Chemical-Mechanical Polishing for Fabricating Patterned W Metal Features as Chip Interconnects

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ABSTRACT

Interconnect features of W metal, recessed in an SiO_2 dielectric, can be formed using a novel chemical-mechanical polish process. Mechanical action, to continually disrupt a surface passivating film on W, and chemical action, to remove W, appear to be requirements for workability of the process. A trial process chemistry using a ferricyanide etchant is described. Removal of the W is discussed in terms of competition between an etching reaction which dissolves W and a passivation reaction to reform WO₃ on the surface of the W. This novel processing technology is compared with earlier methods of fabricating metal interconnect structures.

It is now generally recognized that planarization of multilevel metal interconnect structures for chips offers significant process advantages such as the elimination of stepcoverage concerns and improvement of lithographic resolution by minimizing depth of field variations (1-7). Two alternative process approaches to achieving planarization can be considered (Fig. 1). One approach relies on dielectric planarization. In this case patterned metal interconnects are conformally covered with an insulator film. This is followed by a planarizing operation to eliminate the topography in the dielectric. Processes that have been used for smoothing include etch-back (8) of the dielectric, deposition of a planarizing polymer film (9), or chemicalmechanical polishing (10). Alternatively, recessed metal schemes (1-5) can be used where the reverse of the metal pattern desired is etched into a planarized film of dielectric, metal is conformally deposited, and then subsequently removed, in a separate step, from the higher blanket areas on top of the planarized dielectric to leave the required metal pattern recessed in the insulator.

An improved low-pressure chemical vapor deposition (LPCVD) process (11, 12) for tungsten, coupled with its high electromigration resistance and dry etch compatibility, makes it an attractive candidate for use in a recessed metal interconnection sequence (3, 5). However, current reactive ion etching (RIE) processes for tungsten which have been used to remove the blanket metal suffer from selectivity concerns due to resist thickness variations over the metal (3). Wet etching techniques are unacceptable due to their inability to preferentially remove topographic features.

Chemical-mechanical polishing (1, 2) could be used to define the tungsten features if a process chemistry appropriate for wafer fabrication were available. Chemicalmechanical polishing of metals has been demonstrated for stainless steels and nickel-based alloys (13), and for copper (2, 14). The mechanism we propose for the process (Fig. 2) requires the action of a metal etchant and a metal passivating agent with an abrasive agent. This combination could result in the high spots continually having the passivated film (anisotropically) etched away, while the low spots are protected. For the majority of metals, the oxide may be used as a simple passivant. During the process, the protective film is removed by the mechanical action of the abrasive slurry. This is followed by a rapid reformation of the protective film. Continuous cycles of formation, removal, and reformation of the passivating layer continue until the desired final thickness of metal is achieved.

Consideration of the proposed mechanism and Fig. 2 suggests that the minimal requirements for the proposed process chemistry for W removal are: (i) materials selectivity, a significantly faster removal rate for the W than either the dielectric surface, which forms the structure, or a sacrificial etch stop; (ii) topographic selectivity, a metal removal process which selectively removes metal from the

"high" spots while leaving it protected in the low spots; (*iii*) the overall process should be noncorrosive, (*iv*) the process should leave the wafers clean enough to be compatible with further semiconductor processing in a clean room.

This paper will describe a process chemistry that has been successfully used in the chemical-mechanical polishing of tungsten to form chip interconnect structures using a recessed metal process sequence. Given the nature of the polish mechanism described, we suggest that the particular etchant-passivator combination discussed here is not the only chemical system that could be used for successful fabrication of interconnect structures. Preliminary descriptions of the application of this technology to form a fully planarized interconnect structure with 1.2 μ m contacts, as applied to a 64 Kb complimentary metal oxide semiconductor static random access memory (CMOS SRAM), and, using x-ray lithography to achieve 0.5 μ m on all interconnect levels, have previously appeared (6, 7).

Experimental

Materials and preparation of slurry.—All chemicals were used without further purification. Typical asreceived purity levels were at least 98%. Water used in the preparation of the slurry had resistivities greater than 18 MΩ and was filtered through a Millipore filter system, type RO to remove ionic and organic contaminants and particles less than 1 μ m in size.





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Fig. 2. Proposed mechanism of planarization of a patterned metal feature by chemical-mechanical polishing (see text for details).

All slurries were prepared by adding the chemical constituents to a rapidly stirred solution of water. Addition of the abrasive to the solution typically resulted in the solution becoming optically opaque. To insure homogeneity of the polishing slurry, the resulting suspension was stirred during use. Although there was evidence for precipitate formation in the slurry after several hours, polish results were not found to change after storage for times up to 8 h. All slurries were delivered to the polish table using a peristaltic pump.

Slurry particle size analysis.—A Coulter Model LS 130 laser diffraction particle size analyzer was used to measure the particle size of the free abrasive in the polish slurry. Size calibration was checked using appropriate standards in the 0.1-100 μ m particle size regime. Polish slurry particle sizes were determined on flowing samples (recirculation rates of 0.5 liter/min) using dilution factors (in water) in the 0.1 to 0.5 range.

Chemical-mechanical polish tools, fixtures, and operating parameters.-In general, we expect the same polish tools and fixturing traditionally used in the chemicalmechanical polishing of blanket Si wafers, to be applicable to the metal polishing process described in this work. In Fig. 3, is shown a schematic of the key components of the polish technique. The wafer (1) is placed in a holder (2) with the wafer surface in direct contact with a pad-covered table (4). During the polish experiment, the abrasive slurry (3) flows onto the surface of the pad and the rotation speed of the table and of the holder can be independently varied. Pressure at the wafer-slurry-pad interface is controlled via an overarm mechanism which allows pressures in the 1 to 5 kg/cm² range to be applied to the wafer holder. Most of the data presented here was obtained on Strasbaugh Model 6CA and 6DQ polishers but polish tools from other manufacturers are expected to be roughly equivalent in overall performance. Several different types of polish pad materials and wafer template assemblies obtained from Rodel were found to provide acceptable and reproducible results. Fixturing provided by other suppliers is expected to give similar results.



Fig. 3. Schematic of chemical mechanical-polishing technique, top and side views. For clarity, overarm mechanism connected to wafer holder has been omitted. Wafer (1) is held in holder (2) using commercially available template; slurry (3) flows between wafer surface and pad (5) covered table (4). See text for further details.

tered Ti/TiN adhesion layer. W was deposited using an LPCVD process (11, 12) in a batch CVD reactor (Genus 8402). Patterned wafers (6) for demonstration of the recessed W chemical-mechanical polishing process were prepared either by the Yorktown or East Fishkill Si Facility. These wafers had W deposited into RIE patterned features of a planarized SiO₂, with the planarization achieved using a previously described (10) chemical-mechanical polish process to smooth topographic variations in the SiO₂ insulator. Metallization consisted of RIE patterned Ti/Al(2.5%Cu)/Si deposited by dc magnetron sputtering. Argon sputter-cleaning was used prior to the metal deposition to insure adequate adhesion to the W and to the insulator surfaces.

W film thickness' determination.—Average metal thickness and standard deviation was determined using either a manual four-point sheet resistivity probe apparatus or an automated four-point Prometrix Model 20 measurement tool. Thicknesses of the films were calculated from the experimentally measured sheet resistivity using the previously determined value for the layer resistivity (12) of the LPCVD W.

Determination of wet etch rates.—Removal rates of W were determined by comparing initial thicknesses (as determined above) of the W films *vs.* the thickness remaining after 1-24 h of contact with the liquid of interest. All results were determined for static solutions at 20°C.

Results and Discussion

In the absence of complexation reagents or substances which form insoluble salts, W becomes passivated at acidic pH values less than 4 (15) due to the formation of WO₃. However, in the presence of an oxidant such as $K_3Fe(CN)_6$ and weak organic base complexing agents, the range of pH in which W gets passivated is extended to 6.5 (16). Solutions were formulated containing the weak oxidant $K_3Fe(CN)_6$, and it was observed that by adjusting the pH, both low static wet etch rates and chemical-mechanical polish removal of W films could be simultaneously achieved, see Table I.

We observed that when the chemical formulations were combined with silica or alumina abrasive particles, W films could be chemical-mechanically polished at removal rates as high as 400 nm/min. For the formulations listed in Table I with pH values less than 6.5, the W removal rates

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Table I.

Formulation (Note 1)	pH	Wet etch rate (nm/sec)	CMP observed (Note 2)
 К-F	5.0	8	+
K-F-En	6.5	$\tilde{6}$	+
F-En	10.5	43	

 $F=K_3Fe(CN)_6;\,K=KH_2PO_4;\,En=Ethylenediamine.$ 1. Formulations typically contain 0.1-5 weight percent of chem-

i. Formulations typically contain 0.1-b weight percent of chemicals.

2. Chemical-mechanical polishing (CMP) and pH determined on formulations containing abrasive. (See text for details.)

loading of abrasive. In the absence of applied pressure or free abrasive, the W removal rates were found to be minimal. This implies that areas of recessed metal would only be slowly removed. However, for the chemical formulation with pH 10.5, significant removal rates were found in the absence of added slurry or applied pressure suggesting, in this case, a removal process which is hydrodynamically assisted wet etching rather than chemical-mechanical polishing.

The optimum particle size and particle size distribution for the abrasive additive has not been determined. However, we have observed that commercially available colloidal silica or dispersed alumina in the 0.2-2.0 µm size range (see Experimental section) yield W surfaces after polishing that are highly specular and which appear scratch-free to the human eye. In Fig. 4 is shown particle size data for an alumina dispersion. We find that for any of the process chemistries in Table I, the measured particle size and distribution stays relatively constant. Although the alumina is specified as a deagglomerated solid with 0.05 µm average particle size, the light scattering measurements suggest that in the flowing, liquid state, the average particle size is considerably larger. Whether alumina aggregates form in solution and subsequently break apart during the polishing operation is not known at present. We have observed that addition of a coagulation agent to the slurry results in immediate settling of the normally dispersed fine particulates. Particle size analysis performed on these coagulated samples show significantly larger measured particle size, in addition to a distinct increase in surface scratching of the metal films polished under these conditions. This suggests a potentially important relationship between average particle size at the wafer surface and the incidence of polish-induced damage at that surface.

Further distinction between removal of a blanket W film under chemical-mechanical polish conditions vs. wet etching (when process chemistry pH is above the passivation limit) can be made by observing the thickness decrease over the surface of the wafer as a function of polish process time. In Fig. 5 are shown two traces where W thickness is plotted over multiple points on the wafer surface before (a) and after (b) 3 min of polish processing in the chemicalmechanical polish mode. Two effects are observed which reflect the novel aspects of the removal process. It is seen that during the polishing, in addition to a thinning of the metal film as a function of time, the thickness uniformity of the metal can be modified. In the specific example shown in the figure, after 3 min of polishing at 130 nm/min the original film, which was lower in the center, now has a



RM



Fig. 5. Sheet resistivity contour maps of blanket W films, where lines connect measured points of constant resistivity. Bold solid lines give mean resistivity with dotted lines showing areas which have higher (+) or lower (-) sheet resistivity than the mean. (a) Initial film with mean, calculated, thickness of 500 nm; each contour interval (dotted line) has successively 4% higher or lower sheet resistivity than the mean. (b) after 3 min polishing, mean thickness 112 nm with 8% deviation per contour line.

wedge shape with the maximum to minimum thickness variation observed from one wafer edge to another. In general, we have found that many different kinds of final global planarity can be achieved, dependent on the initial thickness uniformity of the metal film and the settings of the polish tool. Experiments done using identical polish tool settings but with the F-En chemistry at pH 10, show an isotropic removal of W, with no change in the global planarity of the film, typical of a wet etching process.

In addition to the sensitivity of the removal process to choice of mechanical parameters during polishing, as mentioned above, we find that the polish rates also depend on the concentration of chemicals listed in Table I and on the temperature of the slurry during processing. This suggests, consistent with the proposed mechanism, that the polish process is driven both by mechanical and by chemical effects. We typically observe that polish rates are proportional to the concentration of chemical constituents listed in Table I. In addition, we find that at any given concentration of chemicals, polish rates increase as a function J. Electrochem. Soc., Vol. 138, No. 11, November 1991 © The Electrochemical Society, Inc.



60 µm

Fig. 6. SEM views of a patterned W in SiO₂ dielectric surface following chemical-mechanical polishing.

tacting the wafer surface can be controlled. Given the hydrodynamic and chemical complexity of the polishing process, we suggest that modeling of the concentration and thermal effects observed will have to take into account a number of competing processes. These include diffusion to and depletion of the chemical constituents at the wafer This process can readily be applied to form W vias in an SiO_2 insulator layer (6, 7). A typical process sequence involves deposition of the insulator layer followed by lithographic patterning of the insulator, sequential deposition of both an adhesion layer and blanket W, followed by polishing to remove the surface W. Figure 6 is a scanning electron micrograph top down view of W patterns that result after processing. We observe that the patterned metal is well defined with no evidence of metal smearing into the adjacent insulator. In addition, the surface of the W is highly specular without any obvious chemical attack or corrosion.

Cross-sectional scanning electron micrograph (see Fig. 7) shows clear evidence for the planarity of the process and indicates that the W seams, unavoidable with a conformal CVD deposition process, have not been degraded by process induced wet etching. In general, we find that the degree of local planarity (metal *vs.* insulator height) and the final thickness and uniformity of the insulator film is dependent on the removal rate ratio of metal-to-SiO₂ insulator.

However, we have observed that subtle changes made to the process chemistry can lead to a degradation in quality of the recessed metal surface. This is an effect only observed on patterned wafers since blanket, polished films are always smooth. On blanket W wafers the K-F chemical system, with or without the addition of ethylenediamine, shows very similar blanket polish rates, and low static wet etch rates suggesting efficient surface passivation. Patterned wafers polished with the K-F-En chemistry show high-quality cross-sectional SEM features similar to those in Fig. 7. However, patterned wafers polished under exactly the same conditions and using the K-F chemistry in the absence of ethylenediamine show distinct signs of surface attack and loss of planarity relative to the adjacent insulator surface. This observation suggests that in the presence of ethylenediamine, and under the dynamic conditions of polishing, surface passivation of W is more efficient than in the absence of the weak amine base. The mechanism proposed for W passivation in the presence of ferricyanide involves (16), see Eq. [1]

$$W + 6Fe(CN_6^{-3} + 4H_2O \rightarrow WO_4^{-2} + 6Fe(CN_6^{-4} + 8H^+ [1])$$

the generation of protons at the surface via a W/W^{+6} redox reaction. The local concentration of protons at the metal interface can be expected to be influenced by the presence of buffering agents and weak base. We, therefore, suggest that static wet etch rates and bulk solution pH values alone do not entirely reflect the dynamic chemical environment which occurs at the metal-solution interface during metal polishing.

Fully planarized, two level interconnect structures, with W studs forming the contacts to the underlying Si device areas and between the wiring levels *i.e.* (W stud1-M1-W stud2-M2), have been recently fabricated (6, 7). The W stud levels were formed in a chemical-mechanical polish process using the K-F-En process chemistry. Overall yields were high and contact resistance values were not affected by the chemical-mechanical polish processing. The data show, see Fig. 8, the contact resistance scales with feature



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