ELSEVIER

Materials Chemistry and Physics 51 (1997) 75–79

MATERIALS CHEMISTRY AND PHYSICS

Chemical mechanical polishing for selective CVD-W

M.T. Wang [a,*], W.K. Yeh, M.S. Tsai [b], W.T. Tseng [b], T.C. Chang [b], L.J. Chen [c], M.C. Chen [a]

[a] *Department of Electronics Engineering, National Chiao-Tung University, Hsinchu, Taiwan, ROC*
[b] *National Nano Device Laboratory, 1001 Ta Hsueh Rd., Hsinchu, Taiwan, ROC*
[c] *Department of Submicron Technology Development, ERSO/ITRI, Hsinchu, Taiwan, ROC*

Received 18 December 1996; revised 15 May 1997; accepted 15 May 1997

Abstract

This work investigates chemical mechanical polishing (CMP) for W-filled contact holes, vias, and trenches by selective chemical vapor deposition. A novel process that combines the CMP technique with selective chemical vapor deposition of tungsten (CVD-W) was employed to remove nail heads due to overgrowth and W-particles on the surface of dielectric due to selectivity loss. The overfilled nail heads and the selectivity loss can be completely removed with very low down-pressure (3 psi) in a very short polishing time (30 s). This indicates that the novel process is very promising for ULSI multilevel interconnection application. The removal rate selectivities of W to thermal oxide, PECVD-TEOS, and BPSG were found to be 47:1, 30:1 and 15:1, respectively, while the selectivities of W to the barrier metals of TiW, Ti and Ta were determined to be 0.6:1, 6:1 and 28:1, respectively. © 1997 Elsevier Science S.A.

Keywords: Selective tungsten chemical vapor deposition; Chemical mechanical polishing; Removal rate selectivity

1. Introduction

As device dimensions in integrated circuits are continually reduced, the requirements imposed on the advanced metallization become increasingly stringent. These include improved photolithographic margin, increased process window, and reduced defect density. Chemical mechanical polishing (CMP) has been proposed as a viable technique to meet the above requirements [1,2]. For industrial application, poor productivity is the main disadvantage of the W-CMP planarization process. The low throughput of W-CMP results in high process cost per wafer.

The selective chemical vapor deposition of tungsten (CVD-W) is in principle much simpler than the blanket approach for contact holes or vias filling, because no adhesion layer and etch back process is needed [3–5]. However, the selective-W approach is not adequate for contacts and vias with different heights which occur in many applications. Thus, an appropriate post-deposition treatment is necessary to eliminate the selectivity loss, and to remove the nail heads formed due to overfilled deposition and depth variation of the contacts.

In this study, CMP was employed to eliminate the selectivity loss and to remove the nail heads. A novel process that combines the CMP technique with the selective CVD-W process was developed to promote the productivity of W-CMP. Removal rate selectivities of W to dielectrics (thermal oxide, PECVD-TEOS, and BPSG) and barrier metals (TiW, Ti and Ta) were also investigated.

2. Experimental

Three types of samples were prepared on 6 inch Si wafers in this work: blanket tungsten, selective CVD-W on contact holes, and selective CVD-W on vias/trenches. Blanket tungsten films were directly deposited on bare Si wafers for the purpose of determining the optimal deposition condition as well as the proper W-CMP process. For the contact holes samples, contact holes with sizes ranging from 0.4 to 1.0 μm were patterned on 5000 Å thick thermal oxide using the conventional photolithographic and dry etching technique. For the vias/trenches samples, a four-layer structure of Ti/TiN/AlSiCu/TiN was sputter-deposited on thermally oxidized Si wafers to the thicknesses of 500/1000/5000/400 Å. The samples were then patterned with reactive ion etching (RIE) and subsequently covered, in sequence, with dielectric layers of 2000 Å plasma-enhanced chemically vapor-deposited (PECVD) oxide, 3000 Å spin-on-glass (SOG), and 3000 Å PECVD oxide. Vias with sizes ranging from 0.4 to 1.0 μm were patterned using dry etching technique and the 400 Å

* Corresponding author.

0254-0584/97/$17.00 © 1997 Elsevier Science S.A. All rights reserved
PII S0254-0584(97)01965-2

TSMC 1006

Table 1
General specifications of the slurry for W-CMP

Slurry	W-A355/FE10
Oxidizer	$Fe(NO_3)_3$
pH value	1.7
Abrasive material	Al_2O_3
Solid content	6%
Mean abrasive particle size	100 nm

Table 2
Experimental parameters used for W-CMP

Platen/carrier speed	32/28 rpm
Down-pressure	3.0 psi
Back-pressure	2.0 psi
Slurry flow rate	150 ml min^{-1}
Temperature	29–30°C
Pad	IC-1000/SUBA IV

TiN layer was removed during the RIE. Trenches with widths ranging from 0.6 to 1.2 μm were also patterned by RIE.

Prior to the CVD-W, precleaning of the wafers was carefully performed. The blanket and contact hole samples were dipped in dilute HF solution (50:1) to remove the native oxide. The vias/trenches samples were dipped in a 0.1 M solution of hydroxylamine sulfate [$(NH_2OH)_2 \cdot H_2SO_4$] at 60°C for 2 min with in situ ultrasonic agitation [4]. The wafers were loaded into a load-locked cold wall CVD system within 5 min of precleaning. The selective CVD-W process was used to deposit W on the contact holes and vias/trenches samples. For the contact holes filling, selective CVD-W was performed at 300°C and a total pressure of 100 mTorr, while for the vias/trenches filling, the deposition was carried out at 250°C and a pressure of 8 mTorr. Tungsten was overfilled to ensure that the W-plugs and vias/trenches were stuffed completely.

After selective CVD-W, the overfilled W and the selective loss were removed by W-CMP using a commercially available CMP polisher [Westech-372M]. Table 1 shows the general specifications of the slurry for W-CMP, and Table 2 shows the experimental parameters of W-CMP used in this work.

A scanning electron microscope was employed to characterize surface morphology, step coverage, and deposition rate of W. The tungsten films' crystalline phase was examined by X-ray diffraction analysis. To evaluate removal rate selectivities of W for various dielectrics (thermal oxide, PECVD-TEOS, and BPSG) and barrier metals (TiW, Ti and Ta), CMP was performed under the conditions shown in Table 2. An ellipsometer was used to measure the thickness of the dielectrics. For barrier metals, a four-point probe was used to measure the sheet resistance at 13 points across the wafer, and the change of sheet resistance before and after CMP was used to determine the removal rate of metals.

3. Results and discussion

The resistivity and deposition rate of the blanket W-film varies with WF_6/SiH_4 flow rates. Fig. 1 shows that both the resistivity and the deposition rate increase with increasing SiH_4 flow rate at a constant WF_6 flow rate of 20 sccm. The larger amount of Si incorporated into the W-film is presumably responsible for the higher resistivity [6]. Grain structures of the blanket W films deposited with different WF_6/SiH_4 flow rate ratios are fairly different, as shown in Fig. 2. The X-ray diffraction patterns of the W films deposited on bare Si with different WF_6/SiH_4 flow rates are illustrated in Fig. 3. The α-W signals were detected on the films deposited with lower the SiH_4 flow rate of 10 sccm (Fig. 3(a)), while the β-W signals were detected on the films deposited with the higher SiH_4 flow rate of 30 sccm (Fig. 3(b)). The α-W phase, which has the b.c.c. type-A2 lattice, is desirable for ULSI application because it is thermodynamically stable and exhibits the lowest resistivity among all crystalline phases of tungsten [7]. The β-W phase has a cubic, type-A15 lattice, and is metastable below 630°C [8]. For the WF_6/SiH_4 flow rate ratio less than 1/2, amorphous WSi_x film was obtained (Fig. 3(c)) and the deposition selectivity was lost completely [9].

Fig. 4 shows the half-filled and overfilled contact holes of 1.0 μm size using selective CVD-W technique. Excellent selectivity, good uniformity, and low resistivity W can be

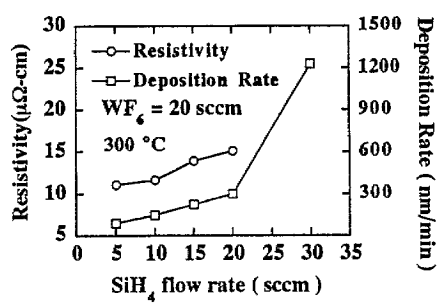

Fig. 1. Resistivity and deposition rate of blanket CVD-W vs. SiH_4 flow rate at a fixed WF_6 flow rate of 20 sccm.

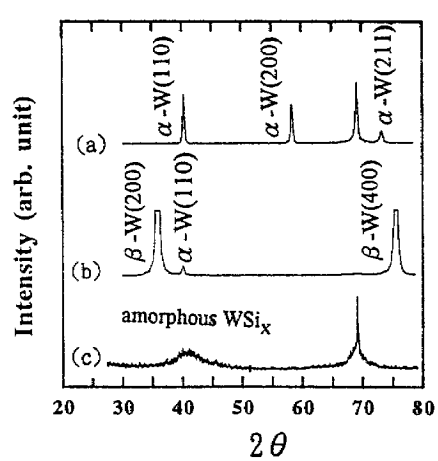

Fig. 2. XRD spectra of tungsten films deposited on bare Si substrate with WF_6/SiH_4 flow rates of (a) 20/10, (b) 20/30 and (c) 2/30 sccm, respectively.

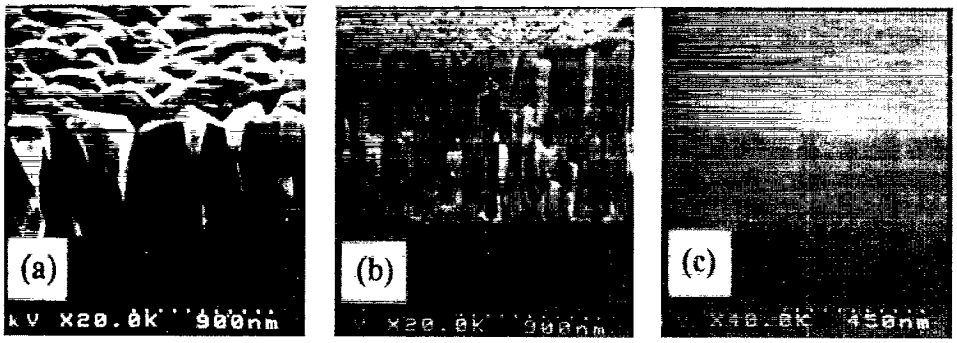

Fig. 3. Scanning electron micrographs showing different structures of blanket tungsten films deposited with WF_6/SiH_4 flow rates of (a) 20/10 sccm, (b) 20/30 and (c) 2/30 sccm, respectively.

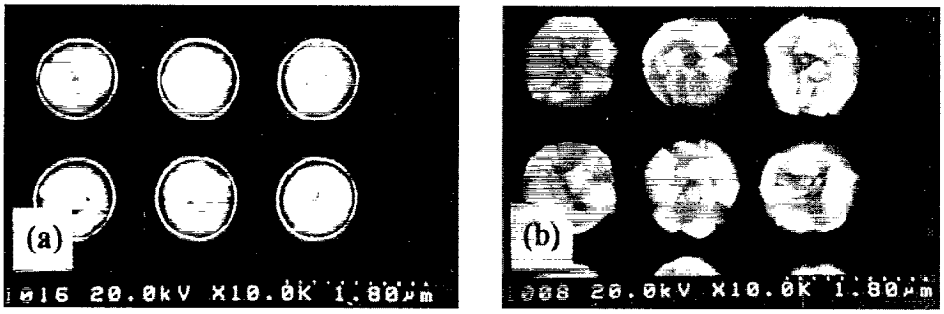

Fig. 4. (a) Half-filled and (b) overfilled contact holes with size of 1.0 μm using selective CVD-W process.

obtained with the WF_6/SiH_4 flow rate of 20/10 sccm. Thus, the WF_6/SiH_4 flow rate of 20/10 sccm was used to deposit tungsten for CMP study in this work. Compared with the blanket W, the total volume of W that needs to be removed during the W-CMP process is very small for the selective CVD-W, as shown in Fig. 5. Only a small volume of the nail heads due to overgrowth and some W-particles on the surface of dielectric due to selectivity loss need to be removed. The belly-shaped vias were formed as a result of the different etching rate between SOG and PECVD oxide during the via etching [4].

CMP was firstly applied to polish the blanket tungsten films with a thickness of 8000 Å. A very smooth surface can be obtained at a down-pressure of 3 psi and a back-pressure of 2 psi with a removal rate of 1400 Å min^{-1}. Before CMP, the tungsten film has a rough surface, as shown in Fig. 6(a); the surface roughness was flattened and a scratch-free surface was obtained after the CMP process, as shown in Fig. 6(b).

Fig. 7 shows the results of a processing series of pattern definition (Fig. 7(a)), selective CVD-W (Fig. 7(b)), W-CMP (Fig. 7(c)), and post-CMP cleaning (Fig. 7(d)) in sequence performed for 0.6 μm trenches. The overgrowth of CVD-W on the trenches was completely removed after a 30 s W-CMP process; however, some abrasive particles (Al_2O_3) remained, as shown in Fig. 7(c). A dilute ammonia solution (0.6%) was used to remove the contamination from slurry and/or from dirty CMP environments. With such a post-CMP cleaning step, no abrasive particle residues can be observed, as shown in Fig. 7(d) [10,11]. Increased ammonia content in the solution tends to induce corrosion of tungsten; Fig. 8 shows a serious corrosion of tungsten following a 120 s post-CMP cleaning in a 5% ammonia solution.

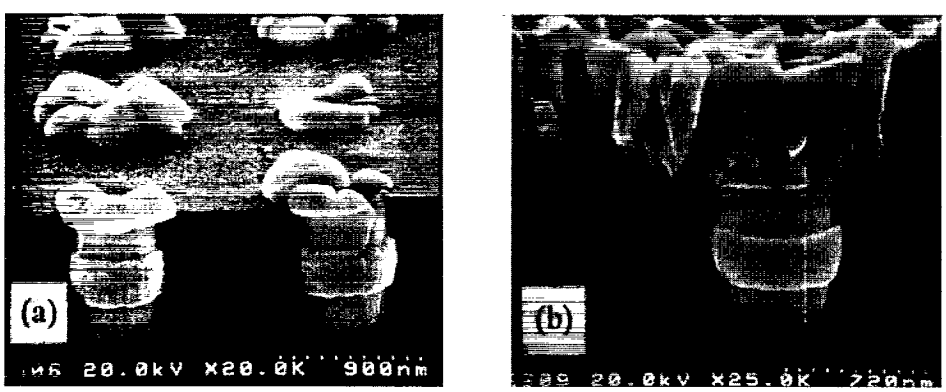

Fig. 5. The total volume of tungsten films needs to be removed during W-CMP process is smaller for (a) selective CVD-W, as compared with (b) blanket deposition.

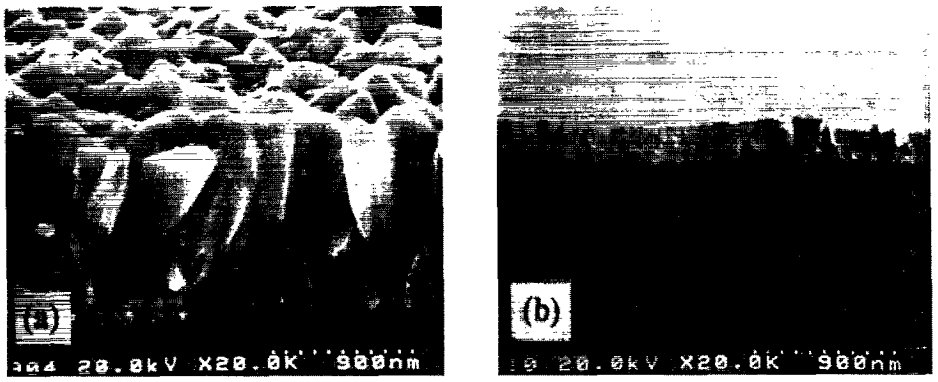

Fig. 6. Scanning electron micrographs showing the surface of a blanket tungsten film (a) before, and (b) after a CMP process.

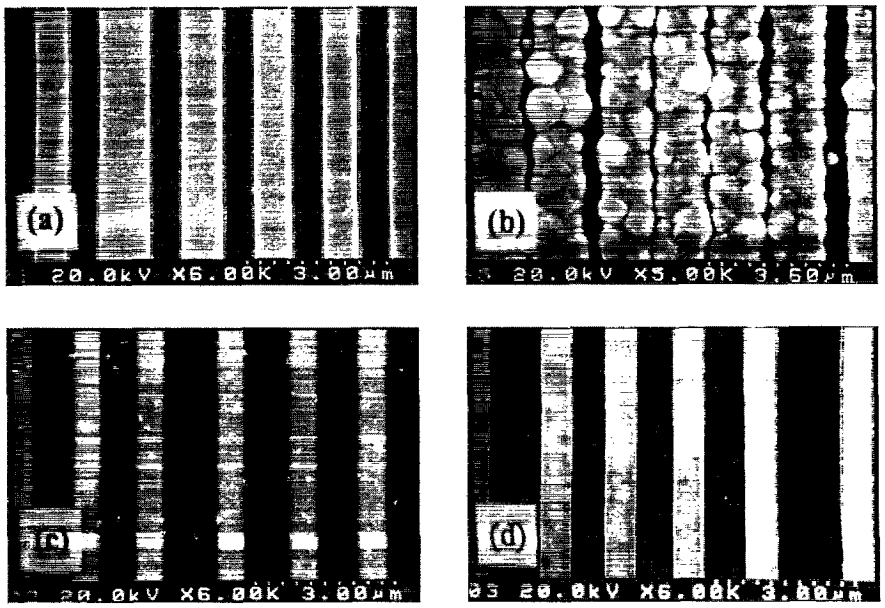

Fig. 7. Scanning electron micrographs showing the results of a series of processing in sequence: (a) pattern definition, (b) selective CVD-W, (c) W-CMP process, and (d) post-CMP cleaning. The pattern contains 0.6μm trenches.

Fig. 8. Scanning electron micrographs showing a serious corrosion of tungsten resulting from a 120 s post-CMP cleaning in a 5% ammonia solution.

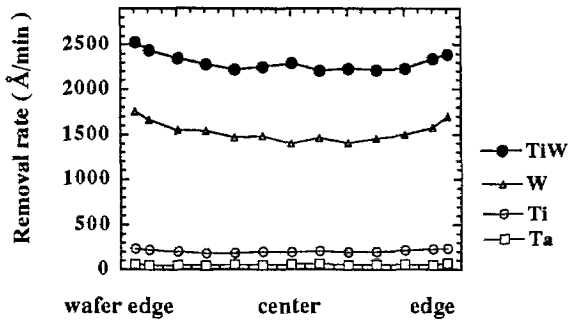

Fig. 9. Removal rate selectivities of W to barrier metals.

The overfilled nail heads of selective CVD-W and the tungsten particles deposited on the surface of dielectric due to selectivity loss can be removed effectively even using a very low down-pressure of 3 psi in a very short polishing time of 30 s. The lower down-pressure of W-CMP reduces surface roughness and extends pad lifetime, and the shorter polishing time promotes throughput of CMP and reduces consumption of slurry; thus, the process cost of W-CMP can be reduced effectively. The removal rate selectivities of W to various dielectrics and barrier metals were also evaluated with the CMP process performed under the conditions shown in Table 2. The removal rates of thermal oxide, PECVD-TEOS, and BPSG were found to be 30, 45 and 95 Å min^{-1}, respectively; thus the removal rate selectivities of W to thermal oxide, PECVD-TEOS, and BPSG were determined to be

47:1, 30:1 and 15:1, respectively. The removal rates of the barrier metals of TiW, Ti and Ta were found to be about 2300, 240 and 50 Å min^{-1}, respectively. These removal rates together with those of W measured at various positions across a 6 in substrate are illustrated in Fig. 9. The removal rate selectivities of W to TiW, Ti, and Ta were determined to be 0.6:1, 6:1 and 28:1, respectively, in the center part of substrate. The results of this study indicate that this novel process is very promising for ULSI multilevel interconnection application.

4. Conclusions

A novel process that combines selective CVD-W with the W-CMP technique was developed. This novel process not only improves the throughput of W-CMP dramatically, but also extends the pad lifetime and reduces the consumption of slurry; thus, the process cost of W-CMP can be reduced. Overfilled nail heads and selectivity loss can be removed effectively using a very low down-pressure of 3 psi in a very short polishing time of 30 s. The lower down-pressure of W-CMP reduces surface roughness and extends pad lifetime, and the shorter polishing time promotes throughput of CMP and reduces consumption of slurry; thus, the process cost of W-CMP can be reduced effectively. Furthermore, the removal rate selectivities of W to thermal oxide, PECVD-TEOS, and BPSG were found to be 47:1, 30:1 and 15:1, respectively. These results indicate that the novel process developed in this study is very promising for ULSI multilevel interconnection application.

Acknowledgements

This work was supported by the National Science Council (ROC) under Contract No. NSC85-2215-E-009-068.

References

[1] F.B. Kauffman, D.B. Thompson, R.E. Broadie, M.A. Jaso, W.L. Guthrie, D.J. Pearson and M.B. Small, J. Electrochem. Soc., 138 (1991) 3460.
[2] M.A. Fury, Solid State Technol., 38 (April 1995) 47; 38 (July 1995) 81.
[3] W.K. Yeh, M.C. Chen and M.S. Lin, J. Electrochem. Soc., 142 (1995) 3582.
[4] W.K. Yeh, T.C. Chang, K.Y. Chan and M.C. Chen, Jpn. J. Appl. Phys., 35 (1996) 1115.
[5] W.Y. Yeh, M.C. Chen and M.S. Lin, J. Vac. Sci. Technol. B, 14 (1996) 1.
[6] J.E.J. Schmitz, Chemical Vapor Deposition of Tungsten and Tungsten Silicides for VLSI/ULSI Applications, Noyes, Park Ridge, NJ, 1991, p. 63.
[7] W.B. Pearson, Handbook of Lattice Spacings and Structures of Metals and Alloys, Pergamon, London, 1958, p. 884.
[8] C.J. Smithells, Metals Reference Book, London, 1955, p. 195.
[9] M.L. Yu and B.N. Eldridge, J. Vac. Sci. Technol. A, 7 (1989) 1441.
[10] S.R. Roy, I. Ali, G. Shim, N. Furusawa, R. Shan, S. Peterman, K. Witt and S. Eastman, J. Electrochem. Soc., 142 (1995) 216.
[11] T.L. Myers, M.A. Fury and W.C. Krusell, Solid State Technol., 38 (October 1995) 59.