

Interfacial reactions during sputter deposition of Ta and TaN films on organosilicate glass: XPS and TEM results

J.A. Wilks^a, N.P. Magtoto^a, J.A. Kelber^{a,*}, V. Arunachalam^b

^aDepartment of Chemistry, University of North Texas, Denton, TX 76203, USA

^bTexas Instruments, Inc., 13121 TI Boulevard, MS 365, Dallas, TX 75243, USA

Received 25 September 2006; accepted 16 January 2007

Available online 21 January 2007

Abstract

The evolution of the interface between organosilicate glass (OSG) and sputter deposited Ta or TaN films has been characterized by X-ray photoelectron spectroscopy (XPS). Cross-sectional TEM (XTEM) was also used to analyze Ta/OSG and TaN/OSG/interfaces for samples formed under different deposition conditions. XPS data show that Ta deposition onto OSG results in formation of an interphase between 1 and 2 nm thick composed of oxidized Ta and C. Metallic Ta is then formed on top of the interfacial region. In contrast, Ta-rich TaN formation occurs with some nitridation of the substrate, but with no significant interphase formation. The XPS data are consistent with the XTEM data. The XTEM results for Ta/OSG indicate a spatially irregular interface over a length scale of ~ 2 nm, while results for TaN/OSG indicate a spatially abrupt region. © 2007 Elsevier B.V. All rights reserved.

Keywords: Diffusion barrier; Dielectrics; Physical vapor deposition (PVD); Tantalum; Tantalum nitride; Transmission electron microscopy; X-ray photoelectron spectroscopy (XPS)

1. Introduction

The integration of Cu in device interconnects requires that a barrier be present to hinder Cu diffusion into the low-k dielectrics under bias/thermal stress conditions. The current International Technology Roadmap for Semiconductors forecast is for diffusion barrier widths to shrink to <4 nm in the immediate future [1]. At such dimensions, the manufacturability of existing TaN_x/Ta barriers becomes increasingly problematic, and a simplified barrier structure – such as pure Ta – becomes more desirable. (The notation “TaN_x” is used here to mean the broad class of Ta nitrides, without implying any specific stoichiometry.) Previous X-ray photoelectron spectroscopy (XPS) studies [2] have shown that Ta deposited by sputter deposition onto an amorphous Si–O–C (a-Si:O:C) substrate reacts at room temperatures to form an “interphase” containing Ta, O, and C, but not Si. Cu will not adhere strongly to this interphase. The thickness of the interphase, ~ 3 nm or more

(determined from XPS data), depending on the Ta deposition rate (~ 1 Å/min– 2 Å/s), implied that pure Ta barriers would not be suitable candidates for further diffusion barrier scaling. Obvious questions posed by those results are:

- (1) Does a comparable interphase form during TaN_x deposition?
- (2) Are laboratory results achieved at lower deposition rates relevant to interfaces formed at the higher deposition rates?

XPS data presented in this report corroborate previous results [2] for interphase formation during Ta deposition on a-Si:O:C and also demonstrate that interphase formation is suppressed by TaN_x deposition. XTEM data are consistent with the XPS results, and show that Ta deposited onto a-Si:O:C dielectric under industrial conditions forms a geometrically irregular interface with an interfacial region ~ 2 – 3 nm thick while TaN deposited under similar conditions yields an abrupt interface. These results demonstrate that further barrier scaling must rely either on TaN_x barriers suitably modified for Cu plating, or on more radical approaches to diffusion barrier design. Stand-alone Ta barriers will pose serious problems, due to the interfacial reaction, as average barrier thicknesses shrink below 4 nm.

* Corresponding author at: Department of Chemistry, University of North Texas, P.O. Box 305070, Science Research Building, Room 232, Denton, TX 76203, USA. Tel.: +1 940 565 3265.

E-mail address: kelber@unt.edu (J.A. Kelber).

2. Experiment

XPS experiments were performed in a combined UHV surface analysis/magnetron sputter deposition system composed of two independently turbomolecularly pumped chambers separated by a gate valve. Sample transport between the UHV analysis chamber (working pressures; $3\text{--}6 \times 10^{-10}$ Torr) and deposition chamber (base pressure, 5×10^{-9} Torr) was accomplished with a magnetically coupled rotary feedthrough. The analysis chamber was equipped with a 100 mm mean radius hemispherical analyzer (VSW) and dual anode polychromatic X-ray source (Physical Electronics) for XPS, reverse view 3-grid LEED optics (Omicron) and a differentially-pumped Ar ion sputter gun (Specs) for sample cleaning. Pressure in the analysis chamber was monitored by a nude ion gauge out of line of sight to the sample. Sample temperature was controlled by a combination of liquid nitrogen cooling and resistive heating between 150 and 1200 K, with sample temperature monitored by a type K thermocouple mounted just below the base of the sample transfer platform. Separate calibration experiments, in which a thermocouple was also mounted to the surface of the sample, were used to characterize the temperature gradient (about 100 K over a broad temperature range) between sample surface and sample mount base. Temperatures reported here are those for the sample surface.

The deposition chamber was equipped with two water-cooled DC-Magnetron sputter deposition sources (Minimack). Ar and N₂ were admitted to the chamber through manual leak valves with gas pressures monitored by nude ion or baratron gauges. Sputter deposition rates were measured by subsequent XPS analysis, and varied by adjustments of source/sample distance or gas pressure. Sample temperature was not controlled during deposition.

XPS spectra were acquired with polychromatic Mg K α x-radiation with the source operated at 15 kV and 300 W and the hemispherical analyzer operated in constant pass energy mode (22 eV). Sample charging was frequently observed during XPS analysis. Binding energies were calibrated by setting the C(1s) level of the substrate at 285.0 eV [3]. XPS data were analyzed using commercially available software (ESCATOOLS) and Shirley background subtraction. In simulating spectra for comparison to experiment, Gaussian–Lorentzian peaks were used. During peak fitting, binding energies and FWHM values were fixed, and only intensities permitted to vary. The FWHM value used in this study for Ta metal (1.33 eV), was determined by measurements on polycrystalline metallic Ta in this system. A different FWHM value was used for Ta oxide and nitride features (1.55 eV) due to a widening of the XPS feature that could not be fit properly using the FWHM for metallic Ta.

Ta sputter deposition was carried out using an Ar plasma with a pressure of 15 mTorr. Ta and TaN_x were deposited from a 99.999% purity Ta target at a deposition rate of $1.3 \pm 0.2 \text{ \AA min}^{-1}$ with the deposition rate controlled by changing the plasma power level. The deposition rate was calculated using the total Ta deposited thickness plotted against deposition time. During TaN_x deposition N₂ was introduced at a partial pressure of 5 mTorr making the total deposition pressure

20 mTorr. All the depositions were performed at room temperature.

The deposition rate was determined, for both Ta and TaN_x deposition, by measuring the attenuation of the Si(2p) substrate signal according to

$$I = I_0 \exp(-d/\lambda) \quad (1)$$

where I_0 and I are the initial and after deposition absolute Si(2p) core-level signal intensities, d the thickness of the attenuate overlayer (Ta, TaN_x, or Ta oxide), and λ is the calculated [4] electron inelastic mean-free path (IMFP) for (in this case) a Si(2p) electron through Ta, Ta₂O₅ or TaN, with values of 14.22, 20.10, or 14.37 Å, respectively. Absolute, rather than relative Si(2p) intensities were measured. The error in measuring absolute intensities in this apparatus has been determined to be <10%. A basic assumption here is that substrate Si will not be incorporated to any significant degree in the deposited reactive layers. This assumption is based on previous studies [2] and verified by our results (see below).

The nonporous organosilicate glass (OSG) wafers used in the XPS portion of this study were 4200 Å thick, deposited on Si substrates, and displayed a dielectric constant of 2.9. Each wafer was cut into multiple pieces $\sim 1 \text{ cm}^2$ and mounted on a Mo sample holder for surface science studies, and inserted into vacuum without further treatment. A combination of liquid-nitrogen cooling and resistive heating of the sample holder permitted a variation of sample temperature between 100 and 1100 K. Temperatures were monitored by a K-type chromel–alumel thermocouple spot-welded near the sample holder.

TEM samples were prepared using similar OSG wafers (dielectric constant = 2.8) Ta and TaN films were deposited into pre-patterned vias with a hollow cathode magnetron (Novellus). A deposition rate of 9.4 \AA s^{-1} was used for both Ta and TaN deposition. Cross-sectional samples were prepared for TEM by focused ion-beam (FIB) milling, employing 30 kV Ga⁺ ions using a final beam current of $\sim 50 \text{ pA}$. Samples were characterized in the TECNAI F30ST analytical TEM operated at 300 keV. High resolution TEM microscopy was performed with the sample aligned for the $z = (1\ 1\ 0)$ axis of the substrate Si.

3. Results

3.1. Ta deposition

Ta was deposited onto an OSG substrate in seven sequential depositions of 3 min each. XPS core level spectra were acquired after each deposition. Fig. 1 shows the change in absolute Si(2p) intensity as a function of deposition time for both Ta deposition and TaN_x deposition. The linear behavior is consistent with the formation, in each case, of an attenuating overlayer with thickness proportional to deposition time. This indicates a constant deposition rate during both Ta and TaN_x deposition. A change in slope is observed for Ta deposition between $t = 6$ and 9 min, and this coincides with the formation of metallic Ta (see below), which has a shorter electron inelastic mean free path. Estimation of deposition rates for a process in

Download English Version:

<https://daneshyari.com/en/article/5365023>

Download Persian Version:

<https://daneshyari.com/article/5365023>

[Daneshyari.com](https://daneshyari.com)