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ABSTRACT

The electrical resistivity of thin films of Ta and TaN has been examined as a function of film thickness and other processing parameters. The films were deposited by magnetron sputtering of Ta cathodes with Ar, or mixtures of Ar and N₂ for the case of TaN. The TaN films were deposited in a broad range of compositions, depending on reactive gas flow and system configuration, and showed strong increases in resistivity as thickness was decreased. Ta films deposited on silicon dioxide or Cu surfaces were always beta-phase, and showed little resistance increase as the thickness was reduced. Ta films deposited on TaN with greater than 30% N and with thickness greater than 2 nm were alpha phase, and showed significant increases in resitivity as thickness was reduced. The application and electrical properties of these films as diffusion barriers in interconnect structures will depend strongly on the thicknesses at the contact points at the bottoms of vias and only weakly on the phase of the Ta. The resistivity of the barrier films on via and trench sidewalls is high enough to be irrelevant to circuit performance.

INTRODUCTION

With the transition from Al(Cu) to Cu metallurgy for interconnect applications, it was necessary to include additional thin film layers in the structure or during the fabrication. The role of these films is to enhance the adhesion of the Cu to the dielectric, to prevent out-diffusion of Cu into the dielectric or elsewhere, to prevent in-diffusion of oxygen-containing species to the Cu, and also to allow subsequent electroplating of the Cu circuit elements; either vias, lines or both. While layers of Ti and TiN were used for some of these purposes in the Al(Cu) metallurgy system, most work in the Cu system has reported the use of Ta and TaN, either individually or as a bi-layer structure (1). These films are generically called diffusion barrier, barrier or liner films, although they can have many roles.

The bi-layer structure adopted by IBM consists first of a deposition of TaN directly onto the dielectric following the appropriate via or trench etching and any required post-RIE cleaning steps. Subsequent to the TaN, a layer of Ta is deposited which is then followed by a Cu seed layer. The TaN provides multiple functions in this case: (a) its adhesion to silicon dioxide and many low-k dielectrics is good, (b) it has moderate diffusion barrier properties, and (c) it tends to allow, in many cases, the subsequent deposition of Ta to occur in the 'alpha' phase (b.c.c.) of Ta, which has 5-7x lower electrical resistivity than the 'beta' (tetragonal) phase of Ta. Directly depositing Ta onto oxide or dielectric surfaces tends to result in the higher resistivity beta phase and only modest adhesion. Surprisingly, the beta phase of Ta is only observed in thin film form and is thought to be metastable. Following the alpha-phase Ta deposition, which serves both as a diffusion barrier as well as an adhesion layer for Cu, the Cu seed layer is deposited, and the

wafer then undergoes thick Cu electrodeposition in a separate system which fills the trenches and vias.

The deposition processes for these three layers has used PVD technology, which has evolved from conventional, planar PVD through collimated sputter deposition, and currently to various forms of ionized-PVD, or I-PVD (2). Each succeeding evolution of PVD has been required due to the increase in the aspect ratio of the interconnect vias and trenches, as well as their declining lateral dimensions. The thicknesses of the TaN, Ta and Cu layers have also tended to scale with the declining feature size. The deposition of these materials is generally measured in the planar or 'field' areas between the inset trenches or vias. Typically, the TaN and Ta field thickness ranges from 15 to 50 nm and the Cu seed layer thickness is in the 70-150 nm range, again depending on feature size and aspect ratio. The functional thicknesses, though, on the sides and bottoms of vias and trenches are usually only 10-20% of the field thickness. This is due to the solid-angle limitations of directional PVD technology and the moderate-to-high sticking coefficients of the sputtered atoms.

The general road map plans for interconnect technology include ever-shrinking lateral dimensions as well as modest increases in the aspect ratio of the vias. Eventually the dimensions of the vias and lines start to approach the mean-free-path for electron scattering, which for Cu is about 38-40 nm. This results in proportionately increased electron scattering from the surfaces of the features and a slow increase in the electrical resistance as line-width decreases, an effect often described as the 'size effect.' This has been an anticipated problem for Cu for many years, and starts to become a significant issue below 100 nm feature size.

It has generally not been recognized, though, that the diffusion barrier and liner films also exhibit size effects. While the electron mean free paths in many relevant barrier materials are not known well, they appear to be in the same range as Cu and Al; tens of nm, based on approximations of the fermi velocity and electron densities, as well as some published work (3,4). This becomes critically important when the film thicknesses are well below the mean-free-path length, which is most likely the case for the TaN and Ta applications cited here. In addition, due to the desire to maximize the Cu conductivity in future interconnect lines and vias, the diffusion barrier, liner and seed layers must be reduced in thickness in succeeding generations, further exaggerating the size effects on the resistance. This paper examines the various size-related effects of TaN and Ta in the thickness ranges relevant to interconnect technology in the sub-100nm generations.

2.0 EXPERIMENTS

Most of the measurements cited in this manuscript are of planar films deposited by magnetron sputtering, known generically in the semiconductor industry as PVD. Parallel work with Ta and TaN films deposited with Atomic Layer Deposition (ALD) is discussed elsewhere (5,6). The magnetron depositions used conventional manufacturing systems, typically single-wafer deposition chambers with circular, planar, rotating-magnet cathode of approximately 30 cm diameter designed for 200 mm wafer applications. The various PVD methods employed included conventional PVD, collimated PVD and ionized PVD; the latter case using rf-coils immersed in the plasma between the cathode and the sample. Deposition rates were determined by measuring scribed steps in thick deposits using a surface profilometer. TEM and SEM cross-sections were

also made of some samples as a means of confirming film thicknesses. For most of the samples, sheet resistance was measured ex-situ immediately following deposition with a conventional 4-pt. Probe. Some electrical resistance measurements were also made in-situ by measuring the current at constant voltage across two Pt electrodes separated by 15mm on a SiO₂ surface.

2.1 TaN

TaN was deposited by reactive sputtering, using a pure (99.95%) Ta cathode and a background gas mixture of high purity (99.9999%) Ar and (99.999%) N2. Unlike the earlier TiN system, TaN has a wide variety of possible compound compositions, such as Ta₃N₄, Ta₄N₅, Ta₅N₆, TaN and Ta₂N, and the result is a significant variation in film composition and electrical resistivity as a function of the nitrogen level in the plasma at constant film thickness (Fig. 1). The XRD theta-two-theta plot of TaN with 30% nitrogen is shown in Fig. 2, and shows a broad peak consistent with multiple TaN_x compounds. To complicate the characterization of the TaN, the presence of shields, wafer clamps and collimators in the deposition chamber can alter the local nitrogen composition at the film, which has an immediate effect on the film composition and resistance. In Figure 1 for example, the addition of shields near the wafer results in significant consumption of nitrogen in the films deposited there (farthest-right, dashed-line curve), reducing the nitrogen level in the film deposited on the wafer substrate. In the deposition system, the nitrogen is introduced underneath the wafer to help facilitate thermal contact of the wafer with the substrate platform. If the substrate holder is moved up into the shielding region (farthest left curve in Fig. 1), the nitrogen level in the film is increased because the nitrogen is now introduced only into the smaller volume inside the shielded area. The implication of these observations is that the deposited films are unsaturated with nitrogen and will absorb (pump) the nitrogen locally, reducing the overall chamber density. Another implication of this is that the nitrogen concentration of TaN films in deep vias or trenches will probably decline farther down into the feature, as the gaseous nitrogen flux is absorbed on the upper sidewalls.

A related effect is observed with I-PVD of TaN, in which due to the ionization and acceleration of nitrogen ions to the film surface, the incorporation rate of nitrogen in the films at a given nitrogen level in the gas is enhanced over collimated or conventional PVD, resulting in higher sheet resistance. In effect, in I-PVD deposition of TaN, the curves in Figure 1 are shifted to the left. This will also have an effect on the local TaN composition in a deep via or trench due to the directional nature of the impacting nitrogen ions. The flat surfaces at the via or trench bottom will be directly bombarded with nitrogen ions, leading to a higher nitrogen level in the film at these locations compared to the lower sidewall areas, which only receive an attenuated nitrogen flux from the gas phase.

The stability of the TaN films in air was good at low (<20%) nitrogen levels. At high nitrogen levels (30% N or higher), and concurrently high electrical resistance, the film's sheet resistance began to increase immediately upon exposure to air as the films most likely absorb oxygen or water vapor (Fig. 3). At the higher nitrogen levels measured, consistent with roughly 50% nitrogen in the films, the sheet resistance increased approximately 2x in less than 1 hour of air exposure. This effect of air exposure will also alter the properties of Ta films deposited on the TaN, as described below.

The sheet resistance of TaN was measured as a function of film thickness (Fig. 4) immediately following deposition for a TaN composition of approximately 30% nitrogen, which is similar to the composition used in manufacturing applications and has a thick-film (>50 nm) resistivity of7 500 micro-Ohm-cm.. Films were only measured to a minimum thickness of 2 nm, but as seen in Fig. 3, the sheet resistance increase from 5 to 2 nm is over an order of magnitude. The thinnest films were very unstable in air and oxidized rapidly.

Because of the instability of TaN in air below 10 nm thickness, in-situ measurements were made of 2-electrode planar substrate structures at constant voltage (5 V). The electrodes were sputter-deposited Pt films on SiO₂/Si substrates separated by 15 mm. Current through the TaN film was then measured in the deposition chamber as a function of film thickness. The initial resistivity for the 2 nm TaN film is 5.5×10^4 micro-Ohm-cm, approximately 40% lower than the ex-situ measurement in Fig. 4 which was taken a few seconds after air exposure. Nevertheless, this is a very large resistivity for a conducting barrier film. The calculated series resistance of a film of this thickness (2nm) at the bottom of a 100 nm diameter via is over 100 Ohms; much too high to be useful in an interconnect application. For comparison, the resistance of a 2:1 aspect ratio Cu via at 100 nm diameter is 0.5 Ohms.

2.2 Ta

Deposited Ta films usually are generally characterized as having either an alpha (b.c.c.) or beta (tetragonal) phase configuration, or occasionally combinations of alpha and beta phases. The

electrical resistivity is strongly dependent on phase: the alpha phase generally has a low resistivity (when sputter-deposited) of roughly 1.5-2x the bulk value of 13 micro-ohm-cm. The beta phase has a resistivity in the 150-250 micro-ohm-cm range (1, 4). It has been observed, first by Finestein et al. (7) in the 1970's and later by others (1), that the deposition of Ta on oxide surfaces tends to lead to the beta, or high-resistance phase. This was also observed in the current study, where Ta films on bare silicon dioxide showed resistivities of 180 to 260 micro-ohm-cm as a function of thickness (Fig. 5) and by XRD showed essentially 100% beta-Ta (002). What is somewhat surprising about this data is the modest increases in the observed resistivity as the film thickness becomes quite thin. At an average film thickness of 1.5 nm, the resistivity has only increased by 40% over the nominal bulk value of 180 micro-Ohm-cm. From the scaling of resistance at small dimensions, this suggests that the mean free path for electrons in TaN is around 1.5-2 nm, which is well below that of conductors such as Cu or Al, which are 20x and 10x larger, respectively. This will make the application of beta-Ta in very thin diffusion barrier films less deleterious than many other materials, such as alpha-Ta or alpha-W.

It was also observed by Finestein et a. that Ta deposited on tantalum nitride or some metal surfaces tended to take the alpha phase (7). The minimum level of nitrogen in the TaN can be inferred from Fig. 6, which shows the sheet resistance of a constant thickness (145 nm) Ta film deposited on top of a 5 nm TaN film where the working flow of nitrogen was varied. The nitrogen concentration is not plotted for each flow rate. However, films deposited in this particular vacuum system without shields showed approximately 30 atomic percent of nitrogen for a nitrogen flow of 30 sccm. The Ta films deposited with too-low N in the TaN were consistent with beta-phase Ta. An example of the minimum thickness of TaN required is shown

in Fig. 7, where at least 2 nm of TaN (30% N), was required to facilitate deposition of the Ta in the alpha-phase.

The resistivity of alpha-Ta, deposited on a constant thickness 3 nm TaN(30%) underlayer was then measured as a function of Ta thickness (Fig. 8) for films down to 3.5 nm. The parallel resistance of the underlying TaN film was several orders of magnitude higher than the Ta, and did not influence the Ta measurement. The dependence of resistance on film thickness is much more consistent with the general model of electron scattering (described below).

The thick-film resistivity of the alpha-Ta films tended to approach about 2x the bulk resistivity of Ta, 13.1 micro-Ohm-cm. This is consistent with earlier reports (1,4) and is likely to be due to the deposition process (PVD) combined with the relatively low deposition temperatures (25C), which are well below 1/3 of the melting temperature. Under these conditions, the films are fairly fine-grained and have significant electron scattering from defects and grain boundaries.

In a related experiment, films of TaN which would have normally facilitated the subsequent deposition of alpha-Ta were exposed to room air for periods of a few seconds to several days prior to Ta deposition. This sequence can simulate production sequences where the TaN is deposited in one tool and the subsequent Ta is deposited in a different tool at a later time. This effect can be seen in Fig. 9, where a 5 nm TaN (30% nitrogen) film is exposed to air prior to the deposition of Ta. For air exposures of up to a few minutes, the subsequent Ta is still deposited in the alpha phase. However, for exposures of greater than a few tens of minutes, the subsequent Ta is formed in mixed alpha and beta phases, or with long enough air exposure, in pure beta phase.

The initial expectation is that the TaN films are either oxidizing or else absorbing water vapor during the air exposure. This sensitivity to air exposure is also a function of both film thickness as well as nitrogen content. For either thinner TaN films, or else a higher level of N in the TaN, the maximum exposure time to air which still retains the alpha structure in the subsequent Ta deposition drops rapidly. For example, 5 nm films of TaN with 40% N can be exposed to air for only 15 seconds or less, or else the films become sufficiently oxidized at the surface to lead to beta-Ta formation in the subsequent deposition.

Finally, films of Ta were sputter deposited onto Cu surfaces. Earlier reports on non-integrated Ta depositions on Cu (i.e, deposited in different chambers with an air exposure) indicate that only beta-Ta is deposited on the Cu (7). It was claimed that the Cu film easily oxidizes (or absorbs moisture) and this leads to the formation of beta-Ta upon subsequent deposition. In the current experiment, Cu films were deposited in-situ just prior to the Ta deposition; both at a base pressure of 10⁻⁸ Torr. For a variety of Cu film thicknesses, from 2 to 100nm, the subsequent Ta films were always deposited in the (002) beta phase (Fig. 10). The Cu films were deposited on an underlying Ta adhesion layer and were continuous above 5 nm thickness and exhibited a (111) texture. There have been reports of heteroepitaxial growth of Cu (111) on (002) beta-Ta, which has a pseudohexagonal atomic arrangement (8). This is consistent with the observation of (002) beta-Ta deposited on (111) Cu in the present experiment, independant of the presence of a water or oxide-contaminated Cu surface.

DISCUSSION

There are several effects which may alter the observed electrical resistivity of a very thin film or line (wire) from the intrinsic or bulk value which is due mostly to phonon scattering. These effects can include impurity incorporation, grain size and grain boundary scattering, surface roughness, as well as the size effect due to electron-surface scattering. The relative values of some of these effects have been predicted for Cu films and lines (9) and a chart showing the general magnitude of these effects in given in Fig. 11. (9). A similar chart could be produced for TaN and Ta if the electron mean-free-paths, grain sizes and surface roughness were known. A general form for the electrical resistivity, ρ , for a line of thickness d, that describes these effects is given as (9):

$$\rho_{\rm l} = \rho_{\rm b} \left(1 + 0.75 \,\lambda(1-p) \,\mathrm{S}\,/\mathrm{d} + 1.5 \,[\mathrm{R}/(1-R)] \,\lambda/\mathrm{g} \right)$$
 Eqn (1)

Where ρ_b is the resistivity of the bulk material, λ is the electron mean free path, S is a coefficient >/= 1 which characterizes the average surface roughness, R is the coefficient describing electron scattering at grain boundaries (typically 0.3 to 0.5 for Cu), g is the grain size, and p is the scattering coefficient at the surface, which varies from 0 for diffuse scattering to 1 for purely elastic scattering. For many thin film materials, it appears that p is purely diffuse and as such, the size-dependent scattering effects are maximized. For a 2-dimensional, planar films similar to the measurements made here, the factor 0.75 in this equation is replaced by 0.38.

For the relatively thin films made in this experiment, the surface roughness coefficient can be assumed to be near 1.0. In general, for as-deposited, sputtered films, the grain size, g, can be assumed to be approximately equal to the film thickness. Therefore, for a typical R value of 0.3, the resistivity of the film scales as roughly:

where t is the film thickness. This suggests that if the film resistivity is measured as a function of declining film thickness, the mean free path, λ , can be estimated as equal to the thickness where the resistivity has increased to 2 times the thick film value.

In the case of TaN (Fig. 4), the thick-film resistivity is 500-600 micro-Ohm-cm for the 30% N level which was used for this chart. From Fig. 4, the mean free path is then estimated to be 20-25 nm. The scaling of thinner films, down to 5 nm, follows the general scaling of Eqn. 2. Below 5 nm, the films appear to oxidize so rapidly that the resistance increases much faster than expected from simple scaling.

For Ta deposited in the beta phase, the size-dependant scaling does not tend to follow the general model. From a thick-film value of 180 micro-Ohm-cm, the resistivity does increase as the film thickness is decreased, but the increase is only 40% for the minimum thickness measured of 1.5 nm which would imply a mean free path of less than 1 nm.

For alpha-phase Ta, the resistivity as a function of film thickness (Fig. 8) suggests a mean-free-path of 20 nm. This is a similar value to Al, and approximately 1/2 that of Cu and 2x a recent measurement of alpha-W films by the same technique (10). These results are summarized in Table 1.

The typical application of diffusion barriers in a dual-damascene inteconnect structure is shown in Fig. 12. In this figure, an electrical connection is made from the buried conductor below the left-most via, up through that via, along the horizontal line, and down through the right-side via to another buried conductor. The diffusion barriers are shown as a solid, conformal line for the TaN and a dotted, also conformal line for the Ta. In this case, the diffusion barriers located at the bottom contact surface of each if the two vias are in-series, electrically, and the barriers located on the sidewalls of the via and the trench are in-parallel.

In Fig. 10, as an example of the scale of the resistances involved, if the via diameter is 100 nm, about 200 nm high, and constructed from near-bulk Cu, the dc resistance of each via is 0.5 Ohm. For the line, which might have a width of 150 nm, a height of 200 nm and a length of 1000 nm, the resistance is 0.6 Ohm. The effective resistivity for the diffusion barrier depends on the relative thicknesses of TaN and Ta. As an example of the best case condition, let the thickness of the barrier be 10 nm with an average resistivity of 100 micro-Ohm-cm. This is consistent with a mostly alpha-Ta barrier. In the via, the resistance of the liner on the sidewalls is 220 Ohms, and along the line where the barrier is only on the sides and bottom the resistance is 180 Ohms. Since these resistances are in-parallel with the Cu conductor, the diffusion barrier plays no part in the net resistance of the line, nor can it function as a redundant, current carrying conductor. At the

via bottom, the series resistance of this same barrier is about 1.2 Ohms. While this is similar to the net Cu resistance of the circuit element, it will be strongly dependent on the final barrier thickness and composition.

The series resistance at the via bottom will depend strongly on the thickness of the barrier films, and in the case of Ta (or W), its phase. The low resistivity, alpha Ta phase is desired in this application. Since it has been observed that Ta deposited on Cu is always in the beta phase, it is necessary to have an adequate thickness of TaN $_{0.3}$ (30% or higher nitrogen level). However, the additive resistance contribution of the thin TaN may greatly exceed the resistance advantage of alpha Ta compared to beta Ta, and in many cases it will be desired to remove the TaN completely from the via bottom.

CONCLUSIONS

Thin films of TaN and Ta have been examined, primarily for the scaling of their resistivity for very thin films. In the case of TaN, the material was found to be multi-phase with a nitrogen composition which strongly depended on the configuration of the sputtering system and geometry, with the implication that TaN deposited in deep features was probably depleted in N. The films were mildly unstable in air and showed a strong resistive increase as the film thickness was reduced, consistent with an electron mean free path in the 20-25 nm range.

Ta films were deposited in the beta phase on silicon dioxide and Cu surfaces. The resistivity was >10 times the bulk, but exhibited only a weak increase as film thickness was reduced to 1.5 nm.

If Ta was deposited on >2nm of TaN (30% N), the material was in the alpha phase with a resistivity of 2 times the bulk. If the TaN was exposed to air for periods of 10 or more minutes prior to Ta deposition, the Ta was deposited in the beta phase. For the alpha-Ta, the resistance increased as the thickness was reduced consistent with an electron mean free pat of 20 nm.

In interconnect circuit elements, diffusion barriers of Ta and/or TaN on the sidewalls of the vias or trench had no effect on the net electrical conductivity of the circuit. Diffusion barrier films at the contact point at the bottom of the via could add 1 or more Ohms to the net circuit resistance, depending strongly on the barrier thickness. If the barrier could be removed completely from the via bottom, then the electrical properties of the barrier materials would be completely irrelevant to the operation of the circuit.

Table 1.

Material	Electron mean-free-path	Referenc
		e
Al	18 nm	3
Cu	39 nm	3
Та	20 nm	
TaN	20-25 nm	
W	10 nm	9

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TABLE AND FIGURE CAPTIONS

Table 1. Electron mean-free-paths for relevant interconnect materials. Cu and Al derive from Ref. 3, W is from Ref. 10.

Figure 1. Sheet resistance of 10 nm TaN film as a function of nitrogen flow during reactive sputter deposition. The solid line (left-most) is for deposition with internal shielding in place and the wafer table elevated, the dotted line (middle) is for no shielding, and the dashed curve (right-most) is for deposition with shields in place and the sample table lowered.

Figure 2. X-ray diffraction, theta-two-theta trace of 45 nm TaN films deposited with approximately 30% nitrogen in the film.

Figure 3. Changes in TaN sheet resistance for 10 nm TaN film as a function of exposure time to room air.

Figure 4. Resistivity of TaN (30% N) as a function of film thickness at 25C on SiO_2 . The circular data-point is for an in-situ measurement.

Figure 5. Resistivity of beta-Ta films on SiO_2 (with no TaN underlayer) as a function of film thickness.

Figure 6. Sheet resistance of 145 nm Ta film as a function of nitrogen flow during the reactive sputter deposition of a 5 nm TaN underlayer.

Figure 7. Sheet resistance of a 145 nm Ta film deposited over TaN films with 30% nitrogen as a function of TaN film thickness.

Figure 8. The resistivity of alpha Ta as a function of film thickness for Ta deposited over 3 nm of TaN(30%). The bulk resistivity of alpha Ta is 13.1 micro-Ohm-cm.

Figure 9. Sheet resistance of 145 nm Ta film deposited on 3 nm TaN (30%N) as a function of air exposure time between the TaN and Ta depositions. The beta-phase sheet resistance is approximately 13 Ohm/sq. The alpha-Ta sheet resistance is 2 Ohm/Sq.

Figure 10. XRD theta-two-theta trace for Ta deposited on 20 nm Cu with a 4 nm Ta adhesion layer under the Cu.

Figure 11. The scaling of resistivity for Cu thin films as a function of film thickness showing the additive effects of surface scattering (the size effect), grain boundary scattering and surface roughness. The electron mean free path for Cu is 39 nm (8).

Figure 12. Schematic of dual damascene interconnect structure.



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