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Size effects on uniaxial tension and multiaxial ratcheting of oligo-crystalline stainless steel thin wires

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ABSTRACT

The uniaxial tensile and multiaxial ratcheting behaviors of oligo-crystalline 316L stainless steel thin wires of two diameters (190 and 90 μm) with less than 3.5 grains across diameter were experimentally investigated. The size effect of "thinner is stronger" was observed on the yield strength, though the ultimate tensile strength and elongation were lower in the thinner wire. Under six differently combined axial stress and shear strain cycling conditions, the ratcheting strains were all found to be relatively suppressed in the thinner wire, accompanied by enhanced shear stress flow. Moreover, unlike in bulk materials, the multiaxial ratcheting strain in both thin wires tended to be loading path independent in the cases with the same shear strain amplitude, axial mean stress and stress amplitude. The mechanisms of these size effects were analyzed to be related to the surface dislocation source limitation and strain gradient hardening.

1. Introduction

The size effects on mechanical properties of metallic materials have become one of the key issues in medical implants, electronic and electromechanical systems to accommodate the needs for reducing device dimensions to the micron or sub-micron scale in these fields [1,2]. As the scale of device structure is reduced to the grain size level, only a few grains are left in the cross-section to bear the load and deformation. In such oligo-crystalline materials, the macrostructural dimension usually interferes with the process of plastic deformation, leading to a distinct mechanical behavior that could significantly deviate from those of the polycrystalline and single-crystalline materials [3]. For example, the well-known Hall-Petch effect revealed a primary strain-hardening process in the plastic deformation that the dislocation glide would interact with grain boundaries and form dislocation pile-ups as obstacles for successive slip [4–6]. Macroscopically, this effect described an inverse relationship of $\sigma_y \sim d^{-\frac{1}{2}}$ between bulk yield strength σ_y and average grain size d for various polycrystalline materials [7–9]. A violation of the Hall-Petch relation was found for oligo-crystalline Cu wires when the ratio of wire diameter and grain size, D/d, reduced to 2.4 due to free surface softening [10]. The less grains across the diameter, the higher fraction of soft near-surface grains appeared, where dislocations were either partly blocked by grain boundaries or could escape from the surface without impinging on a grain boundary. Such intervention of small sample dimension to the strain-hardening process resulted in nearly 30% σ_y reduction. Similar weakening phenomenon was also found on Cu-Al rods [11], nanocrystalline copper pillars [12] and Ni films [13,14]. Besides the free surface softening mechanism, statistical size effects on microstructures were also demonstrated to contribute to the weakening behavior in oligo-crystalline materials. For example, in oligo-crystalline Au wires, the high probability of $\langle 1\,0\,0 \rangle_{//\,\text{tensile axis}}$ "soft grains" would lower their tensile strength and ductility [2,15,16]. Though appeared conflicting with the aforementioned size effects, another way of deviation from the Hall-Petch relation was experimentally evidenced by the strengthening on reducing D of coarse-grain Ag wires with D/d less than 3 [17]. Such influence of sample dimension was neither widely recognized for materials nor clearly explained. Only a limited mechanism was proposed that small sample dimension could reduce the dislocation source density, thus delay the large-area plasticity of the material and elevate the yielding strength [14]. Meanwhile, applying loads with high strain gradients, including torsion, bending and micro/nano-indentation, could further complicate the interference of macrostructural dimension with plastic deformation. The strength of sample was found increasing with reducing dimension for various materials [2,18–22]. It was argued that a smaller geometry for deformation would lead to a steeper strain gradient and a higher geometrically necessary dislocation (GND) density, which would enhance the strain-hardening behavior of the material [18,23,24]. Collectively, the small-scale dimension of macrostructure could interfere with the process of plastic deformation in multiple ways and result in non-monotonic size effects, which would present distinct mechanical deviations in different ranges of size depending on the material,

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microstructure and loading type. The many experimental as well as theoretical studies on size effects so far presented like pieces of puzzle. Experimental studies are necessary to reveal the mechanical behavior of oligo-crystalline materials.

Besides the intrinsic complexity of size effects, another challenge to the design of reliable small-size device is the complicate external loading conditions in the real-world application. While many studies have been conducted on small-size materials under uniaxial and simple cyclic loading conditions [9,25–27], no research is available till date, to the authors' knowledge, on the size effects under multiaxial cyclic loading, which is much closer to the complex stress-strain state of the in-service structure [1,28,29]. Plasticity accumulation in non-symmetric cyclic loading, namely ratcheting, is also important for the integrity of small-size structure, since the thin structure with low stiffness prefers tension stress rather than compression to avoid buckling. In terms of bulk materials, the multiaxial ratcheting behaviors depend on the loading path and related non-proportionality. Under non-proportional multiaxial loading, the plastic deformation is featured by slip multiplicity and heterogeneous substructure as a result of the rotation of maximum shear plane in every loading cycle, which leads to the additional, non-proportional hardening behavior [30–32]. As the macrostructural dimensions decrease and materials become oligo-crystalline, the plastic deformation in the process of multiaxial ratcheting could interact with the size effects, which may cause distinct cyclic mechanical behaviors that could be important for the structural integrity assessments of small-size devices. However, because of the technical limitation of multiaxial cyclic testing on small-size samples, the size effects under such complex loading conditions are almost left blank.

In this work, we investigated the multiaxial tension-torsion ratcheting behavior of oligo-crystalline thin wires of 316L austenite stainless steel, the most commercially available coronary stent material [33], using a novel micro tension-torsional fatigue testing apparatus [34]. The samples shared the same grain size while the wire diameters were reduced from 190 to 90 μm, leaving the number of grains across diameter to range from about 3.5 to 1.6. Uniaxial tension tests were conducted first to identify specific size effects without applying deformation gradient. The specific tension-torsion ratcheting behavior, including ratcheting evolution, flow stress and surface slip traces under different loading paths were then acquired and analyzed to understand the size effects on multiaxial ratcheting of oligo-crystalline thin wires. This work will not only help to fulfill the understanding of the basic tension behavior of oligo-crystalline materials but also fill the gap of multiaxial cyclic behaviors for small-size materials, which is important for the structural integrity assessment on small-size device.

2. Materials and experiments

2.1. Sample preparation

The materials investigated in this study are 316L stainless steel wires of initial diameters of 100 and 200 μm. The chemical composition (in weight fraction, %) of the steel wires is as follows: C, 0.03; Ni, 10; Cr, 17; Mn, 1.5; Mo, 2; Si, 0.9; P, S $<$ 0.035; Fe, balance. The as-received cold-drawn thin wires were first annealed in a vacuum atmosphere at 1050 °C for 1 h to uniform microstructure morphology and release residual stress, and then further electropolished in solution of 10% perchloric acid and 90% ethanol (in volume fraction) to remove the cold-drawn traces and defects on the wire surface. Excellent surface quality was acquired for both wires by electropolishing, as shown in Fig. 1. The resultant wire diameters measured by scanning electron microscope (SEM) image were 190 ± 5 and 90 ± 5 μm, respectively. The microstructure of the wires of different diameters were examined by SEM after chemical etching with 15% oxalic acid solution, as shown in Fig. 2. The average grain sizes of different diameter wires are both 55 ± 7 μm determined by the linear interception method. The two kinds of wires in the diameter of 190 and 90 μm are hereafter referred to as the 190-μm wire and 90-μm wire, respectively.

2.2. Tension tests

Uniaxial tension tests on the electropolished thin wires were carried out on a micro-uniaxial fatigue testing machine. The tension tests were under deformation control mode at a rate of 0.01 mm s^{-1}, corresponding to a strain rate of 0.001 s^{-1}. The tensile strain was measured for a gauge length of 10 mm by a non-contact displacement detection system [34]. Repeated tests with fracture sites near the center of gauge length were conducted for comparison.

2.3. Multiaxial ratcheting tests

A novel micro-tension-torsion fatigue testing apparatus was applied for the multiaxial ratcheting tests, as shown in Fig. 3. In brief, the apparatus is composed of an axial load frame driven by a linear motor and a torsional load frame driven by a DC motor. A high precision torque transducer combined with a coaxial air bearing enables accurate acquisition of small torque. Detailed information of this apparatus can be found in literature [34], where it was successfully used to measure the axial-torsional stress-strain response of 316L thin wires of diameters of 100 and 200 μm. Cyclic tests under six loading paths were carried out with asymmetric stress control for axial loading and symmetric shear strain control for torsional loading, at an axial stress rate of 18 MPa s^{-1} and a shear strain rate of 0.003 s^{-1}. The shear strain amplitude of all the cyclic tests was 0.8%. The gauge length of the samples was 6.5 mm. The loading paths and test conditions in the axial stress-shear strain plane (σ-γ plane) are illustrated in Fig. 4 and Table 1, respectively. The precise diameter of each sample was measured by SEM image for stress-strain calculation. The shear stress and shear strain were calculated by

$$\tau = \frac{16T}{\pi t^3} \tag{1}$$

$$\gamma = \frac{\theta D}{2L} \tag{2}$$

where τ is shear stress, γ is shear strain, T is torque, D is wire diameter and L is gauge length. The ratcheting strain is defined as

$$\varepsilon_r = \frac{\varepsilon_{max} + \varepsilon_{min}}{2} \tag{3}$$

where ε_{max} and ε_{min} are the maximum and minimum axial strain in each cycle.

The surface morphologies of the ratcheted 190 μm-diameter wire samples were observed by SEM for various loading paths. The surface microstructures resulted from multiaxial ratcheting were further examined by the electron backscatter diffraction (EBSD) for 90 and 190 μm-diameter wires under certain path (Path E). As the wire samples were electro-polished before mechanical testing, no further metallographic preparation was necessary for obtaining EBSD pattern [33]. The EBSD measurements were performed using an FEI Nova 200 NanoLab SEM/FIB equipped with an AztecHKL EBSD system (Oxford instrument). To gain areas with good pattern quality to the largest extent, the wires were mounted with their axis tilting 30° to the incident beam. A step size of 1.5 μm was used for the measurements. Data acquisition and processing were carried out using the HKL Channel 5 software.

3. Results

3.1. Uniaxial tensile behavior

Fig. 5 shows the uniaxial tension true stress-strain curves of oligo-crystalline 316L wires of two diameters. As wire diameter reduces from 190 to 90 μm, with the number of grains across diameter decreasing from 3.5 to 1.6, the average yield strength increases from ∼240 to