



Real-time curvature and optical spectroscopy monitoring of magnetron-sputtered WTi alloy thin films

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ABSTRACT

WTi thin films are known as potential adhesion promoters and diffusion barriers. WTi thin films were deposited by magnetron sputtering from an alloyed target (W:Ti ~ 70:30 at.%). Real-time surface differential reflectance (SDR) spectroscopy and wafer-curvature measurements were performed during deposition to study the growth and the film continuity threshold. SDR measurements during WTi deposition allow the determination of the change in reflectivity of p-polarized light (at Si substrate Brewster's angle) between WTi film and Si substrate in order to monitor layer growth. The comparison between experimental and simulated WTi SDR signals assuming a homogeneous and continuous layer growth shows that film continuity is ensured beyond a thickness of 4.5 ± 0.2 nm. Real-time wafer-curvature measurements allow the determination of the intrinsic stress development in the film. Two regimes are noticed during the growth up to the development of a compressive steady state stress. The early stages of growth are rather complicated and divided into sub-regimes with similar boundaries revealed by both in situ techniques. Deposition of an interfacial continuous layer different from WTi bulk is suggested by both in situ techniques below a thickness of 4.5 nm.

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1. Introduction

WTi alloy thin films are extensively used in microelectronics for Al, Ag and Au contacts. Early studies on WTi were reported by Cunningham et al. [1] on the development of a corrosion-resistant refractory metal–Au contact system. Adding Ti to the base metal allows for an increase in corrosion resistance of the metallization. WTi has been shown to be the best candidate with benefit on adhesion because of its reaction at the substrate–film interface [2,3]. WTi is well known for its good electrical and barrier properties [4–8] and serves as diffusion barrier and contact-to-substrate layer. Refractory metal W films deposited by a PVD process may crystallize either in a stable body-centered cubic (bcc) structure (α -W) or in a metastable cubic A15 structure (β -W). This latter is favored in the early stages of growth [9] and offers lower performance [10]. Miniaturization of interconnection size requires the use of ultra-thin films, while maintaining the film continuity to provide effective barrier performance. Real-time wafer-curvature [11–14] and optical spectroscopy [15–17] monitoring are attractive measurements to characterize the early stages of thin film growth. In situ wafer-curvature measurements of W films have been previously reported [18] with focus on the influence of the working gas pressure on the stress magnitude. However, the initial sequence of stress development has not been explored yet.

In the present study, we report on the early stages of growth of WTi alloy thin films deposited on naturally oxidized Si (001) substrate by magnetron sputtering from an alloyed WTi target (W:Ti ~ 70:30 at.%). Growth and film continuity threshold of WTi films are investigated for a single sputtering condition by coupling in situ and real-time surface differential reflectance (SDR) and wafer-curvature techniques. The results emphasize the complementarity of both techniques and clearly show the continuity of WTi thin films from 4.5 ± 0.2 nm.

2. Experiments

WTi alloy thin films have been deposited at room temperature (RT) by planar magnetron sputtering in DC mode using a working gas of Ar in a high vacuum system (base pressure ~ 1.10^{-5} Pa). The WTi target used in this study was 7.6 cm in diameter, with a purity 4N (i.e. 99.99%), and a W:Ti ratio of 70:30 at.%. Films were deposited on naturally oxidized Si (001) substrates. The plasma discharge was operated at a constant target power of 60 W, using an unbalanced magnetron configuration, at a working pressure of 0.53 Pa, and on grounded static substrates. The target-to-substrate distance was 18 cm. The heating from target or plasma radiation was disregarded because the maximum temperature rise was 5–10 °C, for a deposition at room temperature. Therefore, thermal stress contribution is expected to be minor. Before deposition, the target was sputter-cleaned while substrates were protected by a shutter. The growth rate was determined using X-ray reflectometry (XRR) and mechanical profilometry. Experimental techniques used for ex situ characterizations of WTi film were given in a previous publication [19]. The

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complex refractive index of thin film was characterized ex situ by spectroscopic ellipsometry (GESp5 Sopra™).

Real-time in situ surface differential reflectance (SDR) spectroscopy using p-polarized light at an oblique incidence angle (70°) was performed during deposition. For a p-polarized light, the single-crystal Si substrate has a reflectance minimum (Brewster's angle at 74°) that allows to be more sensitive to differential reflectance in our experiments. A discharge xenon lamp (150 W) was employed as a white light source and the reflected light was collected in the specular direction by a QE65000 spectrophotometer (Ocean Optics™). The reflectance $R(t, \lambda)$ (ratio between the reflected and the incident wave intensities) at a time t was recorded at a rate of one spectrum per second within the spectral range $\lambda = 350\text{--}800\text{ nm}$. The SDR signal was obtained from the differential change of the reflected intensity:

$$\frac{\Delta R}{R_0}(t, \lambda) = \frac{R(t, \lambda) - R_0(\lambda)}{R_0(\lambda)} \quad (1)$$

with $R_0(\lambda)$ the reflectance of the bare substrate being used as a reference. The optical configuration was presented in a previous publication [17].

Real-time in situ wafer-curvature evolution using a multi-beam optical stress sensor (MOSS) designed by kSA was performed during deposition. The measurement is based on the curvature evolution of a thin substrate due to stress build-up in the thin film. The curvature is measured from the bending of the substrate, using the deflection of a 2D array of parallel laser beams (created using highly reflective X and Y etalons). The reflected beams are recorded on a high resolution charge-coupled device (CCD) camera with a typical acquisition rate of 5 Hz. Changes in the substrate curvature produce changes in the angular divergence of the beam array, and therefore changes in the spacing between adjacent laser spots on the CCD camera. 100 μm -thick Si substrates were mounted on a specially designed substrate holder in order to allow unconstrained bending of the substrate. The experimental set-up has been described in details elsewhere [20]. The average stress was determined from the film force per unit width using the Stoney equation. Assuming the substrate is much thicker than the deposited film and a state of plane stress in the film, Stoney's formula can be applied [21]:

$$\sigma = \frac{M_s t_s^2}{6 t_f} \Delta \kappa \quad (2)$$

with M_s the biaxial elastic modulus of the substrate, σ the macroscopic average residual stress in the thin film, t_s and t_f are the thicknesses of the substrate and the film respectively, and $\Delta \kappa$ the change in curvature. As single crystal substrates are generally elastically anisotropic, the biaxial elastic modulus, M_s , must be computed from the stiffnesses or compliances for that crystal, i.e. $M_s = 180.5\text{ GPa}$ for Si (001) [22, 23]. Noticeably, Stoney's formula requires only the elastic constants of the substrate.

In situ curvature and optical spectroscopy have been carried out separately due to a difference in experimental configurations, but using the same deposition conditions. Deposition rate at an Ar working pressure of 0.53 Pa was estimated at 0.053 nm/s using XRR.

3. Results

3.1. Ex situ measurements of WTi film

Table 1 shows ex situ characterizations obtained on WTi ultra-thin (9.5 nm) and thin (180 nm) films deposited at an Ar working pressure of 0.53 Pa. XRD analyses have shown that both films have a single α -W structure. It can be observed from Table 1 that magnetron-sputtered WTi films are Ti deficient compared to the nominal target composition. This can be explained by the difference in atom transport and, to a

Table 1

Ex situ results for 9.5 and 180 nm thick magnetron-sputtered WTi films at a working pressure of 0.53 Pa. Table shows atomic composition, texture, electrical resistivity (p) and residual stress determined by ex situ curvature method (σ) for both thicknesses.

t_f (nm)	W:Ti (at%)	Texture	p ($\mu\Omega\text{ cm}$)	σ (GPa)
9.5	NA	{110} fiber	59.7	−0.75
180	79:21	{110} fiber	86.1	−2.66

lesser extent, to resputtering [19]. Working pressure is an important parameter affecting the film microstructure [24]. For both ultra-thin (9.5 nm) and thin (180 nm) films, a stress transition from tensile-to-compressive is observed as the working pressure increases, as determined by ex situ curvature and XRD methods [19]. Residual stress transition from compressive to tensile stress state is determined to be roughly at the same working pressure around $0.75 \pm 0.05\text{ Pa}$ for both thicknesses. Considering these ex-situ results, WTi deposition was carried out at a working pressure of 0.53 Pa for in situ monitoring to obtain ultra-thin WTi film with both dense microstructure and low compressive residual stress.

Ellipsometry measurement was performed ex situ to obtain the complex refractive index of WTi films ($\tilde{n}_{\text{WTi}} = n + ik$). An experiment was performed on 106 nm thick WTi thin films (optical opacity). Fig. 1 shows the spectral variations of the real (n) and imaginary (k) parts of the complex refractive index of this WTi film. In addition, n and k spectral variations of an optically opaque pure W film was also measured and is represented on Fig. 1 for comparison. Complex refractive indexes of WTi and W are very similar revealing that W dominates the optical response in WTi alloy film.

3.2. In situ SDR measurements of WTi film

Spectral measurements were obtained from in situ surface differential reflectance (SDR) spectroscopy. Fig. 2 shows the SDR signal in false colors obtained for the WTi deposition as a function of wavelength (λ) and deposition time (t). SDR signal was acquired from the beginning of deposition up to 2000 s. Evolution of the SDR signal is induced by the growth of WTi film, which has a higher reflectance than the bare Si substrate. From a film thickness of $t_f \sim 80\text{ nm}$ (i.e. deposition time of 1450 s), the SDR signal remains unchanged up to the end of deposition which is induced by the WTi film optical opacity in all the visible range.

SDR signal is compared with a simulated spectral response assuming a homogeneous and continuous layer growth which takes into account the Fresnel reflectivities at each interface, the absorption and the multiple reflections inside the layer. For a p-polarized light, the Fresnel reflection coefficient r_{ij} (ratio of the reflected and incident electric field amplitudes) at the interface between media i and j is given by:

$$r_{ij} = \frac{n_j \cos \theta_i - n_i \cos \theta_j}{n_j \cos \theta_i + n_i \cos \theta_j} \quad (3)$$

with n_i , n_j the refractive indexes of media i and j , and θ_i , θ_j the propagating angles within these media related to the Snell–Descartes's law: $n_i \sin \theta_i = n_j \sin \theta_j$. Assuming a film thickness of t_f (medium 1) included between vacuum atmosphere medium (medium 0) and a semi-infinite substrate (medium 2), the reflectances R_0 and R are given by [25]:

$$R_0 = |r_{02}|^2 \text{ and } R = \left| \frac{r_{01} + r_{12} e^{j2\beta_1}}{1 + r_{01} r_{12} e^{j2\beta_1}} \right|^2 \quad (4)$$

with $\beta_1 = \frac{2\pi}{\lambda} t_f n_1 \cos \theta_1$ the phase difference induced by crossing the film. Simulated continuous layer spectra are computed from Eqs. (3) and (4) assuming both constant deposition rate and constant refractive index throughout the deposit. The refractive index of WTi thin film dependence on the wavelength used for simulations was taken from the ex situ spectroscopic ellipsometry (data reported on Fig. 1). The best

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