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Individual strain gradient effect on torsional strength of electropolished microscale copper wires

Song Guo, Yuming He *, Jian Lei, Zhenkun Li, Dabiao Liu *

Department of Mechanics, Huazhong University of Science and Technology, Wuhan 430074, China

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ABSTRACT

Torsion experiments on copper wires with different diameters but the same grain size are performed to figure out the individual contribution of plastic strain gradient to size effect. The samples are prepared by electropolishing the wires with an initial diameter of 50 μm at room temperature for different time periods. A significant strain gradient effect is confirmed in wire torsion. Tensile results on the same wires verify that the tensile strength is not significantly affected by the diameters. The Fleck-Hutchinson strain gradient plasticity theory is employed to analyze the torsional responses, giving a material length scale of about 3 μm.

1. Introduction

Experiments on materials at small scales show size effect, manifesting the strength is enhanced with decreasing the sample dimension or the stressed volume [1,2]. Generally, the strain gradient strengthening as a consequence of non-uniform deformation, the grain size effect (i.e. the Hall-Petch effect) caused by internal obstacles to dislocation motion, and the size effect due to geometrical limitations of external dimension are primarily the most common mechanical size effects observed in experiments. Examples of these experiments include wire torsion [3–8], foil bending [9,10], micro- and nano-indentation [11–14], and compression or tension of micro- and nano-pillars [15–17], etc. In addition, pioneering works by Hall [18] and Petch [19] on iron and steel materials have led to a relation, referred to Hall-Petch effect, between the yield strength of metals and the grain size. That is $\sigma_y \propto d^{-1/2}$, where d is the grain size. An inverse Hall-Petch effect has also been observed in uniaxial tensile tests on 30-50 nm thick freestanding aluminum films [20]. Recent tensile experiments on thin metal wires show that the Hall-Petch relation still holds at the micron scale [21–23]. In fact, the grain size effect and the strain gradient effect are not independent, they are often observed to superimpose each other [3,23,24]. For example, in the wire torsion performed by Fleck et al. [3], the interaction between the Hall-Petch effect and strain-gradient effect must exist since the larger diameter wires have the larger grain size. To date, only a few studies have been devoted to unraveling such an interaction [10,23,24].

The main aim of this paper is to separate the strain-gradient effect from the Hall-Petch effect by using the experimental data on the torsion and tension of thin copper wires. As indicated by Gan et al. [23], one of challenges to do so is how to prepare wires with the same grain size while varying the diameter. Several methods for controlling grain size or reducing cross-section of thin metal wires have been developed in literature. For example, Yang et al. [22] used the electropolishing technique to reduce the cross-section of copper wires in a commercial electrolyte under a potential of 30 V for several seconds to obtain the samples with the same grain size. Chen and Ngan [21] etched Ag wires with an initial diameter of about 52 μm by nitric acid for different time periods to thin the diameter. More recently, Gan et al. [23] and Liu et al. [25] prepared the quasi-single-crystal Au wires for torsion and tension tests by annealing the wires at 750 °C for about 12 h. They concluded that, in the absence of grain size effect, the elevation of the torsional yield strength is considered to be result from the plastic strain gradient. However, the grain size cannot be controlled precisely by using the heat treatment. So, it is hard to guarantee that the grain size is completely consistent for the samples with different diameters. Compared with the heat treatment, the electropolishing is a better method to keep the microstructure and the grain size unchangeable since it only polishes the wire surface.

In this paper, we devote to figuring out the size effect individually induced by plastic strain gradients in wire torsion. To that end, we performed torsion tests on the copper wires with different diameters but with the same grain size. Besides, different experimental phenomena, e.g., constant tensile strength [22] or elevated tensile strength [21] with decreasing the sample dimension, have been observed in tension tests on thin mental wires. Thus, tension tests on the same samples

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* Corresponding authors.
E-mail addresses: ymhe01@sina.com (Y. He), dbliu@hust.edu.cn (D. Liu).

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are also conducted to study the influence of the sample dimension on the tensile strength. Finally, the Fleck-Hutchinson strain gradient plasticity (SGP) theory [3,26] is employed to analyze the observed size effect.

Here, the electropolishing is used for smoothing and reducing the cross-section of copper wires. The equipment for electropolishing is shown in Fig. 1. A stainless steel frame with the four long rods and the eighteen short ones serves as the anode plate. These rods are arranged symmetrically at the right and left sides of the frame (see Fig. 1). Copper wires of length about 100 mm are winded on these rods from left to right. The anode plate with copper wires is attached to a polytetrafluoroethylene (PTFE) plate with a thickness of about 10 mm. The other side of the PTFE plate is attached to a cathode plate made of stainless steel with the long rod. During electropolishing, the whole frame including both anode plate and cathode plate is immersed in an electrolyte solution, but a section of the long rods in both anode and cathode plates is out of the electrolyte. The electrolyte used here is mixture of alcohol and phosphate (H_3PO_4:C_2H_5O = 9:1 solution). The four long rods in the anode plate are connected to the positive terminal of a DC power supply, while the negative terminal is attached to the long rod of the cathode plate. A constant current of 3.30A passes from the anode, where copper on the surface of the wires is oxidized and dissolved in the electrolyte, to the cathode. At the cathode, a reduction reaction occurs, which produces hydrogen. The as-received polycrystalline copper wires with an initial diameter of about 50 μm were electropolished at room temperature for different time periods to obtain samples with different diameters but the same grain size. Then, the diameter, surface and microstructure of the wires were examined by a Scanning Electron Microscope (SEM), as shown in Fig. 2. One can see that the diameter of the wire is uniform along the sample length. The scatter for each wire diameter is within 0.5 μm. In order to determine the grain size precisely, the electropolished wires were cut through cross-section by Focused Iron Beam (FIB) in a Quanta 3D Dual Beam system. It is found that the average grain size of the wires with different diameters keeps a constant value of about 3.72 μm, implying that the electropolishing does not change the microstructure. Therefore, by testing these wires, one can determine the strain-gradient effect individually. The competition between the strain gradient effect and the Hall-Petch effect has been studied in our other works [23].

In order to study the influence of wire diameter on the tensile strength, the tensile experiments on the same samples were conducted by employing a micro-tensile apparatus [5,25]. All tests were carried out with a 10 mm gauge length at a strain rate below $0.3 \times 10^{-3}/s$ at room temperature. For each diameter of wire, we performed at least five tests. Each response was obtained by averaging the experimental data of these specimens. The true strain-stress curves of the electropolished copper wires are depicted in Fig. 3(a). Since these wire samples have the same grain size, the Hall-Petch effect is eliminated. Contrary to the conclusion that the tensile strength increases with decreasing diameter drawn by Chen and Ngan [21], we find that the yield strength of the wires with different diameters is almost the same. This conclusion coincides with that given by Yang et al. [22]. From Fig. 3(a), one can see that the plastic flow stress slightly elevates with increasing the diameter of wires. Such a trend is similar to that observed by Stölken and Evans [9] on thin annealed foils. However, the reason for the decrease in flow stress with decreasing wire diameter is not well understood. It may result from the existence of defects on the wire surface introduced by the electropolishing. The smaller is the wire diameter, the stronger is the influence of the defects on the flow stress.

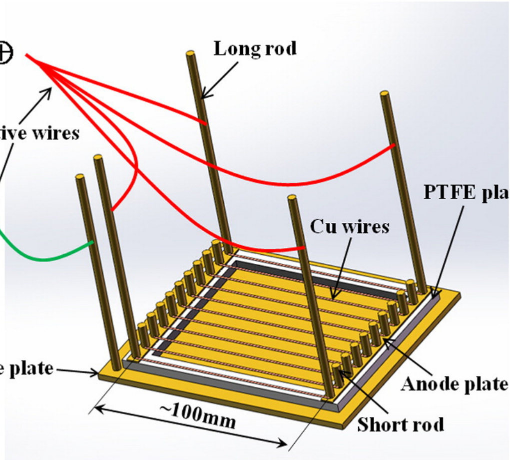

Fig. 1. Schematic diagram of the apparatus for electropolishing the copper wires. During electropolishing, the whole setup is immersed in an electrolyte solution, with the four long rods in the anode plate being connected to the positive terminal of a DC power supply and the long rod in the cathode plate being connected to the negative terminal.

To figure out the strain gradient effect individually, torsion experiments on copper wires with different diameters but the same grain size were also performed by an automated torsion tester [6,25]. Following Fleck et al. [3], we present the torsional data in the form of normalized torque Q/a^3 versus surface shear strain κa. Here, Q denotes the torque, a the wire radius and κ the twist per unit length. The normalized torque-twist curves for the electropolished copper wires are shown in Fig. 3(b). It is found that the normalized torque in both the initial yielding and the plastic range exhibits a significant size effect, which is obviously elevated with decreasing the diameter of wires. For instance, when $\kappa a = 0.01$, the value of Q/a^3 for $2a = 20$μm is approximately 1.9 times that of $2a = 50$μm. This means that the strain-gradient strengthening plays an increasingly dominant role with decreasing wire diameter. As we mentioned above, the tensile flow stress slightly increases as the wire diameter increases (see Fig. 3(a)). Based on this consideration, the strain gradient effect in wire torsion is substantially significant. It is noteworthy that since the Hall-Petch effect is absent, the size effect in both the initial yielding and the plastic range of torsional response is mainly due to the strain-gradient strengthening.

The formalisms as well as the notion of strain gradient plasticity introduced by Fleck and Hutchinson [3,26] are used to account for the size effects observed in the wire torsion. The relation between the normalized torque Q/a^3 and the surface shear strain κa based on the Fleck-Hutchinson SGP theory has been derived by Fleck et al. [3], i.e.

$$\frac{Q}{a^3} = \frac{6\pi\Sigma_0(\kappa a)^N}{N+3}\left\{\left[\frac{1}{3}+\left(\frac{l}{a}\right)^2\right]^{(N+3)/2} - \left(\frac{l}{a}\right)^{N+3}\right\} \tag{1}$$

where Σ_0 denotes the reference stress in the absence of strain gradient, N represents a strain-hardening exponent, and l is a material length scale associated with the material microstructures, such as the mean free path of dislocations [27]. The values of Σ_0 and N are determined by fitting the tensile data for each diameter in Fig. 3(a) with the form

$$\sigma = \Sigma_0 \varepsilon^N \tag{2}$$

After obtaining the values of Σ_0 and N, the material length scale l can be obtained by fitting Eq. (1) to the torsional data in Fig. 3(b). The comparison between the theoretical predictions and the experimental data is depicted in Fig. 4. Here, we use $\Sigma_0 = 290-299$MPa, $N = 0.21-0.28$ and $l = 3$ μm. For the magnitude of the material length scale for copper, Fleck et al. [3] estimated that $l = 2.6-5.1$μm with a mean value of 3.7 μm, and Liu et al. [6] gave $l = 2.05-4.02$μm, which are essentially consistent with our results. It is believed that the material length scale in SGP theory can be considered by itself as an internal variable representing the dislocation cell structure and grain size [27]. A non-fixed material length scale has been adopted elsewhere [6,27–30] which indicates that it depends on the microstructure evolution during deformation. Here, a fixed material length scale is used since we assume that the electropolishing would not change the internal microstructure