# GROWTH AND STRUCTURE OF METALLIC BARRIER LAYER AND INTERCONNECT FILMS I: EXERIMENTS

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## ABSTRACT

We present experimental results directed at understanding the growth and structure of metallic barrier layer and interconnect films. Numerical simulation results associated with this experimental work are presented in an accompanying paper in these proceedings. Here, thin films of Al, Ti, Cu and Ta have been grown by magnetron sputtering onto oxidized Si substrates. Using a specially-constructed substrate holder, the orientation of the substrate with respect to the growth direction was varied from horizontal to vertical. Films were grown at both low and high argon pressure; in the case of Ta, the cathode power was varied as well. The film structure and in particular the surface roughness was measured by X-ray reflectance and also by atomic force microscopy. We find that the surface roughness increases markedly with orientation angle in the case of Ta and Cu films, and in Ti films grown at high argon pressure. At low pressure, however, the Ti film surface roughness remains constant for all substrate orientations. No variation in roughness with either orientation angle or argon pressure was observed in the Al films. These results suggest that, under certain circumstances, shadowing effects and/or grain orientation (i.e., texture) competition during growth can give rise to lower density, more porous (and thus more rough) films, particularly at large orientation angles, as on sidewalls in sub-micron trenches.

## INTRODUCTION

The electronic properties of metal films such as Al and Cu used as interconnects in integrated circuits depend on film microstructure. In particular, the resistivity will increase with increasing porosity (as a result of decreasing density). Non-specular electron scattering from surface and sidewall roughness might also increase resistivity, particularly for sub-0.1-micron wires. Increased porosity can also compromise the performance of metallic barrier layer films such as Ta, intended to prevent diffusion of Cu into the underlying material. The purpose of the current work is thus to investigate experimentally how the microstructure of metallic films grown by magnetron sputtering can vary over topography. We also hope to develop improved film growth simulation capabilities using the results of our experiments in order to correctly model and thus find ways to optimize growth over topography (and hence optimize performance and reliability) of metallic barrier layer and interconnect films.

We present here the results of a systematic experimental investigation intended to quantify how the microstructure of sputtered Al, Ti, Cu and Ta films varies over topography. Numerical simulation results associated with this experimental work are presented in an accompanying paper in these proceedings. Thin films of Al, Ti, Cu and Ta were grown by magnetron sputtering in argon onto Si substrates containing a native oxide. Films were grown at both low and high argon pressure, and in the case of Ta, the cathode power was varied as



Mat. Res. Soc. Symp. Proc. Vol. 564 © 1999 Materials Research Society

well. Using a specially-constructed substrate holder, the orientation of the substrate with respect to the growth direction was varied from horizontal to vertical, in order to simulate sidewall growth while also producing macroscopic samples conducive to precision X-ray reflectance analysis and atomic force microscopy. The results of these experiments are described below.

## EXPERIMENT

The films described here were grown by DC magnetron sputtering in argon (99.999% purity), using a deposition system shown schematically in Figure 1. Both a turbo-pump and a cryo-pump are used, and the consequent background pressure in the chamber prior to deposition was in the range $5 \times 10^{-7} - 5 \times 10^{-6}$ Torr in all cases. A variable orifice throttle valve separates the two pumps from the main chamber, and is operated in conjunction with a closed-loop gas-flow system using a capacitance manometer and a mass-flow controller in order to control the argon pressure during deposition. S-Gun[1] cathodes using 1.85-inch-diameter cylindrical targets of either Al (99.999 % purity), Ti (99.995% purity), Cu (99.999% purity) or Ta (99.9% purity) are mounted in the base plate of the vacuum chamber. Substrates are mounted on a platen that faces downward, located 110 mm above the top surface of the target; film thickness is adjusted by varying the (computer-controlled) rotational velocity of the substrate platen as it travels over the cathode. An aperture located 95 mm above the target is used both to improve source collimation and coating uniformity.

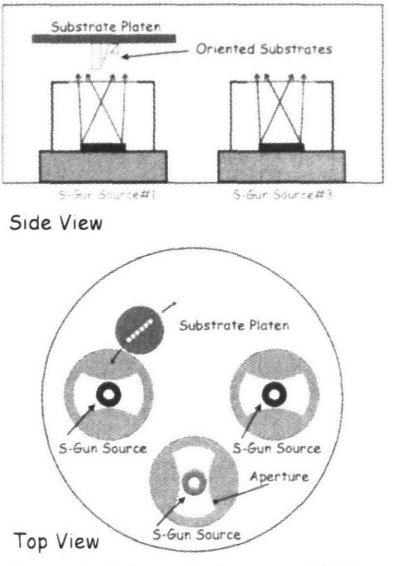


**Figure 1.** Schematic diagram of S-Gun sputtering system.

Films were grown on (unheated) Si (100) wafer sections (~1.4 x 1.4 $cm^2$) having a thin (~2-3 nm) native oxide layer. The wafer sections were mounted (using double-sided tape) on a holder, shown in Figure 2, consisting of trapezoidal blocks that are used to vary the orientation angle of the substrate relative to the growth direction. Fixed orientation angles of 0° (i.e., horizontal), 30°, 60°, and 85° were used.

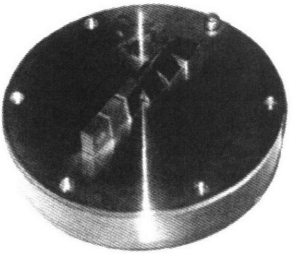

**Figure 2.** Substrate platen for deposition onto oriented substrates.

The power applied to the cathode was fixed at 100 W in the case of Al, Ti, and Cu, and ranged from 100 W to 400 W in the case of Ta. The argon pressure was fixed at either 2 or 10 mTorr. The resulting deposition rates, determined from the film thicknesses (0° orientation angles) deduced by X-ray reflectance measurements (described below) are shown in Table 1.

Film thicknesses for the 0° orientation samples ranged from ~20-35 nm; thinner films were obtained at larger orientation angles, as described below.

| Material | Argon Pressure [mTorr] | Cathode Power [W] | Deposition Rate [nm/s] | XRD |
|---|---|---|---|---|
| Al | 2 | 100 | 0.96 | fcc: (111) |
| | 10 | 100 | 0.73 | |
| Ti | 2 | 100 | 0.53 | hcp: (0002) |
| | 10 | 100 | 0.43 | |
| Cu | 2 | 100 | 2.41 | fcc: (111) and (200) |
| | 10 | 100 | 2.42 | |
| Ta | 2 | 100 | 0.93 | bcc: (110) and (211) |
| | 2 | 200 | 1.76 | |
| | 2 | 400 | 3.27 | |
| | 10 | 100 | 0.82 | |

**Table 1.** Deposition conditions, deposition rates, and diffraction peaks observed by XRD for films deposited at 0° orientation..

X-ray reflectance (XRR) and X-ray diffraction (XRD) measurements were made in the θ-2θ geometry using a four-circle diffractometer with a rotating anode X-ray source having a Cu target, and a pyrolytic graphite monochromator tuned to the Cu-Kα line near 8 keV (1.54Å.) The angular resolution of the diffractometer is ~0.02°. Fits to the XRR data, performed with the IMD software package[2], are used to determine film thickness, surface roughness, and interface widths (i.e., resulting from interfacial roughness and/or diffuseness between the film and the substrate, and between the film and the oxide that forms during exposure to air). With this technique, the measured data is compared with a theoretical reflectance curve computed using an algorithm based on recursive application of the Fresnel equations; the formalism described by Stearns[3] is used to account for the effects of interface imperfections, and an error-function interface profile was assumed. Atomic force microscopy (AFM) was also used to determine surface roughness: measurements were made on selected films using a Digital Instruments Nanoscope III operated in the tapping mode, with both 1.0 micron and 0.1 micron scan lengths.

## RESULTS

Shown in Figure 3 are typical XRR curves, in this case for Ta films grown at an argon pressure of 2.0 mTorr, and with the cathode power fixed at 400 W, at four different orientation angles: the measured reflectance curves are shown as solid

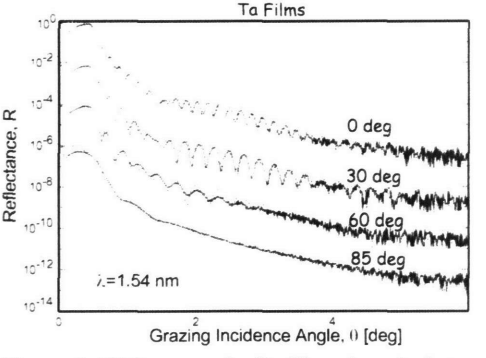


**Figure 3.** XRR curves for Ta films deposited at 400 W cathode power, and with $P_{Ar}$=2 mTorr, at the orientation angles indicated. Fits to the data are shown as dotted lines.

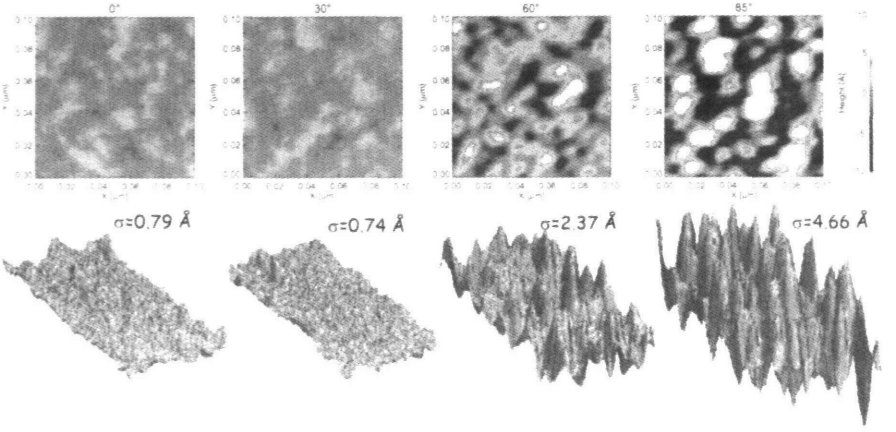


**Figure 4**. Typical AFM data (0.1 micron scan length), in this case for Ta films deposited with $P_{Ar}$=2.0 mTorr (with 100 W of power,) as a function of orientation angle as indicated. The scale is the same in all four cases, and the rms surface roughnesses (σ) are labeled.

lines and the fits to these data as dotted lines. The frequency of the oscillations evident in each reflectance curve depends largely on the film thickness, while the amplitude of the oscillations depends strongly on the surface and interface roughness.[4] Figure 3 shows that (a) the thickness decreases and (b) the roughness increases with increasing orientation angle. The variation in surface roughness is also evident in the AFM data[5] for the Ta films shown in Figure 4.

The measured thickness and roughness values for the Ta films shown in Figs. 3 and 4 are plotted vs. orientation angle in Figure 5(d); equivalent data for Al, Ti, and Cu are shown in Figs. 5(a-c). The grain orientations identified by XRD for films deposited at 0° orientation are indicated in Table 1. A number of trends are evident in these data:

First, the variation in thickness with orientation angle (θ) does not follow the cosθ distribution, as is often assumed.[6] Part of this discrepancy can be due to imperfect source collimation, in spite of the use of the deposition aperture described above.

Second, in all cases except Al, films deposited at high Ar pressure are rougher than those deposited at low Ar pressure. The variation in roughness with Ar pressure observed here is by now widely-known to occur for sputtered films[7,8,9], and results largely from the dependence of the deposition energetics on Ar pressure. That is, at low Ar pressure, neutral Ar atoms reflected from the target can arrive at the surface of the growing film with energies as high as ~100 eV; at high Ar pressure, however, collisions tend to thermalize the gas phase, so that the highest-energy neutrals are no longer incident on the growing film. The larger incident kinetic energies associated with lower Ar pressures tend to produce high density, smooth films through the so-called 'atomic peening' effect.[10] The kinetic energy of reflected neutral Ar atoms also depends on the ratio of the Ar:adatom mass ratio, and this dependence is born out (approximately) in the observed variation in the reduction in roughness with decreasing Ar pressure for the Al, Ti, Cu, and Ta films (0° orientation angle) shown in Fig. 5.

Third, in addition to the increase in roughness with orientation angle in the Ta films described above, we find a similar though somewhat less pronounced result in the case of Cu films, and also in the case of Ti films grown at high argon pressure. At low pressure, however,

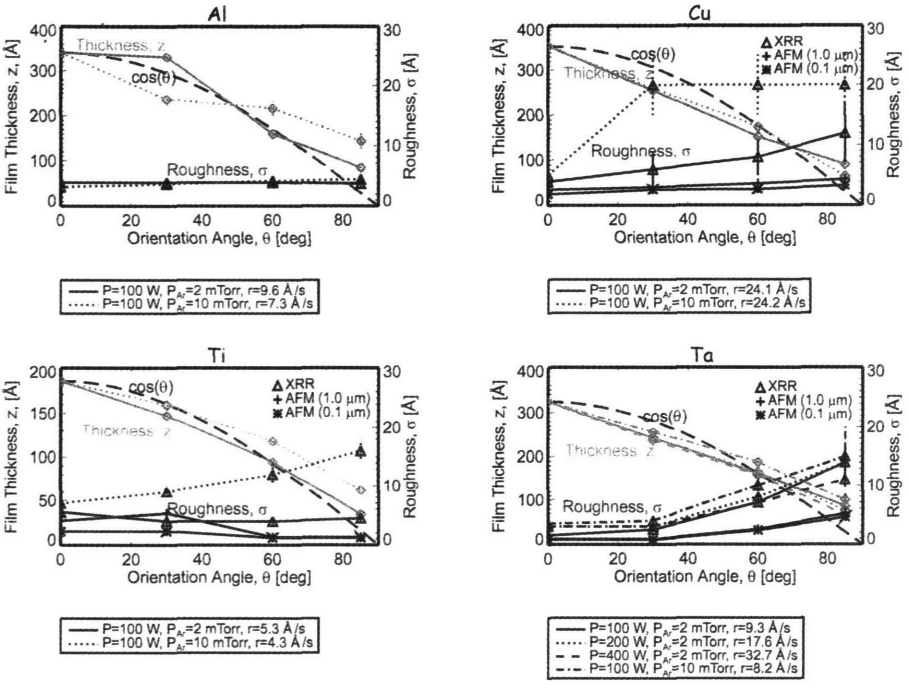


**Figure 5.** Thickness and roughness values, determined from XRR and AFM, for Al, Ti, Cu, and Ta films deposited under the deposition conditions indicated, as a function of orientation angle.

the Ti film surface roughness remains constant for all substrate orientations. No variation in roughness with either orientation angle or argon pressure was observed in the Al films.

The increase in roughness with orientation angle observed in some films (Fig. 5) might be due to shadowing at non-zero orientation angles during growth, an effect which can give rise to lower density, more porous (and possibly more rough) films.[11] The importance of this effect will depend on a number of factors, including adatom surface mobility and wetting, the partial pressure of residual impurity gas atoms (which can effectively reduce adatom surface mobility) present in the vacuum during growth, and the amount of energy delivered to the surface of the growing film by reflected neutral Ar atoms. In addition to the shadowing effect, the surface roughness can also depend on the competition between growth of different grain orientations (i.e., texture,) which in turn can be affected also by surface mobility, residual gasses, and Ar energetics. As is evident from the XRD data in Table 1, neither the Cu nor Ta films are perfectly textured, and so this effect might be important for these materials in particular. In any case, these mechanisms are more fully discussed in the accompanying paper describing our simulation work.

## CONCLUSIONS

We have grown thin films of Al, Ti, Cu and Ta by magnetron sputtering onto oxidized Si substrates, and have varied the orientation of the substrate with respect to the growth

direction from horizontal to vertical in order to simulate growth over topography. We find from X-ray reflectance and atomic force microscopy that the film roughness increases with orientation angle in the case of Ta and Cu films, and also in the case of Ti films grown at high argon pressure. At low Ar pressure, however, the Ti film surface roughness remains constant for all substrate orientations. No variation in roughness with either orientation angle or argon pressure was observed in the Al films. Our results suggest that, under certain circumstances, shadowing effects and/or the competition between growth of different grain orientations can give rise to lower density, more porous (and thus more rough) films, especially at large orientation angles (as on sidewalls). Such films, if used as barrier layers or interconnects, will likely have poor performance and reliability.

It is the objective of our simulation work, described in an accompanying paper, to determine the relative importance of each of the factors (e.g., adatom surface mobility, wetting, the partial pressure of residual impurity gas present in the vacuum during growth, and the amount of energy delivered to the surface of the growing film by reflected neutral Ar atoms) that can influence the shadowing and texture effects proposed above. In addition, we are currently working to produce high-resolution cross-sectional TEM images of the films shown in Fig. 5, which may help elucidate the cause of the observed variation in roughness with orientation angle presented here.

## ACKNOWLEDGEMENTS

We wish to acknowledge the support and encouragement of W. L. Brown, E. Martinez, and C. J. Case of Bell Labs. We also appreciate the support for part of this work provided through an NSF/DARPA VIP contract administered through the University of Illinois, and by the U. S. Department of Energy, Office of Science, Laboratory Technology Division under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp. and contract DE-AC05-76OR00033 with Oak Ridge Associated Universities.

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4. Note, however, that for thicker films, the amplitude of the oscillations are also reduced as a result of the finite instrumental angular resolution.
5. The rms surface roughness values determined by AFM are consistently less than those determined from XRR analysis. This discrepancy is due to the difference in spatial frequencies sampled by each technique. See, for example, D. L. Windt, W. K. Waskiewicz, and J. E. Griffith, *App. Opt.*, 33, 2025-2031 (1994)
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