BTA Free Alkaline Slurries Developed for Copper and Barrier CMP

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With the technology node moving down to below 32 nm, ultra low k materials are routinely used currently. Integration of low k materials in the backend presents challenges in metal CMP processes. This paper reports the development of the alkaline slurries containing a novel additive that can be used for primary and secondary Cu CMP as well as barrier CMP. The slurries exhibited a relatively high removal rate in Cu CMP, especially at low polish pressure, because of the strong affinity between copper and functional groups in the additive. The smooth Cu surface with sub-nanometer roughness can be obtained with the Cu slurries plus the nonionic surfactant, BRI30. The Cu CMP slurries bring about satisfactory results of dishing and oxide erosion in the experiments. The k value of the low k material does not show significant difference after CMP with the barrier slurries. Since the slurries are free of BTA, it will be beneficial for post CMP cleaning of wafers. The low leakage current in the electric test could be attributed to the low level of metal contamination because of unique complexing action of the chelator.

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Chemical Mechanical Planarization (CMP) has become one of the most important fabrication technologies in the semiconductor industry worldwide since it was first started in IBM for the application of integrated circuits in early 1980s.1 Due to the demand of metal wiring transformed from aluminum to copper, copper CMP in dual Damascene process was indispensable (as was first reported in IBM in 1997). The excess material was deposited on top of the useful wiring structure (called “overburden”), and it must be removed by CMP.2

Slurry is one of the key consumables in CMP. Typical copper CMP slurries in use today can be categorized as the following: (1) Ammonium hydroxide based slurry; (2) Nitric acid (HNO3) based slurry; (3) Peroxide based slurries; (4) Carbonate and sorbate based solutions. The discussion of nitric acid and ammonium hydroxide containing slurries is only of academic interest, since these types of slurries have been used in the beginning of copper CMP era and were abandoned by industry mostly. The inhibitor used in most cases is BTA.3

In addition to the copper slurries mentioned above, there were new slurries developed with two complexing agents (glycine and ethylenediamine) and one inhibitor (3-amino-1, 2, 4-triazole). Based on the chemical synergistic effect, they showed good material removal and surface planarity performances as reported.4

For copper/barrier CMP, a certain level of selectivity is expected. IMEC researchers found that, at 2 psi, the polish rate for Ta/TaN is 90 nm per minute; for TiN is 46–52 nm per minute; for copper is 55 nm per minute; for SiO2 dielectric material is 4 nm per minute. EKC9011/9003 barrier slurry was used by them.5

Philippe Monnoyer at Freescale Semiconductor filed a patent in 2007. In this patent, the barrier slurry comprises water, an oxidizing agent such as hydrogen peroxide, an abrasive such as colloidal silica, a complexing agent such as citrate and a corrosion-inhibitor such as benzotriazole. The preferential removal of cap layer material relative to underlying ULK dielectric material can be enhanced by including in the barrier slurry composition the first additive, such as sodium bis(2-ethylhexyl)sulfo succinate. The removal rate of the barrier layer material can be tuned by the second additive, such as ammonium nitrate.6

Hitachi Chemical engineers developed for barrier metal slurry, HS-T815-X, which showed selectivity to Cu, TaN and SiOC is 1:1:1. It is an abrasive-free polishing (AFP) solution.7

In 2006, K. Gottfried, et al. in Fraunhofer Institute Microintegration and Reliability used RHEM barrier slurry for removal TaN barrier layer. The Rohm and Haas barrier slurry composition is made of colloidal silica with particle size < 30 nm, and hydrogen peroxide, with pH 2.7–3.0. The removal rate for different films are: TaN, 100–150 nm/minute; Cu, 40–50 nm/minute; SiO2, 7–8 nm/minute.8

In 2002, Lily Yao, et al. developed hydroxylamine-based slurry for barrier CMP with silica abrasives (pH = 3 to 9). It demonstrated a good selectivity for barrier film, with the removal rate for Cu:Ta/NILD = 1:3–10; ~1.9

J. Schlueter et al. in DA Nano used alkaline slurry for barrier CMP, with formulations containing colloidal silica, organic acid and pH control to between 10.5 and 11.1. They found that some nonionic surfactants and salts of organic acids had a somewhat stronger inhibitive effect to copper.10

Ken Delbridge, etc. in Planar Solutions presented their barrier slurry development work, and they formulated ER807X barrier slurry. It is made of alkaline colloidal silica with solids < 10 wt%, hydrogen peroxide as an oxidizer, and proprietary additives for controlling dishing and oxide loss and adjustable selectivity for barrier, Low-k, and Cu.11

Hebei University of Technology (HEBUT), have been developing Cu/barrier CMP slurries for about two decades. The colloidal silica based slurries have been formulated with addition of a unique alkaline additive with multiple functional groups. The additive can be abbreviated as FA/O-1 or FA/O-2. All the HEBUT CMP slurries are formulated in alkaline media with addition of FA/O.12–15 The structure of the additive is shown in Figure 1. It contains multiple functional groups such as tertiary amine, acetic acid and ethanol, and they can form complex ions with copper. BTA, benzotriazole, is a very common passivating agent in Cu CMP slurry. It is a very effective corrosion inhibitor with a formation of a monolayer adsorbed on Cu surface and a multilayer built on top of the monolayer.16 But the Cu-BTA film is too strong that there are sometimes BTA residues remained on copper surface in post CMP cleaning. These residues are normally attributed as surface defects. Further more, BTA is a carcinogenic compound. The HEBUT slurries do not contain BTA and the chemicals used in slurry formulation are environmentally benign.

It is well known that one way to reduce RC delay is to use low k dielectric film. But its mechanical strength deteriorates due to the porous structure. So it is required to develop a low pressure Cu CMP process for elimination of film delamination. Electrochemical-mechanical planarization (ECMP) is a good option. Feng Liu, et al. at Applied

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Table I. CMP Recipes for Cu Patterned Wafers, Cu Blanket Wafers and Barrier Wafers.

| CMP                  | Platen Speed, rpm | Carrier Speed, rpm | Membrane Pressure, psi | Down Pressure, psi | Inner Tube Pressure, psi | Flow Rate (ml/minute) |
|----------------------|------------------|-------------------|------------------------|-------------------|-------------------------|-----------------------|
| Cu Patterned Wafers  | 35               | 29                | 2.0                    | 2.5               | 2.0                     | 175                   |
| Cu Blanket Wafers    | 43               | 37                | 4.0                    | 4.5               | 4.0                     | 175                   |
| Barrier Wafers       | 60               | 54                | 2.0                    | 2.2               | 2.0                     | 170                   |

Materials, Inc. developed an ECMP process with a down force of 0.3 psi for a medium removal rate of 6000 A/minute. L. Economikos, et al. conducted research on Cu ECMP and they found that the copper polishing rate is voltage controlled and independent of applied down force in the range of 0.2–1.0 psi. But ECMP has not got commercial application yet.

Colloidal silica based alkaline slurries, combined with H$_2$O$_2$, have been formulated for primary and secondary copper polish with addition of FA/O-2 and FA/O-1 additive, respectively. The advantages of the copper CMP slurries are listed as: (1) No BTA is used during polish; (2) The slurries are environmentally benign; (3) For patterned wafers, the polish rate at localized peak area is increased with the help of FA/O chelator for its reaction with copper oxide; in localized valley area, the copper surface is passivated in the presence of chelator.

The barrier slurry has been formulated with addition of FA/O-1. The benefits of this barrier CMP slurry lie in: (1) No hydrogen peroxide is added in the application; (2) Metal contamination is eliminated, based on the strong affinity between FA/O chelator and metal ions; (3) No inhibitor such as BTA is used in formulation of the slurry.

Experimental

All the experiments were performed on an AMAT Mirra Polisher with optical End Point Detector. Stacked IC 1000 pad on Saba IV with a transparent window (Dow Chemical Company) was used for copper polish and it was conditioned by TBW diamond conditioner for 1 minute. Politex pad (Dow Chemical Company) was used for TaN barrier polish. Ontrak Scrubber was used for post-CMP cleaning with 2 minutes per wafer. The polish recipes in the experiments were supplied by ATDF fab in Austin, Texas, USA. In order to make sure the good status of the CMP tool and all the consumables, the CMP tool had been pre-qualified by following ATDF recommended CMP parameters (as shown in Table I), and standard consumable setups before the experiments were started.

HEBUT slurries, used in the experiments, were made up of 40 nm colloidal silica with addition of FA/O-1 and FA/O-2 chelator. The slurry properties are shown in Table II. Two other commercially available slurries were used for comparison, and they were recorded as C-Slurry for copper CMP and P-Slurry for barrier CMP, respectively. The C-Slurry was aluminum oxide based slurry with pH in the range of 7.5–7.9. The P-Slurry was silica based slurry with pH in the range of 9–10.

All the wafers, used in the experiments, are 200 mm in size, and they are listed in Table III.

The patterned wafers used in the experiments are shown in Figure 2. These wafers were used for the measurement of dishing and erosion as well as electrical test after CMP.

Black diamond (BD) film is carbon-doped silicon dioxide, which is one of the low k dielectric materials. Blanket wafers were deposited by CVD on 200 mm Si at 13.56 MHz with a deposition rate of 1200 A/min. The TaN load was 4.2 g/cm$^2$. The TaN was deposited at a deposition rate of 1000 A/min. The BD thickness was 100 A. The wafer was then sputter cleaned in Ar for 1 minute at 3000 A pressure. The wafers were plasma etched with 20 sccm H$_2$ and 1000 sccm CHF$_3$ for 20 seconds to remove the native oxide on the wafer.

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Figure 2. Film stacks of MIT 454 patterned wafers in the experiments.

Table II. Properties of HEBUT Slurries in Copper & Barrier CMP.

| Slurry Name | P1 Cu Slurry | P2 Cu Slurry | Barrier Slurry |
|-------------|--------------|--------------|----------------|
| Chelator Content | 15 ml/l FA/O-2 | 25 ml/l FA/O-1 | 12.5 ml/l FA/O-1 |
| pH          | 10.0         | 9.5          | 9.5             |
| SiO$_2$ Concentration, % | 20.0         | 20.0         | 20.0            |
| H$_2$O$_2$ Concentration, ml/l | 21.5         | 14.8         | /               |

Table III. Wafers used in the CMP experiments.

| Wafer | Wafer Category | Description |
|-------|----------------|-------------|
| I     | Blanket Cu wafer | Copper layer thickness, ~15000 A |
| II    | Blanket TaN wafer | TaN layer thickness, ~900 A |
| III   | Patterned Cu wafer | MIT 454 pattern wafers, as shown in Figure 2. The pattern for measurement is with 50% pattern density (0.1 um line/0.1 um space) |
| IV    | TaN + BD wafer | Black diamond wafers with BD thickness of 10,000 A + 150 A TaN deposited on top. |
with CVD-based black diamond film (k about 3.0) and a TaN barrier layer. The thickness of BD and TaN is shown in Table III.

Then, wafers were baked at 125 °C for 1 hour to remove any moisture in the dielectric film after the wafers were finished with post CMP scrubber cleaning. Four Dimension CV-Mapper was used for k value measurement. The k value was calculated by using the following equation:

$$k = \frac{\varepsilon}{\varepsilon_0}$$

Where $k$ is Dielectric Constant; $\varepsilon$ is 50 points average of measured capacitance; $\tau$ is thickness of BD film; $\varepsilon_0$ is Permittivity of free space; A is the area of the mercury contact.

All the metrology tools for pre and post CMP measurement are listed in Table IV.

### Results and Discussion

**Blanket Cu wafer removal rate.**— We initially investigated the copper polish rate on blanket wafers using HEBUT P1 Copper Slurry and compared it with the rate in a commercially available C-Slurry. The polish rate data are shown in Table V. In normal pressure range, the polish rate of P1 Cu Slurry is slightly higher than that of C-Slurry. Under low pressure conditions, the polish rate of HEBUT Slurry is even much faster, compared to the commercially available C-Slurry.

**Under low pressure conditions, the polish rate of HEBUT Slurry is slightly higher than that of C-Slurry.**

The polish uniformity of HEBUT P1 Copper Slurry is either similar to or even much faster, compared to the commercially available C-Slurry. The Cu surface roughness with HEBUT P1 and P2 Copper Slurry has been demonstrated as a good candidate in polishing copper for the application of low k dielectric materials which low pressure is required for prevention of film delamination.

At alkaline solutions, the copper surface would be predominantly covered with Cu(OH)₂ films, which will be negatively charged. Thus, the interaction of oxidized copper surface will readily take place with the amino groups. The FA/O molecule plays an important role in copper CMP in alkaline media, and it is abbreviated as R(NH₂)₂OH. The freshly exposed copper surfaces in CMP were adsorbed by non-ionic surfactant molecules, while other oxidized copper surfaces were still in the process of polishing.

Dishing and erosion studies.— End point detector was used in CMP experiments, and it is shown in Figure 3. Section I refers to the bulk copper polish. Section II refers to copper CMP close to the barrier layer. Section III refers to the over polish stage, based on experience; in this case, 30 seconds were defaulted as the over polish time in this experiment. On copper polish step, the polished time was identified by end point detector. On baseline barrier CMP process, the patterned wafers were polished for 60 seconds with commercial available P slurry after they had been polished with commercial available C-Slurry on Cu CMP step. On barrier CMP step with HEBUT slurry, the patterned wafers were polished for 30 seconds with HEBUT Barrier Slurry after they had been polished with HEBUT P1 and P2 Copper Slurries separately.

### Table V. Effect of polish pressure on polish rate of 8° blank copper wafer.

| Membrane Pressure, psi | 4.0 | 2.0 | 1.3 |
|------------------------|-----|-----|-----|
| Polish Rate (C-Slurry), A/min | 5321 | 2655 | 1893 |
| Polish Rate (HEBUT P1 copper Slurry), A/min | 5531 | 3871 | 3262 |
| WIWNU (HEBUT P1 Copper Slurry) | 4.5% | 6.8% | 10.2% |
| WIWNU (HEBUT P2 Copper Slurry) | 3.7% | 7.0% | 8.6% |

### Table VI. Post CMP Cu surface roughness on blanket wafers.

| Slurries | Buff Polish step | Wafer ID | RMS (Å) | Standard Deviation (%) |
|----------|-----------------|----------|---------|------------------------|
| C-Slurry | Water Rinse | #1 | 11.3 | 0.96 |
| HEBUT P1 | Water Rinse | #2 | 8.20 | 0.80 |
| Copper Slurry | Water Rinse | #3 | 7.84 | 0.75 |
| HEBUT P2 | Water Rinse | #9 | 7.28 | 1.16 |

Cupric hydroxide, Cu(OH)₂, was not considered because it is less stable than CuO. According to chemical reaction kinetics, with more reactants being formed, the reaction moves to the right hand side. The affinity between cuprous/cupric ion and amine in the FA/O molecule is so strong that the copper reaction rate will be accelerated. In CMP, copper can be removed in an accelerated rate, due to local hot temperature from friction between wafer and abrasives plus pads under a certain pressure. Therefore, the copper removal rate is relatively high with addition of FA/O chelator.

**Blanket Cu wafer surface roughness.**— HEBUT P1 Copper Slurry and P2 Copper Slurry were used for evaluation of copper surface roughness after CMP. The removed copper thickness for wafer #1 and #2 is about 5500 Å/minute; removed thickness for wafer #3 and #9 is about 1600 Å/minute. The Cu surface was first polished for 60 seconds with copper slurry on Platen 1 or Platen 2 and, then, buff polished for 30 seconds on soft Politex pad with water on Platen 3. Then, the wafers were cleaned in Ontrak Scrubber. The results are shown in Table VI. It can be found that the Cu surface roughness with HEBUT P1 and P2 Copper Slurry is slightly lower than that of C-Slurry. The Cu surface roughness with HEBUT P2 Copper Slurry is slightly better than that of HEBUT P1 Copper Slurry, 7.8 Å versus 8.2 Å. The surface roughness of blanket Cu wafer in CMP using HEBUT P2 Copper Slurry is further improved to 7.3 Å, with 5% BRIJ 30 nonionic surfactant addition to water in buffing polish step.

The freshly exposed copper surfaces in CMP were adsorbed by non-ionic surfactant molecules, while other oxidized copper surfaces were still in the process of polishing. The CMP process was accomplished until all the surfaces had been covered with a thin layer of surfactant film. So the surfactant in the CMP slurry or in buff solution can help to produce a smooth Cu surface after CMP. The nonionic surfactant, BRIJ 30, was used in the experiment, and its formula is C₁₂H₂₅(OCH₂CH₂)₄OH. The molecules are oriented in order on the fresh copper surfaces for surface protection. So BRIJ 30 plays a very critical role in producing smooth copper surface.
Table VII. Dishing and Erosion Results for Pattern Wafers in CMP Experiments.

| Slurry                  | Wafer ID | Dishing (nm) | Erosion (Å) |
|-------------------------|----------|--------------|-------------|
| C-Slurry                | 1        | 156          | 604         |
| HEBUT P2 Copper Slurry  | 10       | 49           | 603         |
| HEBUT P2 Copper Slurry  | 12       | 90           | 526         |
| HEBUT P1 Copper Slurry  | 3        | 80           | /           |

The experiment results for the patterned wafers are shown in Table VII. It can be seen that the HEBUT P1 and P2 Copper Slurries bring about less dishing on Cu wire than commercial available C-Slurry. For oxide erosion, HEBUT P1 and P2 Copper Slurries are slightly better than that of C-Slurry.

In copper CMP with HEBUT slurries, the copper surfaces were covered with passivation layers of Cu$_2$O, CuO and Cu(OH)$_2$. So the passivation layers in the trenches help to reduce dishing accordingly.

Electrical test.— Electrical test was used for further evaluation of wafer performance with HEBUT CMP Slurries. Resistivity results are shown in Figure 4. In the figure, Wafer #1 was used for Cu CMP with the commercial available C-Slurry, combined with commercial available P Slurry for barrier CMP. Wafer #10 and #12 were used for CMP with the HEBUT CMP slurries. Lower resistivity values for copper lines polished with HEBUT CMP slurry indicate that the HEBUT Copper Slurries bring about less dishing than commercial available C-Slurry.

Electrical test for leakage current was conducted on two wafers, #1 and #12, and the two wafers were polished with the commercial available slurries and HEBUT slurries, respectively. The result is as shown in Figure 5. The leakage current for the wafer, polished with the HEBUT slurries, is lower than that of the commercial available slurries. It could be related to less metal contamination for the wafers polished with the HEBUT slurries.

Barrier CMP experiments.— The TaN polish rate is about 450 Å/minute with HEBUT barrier slurry. The k value of the underlying black diamond (BD) films was measured after barrier TaN had been completely removed.

In order to remove the TaN film, wafer #2 was polished for 30 seconds with HEBUT barrier slurry, and wafer #10 was polished for 1 minute with commercial available barrier slurry, as shown in Figure 6. The k value for the wafer without CMP is 2.94–2.98. The post CMP k value for film polished with HEBUT Barrier Slurry is 2.99–3.05, while the post CMP k value for the wafer polished with commercial available P Slurry is 2.97–3.02. It shows that the performance of HEBUT Barrier Slurry is similar to that of the commercial available P Slurry for barrier CMP.

The HEBUT Barrier Slurry performs well in barrier CMP for the removal of TaN from BD film. No delamination was observed during the CMP process. And k value was not shown significant difference before and after barrier CMP. The organic molecules with CH$_3$ type are incorporated into the Si-O-Si cage structure, and the amount of CH$_3$ groups in the Si-O matrix mainly determines the insulating characteristics of the low k films. Hydrogen peroxide, used in the slurry during CMP, could possibly attack the CH$_3$ groups in the BD film more or less. Because hydrogen peroxide is not used in the HEBUT barrier slurry, it could be the reason why the low-k degradation is less with the slurry.

Conclusions

HEBUT slurries are used in alkaline media in CMP and they are free of carcinogenic BTA. So they are environmentally benign. The wafers are easily cleaned post CMP free of organic-Cu residues. HEBUT Copper Slurry gives a very high copper polish rate, especially under low pressures, and it is a good candidate in Cu CMP for low k applications. It also demonstrates a very good performance in CMP, such as exhibiting low Cu surface roughness, less dishing and oxide erosion. The FA/O compound plays a critic role in formulation of the CMP slurries since it exhibits a strong complexing effect with copper.
in alkaline media. The HEBUT Barrier Slurry is very effective in removal of TaN barrier layer, but it brings about insignificant change of k value of BD film post CMP. Free of hydrogen peroxide in the barrier slurry could be the reason for explaining stable k value in CMP.

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