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# Post-deposition stress evolution in Cu and Ag thin films

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Evolution of stresses in thin Cu and Ag films after deposition by thermal evaporation in UHV system is studied. Thin films were deposited on 100 μm thick Si substrate at room temperature. Deposition rates for the Cu and Ag films were 0.5 Å/s and 0.9 Å/s, respectively. The total thickness ranged from 7.7 up to 109 nm. The average stress in the films was determined by measuring the radius of samples curvature. The behavior of stress evolution curves is explained by two mechanisms of stress generation: filling grain boundaries and islands coalescence.

Keywords: stress, curvature measurements, thin film.

## 1. Introduction

In the development and manufacturing of silicon devices, the knowledge and the control of thin film stresses are very important. High stresses can cause deformation, delamination, cracking and void formation. The basic materials applied in microelectronics are silicon as a substrate and copper and silver as contacts and diffusion barrier layers. The low resistivity and superior electromigration of those metals in comparison to aluminium have generated great interest [1]. Independently of the applied technique of deposition in the obtained thin films very large stresses develop during the deposition process. Experiments have shown that the stresses in thin metal films can exceed those of the corresponding bulk materials and increase with the decreasing film thickness [2]. Understanding the origin of stresses in thin metal films is, therefore, crucial for predicting the reliability of devices based on thin films. Several techniques are used to study mechanical properties of metallic thin films [3–5]. The mechanical properties of thin films are often investigated by measuring the change in substrate curvature induced by deposited film [6–10].

In this work we continue our earlier studies concerned with the stress in thin films. In previous work [6, 9] we concentrated on stress evolution during deposition of thin films. Now, the results of stress evolutions after the deposition of Cu and Ag thin films are presented. We examined samples with different total thickness deposited at the same rate. The thin film structure was examined by X-ray diffraction.

## 2. Experimental

Copper and silver thin films, with total thickness from 7.7 up to 109 nm, were deposited on the 100 μm thick silicon (001) substrates by thermal evaporation in a UHV system at room temperature. Copper films were deposited at the rate 0.5 Å/s and silver films were deposited with rate 0.9 Å/s. Before deposition the surfaces of Si substrates were rinsed in an ultrasonic washer for 15 min in acetone and for 15 min in ethanol. Then the substrates were dried in nitrogen gas. The dimensions of the samples were 20×10 mm. For all measurements, the base pressure in the deposition chamber was the same: $1.5\times10^{-9}$ torr. The thickness and deposition rate of the film were monitored by a quartz crystal thickness monitor.

The curvature evolution of the substrate was measured with the scanning laser method described by FLINN *et al.* [11] during and after film deposition as a function of time. After deposition of the thin film the curvature of the substrate was monitored up to 30 min. To eliminate thermal effects, the temperature of each sample was kept up as during deposition. Total force per unit width applied by the film on the substrate is directly related to the substrate curvature by the Stoney's equation [12, 13]:

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

where $\sigma_f$ is the stress in the thin film, $E_s$ is the Young's modulus, $\nu_s$ is the Poisson's ratio, $t_f$ and $t_s$ are the film and the substrate thickness, respectively, $R_0$ is the initial radius of curvature before film deposition and $R$ is the measured radius of curvature during or after deposition.

The type of bending (the sign of the radius of curvature) is determined by the direction of movement of a spot on the detector. Positive values are assigned to tensile and negative values to compressive film forces. More details about this method can be found in papers [3, 4].

Structural analysis was made using a Philips X-ray difractometer (MPD) with Cu anode ($\lambda = 1.5458$ Å). The samples were examined in $\theta$–$2\theta$, $\omega$ geometry and pole figure measurement.

## 3. Results and discussion

### 3.1. Structure

Both for Cu and for Ag, $\theta$–$2\theta$ scans revealed two lines from copper and silver, namely (111) and (200). Since the (111) peak is much stronger than the peak attributed to the (200), the preferred crystal orientation is (111) in the growth direction. Example of

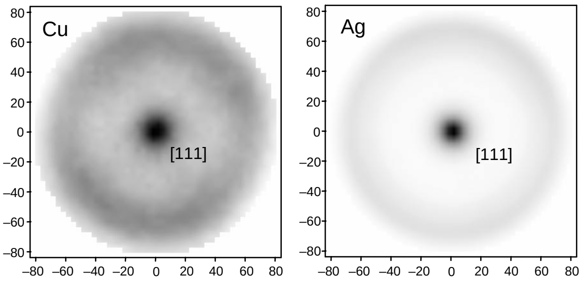

Fig. 1. Pole figure for Cu and Ag thin film.

pole figures of Cu(111) and Ag(111) are presented in Fig. 1. In both cases for Cu and Ag, the (111) planes are parallel to the substrate surface. Narrower and higher intensity rings are observed for Cu thin films. The presence of spread rings means that the thin films have not crystallographic orientation in the film plane.

### 3.2. Stress evolution

The post-deposition stress evolution in Cu and Ag thin films of different total thickness is shown in Figs. 2 and 3. The post-deposition stress evolution data are plotted as a force per unit width versus time and suited on thickness axes. Dependences force per unit width versus thickness during thin film deposition for samples of maximum total thickness for each material are also shown (thin line). In all cases we observe that the stress changes in the tensile direction independent of its initial value. To describe the relaxation process, the exponential function is usually used. Due to the fact that

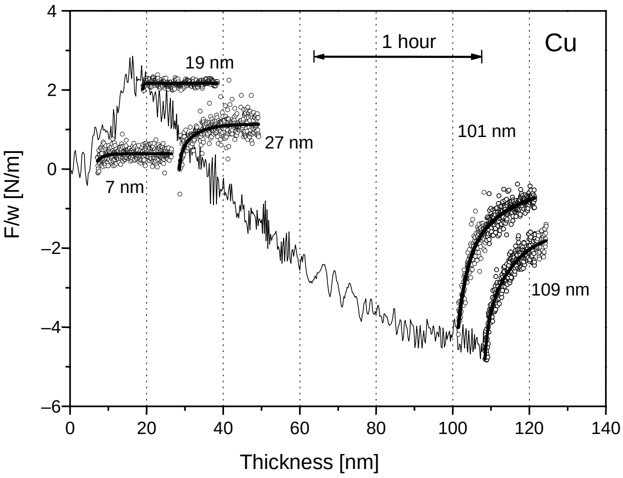

Fig. 2. Evolutions of the stress after deposition of Cu thin films of different total thickness. The experimental data (open circle) and fitting data (black line) are plotted as force per unit width *vs.* time and properly suited on thickness axes. Dependences force per unit width *vs.* thickness (black thin line) show the behavior during growth of thin film to the total thickness 109 nm.

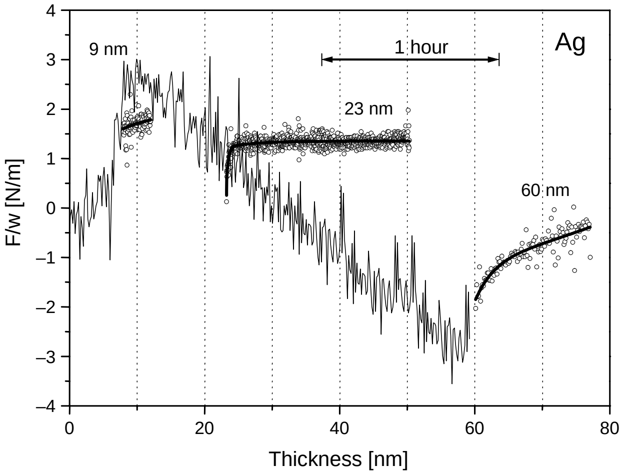

Fig. 3. Evolutions of the stress after deposition of Ag thin films of different total thickness. The experimental data (open circle) and fitting line (black line) are plotted as force per unit width *vs.* time and properly suited on thickness axes. Dependences force per unit width *vs.* thickness (black thin line) show the behavior during growth of thin film to the total thickness 60 nm.

there are two processes that mainly influence stress in thin film, we fit the experimental data by the function consisting of two exponential functions. It was shown that the function consisting of two exponential functions describes well the measured data [15]. For fitting we use the following form of the function [15]:

$$\sigma(t) = \sigma_0 - A_1\left[1 - \exp(-b_1 t)\right] - A_2\left[1 - \exp(-b_2 t)\right] \tag{2}$$

where $A_1$, $A_2$, $b_1$, $b_2$ are the fitting parameters, $\sigma_0$ is the initial stress. We can interpret these parameters easily. Parameters $A_1$, $A_2$ are the maximum stress change and $b_1$, $b_2$ are the inverse of the relaxation time in each process. Positive value of $A_1$ and $A_2$ denotes stress changes in compressive direction, whereas negative value of $A_1$ and $A_2$ denotes changes in tensile direction [15]. For all the samples fitting parameters are listed in Tabs. 1 and 2. For all the samples the values of $b_1$ are smaller than those of $b_2$. It suggests that the part of Eq. (2) connected with indicator 1 represents the process proceeding more slowly. The second part of the equation represents the process proceeding more quickly. In most cases the value of the parameters $A_1$ and $A_2$ are negative, that means the stress of both processes changes in tensile direction. If the parameters $A_1$ or $A_2$ are equal to zero, it means that the equivalent process is inconsiderable.

The interpretation that there are two stress changing processes: filling grain boundaries by excess atoms and islands coalescence is in agreement with the model proposed by CHASON *et al.* [16–18]. In that model, the compressive stress is a consequence of the non-equilibrium state of the film during deposition. This non-equilibrium state causes that the chemical potential of film surface is higher in the

Table 1. Parameters obtained from fitting for Cu.

| | Thickness | | | | |
|---|---|---|---|---|---|
| | 7 nm | 19 nm | 27 nm | 101 nm | 109 nm |
| $A_1$ [N/m] | 0.0 | –0.003 | –0.737 | –1.921 | –2.408 |
| $b_1$ [1/s] | 0.0 | $0.8321\times10^{-5}$ | $2.64\times10^{-3}$ | $0.75\times10^{-3}$ | $1.41\times10^{-3}$ |
| $A_2$ [N/m] | –0.189 | –0.158 | –0.400 | –1.792 | –0.867 |
| $b_2$ [1/s] | $6.21\times10^{-3}$ | $48.13\times10^{-3}$ | $22.28\times10^{-3}$ | $4.25\times10^{-3}$ | $11.98\times10^{-3}$ |

Table 2. Parameters obtained from fitting for Ag.

| | Thickness | | |
|---|---|---|---|
| | 9 nm | 23 nm | 60 nm |
| $A_1$ [N/m] | –0.333 | –0.615 | 1.206 |
| $b_1$ [1/s] | $1.37\times10^{-3}$ | $4.16\times10^{-3}$ | $7.72\times10^{-4}$ |
| $A_2$ [N/m] | 0.0 | –0.526 | –0.798 |
| $b_2$ [1/s] | 0.0 | $56.4\times10^{-3}$ | $10.6\times10^{-3}$ |

presence of the growth flux. Increase in the chemical potential of the film surface causes the migration of atoms into grain boundaries. Extra atoms in the grain boundaries result in compressive stress in the film. Interruption of growth flux results in rapid decrease in surface chemical potential and migration of atoms onto the surface, decreasing the compressive stress. The changes made by this process are quick. According to that model the islands coalescence process generates tensile stress and is independent of the presence of a growth flux. After deposition, this process also occurs. The island coalescence is slower than the process of extra atoms migration from the grain boundaries to the sample surface.

## 4. Conclusions

We measured the force per unit width ($F/w$) after the UHV deposition of Cu and Ag thin films at room temperature. The $F/w$ ratio versus time is determined from *in-situ* measurements of a sample curvature using the optical system. The experimental data were fitted by the formula consisting of two exponential functions. The form of fitting formula is supported by the model that assumes the presence of two processes of stress generation: migration of excess atoms into grain boundaries resulting in compressive stress and islands coalescence causing tensile stress. The curves obtained from the fitting procedure predict well the stress behavior after the deposition.

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