The effects of resist strip processing on a porous low k dielectric oxide

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ABSTRACT: Energy-loss imaging in the transmission electron microscope has been used to characterise localised changes in the densities and compositions of porous low k dielectric oxides formed by two types of optimised resist strip processing.

1. INTRODUCTION

Increased device scaling requires the use of low k dielectric materials in order to counteract the adverse effects of increased capacitance. The k values (<2.4) needed for the 65 nm technology node and beyond can be achieved by incorporating pores into oxide films, although such pores can complicate subsequent device processing. For example, low k oxides that contain pores can be penetrated by precursor gases during the deposition of barrier layers onto oxide trench sidewalls (Kawamura et al 2002), giving rise to an increase in leakage current and in the k value of the integrated damascene stack. Sidewall porosity can also lead to the formation of discontinuities in plasma vapour deposited (PVD) barrier layers (Tokei et al 2001). Such difficulties have highlighted the need for treatments that can be used to modify the sidewall regions of porous oxides, whilst maintaining their favourable bulk dielectric properties.

Optimised resist strip processes have been used to ‘seal’ the sidewalls of porous dielectric oxides (Besling et al 2002), but little is known about their effects on the microstructure of low k dielectric oxides. Here, we examine such sidewall regions using energy-loss imaging in the transmission electron microscope (TEM) in order to characterise changes in the density and composition of the oxide following resist stripping. We compare oxides that have been given N₂/O₂ and N₂/H₂ based strips. The results are discussed with reference to the different electrical properties of the two oxides.

2. EXPERIMENTAL DETAILS

Single damascene stacks consisting of SiC:H etch-stop (50nm), porous carbon doped oxide (Orion® k=2.2) and oxide capping layers were deposited onto Si wafers using a chemical vapour deposition (CVD) cluster tool. The wafers were printed with 0.18 μm trenches in a Nikon deep-ultra-violet (DUV) scanner using a 60 nm Shipley AR7 anti-reflective coating and a 600nm DUV photoresist. The trench etch was performed using a Trikon Omega etch tool using CF₄/CH₂F₂. Subsequent photoresist stripping and polymer cleaning were performed using N₂/O₂ or N₂/H₂ based strips. A TiSiN barrier layer was deposited from tetrakisdiethylaminotitanium (TDEAT) and NH₃ precursors using a low temperature CVD process. Samples of highly uniform thickness were prepared for TEM examination using focused ion beam (FIB) milling. Energy-loss images were acquired at 300 kV using a Philips CM300 field emission gun (FEG) TEM using a Gatan Imaging Filter (GIF) equipped with a 2048 pixel charge-coupled device (CCD) camera. An optimal objective aperture (5 mrad. semi-angle) was used, and chemical maps were calculated from energy-loss images using three-window background-subtracted elemental mapping (Egerton 1996). The pixel size in the chemical maps, which were 1024 pixels in size, corresponded to a distance of 1.56 nm on the sample.
3. RESULTS

The microstructure of a typical region of a damascene stack that contains 0.18 µm trenches is shown in Fig. 1a. The trenches are lined with a conformal TiSiN barrier layer that has a thickness of approximately 8 nm. The trenches are filled with an oxide in order to provide mechanical stability during TEM sample preparation. Part of the low k dielectric oxide layer can be seen at higher magnification in the form of underfocus and overfocus bright field images in Figs. 1b and c, respectively. In these images, phase contrast has been used to enhance the visibility of the pores in this part of the structure. The pore size in the low k oxide is between 2 and 4 nm. Sidewall regions formed during the N₂/O₂ and N₂/H₂ based resist strip processes are shown in Figs. 2a and b, respectively. Fine modified zones can be seen immediately beneath the barrier layer. The bands are similar for each resist strip process, have a typical width of between 5 and 8 nm, and exhibit stronger absorption contrast than the oxides located to their sides.

Fig. 1. a) Bright field overfocus images showing 0.18 µm trenches after N₂/H₂ resist stripping. The etch-stop and capping oxide layer layers are labelled A and B, respectively. The intervening region contains the Orion® carbon doped low k dielectric oxide. b) and c) show underfocus and overfocus images, respectively, of the sidewall regions at higher magnification. Pores are labelled P.

Fig. 2. Bright field images of sidewall regions in the low k oxide, for a) N₂/O₂ and b) N₂/H₂ resist stripping. Fine modified zones are marked X.
Line scans obtained from Ti, O, C and N chemical maps of the dielectric layers and trenches are shown in Fig. 3. There is no evidence from the line scans that the precursor has penetrated the oxide during formation of the barrier layer, and the dielectric contains no measurable Ti. In both samples, the sidewall regions of the dielectric show C depletion and O enrichment relative to the more central parts of the layer.

The sidewall regions of the dielectric oxides were examined in further detail. Chemical signals were projected along the sidewalls over a distance of 1800 nm for each element, after removing the effects of sidewall roughness by using a cross-correlation method to align successive rows of the chemical maps laterally. C, O and N line scans across the sidewalls in the two samples, obtained using this approach, are shown in Fig. 4. The line scans show that the N\textsubscript{2}/H\textsubscript{2} treatment results in an increase in the density of both C and O within the sidewall regions of the dielectric layers, when projected in the electron beam direction, relative to that in the N\textsubscript{2}/O\textsubscript{2} stripped sample. A slight increase in the N signal in the dielectric next to the barrier layer is also evident in both samples (Fig. 4c). Ratios between the C and O signals in the two samples are shown in Figs. 5a and b, respectively. Clear increases in both ratios, which are consistent with the presence of a material of higher density in the dielectric oxide (by up to 10%) after N\textsubscript{2}/H\textsubscript{2} stripping than after N\textsubscript{2}/O\textsubscript{2} stripping, can be observed within a region of thickness ~10 nm next to the barrier layer. This fact that this region is localised suggests that it should not affect the dielectric constant of the oxide appreciably.

Fig. 3. Line scans across two dielectric layers obtained from a) Ti, b) O, c) C and d) N chemical maps for the N\textsubscript{2}/O\textsubscript{2} stripped sample. The line scans were averaged over a distance of 200 nm along the layers.

Fig. 4. Line scans across the TiN barrier layer obtained from a) C, b) O and c) N chemical maps after projecting the signals over 1800 nm. Solid lines: N\textsubscript{2}/O\textsubscript{2} resist strip. Dashed lines: N\textsubscript{2}/H\textsubscript{2} resist strip.
Fig. 5. Ratios of a) C and b) O signals in the sidewall regions adjacent to the TiN barrier layers between the N$_2$/H$_2$ and N$_2$/O$_2$ stripped samples c) t/λ line traces (solid lines: N$_2$/O$_2$ resist strip; dashed lines: N$_2$/H$_2$ resist strip). d) Ratio of t/λ maps between the N$_2$/H$_2$ and N$_2$/O$_2$ stripped samples.

Fig. 5c shows t/λ profiles across the region of interest for both samples, where t is the sample thickness and λ is the mean free path for inelastic scattering. The profiles were generated by taking the logarithm of the ratio of the unfiltered to the energy-filtered bright field zero-loss intensity (Egerton 1996), and provide a measure of t if λ is known, or a measure of λ, which varies with density and composition, if t is known. On the assumption that the difference between the t/λ profiles arises from a change in density rather than from a change in composition or thickness, their ratio (Fig. 5d) is consistent with the dielectric being of higher density next to the barrier layer in the N$_2$/H$_2$ stripped sample. This interpretation is consistent with the C and O ratios shown in Figs. 5a and b.

4. DISCUSSION AND SUMMARY

Localised changes in the density and composition of a porous dielectric oxide have been observed following resist strip processing. N$_2$/O$_2$ stripping promotes the formation of a modified zone in which the C and O densities are lower than in the same dielectric oxide that has been given an N$_2$/H$_2$ strip. The observations are significant in relation to the way O-containing strip chemistries are known to promote C depletion. The significance of the difference in the sidewall regions is also underlined by the fact that higher inter-line leakage currents are observed in N$_2$/O$_2$ stripped than in N$_2$/H$_2$ stripped samples (Donohue et al 2002), indicating that the densified layer formed by the latter treatment provides better protection against degradation during processes such as wet polymer stripping.

REFERENCES