Microelectronic Engineering 170 (2017) 21–28

Contents lists available at ScienceDirect

Microelectronic Engineering

journal homepage: www.elsevier.com/locate/mee

Research paper

Investigation of the barrier slurry with better defect performance and facilitating post-CMP cleaning

Xiaodong Luan [a,b,*], Yuling Liu [a,b], Baoguo Zhang [a], Shengli Wang [a], Xinhuan Niu [a], Chenwei Wang [a], Juan Wang [a]

[a] Institute of Microelectronics, Hebei University of Technology, Tianjin 300130, China
[b] Tianjin Key Laboratory of Electronic Materials and the Devices, School of Electronic Information Engineering, Hebei University of Technology, Tianjin 300130, China

ARTICLE INFO

Article history:
Received 8 April 2016
Received in revised form 9 September 2016
Accepted 3 December 2016
Available online 19 December 2016

Keywords:
Chemical mechanical planarization
Barrier slurry
Particle size
Defect free
Scratch
Galvanic corrosion

ABSTRACT

It becomes very critical for yield enhancement to accomplish defect free global planarization during barrier CMP. Conventional commercial barrier chemical mechanical polishing (CMP) slurries usually contain benzotriazole (BTA) and oxidizer (most frequently H_2O_2) which could cause organic defects and oxide particles on the copper surface for post-CMP cleaning. In this paper, we present a kind of weakly alkaline barrier slurry with lower defect occurrence than conventional ones, and a key feature of the slurry is facilitating post-CMP cleaning. The experimental results obtained from the defect map show it is hard for a simple cleaning solution containing nothing but deionized water and surfactants to completely remove oxide particles and the organic residue on the wafer surface polished with the commercial barrier slurry. On the contrary, the defect map of the wafer polished with the weakly alkaline barrier slurry indicates that there is no surface contamination after cleaning with this simple cleaning solution, but there are still many defects. AFM (atomic force microscopy) graphs demonstrate these defects are scratches, and the tool views them as defects. The polishing results indicate that the root causes for scratches are the large particles exceeding 0.5 μm size in the slurry, and micro-scratches are remarkably reduced after installation of a 0.2 μm size filter without affecting the CMP performance, including dishing, resistance, capacitance and leakage current. Compared with the conventional commercial barrier CMP slurry, the alkaline barrier slurry can also control copper line corrosion due to galvanic reaction.

1. Introduction

Chemical mechanical polishing has been used in the fabrication of Fin Field-Effect Transistor (FinFET) device to obtain global planarization of Cu dual damascene interconnect, and its process becomes more complex as integrated circuit devices shrink to 28 nm and below [1,2]. Cu CMP process is divided into three steps. The 1-step is carried out to remove the excessive amount of Cu and needs a higher removal rate of Cu to eliminate efficiently the step height [3,4]. The 2-step should obtain high Cu to barrier metal removal selectivity to control precisely the endpoint over the barrier metals. However, dishing of Cu line in the 2-step is introduced inevitably because over-polishing is usually required to remove all Cu residues on the barrier metal surface to guarantee the electrical isolation between adjacent circuits [5]. Slurry used in the 3-step is referred as barrier CMP, which is our concern in this paper. The barrier CMP should control removal selectivity as a unity among different materials of Cu, barrier metal, and dielectric to realize surface flatness [6–8]. Since the barrier CMP is the final and the most important process, not only can it generate defects during the process, but it can also bring greater challenges to the post Cu CMP cleaning process [9–11]. Therefore, the novel barrier CMP slurry not only must facilitate the post Cu CMP cleaning process, but also prevent some defects due to CMP.

Defect reduction is a major part of a yield management program because it reduces chip yield and reliability [10,12]. Among various defects from barrier CMP process, organic residues [13,14], micro-scratches [15,16] and galvanic corrosion [17,18] are the significant issues for device failure and yield reduction. BTA, a carcinogenic compound, is a commonly used dissolution inhibitor in the conventional commercial barrier CMP slurry to control the chemical removal of Cu from recessed surface regions [19–21]. Copper ions can form Cu-BTA polymer, and these BTA films are low solubility in aqueous media and act as a physical barrier to inhibit the diffusion of aggressive ions [19]. However, it needs a complex cleaning liquid and cleaning process to removal the Cu-BTA film [22–25]. BTA residues remained on the copper surface can be sublimated at a high temperature, causing a lower reliability of the devices [22,24]. Scratches are the most detrimental defects because these directly affect the yield. Several reports describing several possible root causes of scratch formation have been published and the introduction of filtration is an effective measurement for controlling the large particles and agglomerated particles in the slurry to reduce scratches [26–30]. In addition, galvanic corrosion can occur as the barrier CMP

* Corresponding author at: Institute of Microelectronics, Hebei University of Technology, Tianjin 300130, China.
E-mail address: luanyz1988@126.com (X. Luan).

http://dx.doi.org/10.1016/j.mee.2016.12.002
0167-9317/© 2016 Published by Elsevier B.V.

slurry provides the ionic contact medium for the difference of the electrochemical potential between barriers and copper [31–33]. As a result, the less noble metal will corrode at a higher rate than that of the nobler metal. Hence, the control of galvanic corrosion is another key challenge for barrier CMP slurry.

Our team previously developed a weakly alkaline barrier slurry without BTA and oxidizer, and we named this novel barrier slurry as FA/O barrier slurry and presented its formula [6,8,34–35]. The FA/O barrier CMP slurry has obtained satisfactory results of topography modification and exhibits a stability having a shelf life of at least 30 days in the experiments [6,35]. However, no studies have been done on defect performance between it and the competitive barrier CMP slurry. The after-polished surface defect control becomes the key to the yield enhancement as scaling of gate length below 28 nm. Therefore, additional studies are still necessary for improving the performances of this slurry. In the current work, 300 mm copper pattern wafers were polished with the FA/O barrier slurry and the commercial barrier slurry, respectively. Then the comparison was carried out in terms of organic residues, micro-scratches and copper galvanic corrosion. The defect scan tools were used to inspect and analyze the type of the defects, and we were able to determine the source of the defect. The formations of the defects were discussed in detail and the related solution to eliminate the defect was also put forward.

2. Experiment

The FA/O barrier slurry, used in the experiments, was prepared with 10 wt.% colloidal silica (mean particle size is 100 nm), 1.8 wt.% FA/O chelate agent, nonionic surfactant and 0.5 wt.% guanidine nitrate at pH 8.9 according to previous studies [8,35]. The commercial barrier slurry developed for 45/65 nm technology node was purchased from the base line slurry, and it contains BTA with pH in the range of 4–7. The same slurry and condition were used in step 1 and step 2 for making a true and a fair comparison. In addition, the base line cleaning solution (pH is at 4– 7) and a basic alkaline clean chemical which only contains surfactants were prepared to clean the polished pattern wafers, and all the chemicals used in experiment were of analytical grade.

An AMAT Reflection LK tool from Applied Materials Inc., was used for CMP of 300 mm copper pattern wafers and post-CMP cleaning. The polishing pad for copper polish was stacked IC 1000 pad on Suba IV with a transparent window (Dow Chemical Company), and Politex pad (Dow Chemical Company) was used in the barrier polish. After polishing, the polished wafers were washed in megasonic tank and then cleaned in PVA brush box 1 and box 2. At last, the wafers were blow-dried in an IPA vapor dryer. Table 1 shows summary of details for polishing and post-cleaning conditions. In order to prevent the large particles from slurry inflow, a 0.5 μm m point of use filter was installed in the CMP equipment, which has 80% filtering efficiency for 0.5 μm size particles.

The KLA 2367 inspection tool was used to scan post defect characterization, which showed the amount and map of the defect, and the inspection limitation was the size of 0.16 μm and above. The tool SEMVISION G3 was used to identify types of each defect. The Cu surface was imaged by an Agilent 5600LS atomic force microscope-taping mode. Dynamic light scattering method was applied in NICOMP 380/DLS (Particle Sizing Systems Inc., Santa Barbara, CA) to analyze the mean particle size and the distribution of the particles dispersed in the slurry.

Table 1
Polishing and cleaning recipe.

Parameters	Condition
Working pressure	2.2/1.0/1.0/1.5/1.0/1.8 psi
Slurry flow rate	300 ml/min
Head rotating speed	98 rpm
Plate rotating speed	103 rpm
Polishing time	60s
Wafer rotation	100 rpm
Brush rotation	500 rpm
Cleaning solution flow	700 ml/min

3. Results and discussions

Fig. 1 shows the map of the defects and its type as well remained on the patterned wafer surface polished and cleaned with commercial barrier slurry and the simple cleaning solution, respectively. Fig. 1(a) shows the quantities of the defects reach 16,571 and defects are revealed as either BTA films or copper oxide particles (Shown in Fig. 1(b)). For troubleshooting, the brush and the polish pad are new and stable. Therefore, defects originate solely from the process of CMP with the barrier slurry. These copper oxide particles have the potential for moving through the dielectric to become trace metals and deteriorate the properties of the devices. To enhance the removal, an oxidizer (most frequently H_2O_2) and a complexing agent are commonly used in the commercial barrier slurry, and the copper layer is converted to copper oxides and copper hydroxides by the oxidizers and then the complexing agents react rapidly with the copper ions forming water-soluble complexes [31,36,37]. Since the abraded copper oxides can remain at the interface, the post-copper CMP cleaning process should completely remove them from the polished surface.

The simplest approach of surface cleaning from oxide particles is based on chemical dissolution using organic acids or chelating agents, such as citric acid, glycine, acetic acid and oxalic acid, which have the ability to chelate Cu ions and Cu oxides [38,39]. However, almost all of them show relatively more Cu etch rate than DI water, leading to surface defects and even to surface damage [39]. In this experiment, it is inevitable for the copper contaminants remaining on the polished surface because these copper oxides do not dissolve in an alkaline cleaning liquid which contains no complexing agents. Meanwhile, in order to realize the balance between selective copper removal at the protruded area and targeted copper surface protection at the recessed area, BTA is used in most barrier slurry to form Cu-BTA films at the recessed area to protect the copper surface from the corrosive attack [19,21]. However, the insoluble Cu-BTA films are strong and make the surface hydrophopbic, consequently causing more difficulty in cleaning [20]. Under-etching removal and in combination with megasonics and/or brush scrubbing has been studied in cleaning process to remove Cu-BTA films at high total cost of ownership [22–25]. Cu-BTA films are undercut by etching of copper, lifted by mechanical forces, and then transported away from the wafer. Hence it is clear that a simple cleaning solution used in this experiment can't remove the Cu-BTA films completely.

Fig. 2 shows the map of the defect and its type as well on the patterned wafer surface polished and cleaned with the FA/O barrier slurry and the simple cleaning solution, respectively. We can see from Fig. 2(a) that the quantities of the defects have reached a limit value of 30,000 when only a part of the wafer surface is scanned by the tool KLA 2367. However, we don't find any contaminates on the wafer surface, as shown in Fig. 2(b). Since the FA/O barrier slurry is free of BTA and oxidizers, it is the only objective for the cleaning process to remove the colloidal silica particles. Colloidal silica abrasives will attach selectively onto the copper surface due to the same sign of surface zeta potential between the colloidal silica and dielectric (ILD) surface [40], and a cleaning solution containing surfactants can effectively removal all silica particles [10,41]. Therefore, the results that no contaminations on the surface obtained from Fig. 2(b) are what we have expected. The only probable reason to explain why there are a lot of defects on the map is that the defect inspection tool is sensitive to the copper surface roughness and micro-scratches which the naked eye cannot see, but the tool views as defects. To confirm the above suspicion, some test points (the randomly scanned area is 10×10 μm) of copper lines are selected to evaluate the surface quality. As Fig. 3 illustrates, the copper surface