## Dependence of stress in thin Al films on deposition and post-deposition temperature conditions

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Here we present our study of the stress dependence in Al thin films on deposition conditions. We consider two types of Al 100-nm thick films: E-beam evaporated films and films obtained by magnetron sputtering. We investigate the Al film stress hysteresis in the environment with slowly increasing and decreasing temperature, i.e. during the gradual annealing. We consider the effect of deposition temperature on the film stress and grain size. We conclude that the annealing of Al films results in increased tensile stress component and decreasing of the compressive stress. Additionally, we observe that higher deposition temperature gives higher tensile stress and grain size in the film. In order to recover the film quality and reduce the grain size, one can increase the pressure of the buffering gas during the deposition.

Keywords: e-beam evaporation, sputtering, stress engineering, thin metallic films, microtechnology

In various clean room-based fabrication processes, the deposition of Al thin films requires the best possible surface quality of the film in combination with accurate engineering the intrinsic tensile and compressive stress of Al [1]. For the studies of Al thin film stress and surface quality, we took 380  $\pm$  10  $\mu$ m thick Si wafers (100  $\pm$  0.2 mm diameter, polished, double-sided, crystal orientation 100, resistivity range 0.1 – 0.5 Ohm/cm) and deposited Al thin films on them. We explored several temperature and pressure regimes during the deposition as well as post-processing annealing techniques of Al films on Si substrates.

There are two main ways to deposit Al on the wafer, namely, evaporation and sputtering [2]. Evaporation of any source material occurs at high vacuum (pressure around  $10^{-7}$  mbar). The instruments available at Center of MicroNanoTechnology (CMi) EPFL, namely Alliance-Concept EVA 760 and Leybold Optics LAB 600H, allow Al evaporation using high-energetic (up to 15 keV) electron beam to bombard the Al source, heat it up and evaporate Al atoms to the surface of the wafer. This method allows to get the best possible surface quality; however, the quality dramatically changes when the temperature is higher than RT (see Fig. 1). The second approach is magnetron sputtering, which is available at CMi as well by Alliance-Concept DP 650 and Pfeiffer SPIDER

TABLE I. 100-nm Al film stress and roughness dependence on sputtering temperature.

| T, °C   | $\sigma$ , MPa | $R_{\rm a},{\rm nm}$ |
|---------|----------------|----------------------|
| 20 (RT) | -53            | 2                    |
| 100     | 35             | 10                   |
| 200     | 41             | 15                   |
| 250     | 47             | 17                   |
| 350     | 61             | 20                   |



FIG. 1. Atomic force microscope (AFM) picture of the result of evaporation of 100-nm Al film on Si substrate in Alliance-Concept EVA 760. The temperature of deposition was 200 °C, the roughness parameter is  $R_{\rm a} \approx 60$  nm.

600 instruments. Both of the tools allow to change the temperature of the substrate, thus allowing to modify the intrinsic stress of the material. However, the higher sputtering temperature leads to bigger grain size (see Fig. 2 and Table I), thus, lower quality of the film [3]. In Table I, the negative value of the stress  $\sigma$  designates the compressive stress, positive value is for the tensile stress.

Another way to introduce the additional tensile stress to Al film is to perform so-called thermal cycling or annealing, which can be performed at the Institute of Condensed Matter Physics (ICMP) EPFL. The typical change of the film intrinsic stress upon its heating up to 300°C within one hour, annealing at the same temperature for another three hours and cooling to RT is shown in Figs. 3 and 4. It shows that, even though initially the film has mainly compressive stress, a great amount of tensile stress is introduced after the cycle. This method



FIG. 2. The result of magnetron sputtering of 100-nm Al film on Si substrate in Pfeiffer SPIDER 600 for different temperatures. (left) Photo of the wafers after sputtering of the Al films. (right) Scanning electron microscope (SEM) images the films. One can see the grain size increase due to higher deposition temperature



FIG. 3. Thermal cycle for 100 nm Al film on Si substrate. Tensile stress of the amount of  $\approx 200$  MPa is introduced to the film from initial compressive stress of the amount of  $\approx 25$  MPa.

of stress engineering has though one side effect. During thermal cycling, the Al film material is inevitably exposed to a wide range of temperatures for several minutes or even hours, due to the finite time of heating and cooling. Due to this, according to work [4], the compressive stress is partly lifted.

In work [5], another way to introduce additional tensile stress to  $Al_2O_3$  films was proposed and experimen-



FIG. 4. Thermal cycle for 100 nm Al film on Sapphire substrate. Tensile stress of the amount of  $\approx 200$  MPa is introduced to the film from initial compressive stress of the amount of  $\approx 200$  MPa. Here the Sapphire wafer thickness was 625  $\mu$ m.

tally approved. Before beginning of the sputtering process, the chamber, in which the substrate is placed, is pumped down to high vacuum, and then the buffer gas, usually Ar, is introduced. The approach to increase the stress in  $Al_2O_3$  films consists in increasing of pressure of the buffering gas during sputtering at RT. It turned out that for Al thin films, the same dependence on the buffering gas pressure takes place. Moreover, for greater atmosphere pressures, the grain size reduces from 2 nm to  $\approx 0.7$  nm, which works to the improvement of the film quality.

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