

Draft

## Rapid Selective Annealing of Cu Thin Films on Si using Microwaves

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### Abstract

A major goal of the semiconductor industry is to lower the processing temperatures needed for interconnects in silicon integrated circuits. Typical rapid thermal annealing processes heat the film as well as the substrate, creating device problems. Microwave techniques represent a novel solution to this problem. Selective annealing of metallic thin films or other conductive thin films is possible in the limit where the film thickness is less than the skin depth of the material at microwave frequencies, and is advantageous because the relatively low absorption of silicon deters substrate heating and the metallic thin film shields the rest of wafer,

We present results showing the microwave absorption, Q and temperature, of a silicon substrate as a function of its doping concentration and power input into the microwave cavity. Also, we will microwave anneal sub-micron Cu thin films sputtered on patterned SiO<sub>2</sub>/Si substrates and look at the improvement in trench filling for interconnect metallization.

### Introduction

The minimum feature size in an integrated circuit has been decreasing for an increase in the speed and device density on the chip. This trend is detrimental to the interconnections because the cross sectional area of the interconnection decreases as the device dimension is scaled down, while the length of the interconnection increases as the chip size grows.

Conventional physical vapor deposition (PVD) techniques, such as sputtering and evaporation, have long been used in interconnection metallization. These techniques, however, do not provide a conformal deposition profile, which is essential for the multilevel metallization in high density integrated circuits as sizes enter the sub-micron region and aspect

ratios become greater than 1 (aspect ratio = height/width). In this region, conventional PVD techniques will not fill the vias and trenches on the wafer.

A possible solution to this problem can be found with microwave annealing of interconnect metals. Microwave annealing can be used to selectively heat materials with high microwave absorption coefficients, such as Cu or Al, while leaving the surrounding material with relatively low microwave absorption coefficients, dielectrics or lightly doped Si, unaffected. Hence, a sputtered Cu film on a patterned dielectric can be heated in a microwave cavity and cause 'reflow' of the Cu into the trenches and vias without heating the underlying substrate. This process provides the added benefit of minimizing the interdiffusion of interconnect metals into the surrounding dielectric by lowering the temperature of the dielectric.

### Experimental Procedures

A cylindrical microwave cavity was constructed out of Cu as shown in figure 1. The cavity was designed to have a high-Q and good mode separation for the TMO<sub>10</sub> mode at  $f = 6.67$  GHz. All work was done in this mode. The cavity diameter is 1.350" and the cavity height is 1.855". The unloaded Q of the empty cavity is 13320. The samples to be annealed were cut into 3 mm disks and supported in the center of the cavity with a small quartz 'fork'. An argon getter pump was inserted into the gas flow line to purify the Ar; during heating, the samples were continuously purged with purified Ar. A fast switch ( $\approx 10$  ns) was in the power line from the sweep oscillator to the traveling wave tube (TWT) amplifier. Q measurements during heating were performed by switching the microwave cavity power off for a few microseconds and looking at the reflected power decay. As the sample was heated, the resonant frequency was tracked by a control loop that samples the reflected power and adjusts the frequency to minimize it.<sup>1</sup> An optical pyrometer was aligned through a sealed window in the top of the cavity to measure the temperature of the sample.

Cu films were grown in a load lock-equipped ultrahigh vacuum ion beam sputtering system with base pressure in the low  $10^{-9}$  Torr regime. 0.8  $\mu\text{m}$  to 1.0  $\mu\text{m}$  thick Cu films were deposited onto patterned SiO<sub>2</sub>/Si substrates by Ar-ion beam sputtering at a deposition rate of 0.06 nm/sec as estimated from an oscillating-crystal thickness monitor. These substrates were then taken out of the vacuum system and cut into 3 mm disks and annealed in a microwave cavity for 30 seconds with an input power of 1.2 W.

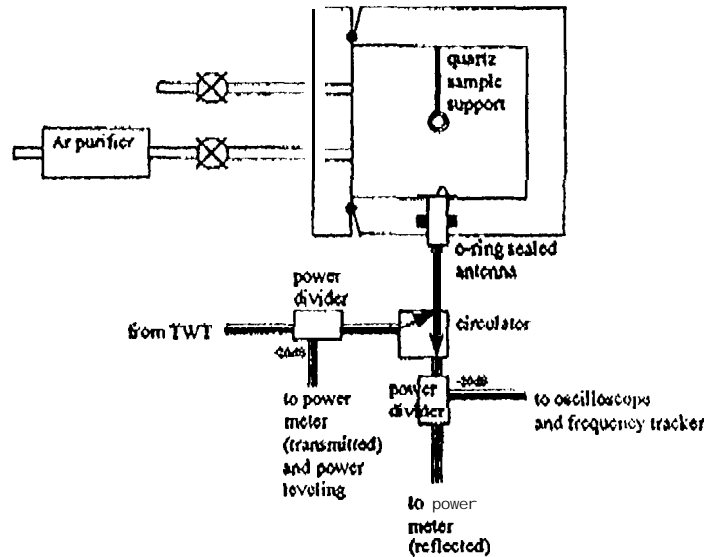


Figure 1: Cross-sectional view of microwave cavity from top. The antenna is sealed with an o-ring and is adjustable to enable critical coupling to cavity. Above and below the quartz sample support are sealed quartz windows to enable temperature measurements with an optical pyrometer.

## Results and Discussion

Silicon samples of two different resistivities were examined to measure their  $Q$  dependence and temperature dependence as a function of power input into the cavity, Figure 2 shows the resulting  $Q$  measurements and temperature measurements for samples with  $\rho = 10.8 \Omega \cdot \text{cm}$  and  $\rho \approx 1000 \Omega \cdot \text{cm}$  as a function of power input into the critically-coupled cavity. The pyrometer's minimum resolvable temperature is  $300^\circ \text{C}$ ; a reading of  $300^\circ \text{C}$  in this figure indicates that the sample temperature was at or below this value. For both samples, an emissivity of 0.73 was used. The sample with  $\rho = 10.8 \Omega \cdot \text{cm}$  has a 'normal' heating curve; as the power input into the cavity increases, the temperature increases and the  $Q$  increases as well. The sample  $Q$  increases at high temperature because the center of the sample is being increasingly shielded as the conductivity of the silicon becomes large. At high temperatures, the rate of temperature increase is quenched because the sample center is no longer being heated due to shielding. The sample with  $\rho \approx 1000 \Omega \cdot \text{cm}$  shows a more neurotic heating behavior. As the power is increased from zero, the  $Q$  of the cavity decreases and the temperature correspondingly stays at or below  $300^\circ \text{C}$ . In this region, the skin depth is

greater than the sample thickness and the absorption of the sample is increasing with increasing temperature, hence the  $Q$  decreases. Abruptly at 1.0 W, the sample undergoes thermal runaway and the  $Q$  drops from 3000 to 1400 and simultaneously the temperature increases from  $\leq 300$  °C to 400 °C. At this point, it is necessary to do a large impedance adjustment (made by adjusting the antenna's position in the cavity) to remain critically coupled. As the input power is increased further, the sample  $Q$  and temperature increase in the same manner as the  $\rho = 10.8 \Omega\cdot\text{cm}$  sample. As the power is decreased from 3.0 W towards zero, a similar catastrophe occurs at 0.4 W. At this point, the  $Q$  of the sample increases from 700 to 2900 and the temperature remains below 300 °C. This hysteretic behavior is shown by the arrows in figure 2. This behavior can be explained by considering the electric field's interaction with a sample that has an exponentially increasing conductivity as a function of temperature, and by including heat transfer to the surroundings as well as the thermal conduction within the sample. A detailed analysis of this hysteretic behavior for thick slabs with exponentially increasing conductivities is presented by Kriegsmann<sup>2</sup>. Fortunately, for the sake of microwave processing of thin films on silicon, the silicon wafers commonly used as substrates in integrated circuits have  $\rho \approx 50 \Omega\cdot\text{cm}$  and have a 'normal' heating curve,

Next we annealed 0.8  $\mu\text{m}$  to 1.0  $\mu\text{m}$  Cu thin films deposited on a patterned  $\text{SiO}_2/\text{Si}$  sample with grooves 0.8  $\mu\text{m}$  deep and widths ranging from 0.2  $\mu\text{m}$  to 1.0  $\mu\text{m}$ . The resistivity of the Si substrate is  $\rho \approx 50 \Omega\cdot\text{cm}$ . A scanning electron microscope (SEM) picture of the initial grooved surface is shown in Figure. 3. Figure 4 shows the patterned sample after Cu deposition. It is clear from these pictures that the Cu filling of these trenches is unacceptable as interconnect metallization; for aspect ratios around 1:1 there are clear gaps in the Cu and as aspect ratios become greater than 2.5:1, there is hardly any Cu in the trenches. Figure 5 shows SEM photos of the Cu films after microwave annealing for 30 seconds with an input power of 1.2 W. During the annealing procedure, the temperature of the Cu surface was being monitored with the optical pyrometer and also by eye. The pyrometer showed the film temperature to be less than 300 °C (using an emissivity of 0.13) and observation of the films in a dark room showed them to be radiating at a wavelength lower than that observable by eye indicating that the temperature was less than 500 °C.<sup>3</sup> Transmission electron microscopy (TEM) photos of the Cu films were taken after microwave annealing. These pictures are shown in figure 6. The average grain size of the Cu is 0.9  $\mu\text{m}$

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