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Nanomechanical response and creep behavior of electroless deposited copper films under nanoindentation test

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Abstract

The nanomechanical response and creep behavior of electroless plated copper (Cu) films have been investigated in this research by using a nanoindentation test. The hardness and elastic modulus of the nanostructural Cu films with a large amount of small grains in size of only 5 nm were measured as 1.5 and 120 GPa, respectively. The Cu films deformed elastically at first and then yielded at a stress of 3.3 GPa. Grain-boundary sliding and grain rotation were expected to dominate the deformation of the Cu films. The Cu films showed a creep strain rate of about $5 \times 10^{-5}\,s^{-1}$ under the nanoindentation test, and the creep strain rate–stress relation exhibited a typical power law expression with a stress exponent of 6.4. The high creep strain rate but low stress exponent of the Cu films implied a fixed creep behavior consisting of grain-boundary sliding and grain rotation by the fast diffusion of Cu atoms through the large amount of grain-boundary.

Keywords: Nanoindentation; Stress–strain curve; Dislocations; Creep

1. Introduction

Electrochemically deposited copper (Cu) has been widely adopted as interconnect metallization in ultralarge-scale integrated circuits to reduce the problem of serious resistance–capacitance delay [1–4]. However, mechanical damages of the Cu films, caused by thermal stresses and chemical–mechanical polishing, etc., severely suppress the processing yield and application reliability of integrated circuits [5–8]. The mechanical properties of the Cu films are thus important to be clarified for the evaluation of the reliability of nanoscaled multilevel interconnects. An instrumented nanoindentation test has been widely applied for the measurement of the mechanical properties of thin films [9,10]. However, only hardness and elastic modulus are generally obtained through the test. True mechanical behaviors of the Cu films relating to the mechanical damages are still obscure even though some related researches have been reported [11–18]. The onset of plasticity, associated with the analyses in true flow stress and yielding strength, provides a quantitative evaluation for the deformation and defect nucleation of Cu films [11–16]. To further effectively avoid the failure of multilevel Cu interconnects, the architecture design can be accordingly modified through the realization of the stress criteria for the permanently plastic deformation of the Cu films.

Especially, electroless Cu deposition which provides good gap-filling and step-coverage capability in finely patterned features is intensively studied as potential Cu metallization for next-generation semiconductor manufacturing [2–4]. Nanocrystalline structures of the electrolessly plated Cu films with ultrafine grain sizes smaller than 10 nm will further change the mechanical properties [4]. Plastic deformation is no longer expected to be determined by dislocation activity but instead by grain-boundary sliding and grain rotation, resulting in a "reverse Hall–Petch effect" [19–22]. Therefore in this research, the nanoindentating mechanical responses including true flow stress and plastic deformation behavior were analyzed to evaluate the more representative mechanical properties of the Cu films [13–16]. Moreover, the stress migration of Cu wires strongly dominates the reliability of multilevel interconnect structures [7]. It is expected to relate to the creep behavior of the Cu films because both of them proceed by the diffusion of Cu atoms. Therefore in this study, the creep behavior of the electrolessly deposited Cu films was also

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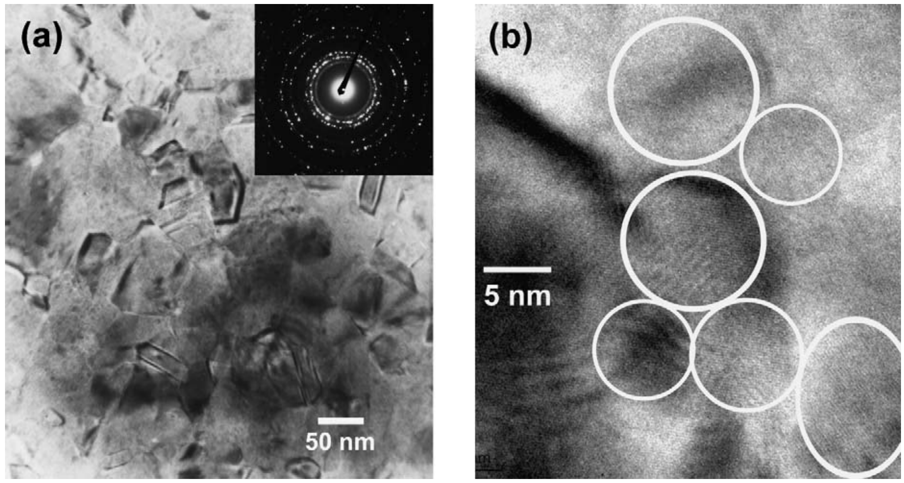

Fig. 1. TEM images of electrolessly deposited Cu films: (a) bright-field image and SAD pattern and (b) lattice image.

investigated by using the nanoindentation test as a prediction of the failure resistance of Cu interconnects to stress migration [17,18].

2. Experimental details

Silicon wafers with a thermal oxide (SiO_2) layer of 550-nm thick and a sputtered tantalum nitride (TaN) layer of 50-nm thick were used as substrates. These Si/SiO_2/TaN substrates were chemically wet-etched using a hydrogen fluoride (HF, 10%) solution at 30 °C for 30 s, catalyzed in an activation solution consisting of palladium dichloride ($PdCl_2$) at 25 °C for 120 s, and then electrolessly Cu-deposited in the solution as reported at a temperature of 65 °C to obtain Cu films of about 2–3 μm thick [2–4]. A transmission electron microscope (TEM, JEOL-2010FXII) with selective area diffraction (SAD) analysis was used to examine the microstructure of the deposited Cu films. From the TEM image and SAD pattern shown in Fig. 1(a), the Cu films were observed as a polycrystalline structure with grain sizes of about 20–50 nm. However, from the more detailed lattice image shown in Fig. 1(b) as well as previous study [4], these grains were composed of clusters of extremely fine nanocrystallites of only about 5 nm.

A UMIS nanoindenter (Based Model, CSIRO, Australia) with a Berkovich diamond indenter (tip radius ~100 nm, edge angle 130.6°) was used to measure the mechanical properties of electrolessly deposited Cu films. A load cell and a displacement-voltage dilatometer (LVDT) were used to control the applied load and to measure the penetration depth of the indenter. The maximum load was set at 3 mN under a loading/unloading rate of 0.01 mN/s, and the indentation depth was controlled below 1/10 of the film thickness to avoid substrate effect. For creep test, the load was hold at the maximum value of 3 mN for 1800 s. A general power law expression was applied to the strain rate–stress relation of the indention creep test to calculate the stress exponent and to realize the creep behavior of the electrolessly plated Cu films.

3. Results and discussion

3.1. Nanomechanical properties and deformation behavior

Fig. 2 shows the load–penetration depth curves of nanoindentation tests of electrolessly deposited Cu films. From the loading/unloading curves and the famous Oliver–Pharr relation [9–11], the elastic modulus and hardness of the Cu films were obtained as about 120 and 1.5 GPa, respectively. Further from the magnified load–depth curve shown in up-left corner of the figure, it was found that, with indentation depth smaller than 6 nm, the loading curve rather matched to the elastic unloading curve in accordance with the "Hertzian elastic relation" [23], revealing the purely elastic response of the Cu films without any plastic deformation under the extremely small strain. As the applied load exceeded 0.11 mN, the curve deviated from the "Hertzian response" after which it was expected that the stress intensity at the indenter tip had accumulated to the critical shear stress for the plastic deformation (yielding) of the Cu films.

As reported for the electroplated Cu films with grain sizes of about several tens of nanometer, plastic yielding occurred

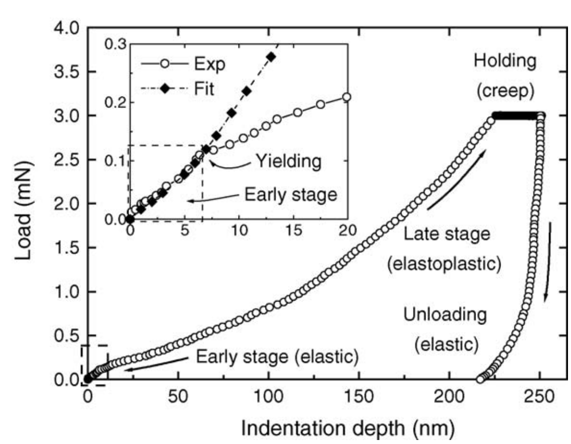

Fig. 2. Typical and magnified load–depth curves of nanoindentation tests of electrolessly plated Cu films (holding 300 s).