Ph.D. Research Project Proposal
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The Role of Complexing Agents in the Chemical Mechanical Planarization (CMP) of Copper Thin Films

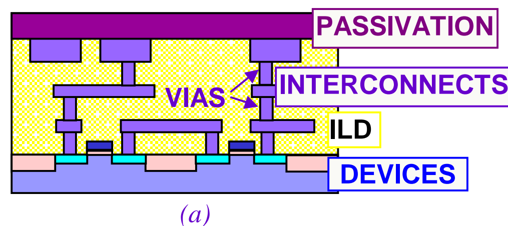
INTRODUCTION

Today copper is accepted as the interconnect metal of current and next generation chips. The first main section of this proposal is reserved to give a comprehensive summary of the technical background on the use of chemical mechanical planarization (CMP) process in manufacture of integrated circuits with copper interconnection lines. Since both the introduction of copper interconnection and CMP process is fairly recent, conciseness in this section is spared to be able to present all the essential descriptions, and terminology concerning these two technological developments. The following section describes the current research project. The goal of the research project is to develop a fundamental understanding for the behavior of copper CMP slurries containing complex agents. The theoretical and experimental techniques used to achieve this goal are explained and the results obtained so far are outlined. Finally the work planned for future is summarized.

TECHNICAL BACKGROUND

Multilevel Metallization Using Chemical Mechanical Planarization (CMP) Process

Today's state of the art integrated circuits (ICs) contain numerous device elements including tens of millions of transistors, capacitors and resistors on a single chip. Interconnects in a chip are the conductor lines, which connect these discrete elements to each other to form a circuit. As chips have become larger and more complex the requirements placed on the interconnect system have vastly increased. With more devices, more layers of interconnect are required for efficient routing. In the past thirty years, interconnects have evolved from a single layer of Al to several levels of copper interconnects, resulting in multilevel metallization (MLM). In the MLM scheme (Figure 1), first layer of interconnection is reserved for making ohmic contacts to the silicon devices or components. An insulating interlevel dielectric (ILD) layer separates the silicon or local interconnection lines from the initial or first metal interconnect layer. In the following metal layers, the linkage from one layer of interconnects to another is provided by vias, metallic conductors in holes between the surrounding ILD. The final layer of dielectric is referred to as the "passivation" layer, which provides physical and chemical protection to the underlying metal and device structures [**1**].



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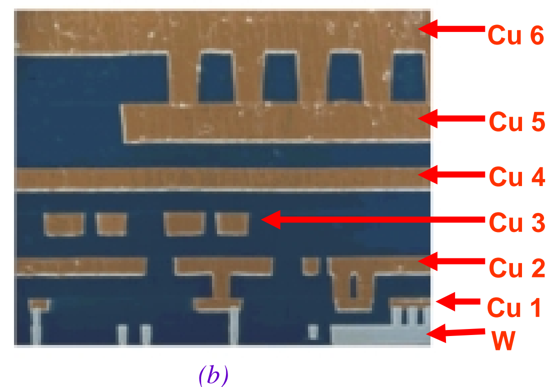


(b)

Figure 1. (a) Schematic cross section of a chip with multilevel metallization (MLM) (b) Interconnect lines in IBM's CMOS 7S chip (picture from IBM's web site)

As more and more layers are built on the silicon surface, it is found that problems with non-planarity become severe. Three that impact yield and performance are poor step coverage of deposited films, depth of focus of high performance photolithography tools and residual material after dry etching [**2**]. The lithography and etching of finely spaced, fine featured interconnect lines require that the underlying dielectric be extremely planner. For pattern resolution below 0.5 µm, planarity needs to be both local (for stringer free etching) and global (over the entire stepper field). Planarization over many micrometers is needed to eliminate metal etch residuals, and planarization over several millimeters is required to alleviate photolithographic depth of focus limitations [**3**]. Planarization is the process of smoothing and plaining surfaces. Processes that have been used for smoothing the ILD layers include etch-back of the dielectric, deposition of a planarizing polymer film, as well as chemical mechanical planarization (CMP). Very few other planarization schemes obtain the global planarity offered by CMP, by far most widely accepted method for achieving global planarity required to meet the depth of field requirements of lithography tools in the sub-0.5 µm regime [**4**].

The CMP process is a polishing process in which surface roughness on a wafer is removed by smoothing and plaining aided by both chemical and mechanical forces. A CMP tool, reduced to its essentials consists of a polishing pad affixed to a circular polishing plate, and a carrier to hold a wafer against the pad (Figure 2). Both the

carrier and the plate are rotated (usually in the same direction) as the front of the wafer is pressed down against the pad. Typical slurry is water-based and contains both abrasive and chemical additives. Sub-micron SiO_2, Al_2O_3 or CeO_2 particles are commonly used as abrasive particles in slurries. The slurry is dispensed to the center of the pad from a slurry feeder. Centrifugal force distributes the slurry across the pad forming a thin sheet of liquid on the pad. The combination of mechanical action from the pressure and velocity applied to the abrasive particles and chemical action from the chemical reagents causes material to be removed from the surface of the wafer. Planarization could only be achieved if higher removal rates are obtained at the protruding regions than recessed regions of the thin film on the wafer surface. When a rigid pad with low compressibility is used most of the mechanical abrasion concentrates on the protruding regions, resulting in higher mechanical removal rate of the high features. Chemical reagents in CMP slurries such as oxidizers, complexing agents and corrosion inhibitors have specific roles in maintaining a low chemical removal (dissolution) rate from the shallow features as well as dissolving the abraded particles in the slurry. When the mechanical and the chemical additives are tailored appropriately and their contributions to the selective removal are adjusted properly the topography of the thin film could be removed effectively due to different removal rates of low and high regions on the wafer surface. As could be realized from this brief discussion chemical mechanical planarization is an extremely complex process. Although a fundamental understanding of the process is still missing, there exist several studies and patents demonstrating that satisfactory levels of planarization of various films could be achieved using well-tailored slurries that are usually prepared empirically at present [**4**].

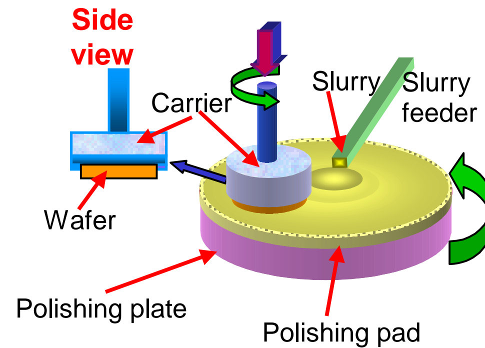


Figure 2. Schematic of Chemical Mechanical Planarization (CMP) Process

Copper Metallization

Today, the demand for faster and more reliable chips with larger scale of integration is bigger than ever. The major reasons for introducing the copper as the new interconnect metal into the next generation ICs stem from the well-established trends in the IC manufacturing. In essence, the goal is to produce ICs with larger scale of integration, higher device density, lower power consumption and faster clock times. There is also an enormous impetus to reduce the process complexity and cost. However, all of these should be achieved without compromising from the reliability.

Achieving these ambitious goals necessitates substantial changes in the interconnection technology. Scaling of the ICs over the years reduced the delay times to a level where major contribution to the total delay today is not due to gate delay but is because of the delays in the interconnection system. The delay in interconnects is characterized by the so-called RC (*Resistance* x *Capacitance*) time constant. The lower the RC, the faster is the propagation rates of electrical signals in the interconnection system. As can be seen from the Figure 3, the role of RC delay would be more significant in the total delay time in next technology nodes [**5**]. Because the interconnect delays become a higher percentage of the total delay time at each new technology node, new materials for both ILD and interconnection should be developed to improve delay times. The interconnect material needs to be a better conductor than Al alloys (alloys of Al, Cu and Si) while we need an ILD with lower dielectric constant than SiO_2 (commonly referred as low-k materials). Since main reliability problem with current Al interconnection is the electromigration at higher current densities, new interconnect

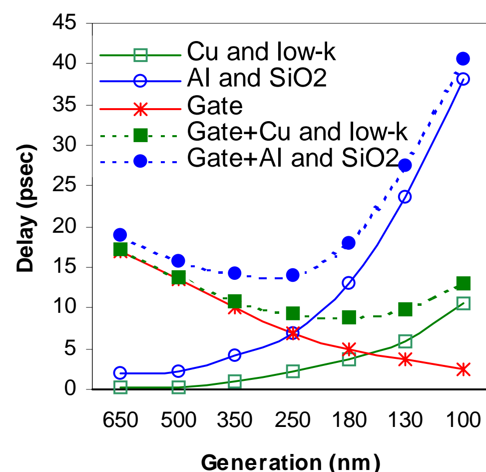


Figure 3. Gate delay and interconnect delay expected using copper and aluminum metallization at current and future generations of ICs (From reference 5)

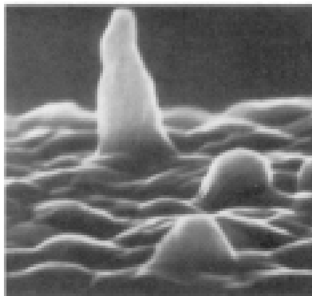
material should also have superior electromigration resistance. At present, semiconductor industry regards copper as the interconnect material of the new millennium while search for the low-k ILD is still continuing.

Copper has several properties that make it very attractive for interconnection. First, copper has a lower resistance compared to aluminum (Table 1) [**4**]. This lower resistance is critically important in high performance ICs, because it enables signals to move faster by decreasing RC constant. Copper enables a decrease in not only the resistance part of the RC delay, but also it can reduce the capacitance part. This reduction stems from the fact that the metal lines can be made thinner using copper. Thinner lines have less total surface area, and thus they lower the capacitance contribution from the metal. Using thinner copper interconnect lines in a low-k matrix one can build ICs which would consume substantially lower amounts of power due to the reduced capacitance [**6**].

Table 1. Comparison of. Properties of Al alloy and Cu (from reference 4)

Property	Al-Cu-Si Alloy	Cu
Resistivity (μΩ-cm)	~3.5	1.67
Electromigration Resistance	POOR	GOOD
CVD, Electroplating	NONE	AVAILABLE
Si Deep Levels	NO	YES
RIE (Dry Etch)	AVAILABLE	NONE

Copper lines can be made thinner as copper has superior resistance to electromigration. Electromigration (EM) is defined atomic diffusion in an electric field created by high current densities. EM damage results from occurrence of electromigration flux divergences at grain boundary triple points [**7**]. It can significantly reduce the reliability of the IC as it can be manifested as hillocks on film surfaces, bridges between two conductor lines or discontinuity in a conductor line as shown in Figure 4 [**8**]. In silicon technology involving Al interconnects, EM resistance can be increased by using so-called bamboo structure (reduces the possibility of grain boundary triple points) and alloying (addition of 1 to 4% Cu to Al) [**9**]. However, even with these enhancements, Al-alloys are not able to answer the reliability concerns as the current densities in interconnects increase with scaling of ICs (more than 10^6 A/cm^2). Metals with higher melting points are less prone to EM as the activation energies for grain boundary diffusion scale well with the melting point of a metal. Copper has a higher melting point (1083 ^{o}C) than aluminum (660 ^{o}C), which leads to greater EM resistance. The reports of EM lifetime testing of Cu interconnect show between one and two orders-of-magnitude greater lifetime for Cu as compared to Al-alloys [**10**]. In essence, high electromigration resistance of copper means that it can reliably handle higher current densities with thinner lines reducing power consumption.



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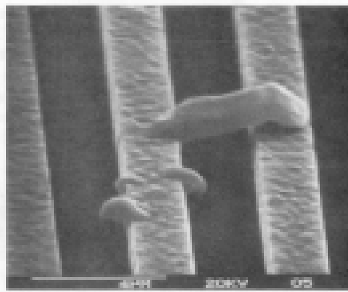

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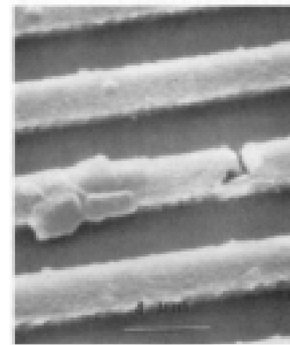


(c)

Figure 4. Manifestation of electromigration damage in aluminum films (a) Hillock formation, (b) whisker bridging between two conductor lines, (c) mass accumulation and depletion (From reference 8).

In reality, the most important benefit with copper interconnection beyond the ability to increase chip speed and reliability and reduce the power consumption is that it can actually lead to lower manufacturing costs compared to aluminum. There are two reasons for the reduction in the costs. The first one is directly related to achieving the tighter packing densities with copper, which is a natural result of using smaller lines. Since higher packing densities can be achieved per level, fewer levels of metallization is needed, resulting in significantly lowered manufacturing costs (see Figure 5). The decline in the number of metallization also results in decrease in complexity. The second reason in the cost reduction is due to the advantages brought by the damascene architecture, which is the new

process to form the copper interconnect lines. Damascene process requires 20-30% fewer steps than traditional subtractive patterning [6]. Especially use of dual damascene (in which both the via and the interconnect are formed at the same time) eliminates the involvement some of most difficult steps such as aluminum etch, and many tungsten and dielectric CMP steps. Figure 6 compares the subtractive aluminum interconnect fabrication process with the copper damascene process [11].

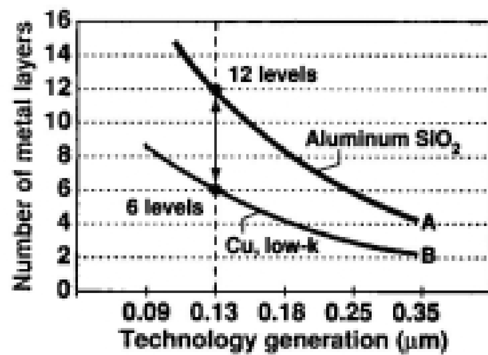


Figure 5. One of the main benefits of copper beyond the ability to increase chip speed and reduce power consumption is that the number of metal levels can potentially reduces as much as half (from reference 6).

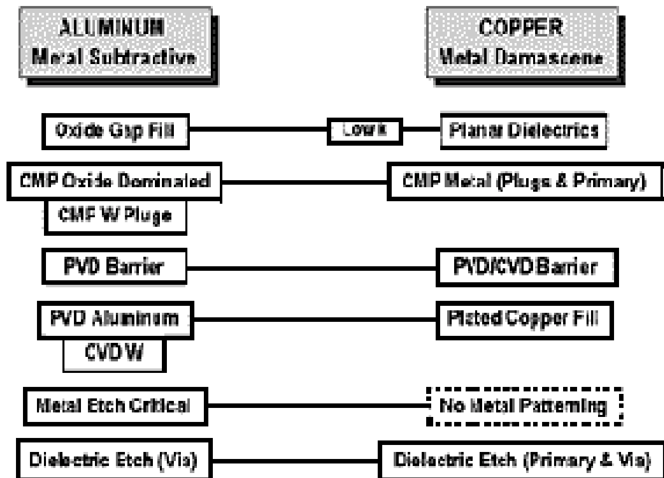


Figure 6. Summary of the major process technology changes required in the transition from the subtractive aluminum interconnect fabrication process to the copper damascene fabrication process (from reference 13).

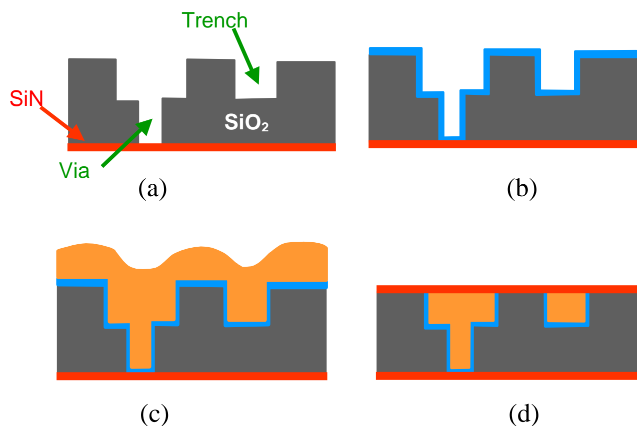


Figure 7. Fabrication steps in dual damascene process (a) Deposition of SiN, SiO_2 and etching trenches and vias in SiO_2 (b) Deposition of barrier layer (c) Copper fill (d) CMP and Deposition of SiN

The traditional subtractive patterning method, which have been used for aluminum is not appropriate for copper interconnection. Patterning Cu using reactive ion etching (RIE) has proved to be extremely difficult due to the fact that volatile copper compounds do not form at the temperatures normally used for RIE (less than 100 ^{o}C) [12]. Copper etch processes necessitates temperatures in excess of 250 ^{o}C, which leads to significant manufacturing problems such as the stability of etch mask [13]. The process flow for copper patterning is well established today. Tungsten plugs are used at first level of metal to contact the source, the drain and gate regions, due to concerns over copper poisoning. After these, the rest of the interconnection including the vias is made of copper using single or dual-damascene structures (Figure 1 (b)). Figure 7 shows the fabrication steps for a dual-damascene process. Here, the first step is the deposition of a thin layer of SiN, which acts as a barrier against diffusion of copper between metal levels. This layer also serves as an etch stop in the ILD etch process that defines the damascene via. Deposition of ILD (in this case SiO_2) follows the SiN deposition. PECVD is the commonly used method in the deposition of both SiN and SiO_2 layers. After lithograph and via/trench etch steps, a conductive barrier layer is deposited. This barrier layer has two purposes. First one is to prevent copper from diffusing into the ILD SiO_2. The second is to enhance adhesion of copper. Now the wafer is ready for copper deposition. Nowadays copper fill is done using electroplating. Copper deposition is fallowed by CMP. Copper CMP is used to remove excess copper and planarize the surface. Finally SiN cap layer is deposited. At this point the wafer is ready to next metallization step.

Planarization Mechanism in Metal CMP Process

The most commonly accepted planarization mechanism in metal CMP is Kaufman's tungsten CMP model [**14**]. In this model, the slurry chemistry is such that it induces formation of a protective passivating film on the metal surface (Figure 8). Passivation could be caused by either formation of native metal oxides on the surface using oxidizers such as H_2O_2, KIO_3 etc. or by forming chemically adsorbed layers using organic inhibitors. For example, benzotriazole (BTA), a common corrosion inhibitor for copper, could be utilized in copper CMP slurries [**15**]. In an ideal CMP process, the protective film is removed by mechanical action of the abrasive slurry only at the protruding regions. Mechanical abrasion in CMP occurs in two modes; Hertzian indentation and fluid-based wear. During Hertzian indentation, the abrasive particles are dragged across the surface and act as cutting tools. During fluid-based wear, abrasive particles impinge on the surface at some velocity and angle. The mechanism is virtually same with the erosion-corrosion process. The energy imparted to the surface from the collisions with the abrasive particles results in strain, weakened bonds and eventually material removal [**4**]. Fluid layer thickness between the wafer and the pad determines whether mechanical removal occurs as Hertzian indentation or fluid-based wear. If fluid layer thickness is low, Hertzian indentation dominates while fluid-wear becomes the main material removal mechanism when the fluid layer thickness is large. Since the thickness of fluid layer varies vastly across the wafer both mechanisms play a role in different regions of wafer [**16**].

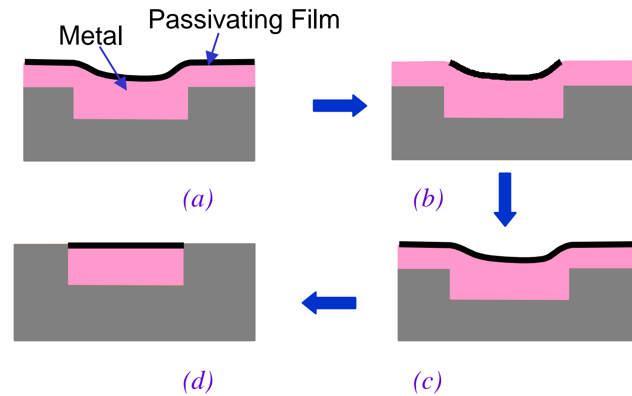


Figure 8. Kaufman's tungsten CMP model: Passivation of tungsten (a), mechanical removal of passivating film at the protruding parts causes wet etch of the unprotected metal (b) until passivating film reforms (c). Planarization is achieved with continuous cycles of the outlined mechanism (d).

The wet etch of unprotected metal occurs at the protruding regions until metal repassivates. Oxidizers and complexing agents in the slurry are responsible for the wet etching (dissolution) of the metal. Oxidizers are chemical reagents that change the oxidation state of the metal. They have a dual function in the slurry. They can form oxides which protects the recessed regions from the wet etch as well as induce dissolution of metal at the unprotected protruding regions. Continuous cycles of formation, removal and reformation of the passive layer continue until the desired final thickness of planar metal surface is attained.

It is plausible to think that planarization in copper CMP should be governed by the mechanism given by Kaufman for tungsten CMP. In copper chemical mechanical planarization (Cu CMP), in addition to wet etching of the unprotected metal surface, the chemical reagents in the slurry should also be able to completely dissolve all the abraded material [**4**]. This is necessary to prevent precipitation of abraded copper particles as well as to avoid redeposition of copper ions back to the wafer. Complexing agents and oxidizing agents are used to increase the solubility of copper to such level that all the abraded particles should be dissolved and stabilized. While increasing the solubility helps dissolving all the abraded particles, it does not necessarily cause higher dissolution of copper film on the wafer, i.e. isotropic etching, due to the size dependence of dissolution process.

It is possible to show size dependence of dissolution process with simple calculations I have outlined below. Considering only the thermodynamics, Kelvin's equation describes the relationship between the solubility constant and the particle size:

$$\ln \frac{K_r}{K_\infty} = \frac{2\gamma V_M}{rRT} \tag{1}$$

where K_r is the solubility constant of a spherical particle with radii of curvature (or radius) of r, K_∞ is the solubility constant of a flat interface with infinite radii of curvature, γ is the interfacial tension between dissolving solid and the solution, V_M is the molar volume of the dissolving solid, R is the gas constant and T is the absolute temperature [**17**]. Assuming that abraded copper is in the form of CuO spheres with V_M=12.59 cm^3/mol and using the reported average value of γ as 650 mJ/m^2 [**18**] I calculated the relative solubility (K_r/ K_∞) and plotted it in Figure 9. As could be seen from the figure relative solubility of a 2 nm CuO particle in a CMP slurry would be over three order of magnitudes larger than CuO film in a recessed area. (Assuming the trench width and the width between the trenches made in SiO_2 is around 1 μm, CuO film in the recessed area could have a radius of curvature of around 1 μm, which would give a relative solubility value of 1.007).

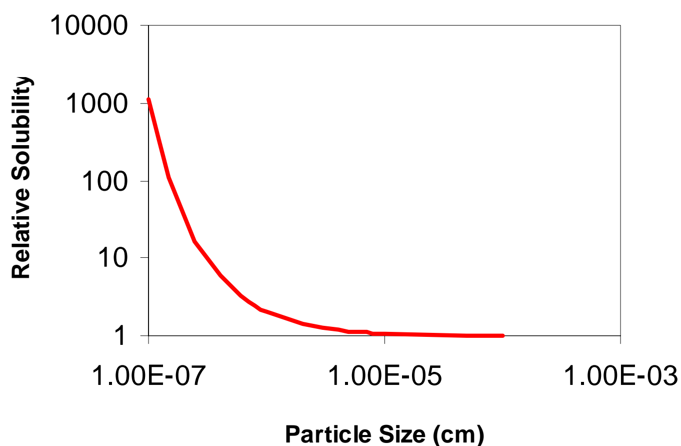


Figure 6. Relative solubility versus particle size plot for CuO using Kelvin's Equation.

Since dissolution in the slurry is controlled by heterogeneous electrochemical reactions the surface area of the dissolving material would strictly control dissolution rate. Since the surface area of material/volume would increase by dislodging it into the slurry we should expects at least eight times of increase in the dissolution rate.

THE RESEARCH PROJECT

Project Objectives

In copper chemical mechanical planarization process (Cu CMP), electrochemical and mechanical mechanisms responsible for planarization and material removal from the wafer surface are not well understood. Although Cu CMP is a key process in the manufacture of more reliable, cheaper and faster chips, the fundamental understanding of the process is still missing due mainly to the proprietary nature of the slurry development research in the microelectronics industry and even at some universities. This is generally true for the slurries that are used in CMP of other thin films as well. Slurries are almost always formulated empirically at present.

In this research project, my main goal is to delineate the role of complexing agents in the chemical mechanical planarization of copper. Although complexing agents are one of the vital chemical additives in preparation of commercial copper slurries, there have been only very limited amount of work examining their behavior and their affects to Cu CMP process [**4**,**19**, **20**, **21**]. The role of complexing agents during Cu CMP process is still unclear. I aim to develop a fundamental basis for the behavior of copper CMP slurries containing complexing agents. I believe that the results of the project will provide the in-depth understanding that is necessary to formulate superior slurries for Cu CMP process. In the present project, I examine a range of organic complexing agents with carboxylate and ammonia groups. The complexing agents under investigation are glycine, ethylene diamine (EN) and ethylenediaminetetraacetic acid (EDTA).

Complexing Agents

In pure water, metal ions are surrounded b polar water molecules, which form coordination bonds with the ion, as exemplified for cupric ion in Figure 10 (a). The solubility of metals in pure water is determined by the ability of water molecules to shield the charge on a given metal ion from the other metal ions in solution. A complexing agent such as NH_3 (dissolved ammonia gas), which has also a polar molecular structure due to the lone electron pairs on the N atom, can replace the water molecules at the coordination sites of metal ions when the metal-ammonia complex is thermodynamically more favorable (Figure 10 (b)). The NH_3 molecules are more effective at shielding the charge of metal ions, therefore complexed metal ions may move closer to each other in solution, allowing more ions in solution and thus higher solubility [**22**].

In the metal complexes, the metal ions are bonded to one or more neutral molecules or anions so as to define the integral structural unit. The molecules or ions bonded to the central metal ion are called ligands. Ligands

are Lewis bases because they always bear at least one atom having a lone pair of electrons accessible for bonding to another atom. A positive metal ion can accept electron pairs from a Lewis base, so the metal ion in the complex is a Lewis acid. As the ligand approaches a metal ion, a ligand lone pair forms a sigma bond with the metal through an interaction between the lone pair orbital and an empty orbital on the metal ion or atom [**23**]. While ammonia has only one Lewis based site, some other complexing agents are attached to the metal ion more than one donor atom in such a manner as to form a heterocyclic ring. For example, glycine ($H_2NCH_2COO^-$) and EN ($H_2NC_2H_4NH_2$) in their anionic forms have two donor sites while EDTA has six donor sites when it is fully deprotonated. Figure 10(c) shows the structure of cupric glycinate complex, $Cu(H_2NCH_2COO)_2$. Complexing agents with multiple donor sites are known as chelating agents.

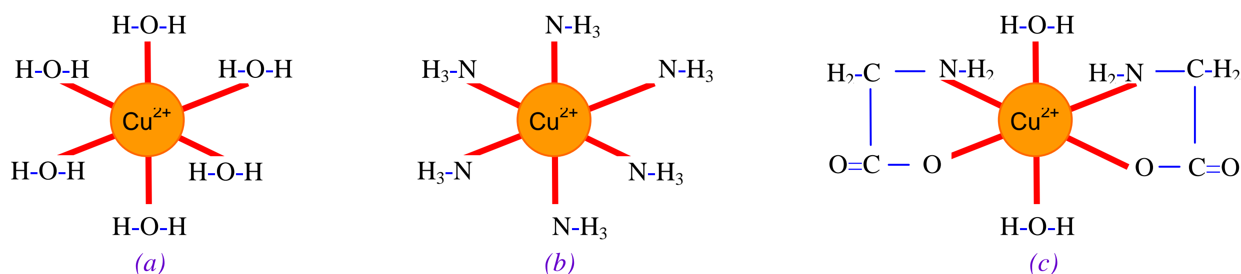
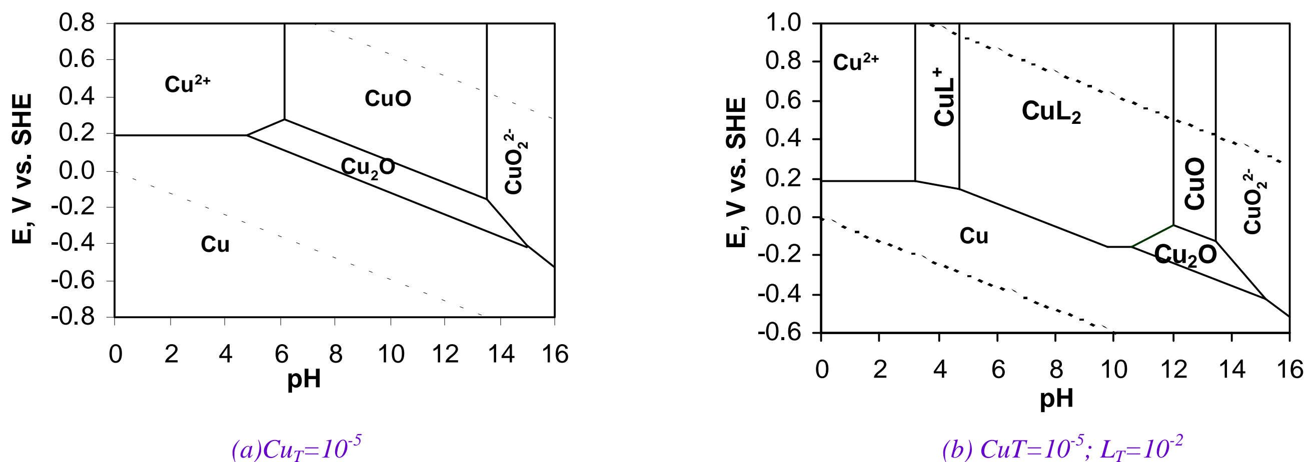


Figure 10. (a) Cupric ion coordinated by water molecules (b) complexation of cupric ion with ammonia, (c) complexation of cupric ion with two anionic glycine molecules.

Theoretical and Experimental Work

In order to understand the role of complexing agents during Cu CMP process it is necessary to examine their chemical and electrochemical behavior. Complexing agents would increase the solubility of copper in an aqueous solution due to their superiority in shielding the charge of copper ions compared to water molecules. However, it is necessary to know the domains in which complexation is thermodynamically more favorable with complexing agents than water molecules. Therefore, I have started the current project with developing theoretical thermodynamic diagrams that would allow me to characterize the interaction between the copper and the complexing agents that I am interested in including glycine, EN and EDTA. I have produced species distribution diagrams versus solution pH and potential-pH diagrams for Cu-glycine-water, Cu-EN-water and Cu-EDTA-water systems using the data available in the literature [**24**]. Figure 11. compares the Cu-water and Cu-glycine-water potential-pH diagram. Figure 11 (b) demonstrates that copper metal is oxidized to form soluble copper (II) glycinate species, either $Cu(H_3NCH_2COO)^{2+}$ [indicated as $CuHL^{2+}$], $Cu(H_2NCH_2COO)^+$ [indicated as CuL^+] or $Cu(H_2NCH_2COO)_2$ [indicated as CuL_2] over a wide range of pH's. The stability regions of cupric oxide, CuO, and cuprous oxide, Cu_2O shrinks with the presence of glycine and they appear at pH's exceeding 10.5.



(a)$Cu_T=10^{-5}$

(b) $CuT=10^{-5}$; $L_T=10^{-2}$

Figure 11. Potential-pH diagrams for (a) Copper-water system with total copper activity, $Cu_T= 10^{-5}$ (b) Copper-glycine-water system with total copper activity $C_{ut}= 10^{-5}$ and total glycine activity, $L_T=10^{-2}$

In the experimental section I have used glycine as the model complexing agent. I use the potentiodynamic polarization technique to experimentally investigate the electrochemical processes involved in the dissolution and oxide formation of copper in the aqueous solutions with and without glycine. The results of the polarization studies are correlated with the potential-pH diagrams. I conduct the polarization experiments using a copper rotating disk electrode (RDE) in a conventional electrochemical cell with a platinum counter electrode and saturated calomel electrode. In these experiments, a computer-controlled potentiostat scans the potential of the copper electrode with a pre-determined rate. The reference electrode measures the potential of the copper electrode and reports it to the potentiostat. Potentiostat also measures the current passed between the copper electrode and the counter electrode, which is necessary to keep the potential at the desired value. Both the potential and the current are recorded and plotted in form of conventional polarization curves (potential versus logarithm of current density).

In order to examine the effects of mechanical components on the behavior of copper during polishing, in-situ electrochemical experimentation is necessary. I use the apparatus schematically shown in Figure 12 to conduct in-situ potentiodynamic polarization experiments as well as other in-situ electrochemical experiments such as measuring the open circuit potential. I designed the apparatus such that I can change the rotation speed and the pressure I exert on the copper work piece. In-situ experiments are conducted in aqueous solutions with and without glycine. The polishing slurry either has abrasive particles at a pre-determined concentration or has no abrasive particles. In the former case, abrasion of copper electrode is due to both the abrasive particles and the pad, while in the later case the polish pad is responsible for the mechanical abrasion of the copper. In this way it is possible to examine the affect of the chemical mechanical polishing on the dissolution and passivation characteristics of copper. In in-situ open circuit potential (E_{OC}) measurements, E_{OC} is monitored during abrasion and compared with its value in the absence of abrasion. The time it takes to repassivate the copper surface after the abrasion is ceased has also been experimentally determined.

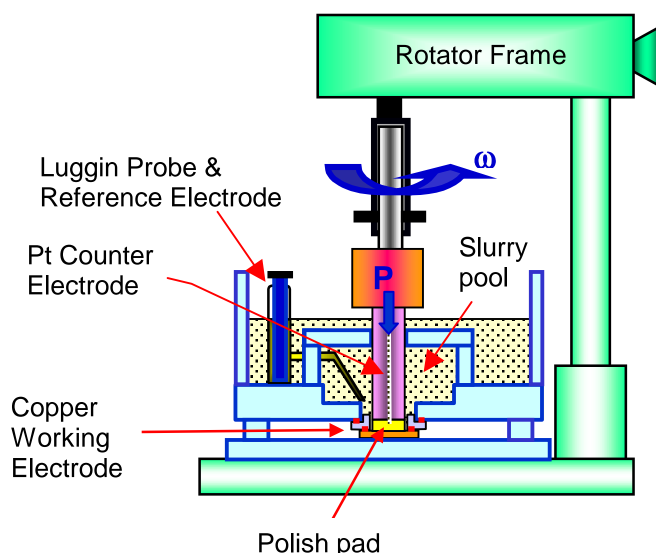


Figure 12. Apparatus used in in-situ measurements

Results so far indicate that experimentally measured polarization curves using copper RDE are in good agreement with the predictions of the potential-pH diagrams for Cu-water and Cu-glycine-water systems. No significant changes in the in-situ polarization are present when copper exhibits active behavior. However, mechanical components significantly affect the in-situ polarization when copper shows active-passive behavior. Experimental results also revealed that Kaufman's tungsten CMP model [**14**] is also valid for copper CMP. Glycine (or complexing agents in general) controls the thickness of the passivation layer of copper. Glycine (or complexing agents in general) may enhance the polishing efficiency between the protruding and recessed regions of a copper thin film on a wafer.

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