Plastic Flow Characteristics of Micro Single-Crystal Copper

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Abstract. In order to study the plastic flow characteristics of micro single-crystal Cu, an in-situ uniaxial loading test was carried out. The slip direction of the test is determined by the Schmidt factor, which is at 1μm~4μm. Within the micropillar size range, the sample has an obvious size effect. This is essentially due to the continuous proliferation of dislocations in the micropillar flowing out of the surface. The smaller the size, the more difficult it is to store dislocations, and the easier dislocations flow out. Therefore, the yield stress significantly increases with the increment of sample diameter. This study provides guidance for the reliability evaluation of microscale components based on the single-crystal Cu.

1. Introduction
With the development of microelectronic devices (such as semiconductor chips) represented by MEMS[1], optoelectronic devices, nanoscience, and technology all put forward urgent demands for the mechanical properties and structural optimization of materials at the micro-nano scale. Under the trend of MEMS and flexible electronic materials becoming the development direction of intelligent electronic industry in the future, the properties of metallic materials applied on a micro-nano scale have been highly concerned in the field of scientific research.

In recent years, it has been found that the mechanical properties of micro-and-nano-structures are closely related to their sizes[2–5]. Brenner[6-7] investigated the tensile strength of Fe, Cu, and Ag single-crystal whiskers with a diameter of 1-20μm. It was found that the smaller the whisker diameter, the higher the strength, and the closer to the theoretical strength. In addition, the dispersion of strength data increases with the decrease in sample size. Dunstan and Bushby et al.[8] conducted an in-depth analysis and discussion on the size effect of the strength of small-size samples and pointed out that the strength was inversely proportional to the characteristic size of the sample. Therefore, to clearly clarify the plastic deformation behavior of microscale single-crystal metals, it is critically important to perform in situ mechanical experiments in electron microscopes.

The purpose of this study is to investigate the deformation behavior of single crystal Cu pillars with different sizes under uniaxial compression. By analyzing corresponding load-displacement curves and stress-strain curves, the size effect of plastic deformation properties of micro-single-crystal coppers was clarified.

2. Experimental details

2.1. FIB sample preparation process
In this study, polycrystalline pure copper samples and base metal samples of 5mm×5mm×3mm were annealed at 1100 °C in a vacuum (10⁻³Pa) for 24h, the grain size of the samples increased significantly.
The Vickers hardness (HV) of the samples before and after treatment was measured, and the Vickers hardness decreased from 330 kgf / mm² to 218 kgf / mm². The surface was polished with different gradient sandpapers of 800 mesh, 1200 mesh, and 2000 mesh, respectively, and then placed on a polishing machine for fine polishing.

The microstructure of the pure copper sample was characterized by an electron backscatter diffraction system (EBSD), as shown in Figure 1. The EBSD analysis indicates that the microstructure of the base metal is condensed into a large single-crystal grain by annealing treatment, and the normal direction of the crystal plane is <111>. The region in the diagram, which is the largest grain size in the pure copper sample, is used as the target region for preparing micropillar samples.

Figure 1. The grain orientation of the sample in IPF Z direction

The micropillars were fabricated in Zeiss FIB / SEM. The bulk sample was placed in a vacuum state of SEM, and the morphology of the upper surface was determined by SEM. In order to ensure that the milling damage of copper micropillar samples is as neat as possible, the cutting process of micron-sized copper pillars adopts the ring delamination strategy. Under the voltage of 30 kV, the ring is cut by a 30 nA large beam, keeping the center of the ring unchanged, by gradually reducing the size of the ring and the beam current to 100 pA, the copper micropillar with pre-determined sizes are finally obtained cut. The surrounding area of the micropillar should be maintained at a certain depth, so as to reserve the observation field space for the diamond probe during the loading test.

In this study, three different sizes of copper micropillars were prepared: 1μm, 2μm, and 4μm. The average diameter of the upper and lower bottoms of the micropillar is \( \phi \), and the height is \( h \). The height-diameter ratio of the micropillar sample \( \phi : h \) remains approximately 1:3. The loading distance \( L_p \) is about 20% of the micropillar height, that is, the strain is set to 0.2. As far as possible to ensure the consistency of the sample and the size stability of the bottom diameter, so as to avoid the influence of micropillar taper on the experimental process. Table 1 lists the geometric dimensions of the micropillar samples in this study.

| Sample number | diameter \( \phi \) (μm) | girder depth \( h \) (μm) | Loading distance \( L_p \) (μm) |
|---------------|---------------------|-----------------|---------------------|
| 1             | 1.035               | 3.480           | 0.500               |
| 2             | 1.211               | 3.751           | 0.700               |
| 3             | 2.018               | 5.203           | 1.00                |
| 4             | 2.169               | 7.103           | 1.400               |
| 5             | 3.972               | 10.920          | 2.000               |
| 6             | 4.048               | 10.580          | 2.000               |

Figure 2 shows SEM images of Cu micropillars of 1μm, 2μm, and 4μm, respectively. No surface voids or pores were observed in the micropillars, and the radial direction of the micropillars is perpendicular to the substrate surface. Since the Cu micropillar was processed by FIB, a certain taper
was formed. Therefore, the taper of the micropillar was controlled within 5° through FIB refining operation.

![Sample images](image)

**Figure 2. Diameters are (a)1μm (b)2μm (c)4μm Cu micropillar specimens**

2.2. In-situ loading process

The compression of Cu micropillars was performed in an in-situ nanoindentation instrument. In this experiment, Bruker's Pi-88 in-situ nanoindentation instrument was used to accurately locate micropillars by the three-dimensional stepping motion control system. The diamond tip of the probe has a circular platform, and the top of the tip can be approximately regarded as a circular plane with a diameter of about 5 μm. It can completely cover the upper and lower surfaces of the micropillar sample, and avoid the influence of local deformation of the contact area on the loading process of the sample. In this study, the displacement control mode was applied to the diamond indenter with a strain rate of $10^{-2}s^{-1}$. The conductive diamond pressure head is used to load through the electrostatic force in the sensor, and the capacitance is used to record time, load, and displacement.

3. Results and discussion

3.1. In situ observation of Cu micropillars during compression experiments

Figure 3 shows the in-situ loading process of sample No. 3. Fig. 3(a) shows the beginning of the compression experiments, and Fig. 3(b) stage is the elastic deformation stage. In Fig. 3(b), the height of the pillar is slightly reduced, and there is no slip band at this time. As shown in Fig. 3 (c), the slip band of the specimen is activated, and the in-situ loading process of the specimen also enters the plastic deformation stage. With the influence of the first slip system, the plastic flow began to occur near the slip band, and the morphology of the slip band became deeper and thicker (Fig. 3 (d)). With the further increment of compression displacement, the slip phenomenon occurs, and the longitudinal compression and transverse expansion process occur (Figs. 3 (e, f)).

![In situ loading process](image)

**Figure 3. In situ loading process of No. 3 Cu micropillar**
Figure 4 shows the displacement-load curve of specimen No. 3 during in-situ loading deformation. Points (a)–(f) marked in the graph are the corresponding phase of the in-situ image in Figure 3, respectively. (a)–(b) stage is the elastic deformation stage. With the increase of displacement, the load rises steadily until reaching point (b), and the load reaches the maximum. At this time, the yield strength point of specimen No. 3 can be obtained. After entering the plastic deformation stage of the sample, and the first large amplitude of the zero load phenomenon at point (c), this part will be discussed in detail later. Point (c) also marks the start of the first slip system of the Cu micropillar. In the (c)–(f) stage, the load decreases sharply several times and even becomes zero. The corresponding zero load condition is accompanied by the thickening and deepening of the slip band of the specimen. In addition, in the (c)–(f) stage, with the increase of displacement, the load also has a slowly increasing trend, which is corresponding to the change process of longitudinal compression and transverse expansion of the sample during in-situ loading.

3.2. Stress-strain curves of Cu micropillar under in-situ loading

Figure 5 shows the true stress-strain curves of Cu micropillars of different sizes. The diameter $\phi$ of the specimens Nos. 1 and 2 is about 1 μm, corresponding to the black curves; The diameter $\phi$ of Nos. 3 and 4 are about 2 μm, corresponding to the red curves; The diameter $\phi$ of Nos. 5 and 6 is about 4 μm, corresponding to the blue curves. It can be found that all tested specimens completed the elastic deformation stage before the critical strain of 0.02. In the subsequent plastic deformation stage, the stress showed a slow and continuous upward trend. In the elastic deformation stage, the slope of the true stress-strain curve of the specimen is consistent, that is, the elastic modulus of the specimen will not be affected by the size of the Cu micropillar specimen. In addition, in the plastic deformation process, the stress changes with the increase of the strain, which is often accompanied by the strain jump[9].

For micro- and nanoscale experiments, the yield strength is usually taken as the measured strain value between 2% and 10% (Uchic, etc. [10]) to avoid the influence of initial micro plasticity, dislocation, and tip fillet (Byer, etc. [11]), especially in micropillars with a small diameter. Considering that strain burst will occur in the stress-strain curve within a certain range, the corresponding average stress value between 6% and 8% strain value is selected as the yield strength of plastic flow $\sigma_y$ of the Cu micropillar in this study.
Figure 5. True stress-strain curves of all tested Cu micropillars during in-situ loading

As shown in Fig. 6, the relationship between the yield strength $\sigma_y$ and the diameter of single-crystal Cu micropillar $\varphi$, where the power-law index $\alpha$ is the linear fitting slope of the curve. The stress of Cu micropillars with different micropillar diameters obviously forms high, medium, and low grades, which means that the yield stress of all tested samples has an obvious size effect.

Figure 6. Relation between the yield strength and diameter of Cu micropillar

4. Conclusion
In this study, the in-situ mechanical tests of Cu single crystal micropillars of different sizes were performed, and the following results were obtained:

1. The sample is taken from the same Cu single-crystal base metal. The slip bands are consistent and independent of the size of the micropillar. In the size range (1μm ~ 4μm) of the Cu micropillar selected in this study, the sample has an obvious size effect.

2. According to the dislocation starvation theory and the dislocation source truncation theory, it can be confirmed that the plastic flow of a single crystal Cu micropillar is essentially a process of the continuous multiplication of dislocations inside the micropillar and the flow out of the surface, which has a dominant role for the size effect of plastic properties of micro-single-crystal copper.

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