MESOMECHANICAL ASPECTS OF THIN FILM FRACTURE

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ABSTRACT

The processes of plastic deformation and fracture of Ag and Ti thin films under thermal treatment and mechanical loading are studied. The evolution of the film surface topography is examined with atomic force and inverted incident-light microscopes. The plastic flow in thin metal films under both thermal and mechanical loading is found to have a similar nature. The processes of plastic deformation and fracture of the films under different external actions are shown to develop sequentially at different scale levels.

1 INTRODUCTION

Currently thin polycrystalline metal films have been received widespread attention because of their extensive application in microelectronics. Although the primary functions of thin metal films are governed by their electrical properties, the mechanical reliability of the films is also of great importance, in particular, because of the heating and cooling cycles that are intrinsic to the fabrication and use of integrated circuits. The thermal-expansion difference between the substrates and interconnection materials leads to high mechanical stresses in thin-film structures [1-3]. These stresses can be even higher than values of the yield strength for conventional bulk metallic materials. Stress relaxation results in the plastic strain of thin films followed by their fracture, e.g., cracking, voiding, hillocking and delaminating. Moreover, internal stresses greatly affect the evolution of the microstructure and texture of thin films that can change their physical properties and, consequently, the operating characteristics of microdevices.

The mechanical behavior of thin films under strain considerably depends on the conditions of loading. Particularly, uniaxial internal stresses develop in thin films under tension, while thermal treatment causes biaxial stresses to appear in films deposited on the substrate. However, the mesomechanical approaches [4] allow considering plastic deformation and fracture of thin films as consecutive stages of the shear stability loss at different scale levels regardless of the conditions of loading.

Advanced techniques such as 3D light microscopy and scanning probe microscopy make possible to characterize in three dimensions the microstructural evolution of strain-induced relief in thin films. The use of the techniques allows one to reveal in details sequential and self-consistent development of plastic deformation and fracture of thin films under different external actions.

The main purpose of the present paper is to study key features of plastic deformation and fracture of thin metal films under thermal treatment and active loading.

2 EXPERIMENTAL DETAILS

The samples studied were Ag and Ti thin films deposited at room temperature by DC magnetron sputtering. The films were grown on SiO_2/Si and polypropylene substrates. The preliminary cleaning of Si substrates involved washing in acetone and twice-distilled water with subsequent drying in isopropyl alcohol vapor. Next, a SiO_2 layer 200 nm thick was grown by thermal oxidation. Finally Ag films 500 nm thick and Ti films 100 nm thick were sputtered in vacuum at a base pressure 3×10^{-3} Torr. For testing under uniaxial tension Ti films were deposited on polypropylene substrates made in the form of a dumb-bell with the following working part dimensions: 33.5×2.6×1.0 mm^3.

The thermal treatment of the films was performed in ambient conditions at temperatures from 373 to 873 K. The experiments included heating to the these temperatures, annealing for 1 hour, and cooling to room temperature. The samples were held at room temperature during about one day before the investigations. The Ti films on the polypropylene substrate were subjected to uniaxial tension at room temperature using testing machine.

The development of strain-induced relief was studied on the plane specimen surface by means of the inverted incident-light microscope Axiovert 25 CA and an atomic force microscope (AFM). The AFM-images were obtained with a silicon nitride tip (Park Scientific Instruments) with a spring constant of 0.06 N/m. All the measurements were performed in air at room temperature and ambient pressure.

Mechanical properties of the films were studied using the NanoTest 600 nanoindenter with maximum imposed loads from 0.5 to 200 mN. Variations of the penetration depth as a function of applied load at the stages of loading and unloading were analyzed according to the method developed by Oliver and Pharr [5].

3 RESULTS AND DISCUSSION

Plastic flow and fracture in thin metal films under both thermal and mechanical loading have a similar nature. In both the cases the processes develop sequentially and self-consistently at different scale levels.

Results of the AFM-studies of Ag thin films before and after thermal treatment at different temperatures are presented in Fig. 1. The as-deposited film is seen to be characterized by fine-grained surface morphology (Fig. 1,a), with an average lateral grain size being equal 100 nm. The surface structure is very uniform and the grain size distribution is monomodal.

The changes in the surface morphology of the Ag films under thermal treatment are observed to be governed by two competing processes: growth of grains and their agglomeration into larger crystallites (Fig. 1,b). The average grain size increases on increasing annealing temperature, and reaches 500 nm at 533 K. In addition, large crystallites with lateral dimensions of up to 2 μm consisting of several grains are observed as early as after annealing at 473 K. At the same time, the formation of grooves at the grain boundaries occurs. The groove depth reaches 200 nm at 473 K.

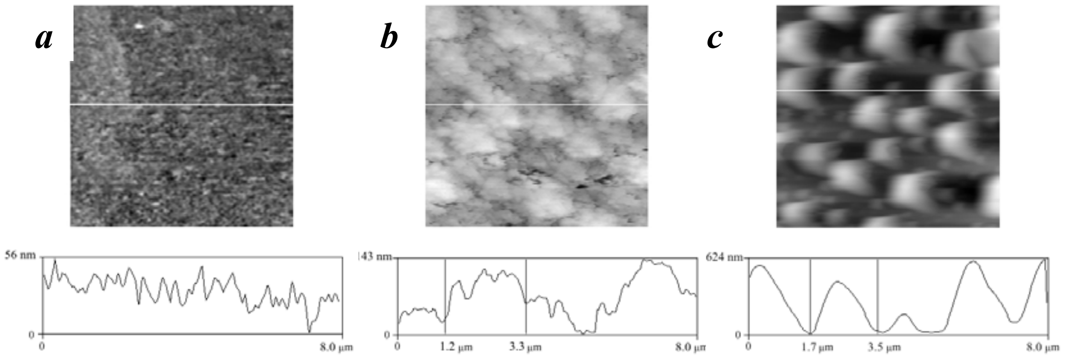

Figure 1: AFM-images and profiles of an Ag film surface before (a) and after thermal treatment at temperatures of 473 (b) and 673 K (c). Observed areas are 8.0×8.0 μm^2.

The fracture stage is observed after thermal treatment at 673 K. The film becomes discontinuous and large separate three-dimensional islands are formed all over the surface (Fig. 1,c). Their lateral dimensions are 1-2 μm and average height is about 500 nm, i.e. higher than the film thickness. The island density is 3.6×10^{7} cm^{-2}. The subsequent increase in the annealing temperature results in increasing the island dimensions, with their density being decreased. Thus, at 833 K the islands are about 2.5 μm in diameter and their density is 0.9×10^{7} cm^{-2}.

The observed changes are appeared to be caused by high internal stresses developed in the Ag films under annealing. If the annealing temperature exceeds the deposition temperature, because of the difference in thermal expansion coefficient $\Delta\alpha$ of the film and substrate, the lateral dimensions of the film during heating no longer match those of the substrate and biaxial stresses are imposed on the film to deform it so that it again fits the dimensions of the substrate. The value of the biaxial stresses can be conveniently calculated by multiplying the biaxial elastic modulus M of the film by $\Delta\alpha$. Since the coefficient of thermal expansion of silver (18.8×10^{-6} K^{-1}) is higher than that of silicon (3.0×10^{-6} K^{-1}), then $M_{\mathrm{Ag}}\Delta\alpha_{\mathrm{Si\text{-}Ag}} = -2.0$ MPa/K and compressive stresses will be developed into Ag films during heating up to annealing temperature. The value of these stresses could be as high as 350 MPa at a temperature of 473 K and 750 MPa at 673 K.

The hardness value H of the Ag films obtained from the analysis of the nanoindentation curves is 1.3 GPa. Using the Tabor relation between the hardness and the yield strength we can estimate a value of the yield strength of the films as $\sigma_{y} = H/3$. This estimation gives a value of 430 MPa. Because a yield strength value considerably decreases on increasing temperature, plastic strain in the Ag films is expected to occur already at 473 K that agrees with the AFM-investigations.

The strain mechanism of thin films under thermal treatment substantially depends on annealing temperature. At low temperatures (typically below a third of the melting temperature, about 410 K for silver), dislocation-mediated plasticity controlled by the thermally activated glide of dislocations has been proposed to be the dominant plasticity mechanism in thin films [6]. In this case, relatively low available thermal energy and work hardening of the film result in higher film strength.

At high temperatures (above 410 K for silver) inelastic deformation in FCC metals were found to be predominantly controlled by diffusional creep [7]. The stresses can be relaxed by the diffusion of matter between the bulk of the film and its surface. It may be realized either by lattice diffusion or by grain boundary diffusion, however, for nanocrystalline thin films with small grain size the contribution of lattice diffusion to the stress relaxation appears to be negligible.

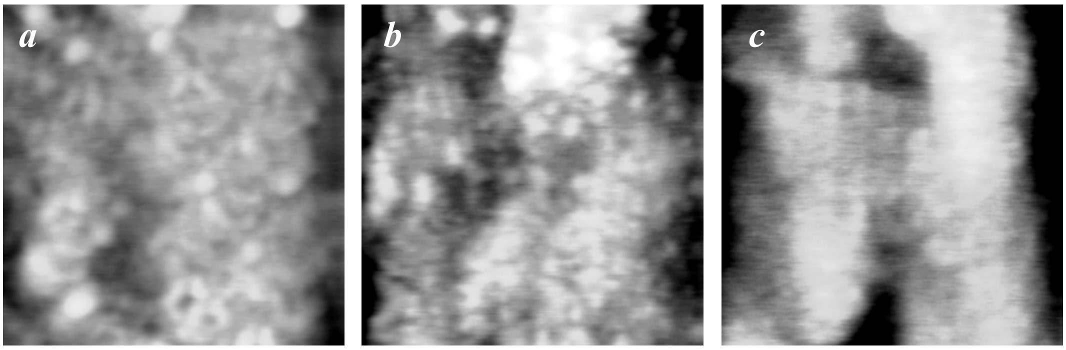

Figure 2: AFM-images of a Ti stripe surface before (a) and after annealing at temperatures of 573 (b) and 773 K (c). Observed areas are 2.0×2.0 μm^{2}.

After being transferred, atoms redistribute at the film surface by surface diffusion leading to surface curvature. Thus, the creep of the films will be determined by the relative mobilities of atoms along the surface and along the grain boundary. The slowest process will control the creep rate of the films. Which mechanism is dominant depends on the ratio lD_b/hD_s [7], where l is the grain dimension in the film plane, h is the grain boundary height, D_b and D_s are the grain boundary and surface diffusion constants. Since growth of grain dimensions results in decreasing the grain boundary length per unit square, the atomic flux from the bulk becomes insufficient that appears to lead to grooving at the grain boundaries. Pinning the grain boundaries by thermal grooving impedes subsequent grain growth that results in decreasing the rate of grain growth in the Ag films at an annealing temperature of 533 K.

Further increasing annealing temperature results in the film fracture and the formation of separate islands, that is the high-temperature limit of the grooving. Intensive diffusion processes cause relaxation of thermal stresses and the decrease of the total energy owing to growth of separate three-dimensional crystallites. In this case, strain relaxation occurs through a series of surface phase transitions before reaching the state of large three-dimensional islands, during which islands transform and grow.

In contrast to the Ag films, thermal stresses in the Ti films deposited on the silicon substrate even on heating up to 773 K reach only 380 MPa ($M_{Ti}\Delta\alpha_{Si\text{-}Ti} = -0.8$ MPa/K), whereas, according to the nanoindentation data, the yield strength of these films is 2.5 GPa. Therefore annealing of the Ti films in the temperature range from 373 to 773 K does not result in their plastic strain and fracture. Moreover, an average grain size in the films is nearly invariable (100 nm).

At the same time, the similar films deposited on the silicon substrate in the form of narrow stripes are plastically strained under thermal treatment. This becomes apparent in smoothening the stripe edges because of its tendency to the minimum surface energy. In addition, already upon annealing at a temperature of 573 K folded structures are formed on the stripe surface (Fig. 2). On increasing annealing temperature the fold dimensions grow and an average fold width reaches 700 nm at 773 K.

The similar folded surface topography is developed under uniaxial tension of Ti films deposited on the polypropylene substrates (Fig. 3,b). The folds are oriented along the directions of maximum tangential stresses. The transverse dimension of the folds is equal to 1 μm that coincides with an average grain size in the films. The strain relief develops only on the film surface rather than on the substrate one. The following loading results in the film fracture. The fracture stage is

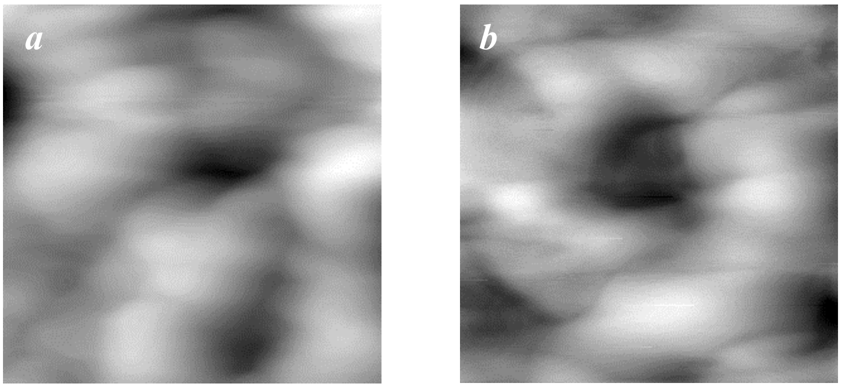

Figure 3: AFM-images of a Ti film surface deposited on the polypropylene substrate before (a) and after tensile test (b). Observed areas are 2.8×2.8 μm^2.

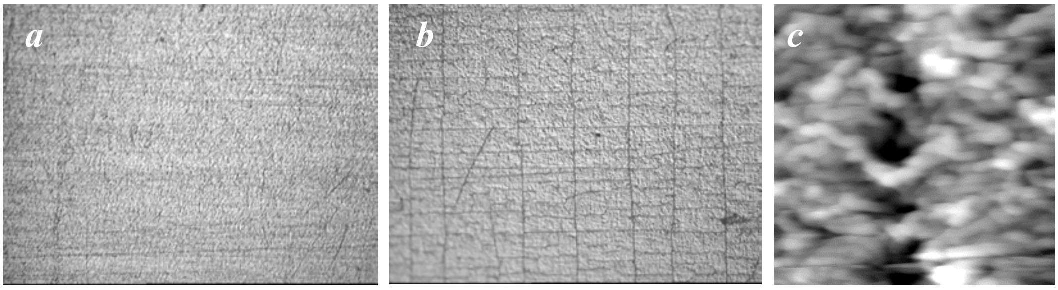

Figure 4: Optical images (a,b) and an AFM-image (c) of a Ti film surface deposited on polypropylene before (a) and after tensile test (b,c). Observed areas are 600×450 (a,b) and 11.2×11.2 μm^2 (c).

characterized by the formation of transverse and longitudinal cracks (Fig. 4). The distance between the cracks is 30-50 μm. It should be noted that the folded structures do not fractured under cracking the films that is clearly seen in Fig. 4,c.

4 SUMMARY

The processes of plastic strain and fracture of magnetron sputtered Ag and Ti thin films under thermal and mechanical loading were shown to develop sequentially and self-consistently at different scale levels. The changes in the surface topography of the Ag films are governed by two competing processes: growth of grains and their agglomeration into larger crystallites. At high temperatures pinning the grain boundaries owing to thermal grooving impedes subsequent grain growth. As a result the continuous film transforms into the island one.

The Ti films are plastically strained under thermal treatment in the temperature range studied only if they are deposited on the substrate in the form of narrow stripes. In this case, smoothening the stripe edges and the formation of folds on their surfaces are observed.

Plastic flow of the Ti films under uniaxial tension is also accompanied by the formation of folded structures oriented along the directions of maximum tangential stresses on their surface. The film fracture results from the formation of transverse and longitudinal cracks.

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References

[1] Nix W.D. Mechanical properties of thin films, Met. Trans. A. 20, pp. 2217-2245 (1989)

[2] Vinci R.P., Zielinski E.M., Bravman J.C. Thermal strain and stress in copper thin films, Thin Solid Films 262, pp. 142-153 (1995)

[3] Smith U., Kirstensen N., Ericson F., Schweitz J.-A. Local stress relaxation phenomena in thin aluminum films, J. Vac. Sci. Technol. A 9, pp.2527–2535 (1991)

[4] Panin V.E. In: Physical Mesomechanics of Heterogeneous Media and Computer-Aided Design of Materials, Panin V.E. (Ed). Cambridge Interscience Publishing, Cambridge, pp. 1-40 (1998)

[5] Oliver W., Pharr G. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments, J. Mater. Res. 7, pp. 1564-1583 (1992)
[6] Kobrinsky M.J., Thompson C.V. Activation volume for inelastic deformation in polycrystalline Ag thin films, Acta Mater. 48, pp. 625-633 (2000)
[7] Thouless, M.D. Effect of surface diffusion on the creep of thin films and sintered arrays of particles, Acta Metall. Mater. 41, pp. 1057-1064 (1993)