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Selective layer CMP process mimicked with atomic force microscope

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In order understand the mechanisms of Chemical Mechanical Planarization (CMP), an Atomic Force Microscope (AFM) is used to characterize polished layer surfaces formed by selective transfer after a set of polishing experiments. The Atomic Force Microscopy (AFM) allows one to examine the effects of applying highly localized stress to a surface. In the presence of solutions tribochemical friction and wear can be investigated. We present results of a study on simultaneous application of chemical agents and mechanical stress involving a model single asperity and a solid surface. We show the consequences of combining highly localized mechanical stress (due to contact with AFM tip) and exposure to aqueous solutions of known pH. The experiment simulates many features of a single particle-substrate- slurry interaction in CMP. To optimize CMP polishing process, one needs to get information on the interaction between the abrasive slurry particles and the surface being polished. To study such interactions, we used all AFM. An AFM tip was used to mimic a single abrasive particle typical of those used in CMP slurry. Surface analysis of selective layer using the AFM revealed detailed surface characteristics of CMP. Studying selective layer in which predominanted copper (in proportion of over 85%) CMP, we found that the AFM scanning removes the surface oxide layer in different rates depending on the depth of removal and the pH of the solution. We show that linear scans and rastered scans display significantly different material removal rates. Oxide removal happens considerably faster than the CMP copper from selective layer removal. This is in agreement with generally accepted models of copper CMP. Quantitative models are presented to explain the observed nanometer-scale surface modifications. Both long-range and the friction forces acting between the AFM tip and surface during the polishing process were measured. The correlation between those forces and removal rate is discussed. In the same time this paper complements recent observations of tip-induced friction and wear and growth in a number of inorganic surfaces in aqueous solution.

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1. Introduction

In spite of the fact that research in CMP has been conducted extensively, there a still needs for a better understanding of tribochemical and mechanical phenomena occuring at the interfaces between the pad and wafer in presence of the fluid slurry [1,2,3,4]. Studies have proved that use of oxidizer helps the formation of an oxide film on selective layer surface [4,5].

A fundamental understanding of surface properties of layers formed from selective transfer on nanoscale level should be generated of knowledge that recentely there are known materials which in optimal functioning conditions form in the contact zones a thin, superficial copper layer, therefore can function in conditions a selective transfer.

These materials are used at various machine couples. These materials have in common that in the friction zones special physical-chemical processes take place, which lead to the forming of a thin copper layer, almost pure, with superior properties at minimal friction and wear [6, 7]. This is a request for any friction couple of high efficiency and also a normal process for the self-adjustment phenomena. In the process of friction of these materials and in the presence of proper lubricants, wear phenomenon itself manifests as a transfer of material from one element of a friction couple to another one, this phenomenon being characteristic to the selective transfer process.

A selective transfer can be achieved for pure in a friction couple lubricated with glycerin or special lubricant if in the friction zone is a material made of copper alloys [7]. This copper is different in its structure from the copper falls out through normal electrolytic procedures.

It is well known that the friction resistance between solids is significantly reduced if lubricated, which in conditions of selective transfer for thin layer copper play the place of lubricant.

Removal of a soft oxide film enables better planarization [8]. In many cases, our understanding of mechanismus of material removal is largely empirical. Nevertheless, it is clear the material removal often involves chemical as well as mechanical stimuli. Copper CMP from selective layer studies in acidic and basic slurries were conducted in past. Use of impressed current during copper CMP has also been reported [9,10,11].

Yet, the synergy of electrochemical, mechanical and tribochemical interactions are important to be understood. CMP is used to remove the excess of metal in damascene processes for copper patterning. Indeed CMP seems to be the only effective technique to achieve both local and global planarization used in modern manufacturing.

At high contact forces, friction and wear can be induced by the tip of an AFM. Under these conditions, the AFM serves as an especially simple friction and wear system involving a single asperity translated across a well-characterized surface.

In recent paper, we have characterized the friction and wear of silocon nitride tips on selective layer surfaces in aqueous solution [12]. In this paper, we monitor the friction and wear of the selective layer substrate in basic solution. As one might expect, the friction and wear of the AFM tip plays affects the friction and wear of the underlying substrate. An understanding of nanometer-scale friction and wear must incorporate the mutual removal of material from asperity and substrate.

2. Experimental procedure

An experimental investigation was conducted for selective layer CMP in alumina containing slurries. Slurry composition was varied for pH and oxidizer-hydrogen peroxide. The way how variation parameters of slurry composition is shown in Table 1.

Table 1. Values of parameters slurry.

Parameter	Values
pH	3...9
Potential	-200... -400 mV
Oxidizer (H_2O_2)	0,09...0,21%

Slurry solution used for selective layer oxidation was prepared as aqueous solutions of 5wt% peroxide and 1wt% of glycine in different pH of 3, 4, 5, 8 and 8.5. The pH of the slurry solution was adjusted with either HCl or KOH with 10 mM ionic strength.

The AFM scanning/scratching was done with no oxidizing agents, and taking place in aqueons solutions of HCl and KOH of 0.01 mol/l mixed to maintain the same pH as in the slurry solution.

The copper from selective layer potential was varied from anodic to cathodic side. Surface characterization using an AFM was conducted post - CMP.

The surface morphology and frictional response to AFM probes were studied that several insights of tribochemical mechanisms. The AFM scanning was done over 5x5 μm area with load force of ca. 20-30 μN. This is about the estimate a force for a single abrasive particle during CMP.

3. Experimental results and discussion

Surface characterization using AFM was conducted after CMP. AFM topography scans were used to find the surface roughness (R_a) values for the scanned areas. The observed $\mathbf{R_a}$ values were considered as an index for planarization quality after CMP.

When the data was analyzed with reference to the variation in process parameters, intersting results were depicted. At low pH of slurry and anodic potential conditions, as well as at high pH of slurry and anodic potential conditions, planarization achieved was of high quality. Whereas with remaining combinations of parameters under study, poor surface planarization was observed. Fig. 1 shows the AFM topography scan for a pit on surface.

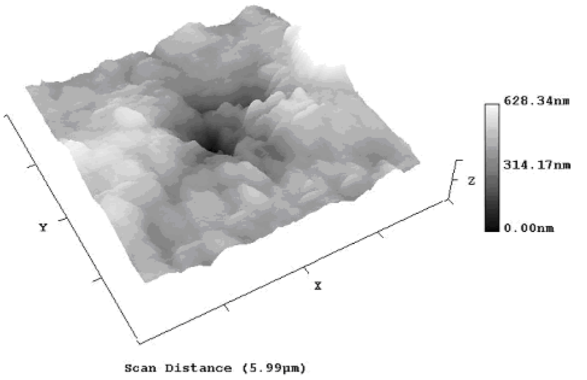

Fig. 1. AFM topography showing pit.

The reasoning for better planarization can be seen as the synergy of the controlled oxidation and subsequent removal process of the oxide at the selective layer surface. In case of excessive mechanical wear or corrosion action, surface planarization is poor. We found that the AFM scanning removes the surface oxide layer indifferent rates depending on the depth of removal and the pH of the solution.

Comparing the obtined qualitative behavior of the removal rates as a function of pH with reported CMP data [13], one can see definite corelation, Fig. 2.

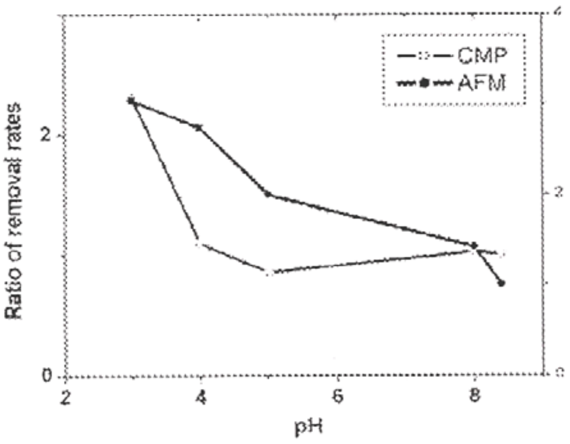

Fig. 2. Comparision of CMP removal rates and the AFM (rates for pH3 taken to be the same).

For the qualitative comparison, we put the same rates for the pH of 3. Quantative comparision reveals the following. Oxide removal with the AFM happens considerably faster than the CMP selective layer removal. This essentially confirms a generally accepted model of copper [13]: originally corrugated/patterned selective layer surface is oxidized while immersed in the slurry solution.

A fast rotating pad and the abrasive particles touch and the remove of oxide layer from the top areas of the corrugated/patterned selective layer surface, because these areas are touched by the pad first. Because the rate of removal of the oxide layer is much higher than the actual CMP removal rate, the rate of oxidation must be much slower that the rate of oxide layer removal.

If we are dealing with polishing rather than etching, the exposed pure selective layer is oxidized faster than the areas passivated by the oxide layer. If the area is still high, the oxide layer is removed again, and the process repeats until the high area disappears, i.e., is polishing away. This result is faster disolution of higher areas than the lower one, i.e., in the planarization.

A serious advantage of the AFM technique is in its stability to measure all forces acting between the slurry particles and the polishing surfaces while polishing/scratching. Because it is plausible to expect direct correlation between the removal rate and the force of friction, between the AFM tip and surface, we measure the friction force.

Friction coefficients were measured for 5-8 differend load forces. Then, the friction coefficients were calculated as the averaged ratio of the friction force to the total vertical force, which include the load force and the force of adhesion.

Fig. 3 shows the friction coefficient (normalized by the lateral spring constant of the cantilever).

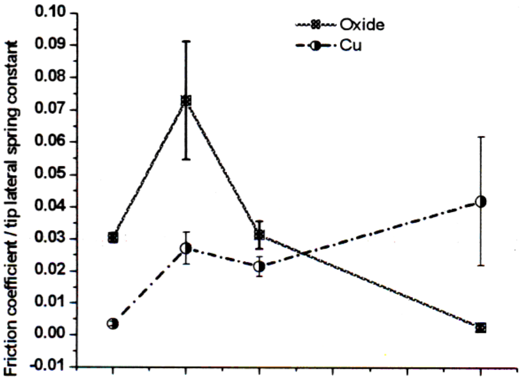

Fig. 3. Dependence of the friction coefficient on the slurry pH for selective layer and oxide layer.

As one see from Fig. 3, the friction coefficient decreases as pH grows from 4 to 8, which confirms hypothesis that the removal rate directly correlates with the friction coefficient. However, the friction coefficient increases while the pH changes from 3 to 4. This happens even though the removal rate is higher for pH 3, as seen in Fig. 2. It is interesting to note that pH 3 is a special case.

As one can see from regular AFM topology images, the oxide is much rougher for the case of pH 3 vs. the other considered pHx.

Analyzing removal noise by the AFM, we can speculate that the oxide is being removed through grinding into nanosize particles of the oxide which are noticeably smaller than in the case of the other pHx. These particles, sliding between the AFM tip and polishing surface, effectively lubricate the tip-surface contact, and consequently, decrease friction coefficient.

Fig. 4 shows a vertical deflection image of a polished, selective layer surface in slurry solution, where the inner 400 x 400 nm^2 square was smoothed by 10 raster scans at a contact force 200 nN.

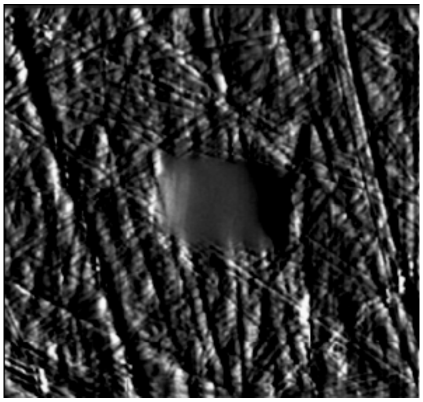

Fig. 4. A 2 x2 μm^2 deflection image of a selective layer surface in basic solution, where a 400 x 400 nm^2 region of the interior has been smoothed by 10 square scans at a nominal contact force of 200 nN.

Deflection images often reveral small surface structures that are obscured in topographic images. The root-mean-square roughness (RMS) of the diamond polished selective layer (outside the central smooth area) was typically 1.8 ± 0.5 nm.

Small area AFM scanning typically reduces the RMS roughness of the surface to 0.24 ± 0.1 nm, about twice the minimum surface roughness of this material due to its atomic structure [12].

During square raster scanning, the shape of the etch pit remains constant, while the shape of the AFM tip evolves constantly. We have shown that, under these conditions the area of tip substrate contact is roughly proportion to time [14]. As noted below, the friction and wear rate drops with time due to this increase in tip area.

Wear measurements during linear scanning are complicated by imagining issues. Since the width of the wear track is often comparable to the tip dimensions, wear track profiles can appear to be narrower than they really are.

Fig. 5 shows track and the tip profiles across the thin dimension of the wear track, perpendicular to the direction of linear scanning.

To compensate for tip size effects, the wear track profiles were manually deconvoluted using the tip profile. In many experiments, as in Figure 5(a) tip wear during linear scanning is asymmetric.

The small contact areas in these cases yield high stress (here about 150 MPa) and high local wear rates. In contrast, the more conformal contact in Figure 5(b) is associated with a high contact area and relatively low stress (here about 25 MPa).

The large difference between these stresses can strongly affect the course of wear. During linear scanning, the area of tip-substrate contact depends critically on substrate wear, in addition to tip wear.

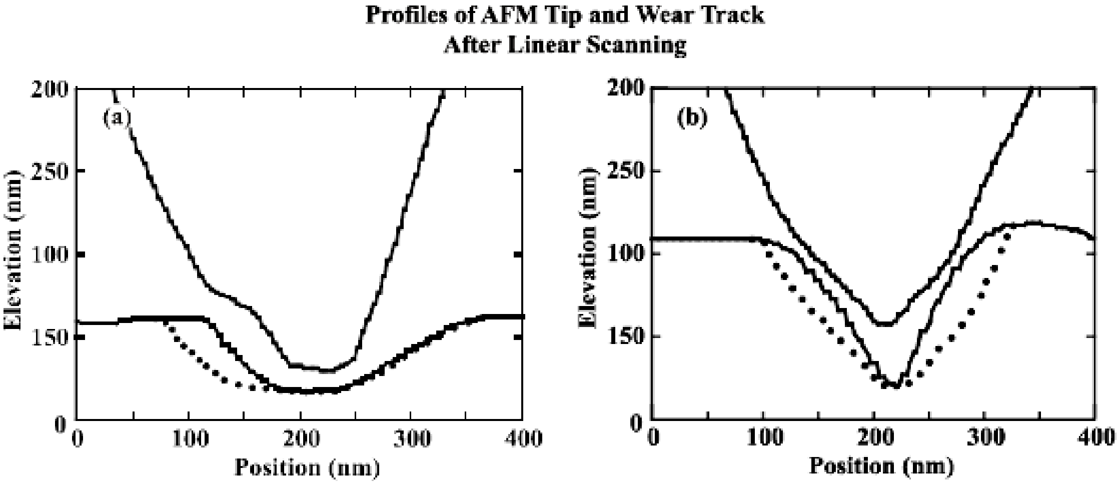
Profiles of AFM Tip and Wear Track
After Linear Scanning

Fig. 5. Profiles of tip and track after linear scanning in basic solution showing (a) strongly asymmetric tip wear and (b) conformal wear tip and track.

The dots indicate the shape of the wear track profile given by deconvolution. Each track was formed by 15 linear scans with 1024 lines/scan at a nominal contact force of 200 nN.

A sequence of wear depth measurements as a function of the number of 1000 × 1000 nm^2 square scans appears in Fig. 6 (a), and as a function of number of 500 nm linear scans in Fig. 6(b).

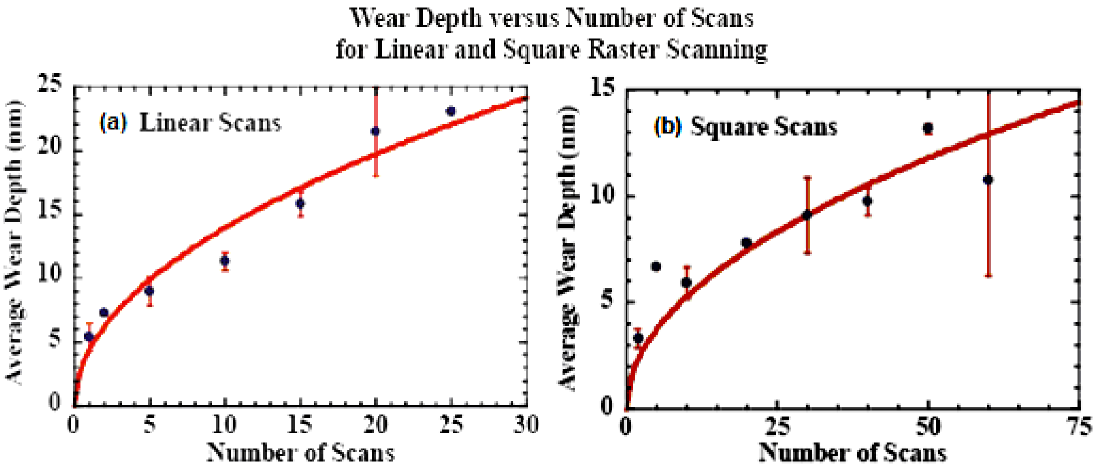
Wear Depth versus Number of Scans
for Linear and Square Raster Scanning

Fig. 6. Wear depth as a function number of scans for (a) 1000 × 1000 nm^2 square raster scans with 512 lines/scan, and 500 nm linear scans with 1024 lines/scan, both nominal contact force of 150 nN.

Each individual depth measurement was made with a new tip; the points with error bars indicate averages of two or more measurements. Both wear rates very approximately with the square root of the number of scans, but wear is much faster for linear scans.

Significantly, wear of linear scans is a linear function of force applied to the tip, while wear for square raster scans varies roughly with the square root of applied force. Models accounting for these results suggest that the local stress is controlled by tip wear during square raster scanning and by substrate wear during linear scanning.

4. Conclusions

* Removal mechanisms in copper CMP in generally and selective layer CMP in particularly can be understood by studying the AFM topography.

* Good planarization can be achieved by controlling the synergy between mechanical wear and electrochemical interaction on the surface.

* AFM technique is ability to measure all forces (inclusive the force of friction) acting between the slurry and the polshing surfaces while polishing/scratching.

* Measurements based on friction in CMP can help to identify the interactions between the pad and different materials (with different friction coefficients) and surface topography as these are exposed and evolve during planarization.

* AFM allows one to examine the effects of applying highly localized stress to a surface and in the presence of slurry solutions, wear can be investigated, wear what appears during linear scans and square scans and they depend of number of scans.

* The friction and wear of silicon nitride tip on selectiv layer surfaces AFM tips in basic solution gradually slows during prolonged linear and square scanning.

*Observations of tip and substrate wear suggest that the tip contact area and stress are principally controlled by substrate wear during linear scanning and by tip wear during square scanning.

* Characterizing the AFM tip before and after wear allows one to incorporate the evolution of the shape of the asperity into microscopic models of single asperity wear and improve our understanding of polishing and micromachining processes.

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