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Effect of copper slurry on polishing characteristics*

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Abstract: The composition of the polishing solution is optimized by investigating the impact of the WIWNU (the so-called within-wafer-non-uniformity WIWNU) and the removal rate (RR) on the polishing characteristics of copper. The oxidizer concentration is 1 Vol%; the abrasive concentration is 0.8 Vol%; the chelating agent of the solution is 2 Vol%. The working pressure is 1 kPa. The defect on the surface is degraded and the surface is clean after polishing. The removal rate is 289 nm/min and the WIWNU is 0.065. The surface roughness measured by AFM after CMP (chemical mechanical planarization) is 0.22 nm.

Key words: copper slurry; chemical mechanical planarization; WIWNU
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1. Introduction

The progressively decreasing feature size and increasing circuit density of circuit components have led to the great-large-scale-integrated circuit (GLSI) and a 65 nm feature size becoming mainstream[1, 2]. However, with severe dimension shrinkage and transistor performance improvements in integrated circuits (ICs), RC delay, electromigration and power dissipation are becoming an important bottleneck in GLSI performance and fabrication[3]. The integration of copper and low-permittivity (k) dielectrics can provide further performance and reliability enhancements to meet the requirements of the next generations of technology[4]. However, the practical use of low dielectric constant materials and more metallization layers causes challenges including film delamination, scratches and deformation of the mechanically fragile films for the traditional CMP technology[5]. To avoid these problems, it is necessary to ensure that polishing pressure is reduced to below 6.89 kPa. At present, the CMP process need higher pressure (> 13.78 kPa). So it is a key issue how to balance the reduced pressure and improve the polished surface.

CMP is a key step in chip manufacture in GLSI. The purpose of CMP is to decrease the bumps formed on the substrate, which provides the essential condition for wirings[6]. The CMP process uses the surface chemical reaction and mechanical function of abrasive particles. However, an abrasive-free process also introduces some defects. For example, scratches on the surface are easily produced by the stacked large abrasive particles and are related to other defects. In addition, the uniformity of the disperse abrasive on the pad determines the removal rate (RR) of copper, where the uniformity is across a complete wafer (the so-called within-wafer-non-uniformity WIWNU)[7]. So it is a problem demanding prompt solution of how to make the abrasive particles disperse uniformly.

$$\mathrm{WIWNU}=\frac{M_{\mathrm{R}}}{\overline{M}}, \tag{1}$$

$$M_{\mathrm{R}}=\sqrt{\frac{\sum_{i=1}^{n}\left(M_i-\overline{M}\right)^2}{n-1}}, \tag{2}$$

$$\overline{M}=\frac{\sum_{i=1}^{n}M_i}{n}, \tag{3}$$

where M_{R} is the standard deviation of removal rate and $\overline{M}$ is the average value of removal rate.

In the experiment, five points on the wafer are selected to test the WIWNU and every point is tested three times.

This paper presents an investigation of 12 inch blanket copper CMP using a 1 kPa working pressure and a newly developed polishing solution. Optimization of the chemistry of the polishing solution and techniques is discussed first. Then the effects on the WIWNU by the polishing behavior are discussed and finally the optimized polishing solution is presented.

2. Experiment

The test structures, shown schematically in Fig. 1, were fabricated as follows. For polishing solution optimization, 12-inch blanket copper wafers were used. A IC1000-A2,050,XY/3/SP2310,24.7’II pad was applied together with

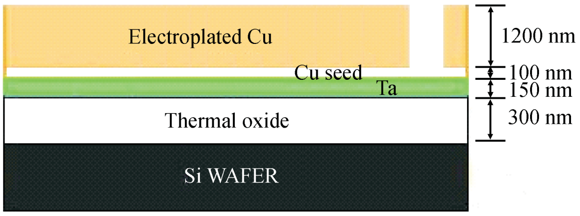

Fig. 1. Cross section of 300 mm Cu blanket wafer.

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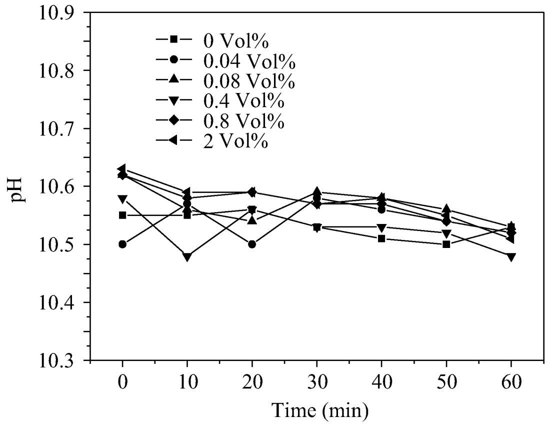


Fig. 2. Effects of concentration of abrasives on pH with different times.

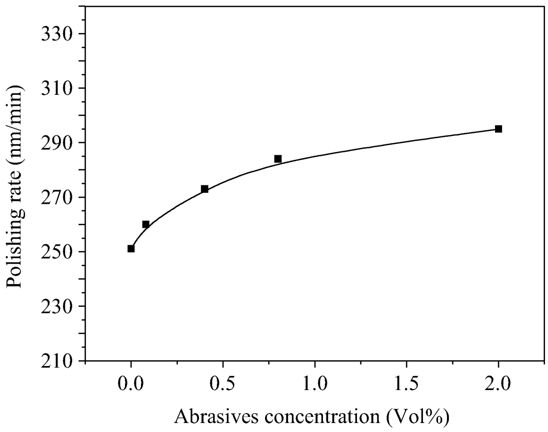


Fig. 3. Relationship between abrasives concentration and polishing rate.

the newly developed polishing solution. The new solution contained the chelating agent. The oxidizer, hydrogen peroxide, was added to the polishing solution in amounts ranging from 1 to 5 Vol%. For optimization purposes, the pH of the polishing solution was varied from 10.45 to 10.65. Polishing runs were carried out using the Alpsitec E460E polishing tool produced in France. The supply speed of the polishing solution was 225 mL/min. The copper surface profile and morphology were investigated using a step profiler (AMBIOS XP-300) and an atomic force microscope (AFM) (Agilent 5600LS). A metaloscope (OLYMPUS BX60M) was used after polishing to observe the surface.

3. Result

3.1. Effect of abrasive concentration

From Fig. 2, we can see that the effects of concentration of abrasives on pH are not large, and the value is stable from 10.45 to 10.65. It can be considered that the slurry is steady when the concentration of abrasives is low. So the low concentration of abrasives in slurry is feasible. The solution contains 2 Vol% hydrogen peroxide and 3 Vol% FA/O chelating agent.

As shown in Figs. 3 and 4, when the concentration of abrasives is super low (0–2 Vol%), the removal rate of copper rises slightly, while the WIWNU is stable. At that moment, the abrasive particles stir the slurry to make the components distribution. At last, the concentration of abrasives is fixed at 0.8 Vol%.

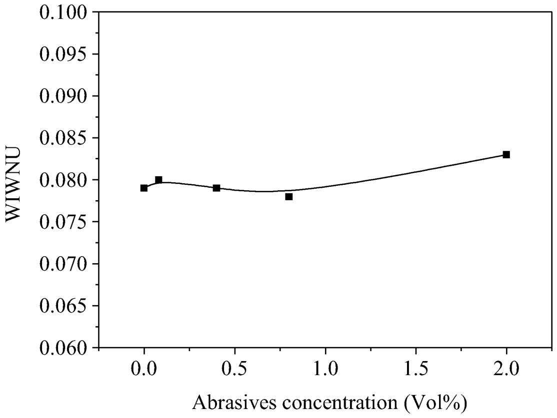


Fig. 4. Relationship between abrasives concentration and WIWNU.

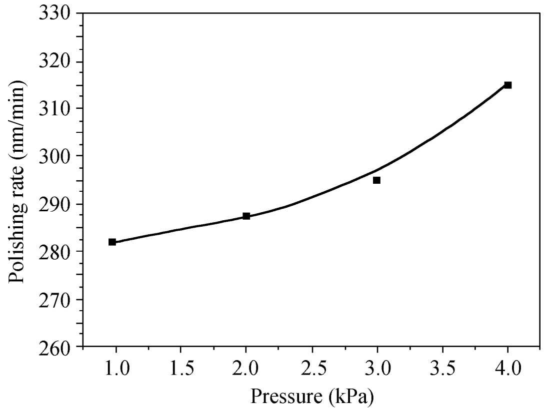


Fig. 5. Relationship between pressure and polishing rate.

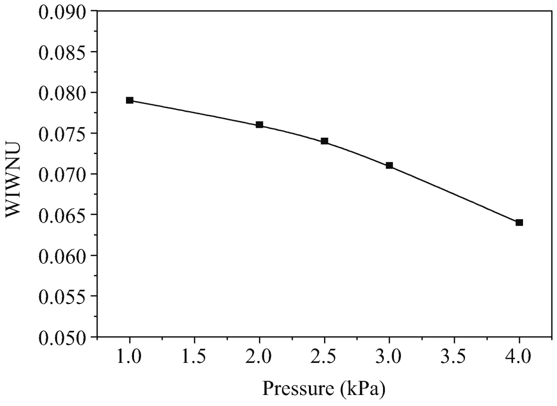


Fig. 6. Relationship between pressure and WIWNU.

3.2. Effect of working pressure

As Figures 5 and 6 show, when the working pressure is 1–4 kPa, the removal rate of copper rises, while the WIWNU changes slightly. Considering the structure of low-k material is fragile, the working pressure is fixed on 1 kPa.

Table 1. Parameters of polisher.

Parameter	Step 1	Step 2	Step 3	Step 4	Step 5
Step duration (s)	10	3	210	2	20
Transition duration to next step (s)	2	2	0	0	0
Head speed (RPM)	30	30	30	30	30
Plate speed (RPM)	33	33	33	33	33
Working pressure (mdaN/cm^2)	−200	45	45	45	45
Back side pressure (mdaN/cm^2)	0	0	43	0	0
Vacuum	1	1	0	1	1
Sweeping external position (mm)	145	145	145	145	145
Sweeping internal position (mm)	155	155	155	155	155
Slurry 1 (mL/min)	300	225	225	0	0
Slurry 2 (mL/min)	0	0	0	300	300

Note: (1) Vacuum: 1 refers vacuum, 0 refers no vacuum.
(2) Slurry 2: 1.5 Vol% surfactant in polishing slurry.

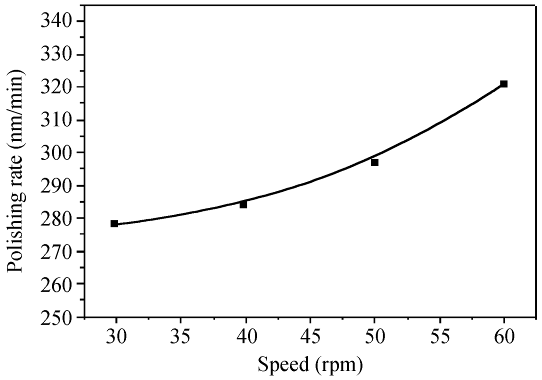


Fig. 7. Effect of speed on polishing rate.

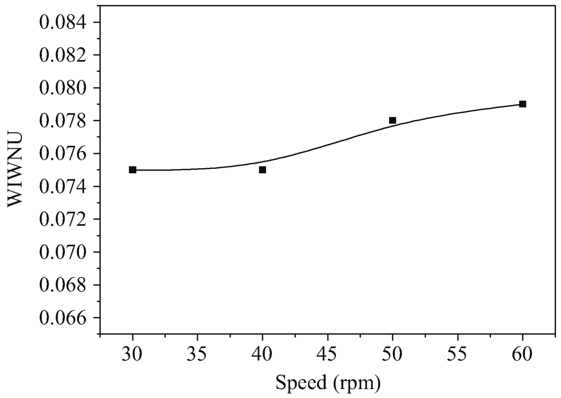


Fig. 8. Effect of speed on WIWNU.

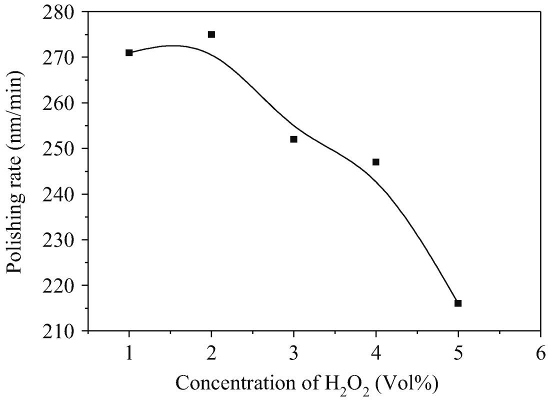


Fig. 9. Effect of oxidizer on polishing rate.

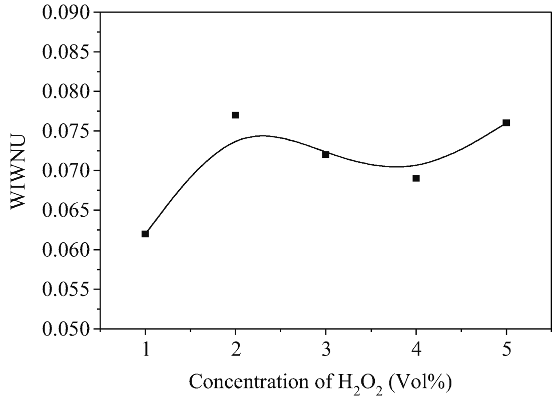


Fig. 10. Effect of oxidizer on WIWNU.

3.3. Effect of speed

The E460 polishing tool uses the active rotation of the head and plate. When the palstance of plate is larger than the palstance of the head, the WIWNU is better. When the palstance of the plate is the same as the palstance of the head, the WIWNU is the best. So the speeds of the plate and head are both 30 rpm.

The results are shown in Figs. 7 and 8. Because the size of 12-inch blanket copper is large, the distribution of slurry and speed on the surface must be the same that can obtain the low WIWNU. While the pressure is low, the speed and the distribution of slurry are the most important factors. Finally, the speed is 30 rpm.

Based on the pressure and speed, the parameters of polisher are fixed, as shown in Table 1.

3.4. Effect of oxidizer concentration

The concentration of the abrasive is very low, so the oxidizer can take part in the chemical reaction effectively. However, the oxidizer consumes the chelating agent. When the con-

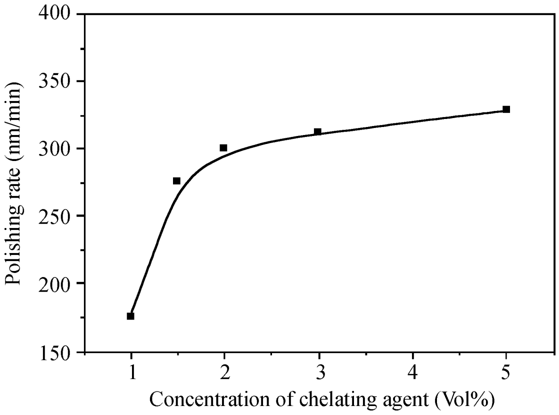


Fig. 11. Effect of chelating agent on polishing rate.

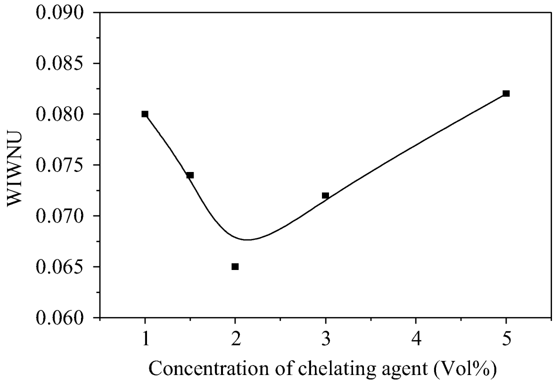


Fig. 12. Effect of chelating agent on WIWNU.

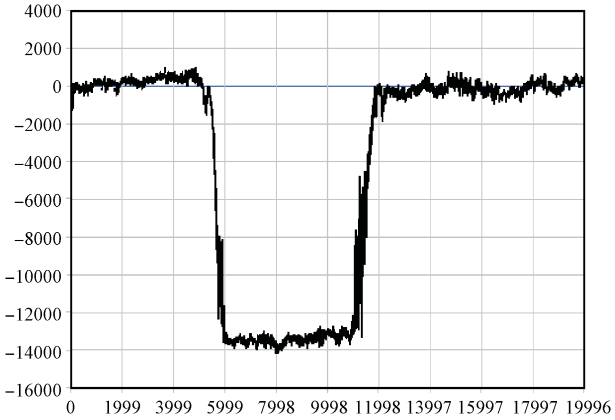


Fig. 13. Thickness of copper thin before CMP.

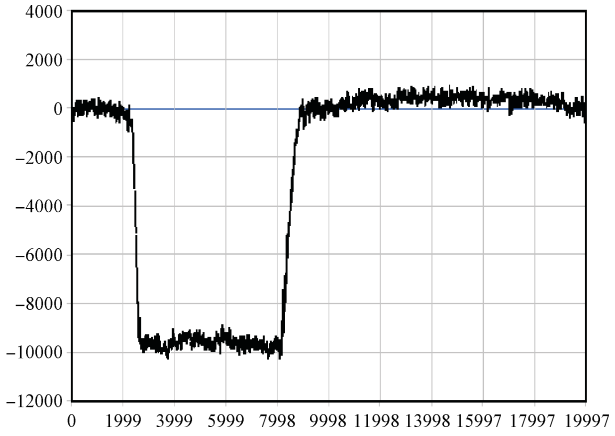


Fig. 14. Thickness of copper thin after CMP.

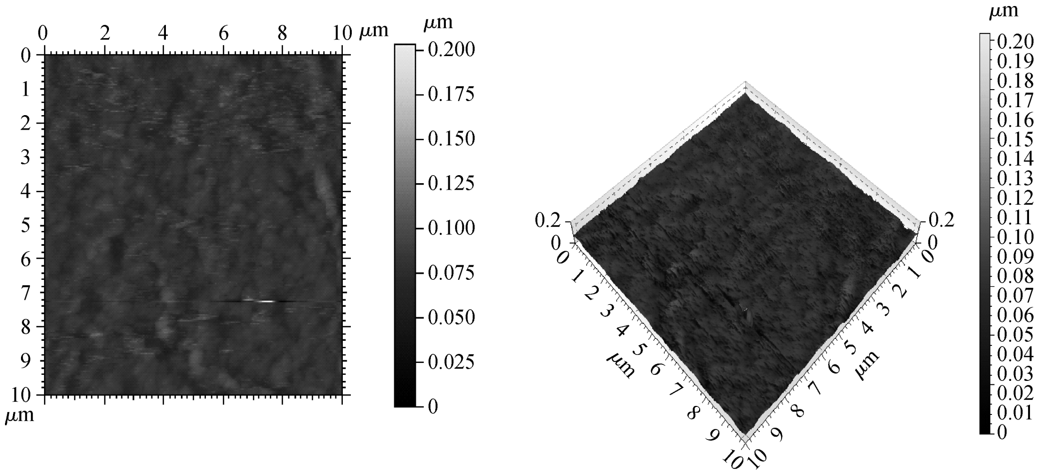


Fig. 15. Surface roughness measured by AFM before CMP.

centration of chelating agent decreases, the polishing rate is also degraded and WIWNU increases. The result is shown in Figs. 9 and 10. The concentration of oxidizer is 1 Vol%.

3.5. Effect of chelating agent

As shown in Figs. 11 and 12, the concentration of the FA/O chelating agent is high, the polishing rate and WIWNU are deteriorative. So the concentration of the FA/O chelating agent is selected as 2 Vol%.

3.6. RR and WIWNU

The rotation speed of the plate/pad and that of the wafer carrier is set at 30 rpm. The supply speed of the polishing solution is 225 mL/min. The optimal conditions obtained from the compromise of the two desirable responses, RR and WIWNU, are silica sols concentration of 0.8 Vol%, H_2O_2 concentration of 1 Vol% and FA/O chelating agent concentration of 2 Vol%, respectively. Several blanket copper test wafers are polished using the optimized polishing slurry. After polishing, the sam-

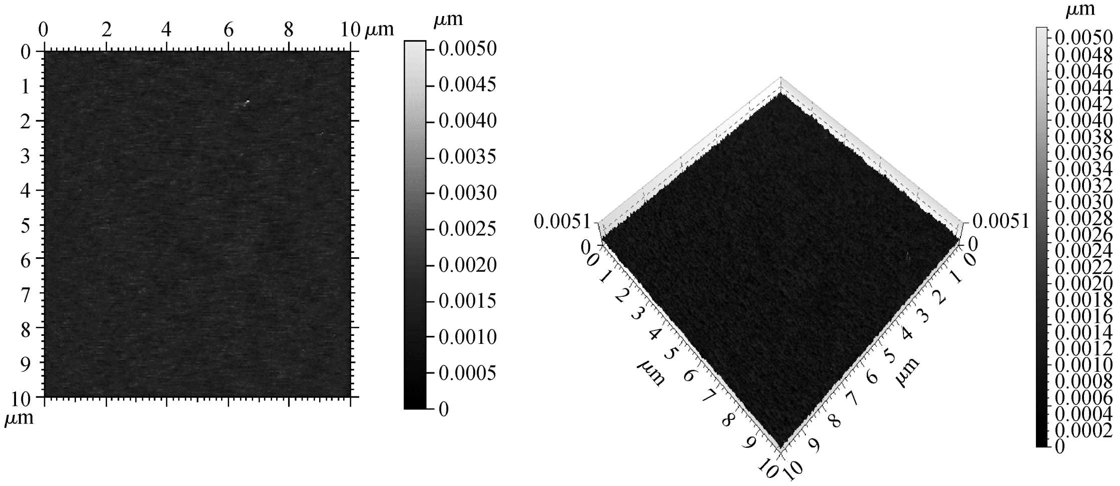
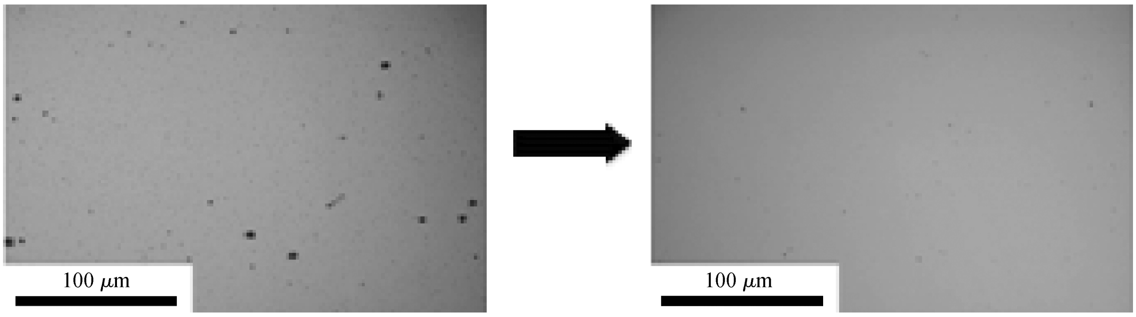


Fig. 16. Surface roughness measured by AFM after CMP.



(a) (b)

Fig. 17. Surface (a) before and (b) after CMP measured by OLYMPUS microscope.

ple is cleaned by rinsing in fresh de-ionized (DI) water. The removal rate of copper is 289 nm/min and WIWNU is 0.065. The results of thickness are shown in Figs. 13 and 14.

3.7. Surface roughness

The copper surface roughness depends strongly upon the chemistry of the polishing solution.

Figure 15 presents an AFM image of a test structure ($10 \times 10\ \mu m^2$) before polishing. The surface roughness is 7.98 nm. Figure 16 shows that when compared with the sample polished by the slurry, the AFM image shows that the surface roughness is 0.22 nm ($10 \times 10\ \mu m^2$).

3.8. Topography

More details are revealed by metaloscope in Fig. 17.

4. Conclusions

The polishing solution, which consists of hydrogen peroxide as oxidizer and chelating agent, is optimized. Experimental results show that the optimized polishing solution has a pH of 10.5 and a hydrogen peroxide concentration of 1 Vol% and chelating agent concentration of 2 Vol%. The silica sols concentration is 0.8 Vol%. Investigations of copper wafers using the optimized polishing solution show a considerably copper removal rate of 289 nm/min and a WIWNU of 0.065.

References

[1] Kulkarni M, Ng D, Baker M. Electropotential-stimulated wear of copper during chemical mechanical planarization. Wear, 2007, 263(7–12): 1470

[2] Han S J, Seo Y J. Voltage-induced material removal mechanism of copper for electrochemical–mechanical polishing applications. Transactions of the Nonferrous Metals Society of China, 2009, 19(1): 262

[3] Ein-Eli Y, Starosvetsky D. Review on copper chemical–mechanical polishing (CMP) and post-CMP cleaning in ultra large system integrated (ULSI)—an electrochemical perspective. Electrochimica Acta, 2007, 52(5): 1825

[4] Tiley J, Shiveley II K, Viswanathan G B. Novel automatic electrochemical–mechanical polishing (ECMP) of metals for scanning electron microscopy. Micron, 2010, 41(6): 615

[5] Bhagavat S, Liberato J C, Chung C. Effects of mixed abrasive grits in slurries on free abrasive machining (FAM) processes. International Journal of Machine Tools and Manufacture, 2010, 50(9): 843

[6] Zhang L, Subramanian R S. A model of abrasive-free removal of copper films using an aqueous hydrogen peroxide–glycine solution. Thin Solid Films, 2001, 397(1/2): 143

[7] Zhang W, Lu X C, Liu Y H. Inhibitors for organic phosphonic acid system abrasive free polishing of Cu. Appl Surf Sci, 2009, 255(7): 4114