

Construction of a Substrate for Characterizing the Real-Time Electrical Conductivity of Thin Films as Deposited

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Abstract:

Atomic layer deposition (ALD) is presently employed in important semiconductor manufacturing processes, which include the deposition of ultrathin layers for gate dielectrics and diffusion barriers for interconnects. As many of the exciting applications of ALD involve forming ultrathin, atomically thin films, depositing a conformal and continuous ALD film with a controlled thickness is critical to performance. Moreover, the ability to assess the film properties *in situ* is essential to avoid the complications that arise when samples are exposed to laboratory air. In this work, we designed and fabricated a substrate with patterned electrodes, which allowed *in situ*, real time electrical measurement of ALD films being deposited in a custom-built microreactor. Fabrication of the substrate consisted of two patterned layers on thermal oxide: electron beam evaporated gold (with a chromium adhesion layer) followed by plasma-enhanced chemical vapor deposition (PECVD) of silicon dioxide (silica) as an insulating, protecting layer. Feature size and film morphology were confirmed by profilometry and scanning electron microscopy (SEM), respectively.

Introduction:

A typical atomic layer deposition (ALD) process starts with exposing a surface to a gaseous organometallic precursor, followed by an inert gas purge. This process reacts with the surface and thus leaves a surface layer of reacted molecules. This layer of atoms is then exposed to a co-reactant such as water or oxygen, followed by a second purge cycle. This process repeats until the desired thickness is produced. The ALD process does not necessarily produce a continuous monolayer every cycle. Having a continuous film is important when making electrical contacts or diffusion barriers. Our project aimed to detect the number of cycles of ALD needed to produce a continuous film using non-linear four-point probe electrical conductivity measurements as outlined in Figure 1.

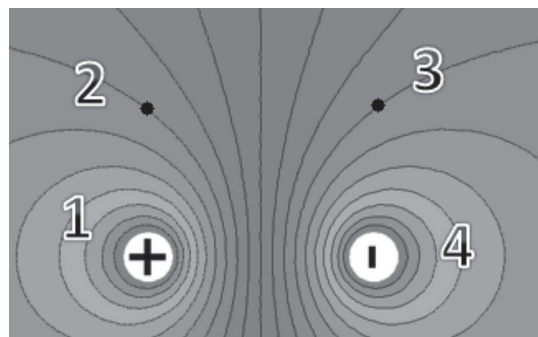


Figure 1: Non-linear four-point probe setup for substrate showing equipotential lines and points of measure. Image Courtesy zureks@gmail.com.

Experimental Procedures:

The custom-designed microreactor in the Engstrom research group allows real time electrical measurement during vapor-phase deposition/reaction, including chemical vapor deposition (CVD) and ALD. The 4-point probes are in a rectangular geometry of 3.8 cm by 2.2 cm. We therefore designed a patterned electrode on a 4-inch wafer based on the probe geometry shown in Figure 2.

Four gold pads (1 cm × 1 cm) were contacts to the probes, and each pad had a lead wire (50 μm wide) heading to central reaction zone (0.1 cm × 0.1 cm). The reaction zone provided a flat substrate surface for a continuous ALD film to be deposited on without possible effects due to the topology, which could then be

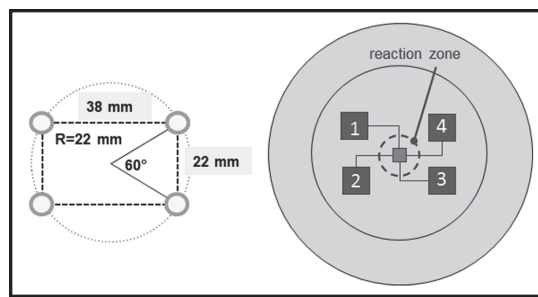


Figure 2: Substrate layout for four-point probe with contact pads with wires connecting to electrodes in the reaction zone.

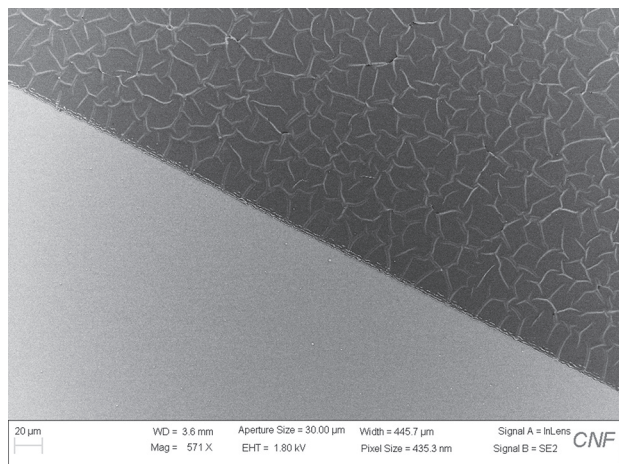


Figure 3: Silica deposited via electron beam evaporation on gold with no adhesion layer.

used to accurately observe the point where an ALD film was continuous on a two-dimensional surface.

Substrates were produced with 100 mm silicon wafers with a surface layer of 500 nm of thermal oxide. The wafers were then coated with a lift-off resist and baked at 180°C for 300 s, followed by coating with photoresist and baking at 115°C for 180 s. The substrates were then UV exposed on an ABM contact aligner to produce a pattern for the substrate electrodes and developed for 90 s with MIF-726. Substrates then were coated with 10 nm Cr|100 nm Au|10 nm Cr via electron beam evaporation, and soaked in 1165 stripper for not less than six hours to lift off the metal.

Similar processes were employed for the deposition of a 500 nm layer of plasma-enhanced chemical vapor deposited (PECVD) silica, after which the substrates were submerged in chrome etch solution to remove the exposed chromium adhesion layer.

Results and Discussions:

Profilometry of the samples revealed that thicknesses of the samples after evaporating chrome and gold layers varied within 11% of the target thickness, which suggested good reproducibility of the process. Optical microscopy of the metal films revealed small features on the surface of the film, indicating metal spitting occurred during evaporation. SEM images of the gold electrodes reaffirmed that spitting was occurring during evaporation of the gold. By fine tuning the process parameters, the quality of the e-beam evaporated metal layer was greatly improved: Instead of using a single deposition rate throughout the evaporation process, we found that a smooth surface could be generated by slowing down the deposition rate from 1.0 Å/s to 0.3 Å/s for the last 20 nm.

Evaporated silica was initially chosen for depositing dielectric layer on top of the electrodes, and scanning

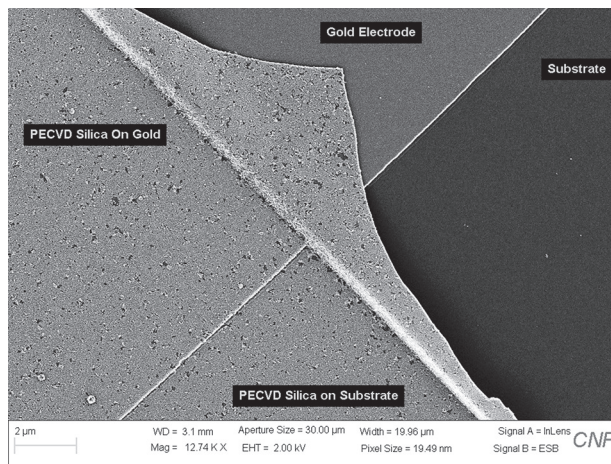


Figure 4: PECVD silica on gold with 10 nm chromium adhesion layer, after lift-off and prior chromium etching.

electron microscopy (SEM) images displayed in Figure 3 revealed crazing of the oxide on gold after deposition. We demonstrated that the craze can be alleviated by an additional chromium adhesion layer on top (i.e., $\text{SiO}_2|\text{Cr}|\text{Au}|\text{Cr}|\text{SiO}_2$).

Furthermore, PECVD deposition of silica gave a denser film that adhered better despite being more difficult to lift-off and required damaging the film manually with tweezers. Floating silica left behind after lift-off was observed by electron microscopy shown in Figure 4, which again demonstrated the process difficulty resulting from the density of PECVD silica layer. The dimension of the floating silica is three orders of magnitude smaller than that of the reaction zone, thus was of no concern to the performance of the substrate.

Conclusions and Future Work:

With the above characterization, we show that substrates with patterned electrodes for real time electrical measurement of ALD film were made as designed. Deposition of thin films should be performed *ex situ* using the IV-probe station in the clean room and *in situ* electrical measurements in the Engstrom lab to complete the objective of this work.

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