Chemical Mechanical Polishing and Planarization of Mn-Based Barrier/Ru Liner Films in Cu Interconnects for Advanced Metallization Nodes

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Mn-based (referred to simply as Mn in the following) barrier/Ru liner stack has been proposed to replace Ta/N/Ta barrier-liner in Cu interconnects for 7 nm and 5 nm technology nodes. During chemical mechanical planarization (CMP) of the associated Cu/Ru/Mn/SiCOH pattern structures, the usual galvanic corrosion and removal rate selectivity issues need to be resolved. In this study, we investigated the polishing and electrochemical behavior of Cu, Mn, Ru and SiCOH films on the relevant substrates in the alkaline region using silica dispersions containing potassium permanganate (KMnO4), guanidine carbonate (GC) and benzotriazole (BTA) and identified compositions that address these challenges. An XPS study of the as-deposited and annealed Mn films confirmed the formation of amorphous manganese silicate (MnSiOx), a self-forming dielectric layer, at the Mn/SiCOH interface. A cross-sectional TEM analysis of the polished Cu/Ru/Mn/SiCOH patterned wafers (32 nm half pitch Cu lines) with our candidate slurry showed excellent post-polish performance with no corrosion and no post-CMP loss of either Cu or the Mn barrier/Ru liner film.

Materials

All the thin films were prepared at IMEC, Leuven, Belgium, Mn-based films (~1.5 nm thickness) were deposited by chemical vapor deposition on a 100 nm thick tetraethylorthosilicate (TEOS)-based BD layer on 300 nm Si wafers and then annealed at ~450°C in the presence of forming gas in a closed chamber to ensure formation of MnSiOx. The ~100 nm thick BD blanket film itself was deposited using alkylsilane precursors. Ru (~30 nm thick) films were deposited by atomic layer deposition on a 100 nm thick BD layer on 300 nm Si wafers, with an intervening barrier layer of Mn. Ru (1.5 nm) on a 100 nm thick BD layer on 300 nm Si wafers, with an intervening barrier layer of Mn (~1.5 thick Mn film between Ru and BD, Cu (500 nm thick) films were electrodeposited on a Ru (~2 nm)/Mn (~1.5 nm) on a 100 nm thick BD layer, also on 300 mm Si wafers. All these wafers were diced into 1” × 1.14” rectangular coupons for polishing experiments and 0.75” × 0.75” coupons for use as the working electrodes in electrochemical experiments. Reagent grade chemicals obtained from Sigma-Aldrich were used in all the experiments without further purification. The pH of the aqueous solutions containing the additives was adjusted using KOH as necessary.

Experimental

Electrochemical experiments.—An Autolab 100 V potentiostat and a three-electrode glass cell (200 mL volume) with a Pt counter electrode and a silver/silver chloride reference electrode were used to acquire the potentiodynamic polarization and open circuit potential (OCP) data. Experiments were performed using a 1 M KOH electrolyte at room temperature. The coupon anode was exposed to an area of the coupon exposed to the KOH electrolyte. All electrochemical experiments were performed using a three-electrode glass cell (200 mL volume) with a Pt counter and a silver/silver chloride reference electrode.

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As the node sizes continue to diminish, the 5 ~ 7 nm thick Ta/TaN barrier-liner will occupy a significant portion of the Cu trench resulting in higher effective resistance and increased resistance-capacitance delay.1 Also, with the even smaller feature sizes and high-aspect-ratio trenches of these nodes, deposition of a conformal Cu seed layer on the Ta liner is a major challenge with any discontinuities in it leading to voids in the copper fill and limiting the yield.1 Hence, it has become necessary to replace these Ta/TaN barrier-liner films with thinner and more functional candidate materials. One such candidate is a Mn/Mn-based film since it can form a very thin (~1 nm) self-forming and excellent Cu diffusion barrier layer of amorphous manganese silicate (MnSiOx) at the Mn/Ta interface (black diamond or SiCOH with k = 2.9 or lower)4,5 interface. However, this MnSiOx layer is not conductive enough and requires a thin conductive liner for the deposition of a Cu seed layer. A ~2 nm thick Ru film with a resistivity of ~7 μΩ cm, much lower than that of Ta’s ~13 μΩ cm, has been proposed6,11 as such a liner. As always, these films stacks need to be planarized and for effective planarization, as shown in Figure 1, the removal rate (RR) selectivities among the Cu, Ru, Mn and SiCOH or BD layers and the galvanic corrosion between Cu/Ru and Ru/Mn couples need to be controlled. With that goal in mind, we investigated the polishing and electrochemical behavior of the relevant Mn-based barrier/Ru liner stack using silica based dispersions and various additives. For convenience, in the following the Mn-based films will be addressed simply as Mn films.

Cu and Ru polishing behavior12–18 and related corrosion issues19–23 for barrier applications have been studied by several authors. Among these, Amanapu et al.18 showed that the underlying substrate can modify the crystalline orientation of thin Ru films with a corresponding crystalline orientation dependence (RR) selectivities among the Cu, Ru, Mn and SiCOH or BD layers and the galvanic corrosion between Cu/Ru and Ru/Mn couples need to be controlled. With that goal in mind, we investigated the polishing and electrochemical behavior of the relevant Mn-based barrier/Ru liner stack using silica based dispersions and various additives. For convenience, in the following the Mn-based films will be addressed simply as Mn films.

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to the electrolytes. A fresh sample was used for each electrochemical experiment. Reagent grade chemicals supplied by Sigma Aldrich were used in all the experiments without further purification. The pH of the aqueous solutions containing the additives was adjusted using KOH.

**Polishing experiments and removal rate measurements.**—All the polishing experiments were performed on an Alpseitec E460 Mecapol polisher at IMEC. Cu, Ru, Mn and BD coupons (1” × 1.14”) diced out of 300 mm wafers described above were used for the polishing using a custom made carrier that can hold these coupons. A 20” diameter Fujibo pad attached onto a platen was used for all the polishing experiments and was cleaned with a DI water jet for 1 min after every polish. All the polishing experiments were performed at a polishing pressure of 2 psi, with a slurry flow rate of 200 mL/min, and platen/carrier speeds of 72/80 rpm. Colloidal silica abrasives (Nexsil 35A) of average diameter ~35 nm obtained from Nyacol Nano Technologies were used to prepare the dispersions for the polishing experiments.

The removal rates for Cu and Ru films were measured from the change in their thickness determined by measuring the resistance of these films using a four-point probe at three different locations from one corner to another along a diagonal of the coupon and averaged. The RRs of Mn films were determined using the X-ray fluorescence technique described below. Each experiment was conducted at least twice and these data were used to compute the average RRs and the associated error bars.

**Dissolution rate measurements.**—Ru and Mn coupons (1” × 1.14”) were dipped for 1 minute and 5 minutes in 200 mL of the desired solutions stirred at ~200 rpm. The temperature of these solutions was maintained at 40°C. The dissolution rates of Mn films were determined using the X-ray fluorescence technique from the change in the normalized intensity count of Mn atoms before and after the dissolution experiment. Each experiment was conducted at least twice and these data were used to compute the average RRs.

**X-ray fluorescence (XRF).**—The deposition of Mn-based, on BD films (~2 nm thickness) was developed earlier by Jourdan et al. and the thickness of these films was confirmed by Transmission Electron Microscopy (TEM). Based on this deposition technique, ~1.5 nm thick Mn films were prepared and diced to 1” × 1.14” sized coupons. X-ray fluorescence analysis was performed on these coupons using the SPECTRO XEPOS energy dispersive X-ray fluorescence spectrometer and Palladium Kα X-ray source (21.2 keV) available at IMEC. The resulting intensity counts of the Mn atoms present in the film were then used as a basis to normalize the intensity counts obtained with other coupons. Pre and post-polished coupons and those dipped in different solutions to determine dissolution rates were analyzed using this XRF instrument and the Mn film thickness was determined based on the resulting counts normalized by the counts obtained with the ~1.5 nm thick films. The accuracy of these measurements is ~0.25 nm.

**X-ray diffraction (XRD).**—Grazing-incidence X-ray diffraction (GIXRD) (ω = 0.5°) analysis was performed using a Bede Metrix-L diffractometer and Cu Kα radiation with a wave length of 1.54 Å to determine the crystalline orientation of the Ru films deposited on a Mn substrate. GIXRD was chosen since these films are relatively thin (~30 nm).

**X-ray photoelectron spectroscopy (XPS).**—XPS spectra were acquired using a Surface Science Instrument model SSX-100 spectrometer using a monochromatic Al Kα X-ray source (1486.6 eV) at the Cornell Center for Materials Research, NSF MRSEC program (DMR - 1120296). Data reported were obtained using an electron emission angle of 55° and a pass energy of 150 eV for survey scans and 50 eV for high resolution scans.

The scans were made on ~1.5 nm thick pristine Mn films on a ~100 nm TEOS based BD films on a 300 mm Si substrate. The spectral data were analyzed using CASA XPS software. The Shirley background was used in the analysis of the survey scans and symmetric Gaussian-Lorentzian-product peak shape function, with a Lorentzian content of about 15%, was used in the analysis of high resolution scans.

**Transmission electron microscopy (TEM).**—A Tecnai F30 ST TEM from FEI with a FEG electron source and operating at 300 kV was used to investigate the polished Cu/Ru/Mn/BD patterned stack coupons. Focused Ion Beam (FIB) cut samples were prepared using Helios 450HP from the post-polished coupons. EDS mapping and atomic concentration profiles at a particular location on the FIB cut sample were obtained using an EDS detector. All these measurements were made at IMEC.

**Results and Discussion**

**RRs, DRs and XRD analysis of Ru on Mn substrates.**—As stated earlier, in our experiments here, we used the same 10 mM KMnO4 + 1 wt% guanidine carbonate (GC) + 1 mM BTA (referred to as the reference solution the following) solutions at pH 10 that was shown by Sagi et al. to minimize galvanic corrosion between Ru and Cu and as well yielded adequate RRs of Ru on TiN and Cu films when 5 wt% silica abrasives were added to the above solution. The measured RRs and DRs of Ru on Mn/BD films with this same dispersion are ~17 nm/min and <1 nm/min, respectively, similar to rates reported by Amanapu et al. and Sagi et al. for Ru films on TiN substrate.

So, not surprisingly, when the crystalline structure of the Ru film on Mn/BD substrate was determined using GIXRD technique, we found the same mix of (002) and (101) orientations (see Figure 2) that was also reported by Amanapu et al. Thus it appears that the
**Figure 2.** GIXRD analysis of as-deposited Ru film on Mn substrate.

Crystalline orientation is the primary factor in determining the RRs of the Ru films in this particular slurry.

**XPS analysis of Mn based film surfaces.**—Figure 3 shows the high resolution scans of O 1s, Si 2p and Mn 2p for ~1.5 nm Mn films on BD substrate. Deconvolution of the O 1s high resolution scan confirmed the presence of three different peaks at 529 eV, 531 eV and 532.5 eV. The peak at 532.5 eV corresponds to the SiCOH component and the other two peaks on the lower binding energy side at 531 eV and 529 eV can be attributed to the presence of MnSi6Oy at the Mn/BD interface and MnOz, respectively, consistent with the reports of several other authors.6–8,10,26–28 The presence of manganese silicate is also confirmed by the Si 2p and Mn 2p spectra at 101.5 eV and 643 eV, respectively. The presence of MnOz oxide in the Mn film is likely due to its growth from available physisorbed moisture on BD and exposure to the ambient atmosphere and was reported already.7,28

It was reported by several authors29–31 that this MnSi6Oy and MnOz bilayer, where Mn is present in the oxidized state of +2 at the Mn/BD interface, behaves as a dielectric film with a dielectric constant $k \approx 11$. Hence, the potential for galvanic corrosion between Ru and Mn is non-existent. Also, since Mn oxide is known to act as a passive layer that inhibits the corrosion of various metal films during polishing,23,32,33 we can also expect minimal corrosion behavior as well as no film loss due to dissolution. This is confirmed by the results described below.

**RRs and DRs of Mn films.**—The RRs of the Mn films obtained using dispersions containing 5 wt% silica abrasives and various additives at pH 10 are shown in Table I. The very thin nature of these films (~1.5 nm thickness and less) poses a difficult challenge for determining the precise values of the RRs. The entire 1.5 nm thin film, comprising of MnOz and MnSi6Oy bilayer, was removed within five

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**Figure 3.** High resolution O 1s, Si 2p and Mn 2p XPS scans of pristine Mn film.
Table I. Removal rates and dissolution rates of Mn and BD films in solutions of DI water, 10 mM H2O2, 10 mM MnO4 - and 10 mM KMnO4 + 1 wt% guanidine carbonate (GC) + 1 mM BTA (reference solution), all at pH 10.

| Slurry (5 wt% Silica + additive(s)), all at pH 10 | DI Water | KMnO4 | Reference solution |
|-------------------------------------------------|----------|-------|--------------------|
| Removal rate (nm/min) Mn film                    | >18      | >18   | >18                |
| BD film                                         | ~25      | ~25   | ~25                |
| Dissolution rate, 1 min (nm/min) Mn film         | <1       | <1    | <1                 |
| BD film                                         | <1       | <1    | <1                 |

Table II. Normalized intensity counts of pristine Mn film coupons (~1.5 nm thickness) and post-polish/post-dissolution coupons for experiments performed with reference solution and with silica abrasives at pH 10.

| Mn sample                  | Normalized Intensity count of Mn atoms |
|----------------------------|----------------------------------------|
| Pristine (~1.5 nm thick)   | 30 ± 3                                 |
| post-polished              | < 2                                    |
| post-dissolution           | 24 ± 2                                 |

As reported earlier by Ablett et al. and Casey et al., a Mn film that is thicker than ~2 nm (let alone a disk) will contain unreacted metallic Mn and MnO2 over the MnSiO4 layer formed at the Mn and BD interface. Hence, the RRs and corrosion properties of thicker Mn films will be quite different from the thin ~1.5 nm films we investigated in this study.

We have also performed the OCP measurements on the ~1.5 nm Mn film on BD and the BD films themselves with various other solutions and the results are shown in Figure 4. As was the case with RRs and DRs, the OCP behavior of Mn converted to MnSiO4 and BD films is very similar for all the solutions tested at pH 10. Also, the potentiodynamic polarization behavior of Mn films in solutions other than reference solution at pH 10 remained the same without any faradaic signal (data not shown).

Potentiodynamic data were obtained in the potential range of EOC ± 300 mV at a 5 mV/s scan rate to determine the corrosion behavior of Cu, Ru and Mn films in the presence of the reference solution using a rotating (~150 rpm) disk electrode and are shown in Figure 5.

RRs selectivities and galvanic corrosion during polishing of the Cu/Ru/Mn/BD Stack.—The RRs of Cu, Ru, Mn and BD films were measured in dispersions containing 5 wt% silica and the reference solution at pH 10 and are shown in Table III. The RRs of Cu and Ru are ~22 mm/min and ~17 m/min, respectively. This RR ratio of ~1.2 minimizes the potential for dishing of Cu considerably. Also, since the ~1.5 nm thin Mn film is the last layer to be removed, it will not pose any galvanic corrosion or corrosion problems.
Mn layers, there would be no problem even if the Mn layer RR is considerably higher than the lower limit of 18 nm/min. The RR of the underlying BD layer is ~25 nm/min. Finally, since no dissolution was observed for all these films in the presence of reference solution at pH 10 even at 40°C, effective planarization of patterned structures should be realized, as shown later.

**Polishing results of Cu/Ru/Mn/BD pattern stack.**—It is well known that the polish results obtained on blanket films do not always carry over to patterned structures. Hence, to validate the usefulness of the composition identified here, it is necessary to planarize Cu/Ru/Mn/BD patterned structures and determine the results. A 300 mm diameter test wafer was used for this purpose. It consisted of ~32 half pitch Cu lines with Cu/Ru (~2 nm)/Mn (~1.5 nm) patterned structures with ~500 nm thick Cu electrodeposited on 100 nm BD layer with an intervening Ru (~2 nm)/Mn (~1.5 nm) stack on the Si substrate and was initially polished to leave only ~25 nm of Cu overburden with a CSL9044C Cu clear slurry, a commonly used but not a standard slurry. The patterned wafer was then diced into 1” × 1.14” coupons that were polished either for 45 seconds or 60 seconds using the dispersion identified in this work, i.e. 5 wt% silica in the reference solution at pH10. After polishing, the coupons were sonicated in the reference solution at pH 10 for 5 minutes to ensure that the surface is free of contamination. TEM analysis of these polished and sonicated coupons showed that the coupon polished for 45 seconds had a few nm of Cu overburden remaining (picture not shown) but the Cu overburden was completely removed in the coupon polished for 1 min. Hence, only these coupons were further analyzed and some typical cross-sectional TEM images and associated EDS chemical maps along with the atomic concentration profiles are shown in Figures 6 and 7, respectively. The cross-sectional TEM images show a very good polished surface with a Cu dishing of ~3 nm and with no barrier or liner loss at the Cu/Ru/Mn/BD interfaces in the ~32 nm half pitch Cu lines. No polish residue was observed on the interlayer dielectrics in the cross-sectional TEM images across various locations on the FIB cut sample (as shown in Figure 6). Also, there was no loss of Ru or Mn, supporting the effectiveness of the dielectric MnSiO_x/O_y bilayer.

The EDS chemical map (Figure 7) of the polished coupon at one particular location opened up by FIB clearly differentiates the Mn and Ru layers and confirms a continuous and conformal deposition of these layers along the sidewalls of the Cu filled trench. The maps and the line scan profile also confirm that there is no Cu diffusion into the surrounding dielectric region suggesting that the MnSiO_x layer is an excellent barrier layer. The formation of this MnSiO_x layer is also confirmed by the presence of small atomic concentrations of Si and O along with the Mn atoms, as can be seen in the EDS line scan profile shown in Figure 7.

**Conclusions**

The RR of Ru on Mn substrate and of Mn on BD substrate films were measured using a dispersion of 5 wt% silica + 10 mM KMnO_4 + 1 wt% GC + 1 mM BTA at pH 10, a dispersion that was shown earlier to be effective in minimizing galvanic corrosion of the Ru/Cu couple. XPS analysis confirmed the formation of a manganese silicate barrier layer and the electrochemical data suggest that this layer acts as a dielectric and hence eliminates galvanic corrosion between Ru and Mn. Finally, patterned test coupons with Mn as barrier and Ru as liner (Cu/Ru/Mn/BD stack) and 32 nm half pitch Cu lines were polished and the results were analyzed using TEM analysis and EDS mapping. The data showed very good post-polish performance with no barrier or liner loss at the Cu/liner/barrier interface. Cu line dishing was limited to ~3 nm.

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