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To cite this article: T. B. Zubi et al 2019 ECS J. Solid State Sci. Technol. 8 P3022

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### JSS FOCUS ISSUE ON CMP FOR Sub-10 NM TECHNOLOGIES

# Unraveling Slurry Chemistry/Nanoparticle/Polymeric Membrane Adsorption Relevant to Cu Chemical Mechanical Planarization (CMP) Filtration Applications

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Due to the emergence of sub-10 nm technologies, next generation slurries have continued to increase in complexity to meet stringent device performance demands. Prior to the chemical mechanical planarization (CMP) process, point-of-use filtration (POU) is implemented in order to limit particle aggregates and ultimately decrease surface defects. This study probes the non-covalent interactions at the interface of a fundamental Cu slurry and a polyamide and polypropylene-based membranes. Results indicate that independent of the membrane used, material removal rate (MRR) showed a subtle decrease as a result of filtration (time and  $\Delta P$ ), demonstrating that the synergistic balance between the nanoparticle and slurry additives is disrupted during the filtration process. Corrosion current measurements (I<sub>corr</sub>) decreased by at least 85% post-filtration, indicating a rapid adsorption of glycine to the filter crystal nanobalance (EQCN) technique. Since Cu-glycine complexes are integral in controlling MRR, a widely reported method of tracking \*OH production was employed. Results show a decrease in the concentration of \*OH, which in turn can be correlated to a decrease in the Cu-glycine complexes, altering the overall Cu MRR.

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Manuscript submitted November 27, 2018; revised manuscript received December 28, 2018. Published January 12, 2019. *This paper is part of the JSS Focus Issue on CMP for Sub-10 nm Technologies.* 

Chemical Mechanical Planarization (CMP) has emerged as a critical process step used to achieve global planarity of complex semiconductor devices that has led to the extension of Moore's Law.<sup>1,2</sup> Coupling the increased amount of transistors used for integrated circuit (IC) development with a decrease in feature size has made the planarization process evermore important to meet stringent postprocessing device requirements.<sup>3</sup> These devices are typically comprised of Cu/low-ĸ layers due to its low resistivity and high electromigration resistance, in addition to the ability to eliminate current leakage within the device.<sup>4–9</sup> In order to optimize the efficiency of these interconnects, the planarization of Cu requires a synergistic balance between the chemical action (slurry) and mechanical force to effectively remove bulk material while controlling defectivity and postpolish wafer topography. More specifically, defects/topography are categorized as micro-scratches, particle contamination, galvanic corrosion/pitting, line dishing, and edge-over-erosion, to name a few.<sup>10-17</sup> To control defectivity during the CMP process for advanced technology nodes, a complex dispersion will be employed, consisting of: functionalized or non-functionalized nanoparticles coupled with chemical components such as complexing agents (small molecules and macromolecular), organic corrosion inhibitors, oxidizing agents, rheological modifiers (i.e. surfactants), and pH adjusters.<sup>13,18-22</sup> Based on the nanoparticle and additive synergy within this complex dispersion, there is an increased amount of non-covalent interactions that can lead to the formation of rogue particle aggregates, which have been known to induce defectivity. Therefore, in-line and point-of-use (POU) filtration can be implemented as a form of process control to maintain a monodispersed nanoparticle dispersion.<sup>23-25</sup> The effectiveness of the polymeric filtration membrane is dependent on its structure, pore size, and surface energy; each of these characteristics can impact the slurry integrity and overall CMP performance. Consequently, it is necessary to investigate the non-covalent interactions, such as electrostatic interactions, pi interactions, hydrogen bonding, or dispersion forces that take place between the slurry formulation and polymeric filtration membrane. It is proposed that the main mode of adsorption/desorption is related to the ability of the slurry, nanoparticle and chemistry to hydrogen bond to the membrane surface which will directly impact the delicate balance needed to obtain optimal Cu CMP performance (i.e. film formation and defect control). Due to the increasing complexity of slurry chemistry, there is currently limited understanding as to how slurry components interact with polymeric filtration membranes. Through non-destructive testing and real-time analysis, this work investigates key adsorption mechanisms of a widely studied silica (SiO<sub>2</sub>) dispersion to both polyamide and polypropylene-based membranes.<sup>2,26–29</sup> Unraveling the non-covalent interactions between the nanoparticle dispersion and filter membrane revealed a change in slurry integrity, which ultimately correlated to the overall CMP performance.

#### Experimental

*Cu slurry formulation.*—The slurry used in this study was composed of 0.5 wt% colloidal SiO<sub>2</sub> (PL-3 obtained from Fuso Chemical Co.), 60 nm in size, 1 wt% glycine (99%, Sigma-Aldrich), 100 ppm benzotriazole (BTA) (99.5% ACS grade, Sigma-Aldrich), and 1 wt%  $H_2O_2$  (30 wt%, JT Baker), along with pH adjusters such as KOH and HNO<sub>3</sub>. The pH of the fully formulated slurry, particle only dispersions, and solutions containing chemistry only (i.e. 1 wt% glycine) were adjusted to 5.8.

*Filtration.*—A Cole Palmer MasterFlex L/S Economy Drive pump was calibrated to 15 mL/min and was attached to a simulated filtration unit. The 47 mm polycarbonate in-line filter holder (provided by Pall Corporation) housed a polymeric filtration membrane and was connected to a Vernier pressure sensor in order to monitor the pressure as a function of time. The pre-filtered slurry travel distance from the pump to the filter membrane capsule was approximately 50 cm, and the distance of the filtrate to the polishing pad was also 50 cm which is consistent with current POU processes. The filtration membranes (provided by Pall Corporation) used in this study were polyamide and polypropylene with nominal pore sizes of 0.2  $\mu$ m and 0.45  $\mu$ m, respectively. The pore sizes selected for this study were based on recommended filter membrane that mimic standard POU filtration conditions implemented during Cu CMP.

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*Particle size and zeta potential.*—Particle size and zeta potential were characterized using a Malvern Zetasizer NANO-ZS dynamic light scattering (DLS) instrument throughout the filtration process.

*Material removal rate (MRR).*—Polishing data was obtained using a Struers LaboPol-5 polisher and 3 mm thin copper disks with a cross sectional area of 7.5 cm<sup>2</sup>. The downforce of the platen on the wafer was set to 3 psi and the platen speed was 100 rpm. The flow rate of the slurry was set to 150 mL/min and was stirred with a magnetic stirrer to avoid the formation of large aggregates. Polishing was performed using an IC-1000 pad, which was conditioned for 10 minutes, prior to each trial using a diamond conditioner. To determine the MRR, the weight of the Cu disk was measured pre- and post-polish. It should be noted that there may be differences in MRR for various Cu samples (i.e. disk vs. wafer), however this study focuses on relative differences.<sup>30–33</sup>

**Potentiodynamic scans.**—Tafel plots, pre- and post-filtration, were obtained using a Gamry Reference 600 potentiostat. In a threeelectrode system, a copper rotating disk (150 rpm) was used as the working electrode while a platinum and saturated calomel electrode were used as the counter and reference electrode, respectively. The equilibration time was set for 15 seconds and the induced voltages ranged from 0.25 V to -0.25 V, referencing against the open circuit potential, with a scan rate of 2 mV/second. The sample area was calculated to be 1.327 cm<sup>2</sup> with a density of 8.96 g/cm<sup>3</sup>. 1 mL of 1 mM KNO<sub>3</sub> was added to the sample prior to each trial. The corrosion current (I<sub>corr</sub>) values were obtained by measuring the current at the intersection of the tangent lines of the cathodic and anodic curves. These values were normalized using the calculated sample area mentioned above.<sup>34,35</sup>

*Electrochemical quartz crystal nanobalance (EQCN).*—Using an ELCHEMA model EQCN-700, a reference point was established by depositing a thin film of polymer resin (provided by Pall Corporation) onto the surface of a gold electrode.

The polyamide resin was dissolved in concentrated formic acid (97%, Alfa Aesar) while the polypropylene was dissolved in toluene (Flinn Scientific). A 40  $\mu$ L sample of 1 wt% glycine (pH of 5.8) was dropped onto the electrode and the change in frequency was monitored over time. In order to solely measure the interaction occurring between the filter membrane and the complexing agent, control measurements were completed with pH 5.8 DI water.

*Hydroxyl radical (\*OH) trapping.*—A well-known \*OH trapping agent, p-nitrosodimethylaniline (PNDA) (97%, Sigma-Aldrich), shows a strong absorption peak at 440 nm, however when PNDA reacts with \*OH, the intensity of this peak decreases.<sup>36</sup> Therefore, when \*OH were generated by mixing 0.01 M  $H_2O_2$  with 1 wt% glycine and cupric ions at pH 8.4 pre- and post-filtration, the concentration was indirectly measured by tracking the change in absorbance at 440 nm using a UV-Vis Red Tide Spectrophotometer.

#### **Results and Discussion**

In order to unravel the dynamic synergy between the slurry formulation and filtration membrane it is essential to investigate the role of the membrane surface energy on filtration performance. More specifically, polyamide and polypropylene filtration membranes were chosen to deliberately alter critical non-covalent interactions that control slurry/polymer adsorption. The primary adsorption mode for the polyamide membrane is hydrogen bonding between the amide functional groups with the surface hydroxyls of the slurry nanoparticle and the amine and carboxyl group of the glycine. It must also be noted that a complex interaction between surface adsorbed glycine on the slurry nanoparticle may further enhance hydrogen bonding to the polyamide membrane. For the polypropylene membrane, due to its hydrophobic nature, the main non-covalent interaction would be weak dispersion forces. Figure 1 shows the pressure response, for



Figure 1. Effect of filtration membrane on time dependent pressure for model Cu CMP Slurry.

polypropylene and polyamide under continuous flow, single-pass conditions for the model Cu CMP slurry described above. For this work  $\Delta P$  is defined as a change in absolute pressure as a function of time.

Results show that there is a difference in pressure response depending on the hydrophilicity and adsorptivity of the filtration membrane used. The polypropylene membrane exhibits no significant change in pressure as a function of time due to its non-polar characteristics that limit the non-covalent interactions between the membrane and slurry. Because the structure does not allow for hydrogen bonding, the slurry will not adsorb to the surface. However, the polyamide-based membrane shows a greater affinity to the slurry causing a rapid increase in pressure, potentially diminishing filter life. This increased interaction is caused by the ability of the membrane to hydrogen bond with the hydroxyl groups on the particle. Additionally, other components of the slurry such as the complexing and passivating agents have the ability to non-covalently interact with the filter. These interactions cause a buildup of particle within the porous membrane resulting in an increase in the rate of adsorption. More specifically, these interactions can be described as enhanced glycine adsorption to free SiO<sub>2</sub> particles via hydrogen bonding. Based on the aforementioned non-covalent interactions, one would speculate that there should be significant differences in the CMP performance upon filtration with membranes of different surface energies. Therefore, a validation experiment to understand the relationship between the slurry chemistry and filtration membrane as a function of pressure was conducted. Figure 2 is a summary of the Cu MRR as a function of  $\Delta P$ .

In theory the filtration process should solely remove rogue (soft and hard) aggregate particles, which have limited impact on removal rate and film formation but should have a significant effect on defectivity (i.e. scratch and particle count). Results show that regardless of filtration performance the MRR decreases as a function of increasing pressure, which can be related to a change in the integrity of the slurry. It must be noted that the pore sizes between polyamide and polypropylene are different, however based on data previously reported, the pore size does not appear to affect the change in MRR or  $\Delta P$  profiles.<sup>37</sup> The increasing standard deviation indicates that the slurry composition is altered (i.e. degradation of productive passivation equilibrium) when exposed to the filtration membrane. That is to say the non-uniform adsorption of the slurry components to the polymeric filtration membrane via non-covalent interactions has increased. In order to unravel key interactions of the slurry with the filtration membrane, it is necessary to analyze subtle changes in slurry integrity. Historically, changes in removal rate, defectivity, and topography control have been correlated to either changes in nanoparticle concentration or degree of aggregation. Utilizing DLS, the changes to the particle size distribution (PSD) can be evaluated as a function of the filtration process to determine the effect of polymeric membrane



Figure 2. Effect of filtration  $\Delta P$  on Cu CMP MRR.

type on post-filtration particle aggregation. Figure 3 shows the PSD of polypropylene and polyamide-based filter membranes as a function of  $\Delta P$ .

Polypropylene-based membrane results revealed that the particle size remains unmodified after implementing filtration, which is expressed by the minimal change in intensity and a conserved monodispersed system. On the other hand, the polyamide filter shows a significant change in particle size characteristics, which can be correlated to the composition of the polymeric membrane. The decrease in intensity and increase in the PSD, as a function of  $\Delta P$  is indicative of particle aggregation. This finding demonstrates that the non-covalent interactions between the nanoparticle and the polyamide filtration membrane are significant enough to alter the integrity of the slurry post-filtration. Additional validation of this can be seen by evaluating zeta potential pre- and post-filtration as shown in Table I.

Results show that regardless of the filtration membrane used, the net surface charge of the slurry nanoparticle statistically decreases. The post-filtration zeta potential measurements for both membranes were taken when the polyamide filter reached the point of saturation (t = 100 min). Historically, this decrease in surface charge can be

Table I. Effect of polymeric filtration membrane on zeta potential of the model Cu CMP slurry.

	Zeta Potential (mV)
Pre-Filtration	$-25.2 \pm 5.0$
Polyamide Filtration	$-17.9 \pm 2.2$
Polypropylene Filtration	$-19.8 \pm 3.1$

attributed to an increase in particle aggregation, however referring to Figure 3, there is minimal aggregation present in the PSD. It is believed that this change in zeta potential is directly related to a modification of the chemical environment (i.e. local complexing and passivating agent concentrations) present in the slurry, which may be the main factor for the aforementioned MRR reduction. The current results have established there is no change in PSD but a change in surface charge, making it necessary to probe the interaction of the Cu slurry chemistry and its effect on film formation. More specifically, subtle changes in chemistry impact concentration gradients which are correlated to the difference in non-covalent interactions overall



Figure 3. Effect of polymeric filter membrane [a) polypropylene and b) polyamide] on the PSD using DLS during critical stages of the filtration process.



Figure 4. Effect of  $\Delta P$  on I<sub>corr</sub> using a model Cu CMP Slurry.

influencing the dynamics of the Cu film, resulting in changes in MRR. Therefore, potentiodynamic scans (Tafel Analysis) can be used to track the changes in corrosion current ( $I_{corr}$ ) and its effect on film formation. Figure 4 is a correlation of  $I_{corr}$  to  $\Delta P$  for polyamide and polypropylene-based membranes.

The chemical balance between glycine, BTA, and H<sub>2</sub>O<sub>2</sub> affects the dynamics of film formation and can ultimately drive MRR. As previously reported, BTA and BTA-like compounds are known to form passivation films and protect against corrosion. Conversely, Cu-glycine complexes promote the formation of \*OH via the Fenton decomposition of H<sub>2</sub>O<sub>2</sub>, which increase the chemical activity (i.e. corrosion rate) of the slurry. If the slurry health is adversely affected by the filtration process one should see impact to the overall film formation equilibrium, which can be monitored via Icorr measurements. Results in Figure 4 show that there is an initial decrease in Icorr, independent of the filtration membrane used, which can be correlated to the decrease in MRR, previously reported in Figure 2. It has been speculated that the drop in removal rate can be related to a change in the PSD, however as seen in Figure 3, particle aggregation is limited. Therefore, the slurry integrity is compromised as the chemical balance, more specifically a decrease in glycine concentration, is disrupted during filtration. Upon reaching saturation, the polyamide filtration membrane was able to capture particle as a result of glycine adsorption, however in the case of polypropylene this did not occur. Thus, when the maximum saturation level is reached for the polyamide-based membrane, the  $I_{corr}$  values level off because the concentration of glycine is stabilized in the system. In order to further validate the adsorption of glycine to the polymeric membranes, the complexing agent was filtered independent of SiO<sub>2</sub> and the filtrate was incorporated into the model Cu CMP slurry. From this slurry, I<sub>corr</sub> measurements were run as a function of glycine filtration to track the change in film formation dynamics, as shown in Table II.

The results show that there is a significant difference in the  $I_{corr}$  value pre- and post-filtration independent of the membrane used. While actual concentrations were not measured, this is a clear indication that glycine has a rapid initial adsorption on filter membrane that has been pre-wet with water. Further validation of complexing agent adsorption to the polymeric filtration membrane was determined using EQCN as shown in Figure 5.

Table II. Effect of filtered glycine on model Cu CMP Icorr values.

 $I_{corr}$  Values ( $\mu$ A/cm<sup>2</sup>) for Untreated Membrane

	Pre-Filtration	Post-Filtration at 10 minutes	Post-Filtration at 60 minutes
Polyamide Polypropylene	$10.14 \pm 1.76$ $10.14 \pm 1.76$	$\begin{array}{c} 1.36 \pm 0.75 \\ 1.50 \pm 0.26 \end{array}$	$\begin{array}{c} 1.07 \pm 0.34 \\ 1.50 \pm 0.26 \end{array}$

Upon injection of a glycine solution on the as-deposited thin film (i.e. polypropylene and polyamide), two distinct regions of adsorption exist. The initial rate of adsorption for both films appears to occur at roughly the same time (i.e. 10 s) validating rapid uptake of glycine on the polymer. Additionally, one can clearly see that the magnitude of glycine uptake on the polyamide thin film is significantly higher, which is consistent with the argument of non-covalent interactions. In order to study the synergy between glycine-polymer and glycine-particle interactions an experiment was conducted to simulate filter lifetime conditions. More specifically, a 1.0 wt% SiO<sub>2</sub> nanoparticle dispersion was first filtered through the polymeric membrane, to embed particle in the matrix of the filter. It must be noted that this embedded particle matrix was filtered with water to remove physio-adsorbed particles. Glycine was then filtered and incorporated into a model Cu CMP slurry as previously described to measure Icorr values as a function of filter time, as seen in Table III.

Similar to Table II, results show that there was a decrease in  $I_{corr}$  values post-filtration regardless of filter time and filtration membrane used. This clearly indicates an imbalance in slurry formulation

Table III. Effect of filtered glycine through SiO\_2-embedded polymeric membranes on model Cu CMP  $I_{\rm corr}$  values

Icorr V	Values	$(\mu A/cm^2)$	) for SiO	2 Seeded	Membrane
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	Pre-Filtration	Post-Filtration at 10 minutes	Post-Filtration at 60 minutes
Polyamide Polypropylene	$10.14 \pm 1.76$ $10.14 \pm 1.76$	$0.78 \pm 0.09 \\ 1.22 \pm 0.20$	$0.63 \pm 0.08 \\ 0.88 \pm 0.01$



Figure 5. Validation of glycine adsorption via a modified EQCN technique.

which can be correlated to a decrease in glycine concentration. It must be noted that the presence of particle initially shows little to no difference to the untreated filter, but may show an effect as a function of filter lifetime. While not a focus of this study, filtering glycine longer shows a decrease in Icorr which could be a direct effect of glycine-particle adsorption resulting in ligand bridging, further reducing its local concentration. Furthermore, this may also explain why the polyamide membrane showed significant plugging (i.e. increase in  $\Delta P$ ) due to an increase in non-covalent sites for particle adsorption. To this point, results have shown the filtration process has a significant impact on the slurry integrity, which is related to changes in the complexing/passivating agent synergy. More specifically, it is believed to be related to the concentration of glycine preand post-filtration, which has a direct impact on the chemical activity during the Cu CMP process. To validate the change in glycine concentration as a driving factor, this work employs the known method

of \*OH trapping described above. Understanding the \*OH kinetics is an indirect method to track the depletion in Cu-glycine complexes. Figure 6 shows the effect of the filtration membrane on the pseudo first order kinetics for the model Cu CMP slurry used in this study.

Independent of filtration membrane, there is a clear decrease in the pseudo first order rate constant, k, post-filtration, where k can be determined by taking the initial slope of  $\ln[C/C_0]$  vs. time as seen in Figure 6. The equation below was then used to determine the [\*OH] where k<sub>2</sub> = 1.25E10 mol<sup>-1</sup> s<sup>-1</sup> which represents the initial steady state rate constant of H<sub>2</sub>O<sub>2</sub> and PNDA alone.<sup>36</sup>

 $\mathbf{k} = \mathbf{k}_2 [*OH]$ 

As summarized in Table IV, it is clear that there is a decrease in the calculated concentration of \*OH post-filtration regardless of membrane type. As previously mentioned, literature supports the role of



Equation	y = a + b*x		
Weight	No Weighting		
Residual Sum of Squares	0.00835	0.00728	0.00779
Pearson's r	-0.98957	-0.98966	-0.99366
Adj. R-Square	0.97915	0.97932	0.9873
		Value	Standard Error
Polyamide Filtration	Intercept	0.00958	8.51E-04
	Slope	-1.62E-04	1.62E-06
Polypropylene Filtration	Intercept	0.01115	7.94E-04
	Slope	-1.52E-04	1.51E-06
Pre-Filtration	Intercept	-0.01667	8.22E-04
	Slope	-2.02E-04	1.57E-06

Figure 6. Pseudo first order kinetics of \*OH formation pre- and post-filtration, where  $C_0$  is the initial concentration of PNDA.

Table IV.	Effect of filtration	on *OH formation.
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	[*OH] (M)	Uncertainty
Pre-Filtration	1.62E-13	1.25E-15
Polyamide Filtration	1.30E-13	1.30E-15
Polypropylene Filtration	1.22E-13	1.21E-15

Cu-glycine complexes on the formation of \*OH via Fenton decomposition to enhance MRR. Therefore, the decrease in concentration of \*OH indirectly indicates a change in glycine concentration, resulting in an adverse effect to the MRR.

#### Conclusions

This work investigated the impact of POU on a fundamental Cu CMP slurry to unravel interactions at the polymeric membrane interface for both a polyamide and polypropylene-based membrane. Results confirm that regardless of the filtration membrane used, there is a decrease in MRR, which indicates a change in slurry integrity. This was further validated using potentiodynamic scans and the change in [\*OH], which ultimately determined that glycine is adsorbing to the filter membrane. This study has shown that filtration is not only attributed to the removal of large particle aggregates, but it can also be associated to the adsorption of slurry components. This change in slurry integrity can ultimately be correlated back to the overall Cu MRR.

#### Acknowledgments

This work was generously supported by a multi-year research grant from Pall Corporation.

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