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Measurement of stress as a function of temperature in Ag and Cu thin films

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Stress measurements of 23 nm copper films and 93 nm silver films on Si (100) have been performed during thermal cycling between RT and 450°C. The changes in stress versus temperature are interpreted. The effects of treatment on microstructure and composition are studied by X-ray diffraction.

Keywords: annealing, stress measurements, thin films.

1. Introduction

Copper and silver are more and more commonly used as interconnecting lines in microelectronic devices replacing aluminium. The ratio of the electrical and thermal conductivity of Cu and Ag is better than that of Al improving electrical performance and causing better heat transport and greater resistance to stress and electromigration [3]. It is possible to modify stress in the thin films. Thermal treatment is one of the modification methods. The stress measurements durig thermal cycles are valuable in providing quantitative data about the mechanical stability of thin films subjected to structural and morphological changes [2–4].

This paper reports on stress modification during thermal vacuum annealing. The experiments have been performed using the curvature measurement optical system [2, 3].

2. Experimental

Figure 1 shows a scheme of apparatus used in the experiment. The measuring technique (radius of the sample curvature) is based on Flinn’s laser scanning method. A laser beam reflected on the rotating mirror sweeps the beam over the sample surface.

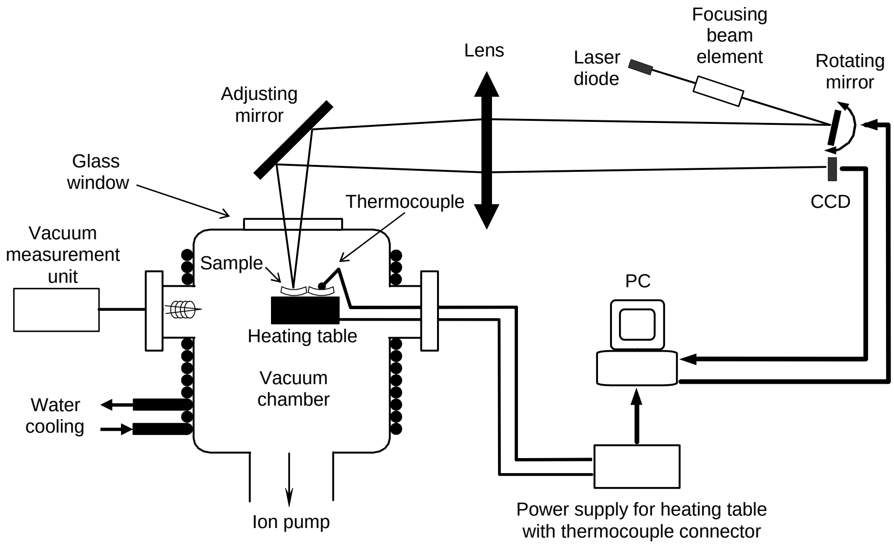

Fig. 1. Scheme of a setup for the curvature radius measurement.

The exact position of the reflected beam as a function of mirror angle allows the radius of the sample to be calculated. Curvature stress in films is then calculated by measuring the change in wafer curvature and using the following equation [2]:

$$\sigma_f = \frac{E_s t_s^2}{6t_f(1-\nu_s)}\left(\frac{1}{R}-\frac{1}{R_0}\right) \tag{1}$$

where E_s is the Young modulus, ν_s – the Poisson ratio, t_s – the thickness of the substrate, t_f – the thickness of the film, R – the radius of curvature of the substrate during deposition, while R_0 – the radius of curvature of the wafer before deposition [2].

We measured the radius of sample curvatures before, during and after deposition [5]. Futhermore the annealing and cooling cycles were realized.

When the temperature of a film is changed, a strain is introduced because of the difference in thermal expansions in film and substrate

$$\Delta\varepsilon_{\mathrm{th}} = \int_{T_1}^{T_2}(\alpha_s-\alpha_f)\,\mathrm{d}T. \tag{2}$$

The thermal stress is due to the difference in thermal linear expansion coefficients of the film and substrate (α_t and α_s, respectively) according to [4]:

$$\sigma_{\mathrm{th}} = \frac{E_f}{1-\nu_f}(\alpha_s-\alpha_f)\Delta T. \tag{3}$$

If the deposited layer has a coefficient of thermal expansion different from that of the substrate, a plane and isotropic deformation will be induced by the substrate.

The films were deposited by evaporation onto 100 μm (100) oriented silicon substrates at room temperature in the ultrahigh vacuum system. The base pressure was about 2×10^{-9} hPa. Before deposition the substrates were subjected to chemical cleaning by being washed for 15 min in acetone and ethanol in ultrasonic washer and dried in nitrogen gas. The thickness of the deposition film was 93 nm for Cu, and 23 nm for Ag. The sample analysed was positioned horizontally onto a heating table. The temperature was controlled by a thermocouple clamped onto silicon substrate close to a sample. This method allows avoiding the effects of stress which is related to a fixed thermocouple on the sample. At the same time constant temperature conditions were provided. The tests show that the reference temperature of the sample is only a few degrees different from that of the silicon substrate in the whole temperature range [6]. The base pressure in the annealing chamber was 2×10^{-7} hPa and during the experiment about 10^{-6} hPa. The measurements were performed during the thermal cycles of heating from RT to 450°C at the rate of 10°C/min and cooling down to RT at the rate of 20°C/min.

3. Results and discussion

Figures 2 and 3 ilustrate the evolution of stress as a function of temperature in Cu and Ag films deposited onto silicon substrate at room temperature. After deposition the Ag film is under the tension stress of about 0.6 GPa. When temperature is increased, the film tends to expand. At about 200°C the stress reaches the maximum (1 GPa). The deviation from linear behaviour is the result of plastic deformation in the film. Further heating causes that stress becomes compressive reaching the minimum (2.75 GPa) at 375°C. During cooling the film contracts faster than the substrate. In this case, the film stress changes to tensile direction and the stress evolution is a linear function of temperature. As the temperature decreases, the plasticity is reduced resulting in the increased film strength [6]. Finnally, when the temperature is again equal to room temperature, stress returns to the starting value. A small loop between 325°C and 425°C was observed. The reason for this effect is probably small thickness of the film and the increase of grains size [6].

After deposition, the Cu film is under compressive stress of about 0.3 GPa. For the Cu layer we observed a rapid increase of tension stress up to 0.87 GPa at 50°C and next a sudden decrease to 0.3 GPa at 100°C. Further increase of temperature causes that the stress decreases linearly in compressive direction. During cooling the stress reaches the minimum (1.3 GPa) at 350°C and rapidly changes direction tending towards zero. From this point, we observed almost linear evolution of the stress according to $\sigma(T) = 0.1$.

As Gardner and Flinn [2] state, the curve representing the stress versus temperature on heating should be close that representing the behaviour of the sample

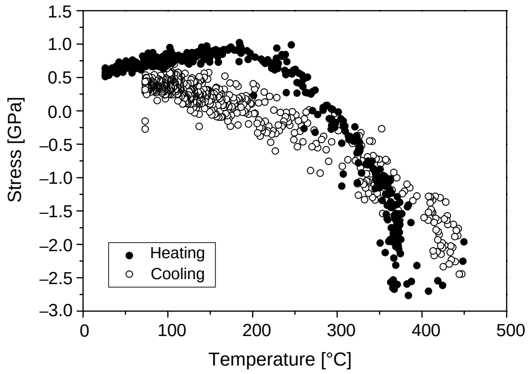

Fig. 2. Stress as a function of temperature evolution for the Ag film.

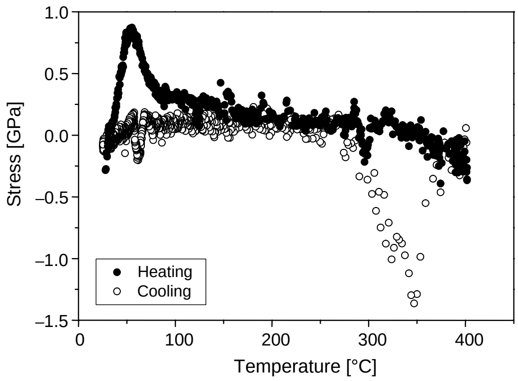

Fig. 3. Stress as a function of temperature evolution for the Cu film.

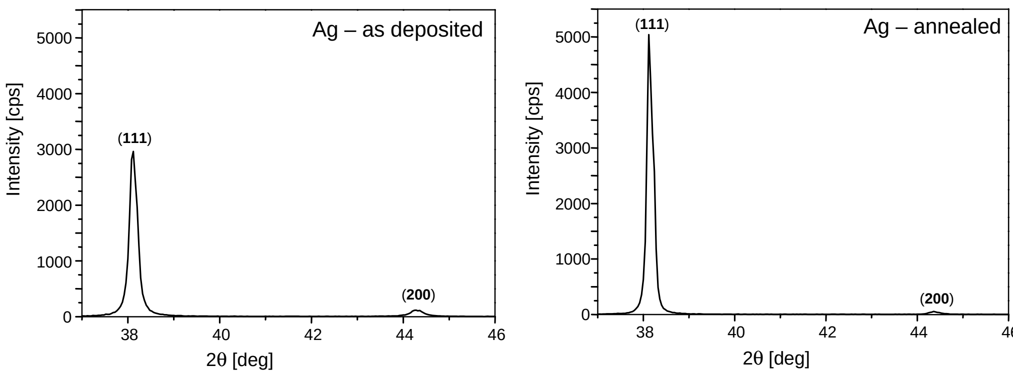

Fig. 4. X-ray θ–2θ scans for the Ag film before and after annealing.

on cooling after deposition and should have the shape of a hysteresis loop. The shape of the $\sigma(T)$ curve in the Cu film is difficult to explain and therefore some investigations will be carried out.

Before and after annealing the Cu and Ag samples were analyzed using X-ray diffraction (Figs. 4 and 5) to identify structural changes in the film. For both materials

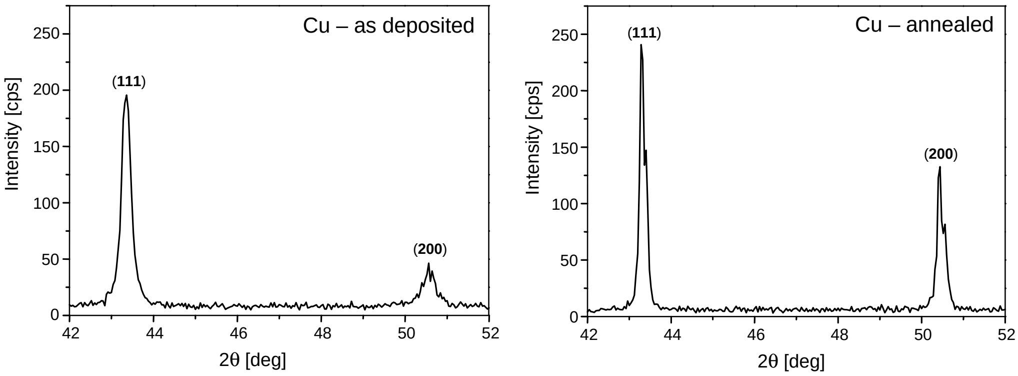

Fig. 5. X-ray θ–2θ scans for the Cu film before and after annealing.

diffracting peaks (111) and (200) were observed. An increase in the intensity of both (111) and (200) peaks in the Cu films after annealing was observed. In the Ag film, an increase in intensity of only the (111) peak was observed but a (111) texture was observed on both types of samples. The grain size increases with temperature. The proportion in intensity of the peaks permits us to state that the structure after annealing is still polycrystalline.

4. Conclusions

The results of *in situ* measurements of curvature and X-ray stress on thin Ag and Cu films strained during thermal cycling are presented. The shape of the $\sigma(T)$ curve in Ag film may be explained by plastic deformation in the film. The obtained shape of the $\sigma(T)$ curve in Cu film has not been reported so far in literature, and cannot be explained in terms of plastic deformation. Futher mechanical and structural investigation of the Cu films is required.

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References

[1] VINCI R.P., ZIELINSKI E.M., BRAVMAN J.C., *Thermal strain and stress in copper thin films*, Thin Solid Films **262**(1-2), 1995, pp. 142–53.

[2] GARDNER D., FLINN P.A., *Mechanical stress as a function of temperature in aluminum films*, IEEE Transactions on Electron Devices 35(12), 1988, pp. 2160-9.

[3] FLINN P.A., GARDNER D.S., NIX W.D., *Measurement and interpretation of stress in aluminum-based metallization as a function of thermal history*, IEEE Transactions on Electron Devices **34**(3), 1987, pp. 689–99.

[4] BERGER S., RASLIN O., *Stresses in Al/TiW/Si(100) contacts during thermal cycling*, Thin Solid Films **333**(1-2), 1998, pp. 264–71.

[5] PIENKOS T., PROSZYNSKI A., CHOCYK D., GLADYSZEWSKI L., GLADYSZEWSKI G., *Stress development during evaporation of Cu and Ag on silicon*, Microelectronic Engineering **70**(2-4), 2003, pp. 442–6.

[6] BOSTROM O., *Wafer shape control study of the reactivity in Ti/Al dual layers and its effect under stress*, PhD Thesis, Universite Aix-Marseille III, 2001.

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