effect of grain size and strain gradient, uniaxial tension test were also performed on gold wires annealed at 330, 360, 390, 420, 450 and 500 °C for 4 h by using a micro-tensile apparatus [19]. The tension results for the gold wires are shown in Figure 3, which have a similar trend with torsion tests in Figure 2. These measurements are convincible since the Hall–Petch relation still holds at the micron scale. The Coplan method [25] was used to determine the yield strength of each sample. The experimental data including annealing temperature, grain size and yield strength are given in Table 1.

The relations between grain size and yield strength in both tension and torsion are given in Figure 4. We can see that grain size has a significant impact on the yield strength of polycrystalline gold wires. For example, as annealing temperature increases from 330 to 500 °C, the value of tensile yield strength drops to nearly one-sixth for $2a = 20$ and 50 μm. These measurements agree well with the Hall–Petch relation, i.e., the yield strength of a polycrystal increases linearly with $d^{-1/2}$.

Figure 4 shows that both grain size and strain gradients contribute to the strengthening of thin metal wires in torsion. Since the Hall–Petch effect is negligible for quasi-single crystal wires, the size dependence of the normalized torque-twist relation in wire torsion (see Figure 2(c)) is attributed to strain gradients. This supports the idea that strain gradient effect plays an increasingly dominant role with decreasing wire diameter. On the other hand, we focus on the wires with $2a = 20$ and 50 μm annealed at 330 °C, respectively. They have a similar grain size, i.e., 0.63 and 0.49 μm, respectively. However, we find that the thinner wire has the lower flow stress (see Figure 2(a) and (b)). This phenomenon can be explained by the interaction between the grain size effect and strain gradient effect [17,23]. Because the grain size here is very small (<2.5 μm), and the Hall–Petch effect is relatively strong in this range. That is, even a small change in grain size will produce a large change in flow strength. So, in this case, the Hall–Petch effect surpasses strain gradient effect. However, when the grain size becomes larger (around 2.5–5 μm), the size effect due to limitations by the grain size reduces, and