Fatigue 2010

Cyclic loading behavior of micro-sized polycrystalline copper wires

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Received 27 February 2010; revised 11 March 2010; accepted 15 March 2010

Abstract

Micro-sized polycrystalline copper wires of diameters ranging from 5 µm to 36 µm were cyclically loaded under stress-control in many steps with increasing the applied stress amplitude after a saturation state in plastic strain is reached. It is observed that the thicker wires show smaller plastic strain at saturation as well as smaller creep strain compared to the thinner wires. The results were discussed combined with the monotonic tensile results of these micro-sized wires.

Keywords: Cyclic loading; copper; size effect; tensile test; stress–strain

1. Introduction

It is known that the mechanical properties of metals are influenced not only by their internal length scales such as grain size, defect density, obstacle spacing etc., but also by the external length scales \cite{1,2}, e.g. the plastically deformed volume \cite{3–6} and the thickness of specimens tested \cite{7–13}. While most of the size effects mentioned above are observed in experiments performed in a monotonic loading mode, studies on the cyclic loading behavior, especially the influence of specimen thickness on the miniaturized samples, are rarely reported \cite{7,14,15}. One major challenge is that performing such experiments on micro-sized specimens is not straightforward due to the lack of suitable testing equipment. The usual instrumentations for testing large specimens are generally difficult or impossible to be directly applied for miniaturized samples owing to their insufficient accuracy to measure the small loads and displacements. Motivated by what is mentioned above micro-sized polycrystalline Cu wires with different diameters were cyclically loaded under stress control in this work. The cyclic loading results are discussed combined with the monotonic tensile behavior of these micro-sized wires.

2. Experimental details

For tensile and cyclic loading testing a recently developed fiber tensile module, which has been described in detail in \cite{11,14} was used. Commercial Cu wires of high purity (>99.998%) with diameter of 50 µm from W.C.

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doi:10.1016/j.proeng.2010.03.100
Heraeus GmbH were annealed at 400 °C for 2 hours in a vacuum furnace (6×10^{-6} mbar). Cross-section of the annealed wire along the drawing direction was prepared by means of a focused ion beam (FIB) workstation (LEO XB 1540) to reveal the microstructure (Fig. 1a). An average grain size of 10.1 ± 2.8 µm in the longitudinal direction and 10.7 ± 3.4 µm in the transverse direction was determined by employing the linear intercept method. By selective electropolishing (with a H2SO4 solution under a potential of 10 volt) the 50 µm thick annealed wires were partly thinned to various thicknesses ranging from 5 µm to 38 µm for tensile and cyclic loading tests. The length of the thinner part of the wire is about 16 to 20 times of its thickness. As an example, a scanning electron microscope (SEM) micrograph of the thinner part of a wire, 20 µm thick and 400 µm long, is shown in Fig. 1b.

All tests were performed in situ within an optical microscope (OM) or a SEM at room temperature. The tensile strain rate was fixed at 2×10^{-3} s^{-1}. Engineering stress (σ) and strain (ε) values are calculated from the as-recorded load–displacement curves, with the stiffness of the machine accounted for. Cyclic loading tests were performed under stress control with a constant load ratio, \( R = \frac{\sigma_{\text{max}}}{\sigma_{\text{min}}} = 0 \), where \( \sigma_{\text{max}} \) and \( \sigma_{\text{min}} \) are the maximum and minimum applied stresses in a load cycle. A multiple step loading procedure is used, as schematically shown in Fig. 2. The applied stress amplitude, \( \sigma_a = \frac{\sigma_{\text{max}} - \sigma_{\text{min}}}{2} \), is increased to the next step as a saturation state in plastic strain is observed from the load–displacement curve. The strain rate for loading and unloading was set always the same as the monotonic tensile test, which means the frequency is not constant for all the load steps; it decreases with increasing applied stress amplitude. In this work the frequencies range from 0.012 to 0.08 Hz, as measured from the as-recorded load–time curves.

Fig. 1. (a) FIB image of the cross-section of a 50 µm thick wire annealed at 400°C for 2 hours; (b) a SEM micrograph of the annealed wire after selective electropolishing, with a thickness of 20 µm and a length of 400 µm for the thinner part.

Fig. 2. Stress-controlled cyclic loading test with a multiple step loading procedure. The load ratio R is set to be zero and the applied stress amplitude \( \sigma_a \) is increased to the next step as a saturation state in plastic strain is observed from the real-time load-displacement curve.

3. Results

3.1. Tensile results

Selected engineering stress–strain curves are shown in Fig. 3a. The 0.2% off-set yield stress as well as the flow stress at a plastic strain of 2% and 7%, respectively, are plotted in Fig. 3b with respect to the wire diameter. A decrease in the flow stress with decreasing wire thickness, especially at higher plastic strain range, is observed.

3.2. Fatigue results

The as-recorded load vs. number of load cycles curve of a 36 µm thick wire is shown in Fig. 4a, while the corresponding elongation of the wire vs. number of load cycles curve is shown in Fig. 4b. At the same load step the elongation of the wire increases strongly from the beginning of the cyclic loading (\( E_1 \), the maximum elongation for
the first load cycle); with ongoing cyclic loading this increase in elongation progressively diminishes and after a certain number of load cycles a quasi-steady state of elongation ($E_S$), referred as saturation in the plastic strain, is reached (e.g. see inset in Fig. 4b). The range of elongation ($E_S - E_1$) is used to calculate the creep strain, $\varepsilon_C = (E_S - E_1)/L$ ($L$ is the gauge length), while the elongation at saturation ($E_S$) is used to calculate the plastic strain at saturation, $\varepsilon_S = E_S/L$, and to determine the cyclic stress–strain curve (CSSC, Fig. 5a). The creep strain as a function of the applied stress amplitude for the wires with different thickness is shown in Fig. 5b. It can be seen that the onset of plasticity is not significantly influenced by the wire thickness; however in the hardening regime, the thicker wires (28 and 36 µm) show smaller plastic strain at saturation as well as smaller creep strain compared to the thinner wires (15, 7 and 5µm).

**Fig. 3.** (a) Selected engineering stress–strain curves; (b) the yield stress as well as the flow stresses at plastic strains of 2% and 7% as a function of the wire thickness $D$. A decrease in the flow stress with decreasing wire thickness can be observed, and this effect is enhanced at higher plastic strains.

**Fig. 4.** (a) As-recorded load–number of load cycles curve; (b) the corresponding elongation–number of load cycles curve of a 36 µm thick wire. The inset in (b) shows that the elongation of the wire increases strongly at the beginning of the cyclic loading; with ongoing cyclic loading this increase progressively diminishes and a quasi-steady state is reached after a certain number of load cycles.
Fig. 5. (a) Cyclic stress–strain curves and (b) applied stress amplitude vs. creep strain curves for the wires with different thickness. Smaller plastic strain at saturation $\varepsilon_S$ and creep strain $\varepsilon_C$ for the thicker 28 µm and 36 µm wires compared to the thinner ones can be observed.

4. Discussion

As shown in Fig. 3, a significant effect of the wire thickness on the tensile properties of the micro-sized polycrystalline Cu wires was observed: the flow stress decreases with decreasing wire thickness. The results are similar to what is observed in tensile experiments on materials with larger dimensions such as Cu, Al and Ni [16–20]. Miyazaki et al. [17] found that the flow stress of Cu and Al plates decreases with decreasing specimen thickness when the ratio of specimen thickness to grain size is smaller than a critical value. The reason is that the constraining force is lower in the specimen surface than in the interior region of the specimen. Keller et al. [18,19] pointed out that the reduced flow stress of the polycrystalline Ni samples with decreasing specimen thickness is due to the decrease in the intragranular backstress, in other works [16,21,22] it is argued that the grains located in the surface layer are less restricted than the inner grains.

Fig. 5 shows that both the plastic strain at saturation and the creep strain are higher for the thinner wires. Similarly, Weiss et al. [7] studied the fatigue (under tension–tension loading with load ratio $R = 0$) behavior of heat-treated polycrystalline Cu foils with thickness of 250 µm and 125 µm, and they found that the plastic strain at saturation as well as the creep strain increases with decreasing the foil thickness. In their view this is attributed to the less dislocation activity during cyclic loading in the thinner foil due to the difference in the stress state; at the same time they also pointed out that the ratio of grain size to foil thickness have to be taken into account. We think that the fatigue response of the micro-sized wires is mainly governed by the wire thickness (or the ratio of wire thickness to grain size). The lower plastic strain at saturation for the thicker wires is attributed to, on the one hand, the reduction in the flow stress with decreasing wire thickness (as discussed above), thus the lower plastic strain for the same tensile stress; on the other hand, the shorter creep strain at each applied stress amplitude (as shown in Fig. 5b). As it is known, the movement of nucleated dislocations is affected differently by free surfaces and grain boundaries. Owing to the acting image forces, dislocations near the free surface can easily escape; whereas in the interior of specimens the dislocations are blocked by the grain boundaries. With decreasing the wire thickness, a near-bamboo microstructure of the wires is approached, i.e. almost all the grains are located on the surface. Therefore, the influence of surface-dislocation interaction in thinner wires is enhanced: first and foremost, this leads to a lower dislocation density (which also explains the lower flow stress in thinner wires observed in tension); besides, it makes it difficult to reach a stabilized state between the applied stress amplitude and plastic strain during cyclic loading, which could be responsible for the longer creep strains in thinner wires.

It should be noted that in monotonic tensile, compression or bending tests on miniaturized single crystals (with thickness typically smaller than 10 µm), the strength normally increases with decreasing the specimen size [2,23–
26]. This is opposite to what is observed on the polycrystalline Cu wires investigated in this work; here the flow stress decreases with the wire thickness in the range of 5 µm to 38 µm. Therefore, further investigations are needed to reveal the size effect on the cyclic loading behavior in case that the dimension of the miniaturized specimens (either polycrystalline or single-crystalline) becomes even smaller.

5. Summary

Using a fiber tensile module stress-controlled fatigue as well as monotonic tensile tests were performed on micro-sized polycrystalline copper wires with diameters ranging from 5 µm to 38 µm. The results show that the onset of plasticity is not significantly influenced by the wire thickness; however, in the hardening regime, a pronounced effect of the wire thickness on both the tensile property and the fatigue response is observed. The tensile stress decreases with decreasing wire thickness. The thicker wires show smaller plastic strain as well as creep strain during cyclic loading at each stress amplitude compared to the thinner ones. The enhanced influence of free surface-dislocation interaction could be responsible for the tensile and cyclic deformation behavior observed in this work.

Acknowledgements

Financial support within the research activities of the K2 Competence Centre on “Integrated Research in Materials, Processing and Product Engineering”, operated by the Materials Center Leoben Forschung GmbH under the frame of the Austrian COMET Competence Centre Program, is gratefully acknowledged. The authors thank Prof. Reihard Pippan of Montanuniversität Leoben, for discussion and evaluation of this paper.

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