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Anisotropic mechanical characterization of Cu single crystals and thin films

Satoshi Shimizu, Nobuo Kojima, and Jiping Ye

Research Department, NISSAN ARC, LTD., 1 Natsushima-cho, Yokosuka 237-0061, Japan

1. Introduction

Copper thin films are widely used in multilevel interconnects for reducing RC of ultra-large-scale integrated circuits. Anisotropic mechanical characterizations of Cu materials and thin films are indispensable for improving the mechanical strength of multilevel interconnect structures. A convenient nanoindentation measurement technique based on Oliver's method [1] has been widely used so far for evaluating the hardness and reduced modulus of thin films. This convenient method, however, does not estimate elastic properties and yield stress due to initial plastic deformation of films, although these properties are well known to be important for optimizing Cu film growth process conditions. In our recent studies [2, 3], we developed a spherical indentation method, based on the Hertz contact theory [4, 5] and Tresca yield criterion [6], for estimating the yield stress of thin films quantitatively on a nanometer scale.

In this work, a further study was made to apply this spherical nanoindentation method to different Cu single crystals and Cu films of different crystal orientations for the purpose of characterizing their anisotropic mechanical properties such as modulus and yield stress relative to their crystal structures and microstructures.

2. Experimental procedure

Nanoindentation tests were performed using an indentation instrument with the capability of measuring load-depth curves with 1-nN load resolution and 0.2-nm displacement control. Cu (100) and Cu (111) single crystals and 200-nm -thick Cu thin films fabricated by spattering on Si (100) wafers were examined. The crystal orientations of the Cu thin films were investigated by X-ray diffraction analysis. A spherical diamond tip with a radius of 1 μm was used in all of the spherical nanoindentation tests.

3. Results and discussion

Figure 1 shows the indentation load (*P*) vs. penetration depth (*h*) curves for the Cu (100) and Cu (111) single crystals. The curves at a low maximum indentation load were overlapped under elastic contact induced by spherical indentation. However, the unloading curves did not overlap the loading curves when the indentation load was increased into the range generating plastic deformation in the crystals. The hysteresis loop energy (*Ur*), which is defined as the energy enclosed within the indentation loading-unloading path, is the irreversible energy consumption associated with the plastic deformation. As shown in Fig. 2, the Cu (111) plane exhibited initial plastic deformation in the relationship between the hysteresis loop energy and the maximum contact pressure (P_m) at 0.94 GPa and the Cu (100) plane displayed it at 1.78 GPa. Under the Tresca yield criterion and the Hertz contact theory, the Cu (111) plane was estimated to have reduced modulus of E_r = 99 GPa and yield stress of σ_y = 554 MPa, both of which were smaller than the values of E_r = 68 GPa and σ_y = 291 MPa estimated for the Cu (100) plane. This anisotropic nature of these mechanical properties agreed with the metallurgical considerations of an active f.c.c. slip system. As illustrated in Fig. 3, in the spherical indentation process at a very low penetration depth, the direction of maximum shear stress was oriented at a 45-degree angle with respect to the direction of the applied load. The active slip direction for the Cu (100) and Cu (111) planes was oriented at an angle of 45 degrees and 35 degrees, respectively. This anisotropic nature of yield stress originated from the gap between the active slip direction and the maximum shear stress direction. It was confirmed that the Cu single crystals possessed anisotropic reduced modulus and shearing yield stress due to the different crystallographic orientations present in the film plane.

To investigate the applicability of this method to the materials of multilevel interconnect structures, a 200-nm-thick copper thin film was examined. Figure 4 shows the *P-h* curves and the plots of *Ur* and P_m. Elastic behavior was exhibited under P_{max}=5.3 μN and initial plastic deformation occurred at 1.21 GPa. This copper film displayed reduced modulus of E_r = 66.5 GPa and yield stress of σ_y = 375 MPa. This reduced modulus result is nearly the same as the value obtained for the Cu (100) plane, and yield stress was found to be between the Cu (100) and Cu (111) planes.

Figure 5 shows the pole figures of X-ray diffraction for the copper thin film having the (100) and (111) orientation. The sharpness of the center spot in the pole figure indicates that this film had a very small grain size. The reduced mod -ulus was dominated by the (100) plane in the most compliant direction. Since the yield stress was reinforced by the (111) plane, it was higher than that of the (100) plane.

4. Conclusions

A spherical nanoindentation method was applied to evaluate the reduced modulus and yield stress of Cu single crystals and thin films. The anisotropic mechanical properties of thin Cu films were characterized and substantiated using Cu (111) and Cu (100) single crystals. It was found that the modulus and yield stress of Cu thin films are strongly dependent on their crystal structures and orientations. This method is a powerful technique for evaluating yield strength in an area several tens of nanometers in size in order to optimize the Cu film growth process with respect to the material microstructure.

References

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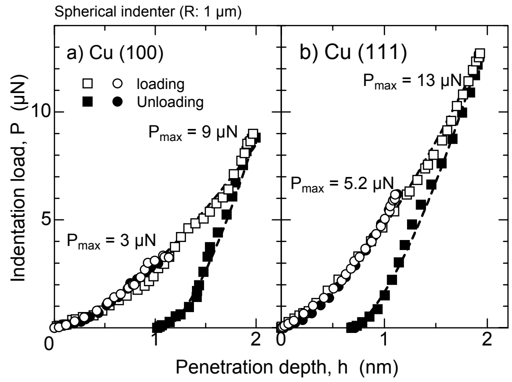

Fig. 1 The relationship between the indentation load (P) and the penetration depth (h) for single crystal coppers.

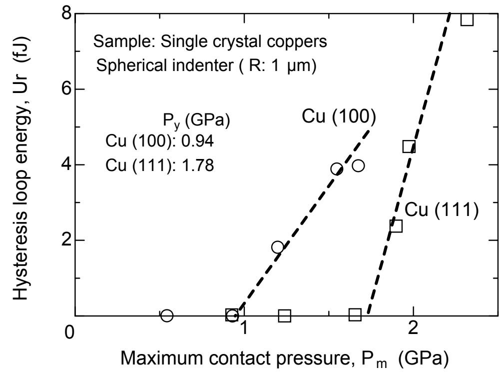

Fig. 2 The relationship between the hysteresis loop energy (Ur) and the maximum contact pressure (P_m).

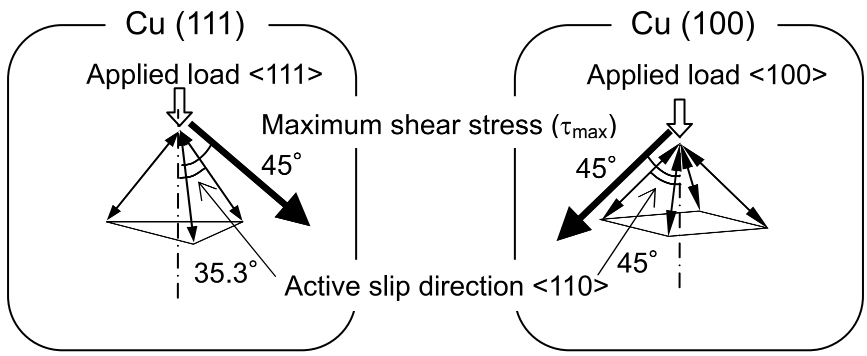

Fig. 3 Anisotropic nature of mechanical properties of single crystal coppers.

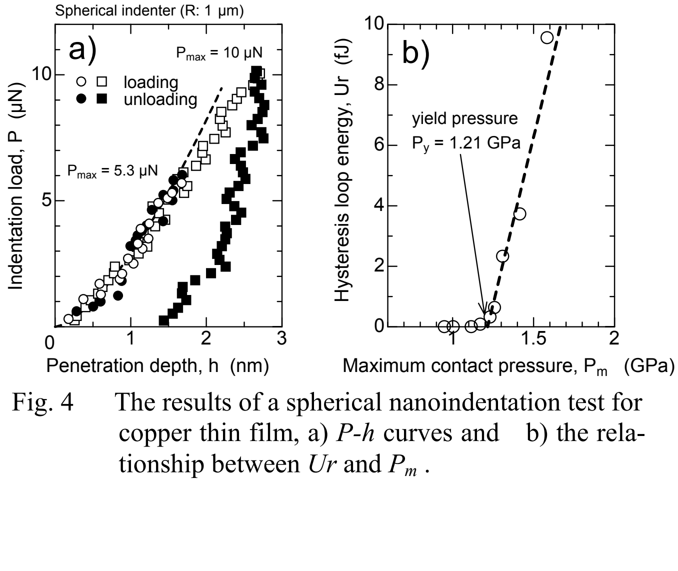

Fig. 4 The results of a spherical nanoindentation test for copper thin film, a) P-h curves and b) the relationship between Ur and P_m .

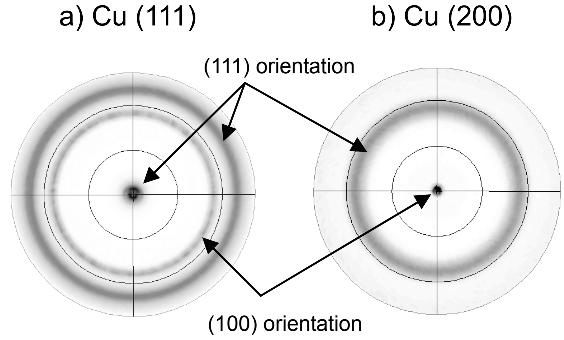

Fig. 5 The pole figures of x-ray diffraction for a) Cu (111) and b) Cu (200).