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Lateral solvent diffusion characterization of low *k* dielectric plasma damage and ALD barrier film closure

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Abstract

The lateral diffusion of toluene solvent molecules was employed to probe the porous structures of plasma damaged low *k* dielectrics and ultra-thin ALD copper diffusion barriers. A change in the pore structure of a microporous CVD low *k* was determined from the absence of diffusion after the plasma treatment of a thin film (<270 nm). This indicated a densification or reduction of the pore structure below the 6.8 Å probe molecule size. Atomic layer deposited layers of WNC and TaN were probed for defect channels when deposited on porous low *k* substrates. Toluene was able to penetrate through microchannels in the films and diffuse laterally inside the underlying porous low *k*. This allowed a non-line-of-site assessment of ALD film surface closure. In both cases the refractive index changes resulting from toluene filling of the low *k* pores was observed optically by ellipsometry or optical microscopy.

Keywords: Solvent diffusion; Low *k*; Plasma damage; ALD

1. Introduction

The use of organic solvent molecular probes for characterizing thin porous dielectric films has been accomplished primarily through the use of ellipsometric porosimetry (EP) [1]. This technique employs the vertical diffusion of probe molecules through a porous structure. However, lateral diffusion of solvents, where solvents molecules diffuse through a porous film beneath a transparent impermeable cap, have been recently employed in our laboratory to characterize low *k* dielectric pore interconnectivity, correlation to pore sealing and to probe PVD barrier defects [2–4]. The use of lateral solvent diffusion has been extended here to the characterization of plasma damage to porous low

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k dielectrics and the assessment of atomic layer deposition (ALD) barrier film growth on porous low *k* films.

Plasma damage of porous low *k* dielectrics has become a major limitation to the retention of the desired low *k* electrical characteristics after patterning with plasma processes. Plasma damage has been implicated in the increase of effective k-values, degradation of current leakage and decreased dielectric breakdown lifetimes [5]. This damage negates the reduction in RC delay that is needed for future IC interconnects. The creation of SiO_2-like damaged layers and carbon depletion have been the primary signals seen in physical and chemical analysis. However, assessment of the true degree of damage has been challenging with each technique returning different results depending on the underlying physics of the measurement.

Atomic layer deposition processes have been pursued for IC interconnects to produce conformal ultra-thin (<7 nm) copper diffusion barriers [6–8]. ALD processes have shown to be extremely sensitive to substrate surface chemistry and follow island nucleation and growth [9]. Plasma treatments of low *k* substrates have been investigated to improve nucleation and hasten surface closure [10]. But a simple test for determination of the minimum number of ALD cycles required to achieve a continuous barrier layer on blanket films has been elusive. Line-of-sight analytic techniques, such as TOFSIMS or LEIS, have been employed but may not reveal non-line-of-sight discontinuities [11].

2. Experimental

Four different CVD SiCOH low *k* films were investigated. The k-value and porosity characteristics are summarized in Table 1.

Table 1
Summary of material properties as deposited

Material	*k* value	Porosity, % by EP	Pore diameter (nm)	Porous surface, EP
CVD1	3.0	7	<2	Yes
CVD2	2.7	8	<2	Yes
CVD3	2.7	8	<2	Yes
CVD4	2.4	16	<2	Yes

The assessment of low *k* damage was accomplished with two different CVD SiCOH low *k* films (CVD2 and CVD4) with porosities of 8% and 16%, respectively. Films of varying thicknesses were exposed to plasmas of varied power and chemistry in LAM Excelan and Mattson Aspen III Highlands chambers with O_2/CF_4 and He chemistries, respectively. This exposure caused plasma damage and created a dense surface sealing layer. Samples were cleaved and sidewalls exposed to toluene by immersion for 20 min. The solvent diffused laterally through the undamaged low *k* layer beneath the sealing layer as depicted in Fig. 1. The toluene diffusion front was discerned optically using a microscope.

The assessment of surface closure of ALD films on low *k* substrates involved deposition of WNC and TaN layers on all the four different CVD SiCOH low *k* films (see Table 1). Low power He plasma exposures were conducted in a Mattson Aspen III Highlands chamber on certain samples to modify the surface chemistry but not creating a sealing layer. Samples were exposed to vaporous toluene in an EP chamber. If film closure was not complete the toluene penetrated into the porous low *k* layer and diffused laterally as depicted in Fig. 2(a). This diffusion was then detected as changes in optical angles ψ and Δ by ellipsometry.

3. Results and discussion

3.1. Low k plasma damage characterization

Sufficient power He plasmas have been shown to seal the surface of porous low *k* films to toluene

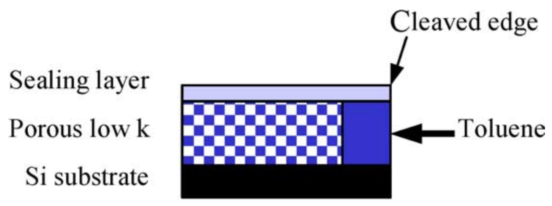

Fig. 1. Schematic side view of toluene diffusion into a porous low *k* film from exposed cleaved edge. Diffusion front is observed from top through transparent sealing layer.