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Organic additives based alkaline alumina slurry for selective removal of barrier layer metals

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In the present work, tantalum (Ta) and copper (Cu) chemical mechanical polishing (CMP) has been carried out using a polishing slurry containing 2 wt% alumina (abrasive), 1 wt% potassium carbonate (oxidizer), and 0.25 wt% each urea and citric acid (additives). The Cu removal rate (*RR*) decreases with an increase in *p*H and is found to be minimum at *p*H 11 while Ta *RR* increases till neutral *p*H and then decreases till *p*H 11 having maximum *RR* at *p*H 7. Ta to Cu removal rate selectivity of 1:1.02 is attained at *p*H 11. Other parameters such as the effect of additives concentration, applied pressure, and platen rotational speed are also studied and results are reported to get a clear scenario. Surface charge analysis reveals that with change in *p*H, zeta potential alters and 30mV zeta potential is found at *p*H 11 which confirms the stability of the formulated slurry. To comprehend the removal mechanism of the metals, potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) are employed. The same trend is encountered for both corrosion current density (I_{corr}) and film resistance (R_{film})values resulting from Tafel and EIS. Based on the research conducted a plausible material removal mechanism is suggested.

Keywords: Alkaline alumina slurry, Barrier layer, Chemical mechanical planarization, Material removal mechanism, Organic additives, Selectivity.

Copper (1.67 μΩ), due to its low resistivity property, has emerged as a doable option for replacing aluminum (2.66 μΩ), which is conventional interconnect metal employed in integrated circuits[1,2]. Cu alone or in stacked form is used as interconnect[3]. High densities, as well as high performance, are needed for future interconnect metal generation. Copper is a highly desirable material as interconnect also accompanies inherent problems associated with it. Those problems are softness, easily deformable, deprived adhesion with accompanying silicon dioxide and other dielectrics layers and beyond 200°C, it diffuses into adjacent surrounding substrate (Si/SiO_2) which ultimately degrades device performance[4,5].

An adhesive barrier layer deposit is required for preventing the Cu diffusion onto the underneath Si/SiO_2 layer. Among the variety of metals investigated, tantalum (Ta) and its nitride (TaN) show a thermodynamically stable behaviour towards Cu. This property of Ta makes it an excellent choice for barrier layer application at high temperatures also[6]. A selectivity issue during chemical mechanical planarization (CMP) is encountered as both metals differ largely in their physical and chemical properties. As compared to Ta, Cu is a mild soft material due to which variation in material removal rate exists. Cu being mild reacts with oxidizer easily. On the other hand, Ta shows inert nature towards the oxidizer. To achieve the final global planarization, an appropriate slurry needs to be designed which results in an identical removal rate for both the materials hence resulting in 1:1 ideal polishing selectivity[7,8]. Polishing slurry formulation being a key aspect of CMP mainly comprises of oxidizer (oxidizes the metal surface), abrasive (abrades the formed oxide layer), and various chemical additives (complexing agent, corrosion inhibitor, surfactant, stabilizer) which assist in providing the desired material removal. Fumed and colloidal silica, alumina is recurrently studied abrasive, while intricately explored oxidizers are hydrogen peroxide (H_2O_2), potassium iodate, and derivatives of quinones[9-13]. To achieve a polishing selectivity of 1:1, very few types of research have been carried out. Vijaykumar *et al.* performed the selectivity studies on Ta barrier layer and Cu with alumina and silica abrasives and H_2O_2 oxidizer[14]. Results show that with the alteration of *p*H and inhibitor, the desired selectivity is achieved at *p*H 4

with silica abrasive and 0.1 wt% BTA[14]. An oxalic acid-based fumed silica slurry containing H_2O_2 oxidizer was investigated for Cu to Ta selectivity. It was reported that by varying the *p*H in a range of 3 to 6 desired selectivity was attained[15]. Numerous chemical additives such as citric acid, tartaric acid, and glycine are investigated to serve as a complexing agent for Ta, and it was reported that they have improved the Ta removal rate (*RR*)[16-18]. Most of the reported work uses hydrogen peroxide as an oxidizer. However, hydrogen peroxide, being a thermodynamically unstable compound, is less widely used in industries[12].

Present work primarily focuses on obtaining the Ta to Cu polishing selectivity of ~1:1 using potassium carbonate as an oxidizer, alumina as abrasive, and urea and citric acid as additives. The slurry formulation reported is unique and has not been studied earlier. Various parameters that affect the polishing rate are also considered i.e., the impact of *p*H, complexing agent concentration, applied pressure change and varied platen rotational speed. Zeta potential and particle size analyses were carried out at odd *p*H values. Electrochemical studies were also conducted with various electrolyte formulations to validate the CMP results. Finally, a plausible material removal mechanism is reported based on the experimental data obtained.

Experimental Section

Polishing experiments

A benchtop polishing machine model Labopol 5/Laboforce 3 (Struers, Denmark) was used for conducting the CMP experiments. A SUBA™ IV polishing pad (8-inch dia), made up of polyurethane material and impregnated with polyester felts, purchased from Eminess Technologies, USA, was used. Ta and Cu coupons (99.9% pure) having a diameter and thickness of 2.5 cm and 1.25 cm respectively, procured from Industrial Engineering Store, India was used for the polishing experiments. A peristaltic pump (Ravel Hitek, India) with a flow rate of 100 ml/min was used to transfer the polishing slurry to the CMP machine. Unless specified, the platen rotational speed and applied downward pressure were held constant at 100 rpm and 40680 Pa respectively. The polishing experiment was conducted for a min.

Alumina and potassium carbonate were used as abrasive and oxidizer respectively. Urea and citric acid were investigated as additives / complexing agents. The formulated polishing slurry contained abrasive (2 wt%), oxidizer (1 wt%), and complexing agents (each 0.25 wt%) was employed. Distilled water was used as aqueous media for preparing the slurry. For modulating the *p*H value of the slurry, potassium hydroxide (KOH) or nitric acid (HNO_3) were used. All the chemicals were purchased from Loba Chemie Private Limited, India. To ensure consistency in results, the polishing pad was conditioned by performing three dummy runs. The metal coupon was washed, dried, and weighed before and after the CMP runs to obtain the weight loss value. The average removal rate (nm min^{-1}) calculated from the weight loss results of the three runs are reported for the individual system along with the standard deviation.

Electrochemical experiments

Potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) studies were performed to get an insight into the electrochemical properties of Ta and Cu. All the experiments were performed using PARSTAT MC 1000 electro chemical workstation (AMETEK, USA)having a three-electrode system. Platinum wire and Ag/AgCl electrodes were used as counter and reference. Working electrodes were made by sealing copper and tantalum rods in Teflon, connected to copper wire to ensure connectivity with the electrochemical system. The electrodes were polished in series with 0.3 µm and 0.05 µm micro polish alumina particles, obtained from Buehler, USA, for 5 min followed by washing and sonication. The four electrolytes formulation studied were: (A) 1 wt% potassium carbonate, (B) 1 wt% potassium carbonate and 0.25 wt% urea, (C) 1 wt% potassium carbonate and 0.25 wt% citric acid, and (D) 1 wt% potassium carbonate, 0.25 wt% urea and 0.25 wt% citric acid. All the electrolytes were at *p*H 11, maintained using HNO_3. Electrochemical experiments were performed in the absence of abrasives.

Preceding each potentiodynamic polarization and EIS, open circuit potential (OCP) measurement was conducted for 5 min to confirm the stability of the system. A potential range of -250mV to +250mV with respect to OCP was applied with a scan rate of 1 mV/s for potentiodynamic polarization experiments. Corrosion current density (I_{corr}) and corrosion potential (E_{corr}) were determined after the extrapolation of the cathodic and anodic branches of the plot. EIS experiments were performed with a frequency range from 100 kHz to 0.1 Hz at OCP value. ZSimpWin software was further used to analyze the acquired

results. The EIS data were validated using Kramers Kronig Transformation (KKT) software.

Surface charge and particle size measurement

Litesizer-500 purchased from Anton Paar was used to measure the zeta potential and average particle size of various slurry samples. The samples were diluted 100 times (dilution factor =100), sonicated for an hour, and used for measurements. The various slurry compositions used for the analysis are coded as follows:

I. 2 wt% alumina
II. 2 wt% alumina + 1 wt% K_2CO_3
III. 2 wt% alumina + 1 wt% K_2CO_3 + 0.25 wt% urea
IV. 2 wt% alumina + 1 wt% K_2CO_3 + 0.25 wt% citric acid
V. 2 wt% alumina + 1 wt% K_2CO_3 + 0.25 wt% urea + 0.25 wt% citric acid

Results and Discussion

Chemical mechanical polishing

The effect of change in *p*H of the polishing slurry on the *RR* of both the metal coupon is represented in Fig. 1(a). As the *p*H of the polishing slurry containing 2 wt% alumina, 1 wt% potassium carbonate is moved from acidic to basic side, a decreasing *RR* is observed for the Cu coupon. A higher removal rate at acidic pH might be due to the formation of a mechanically weak and comparatively soluble cupric oxide (CuO) layer[19]. Ta *RR* is 28.6 nm min^{-1} at *p*H 3, which increases till neutral *p*H and then decreases as the pH was adjusted to the alkaline region. Minimum Ta to Curemoval rate selectivity of 1:2.19 is obtained at *p*H 11. Further experiments were conducted at *p*H 11 to attain the desired selectivity by the addition of additives / complexing agents.

The changes in Ta:Cu selectivity ratio with the variation of additive concentration in the slurry containing 2 wt% alumina and 1 wt% potassium carbonate (PC) are shown in Fig. 1(b). The additives studied were urea (U) and citric acid (CA). In both cases, as the concentration of the additive is increased from 0.1 wt% to 0.5 wt%, the selectivity decreases initially and then increases when additive concentration was increased to 0.5 wt%. Minimum selectivity ratio of 1:1.3 and 1:1.46 was obtained at 0.25 wt% for urea and citric acid respectively.

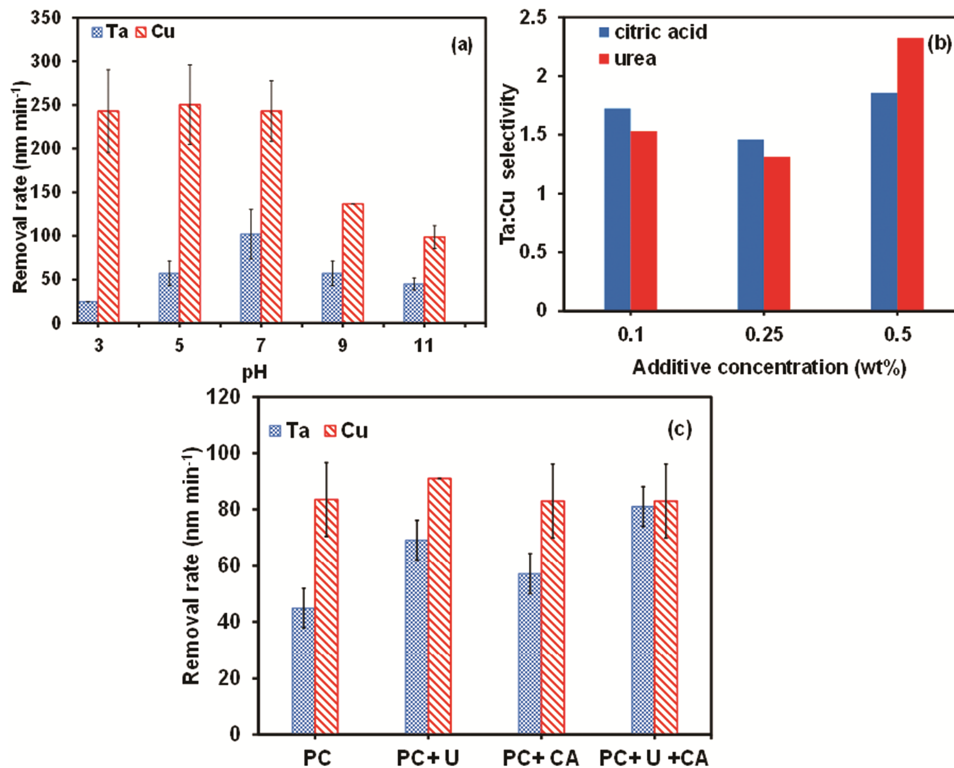


Fig. 1 — (a) Impact of slurry (2 wt% alumina + 1 wt% potassium carbonate (PC)) pH on removal rate of Ta and Cu (b) Ta:Cu selectivity *vs.* additive (urea (U) / citric acid (CA)) concentration in 2 wt% alumina + 1 wt% potassium carbonate (PC) slurry at *p*H 11, and (c) Impact of additive addition on removal rate of Ta and Cu. Slurry consisting of 2 wt% alumina, 1 wt% potassium carbonate (PC), 0.25 wt% urea (U) and 0.25 wt% citric acid (CA) at *p*H 11

A selectivity ratio of 1:2.19 was obtained when the slurry contained 2 wt% alumina and 1 wt% potassium carbonate alone at *p*H 11 and the data is shown in Fig. 1(c). It can be noticed that with the addition of urea or citric acid alone, selectivity decreases considerably. Both urea and the citric acid act as complexing agents for Ta, at the same time it acts as an inhibitor to reduce the removal rate of Cu[20,21]. The addition of both the complexing agents of 0.25 wt% each to the slurry resulted in a removal rate selectivity of 1:1.02.

The effect of applied downward pressure and platen rotational speed on the removal rate selectivity for the slurry containing 2 wt% alumina, 1wt% potassium carbonate, 0.25 wt% urea and 0.25 wt% citric acid was studied and the results are shown in Fig. 2 (a) and (b) respectively. On increasing both the pressure and the platen speed, the selectivity deviates from unity. This observation could be due to the increase in friction force between the exposed metal surfaces, abrasive and polishing pad with the increase in both downward pressure and platen speed[22]. The effect of change in platen speed on removal rate selectivity is relatively less significant as compared to the pressure effect.

Electrochemical analysis

The potentiodynamic polarization plots of Ta and Cu coupons immersed in different electrolytes are shown in Fig. 3(a) and 3(b) respectively. The corrosion current density (I_{corr}) and the corrosion potential (E_{corr}) were obtained after extrapolation of anodic and cathodic branches and the obtained values are presented in Table 1. In the case of Ta, the I_{corr} value for the electrolyte solution B and C is 1.4 µA cm^{-2} and 1.2 µA cm^{-2} respectively, while in the presence of only oxidizer i.e., electrolyte solution A, the I_{corr} value is 0.8 µA cm^{-2}. The anodic branch

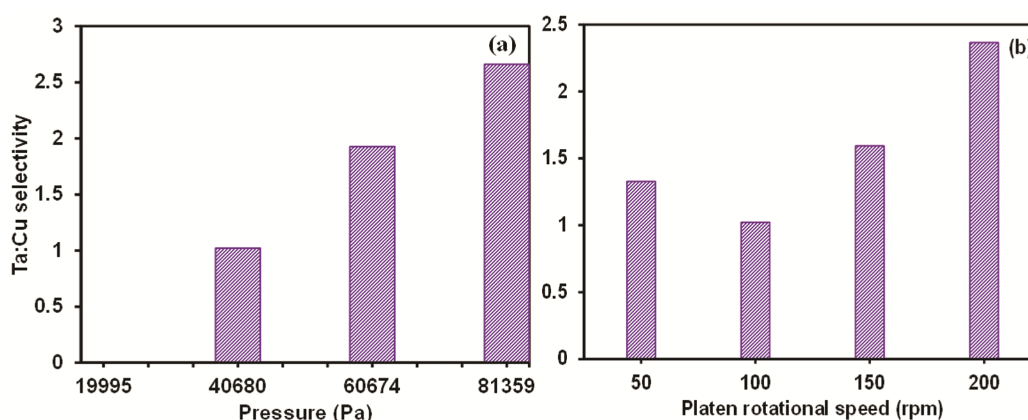


Fig. 2 — (a) Selectivity *vs.* applied downward pressure and (b) Selectivity *vs.* platen rotational speed. Slurry consisting of 2 wt% alumina, 1 wt% potassium carbonate (PC), 0.25 wt% urea (U) and 0.25 wt% citric acid (CA) at *p*H 11

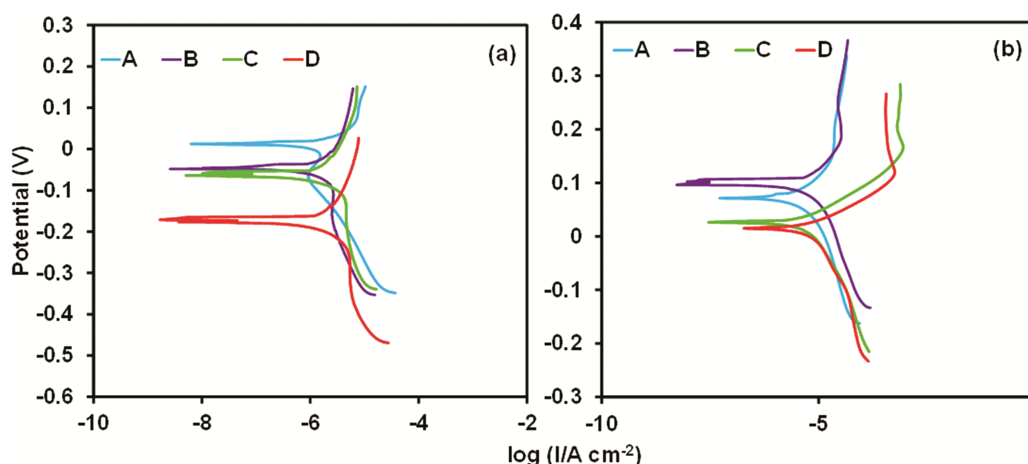


Fig. 3 — Tafel plot of (a) Ta and (b) Cu immersed in various electrolytes

Table 1 — Tafel extrapolation parameters of Ta and Cu immersed in various electrolytes

	Ta				Cu			
	E_{corr} (mV vs. Ag/AgCl)	β_c (mV dec^{-1})	β_a (mV dec^{-1})	I_{corr} (µA cm^{-2})	E_{corr} (mV vs. Ag/AgCl)	β_c (mV dec^{-1})	β_a (mV dec^{-1})	I_{corr} (µA cm^{-2})
A	8.3	64.8	43.6	0.8	70.1	201.7	154.9	1.4
B	-48.6	127.4	132.1	1.4	99.1	154.5	112.2	1.5
C	-52.2	140.0	140.0	1.2	25.1	111.7	57.1	0.9
D	-81.3	102.8	180.2	1.5	15.4	144.1	43.0	1.3

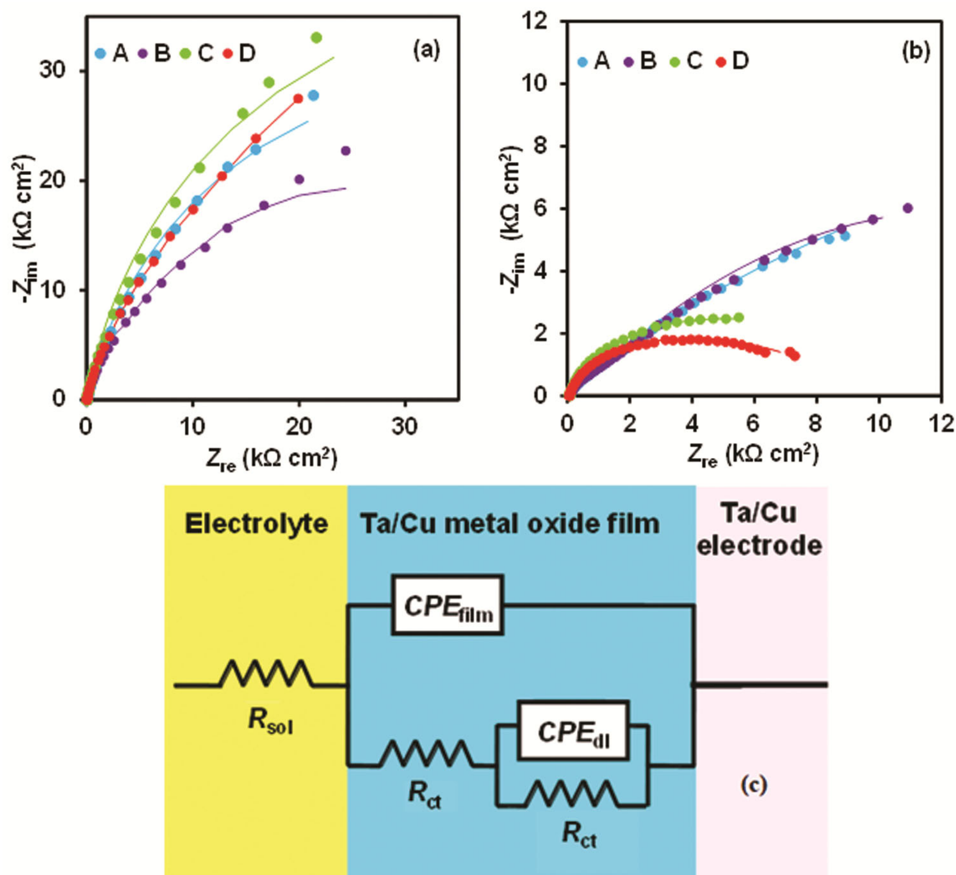


Fig. 4 — Nyquist impedance diagram for (a) Ta, (b) Cu and (c) Electrical equivalent circuit (EEC) fitted in this study

moves towards the right indicating an increase in corrosion current with the addition of complexing agents. This trend is in line with the behaviour observed in CMP studies. The highest I_{corr} value of 1.5 µA cm^{-2} is observed with the electrolyte D.In the case of copper, at the anodic region, a kink is observed for all the electrolytes. This is attributed to the formation of an insoluble oxide layer at the electrode surface. This shows the inhibition mechanism of urea and citric acid on the metal surface[23] and hence, suppression in dissolution, as the I_{corr} value also decreases to 0.9 µA cm^{-2} and 1.3 µA cm^{-2} for the electrolytes C and D respectively.

Galvanic corrosion was evaluated, which arises when different electrochemical materials come into electrical contact while immersed in the electrolyte. Problem due to galvanic corrosion comes into existence when the corrosion potential difference exceeds a range of +100 mV to -100 mV[24]. The difference in corrosion potential of Cu to Ta in the absence of additives is 61.7 mV. Also, with the addition of both the additives, the potential difference was found to be -96.7 mV. This confirms that the addition of both additives has the potential to handle the galvanic corrosion between the two metals.

Nyquist plot for Ta and Cu immersed in various electrolytes is shown in Fig. 4(a) and 4(b) respectively. The presence of a less noticeable single capacitive loop is seen at high frequency and inclined line at low frequency[24,25]. The Nyquist plot presents

the same pattern for all the cases and there is only a slight change in the impedance values upon the addition of complexing agents. For Ta, the Nyquist plot at pH 11 presents a slightly higher impedance value for electrolyte D, when compared to electrolyte A. In the case of Cu, the impedance decreases significantly for electrolyte D in comparison with A.

The electrical equivalent circuit which was used to model the system is shown in Fig. 4(c). Three resistances are present in the EEC, *viz.,*solution resistance (R_{sol}), oxide film resistance (R_{film}), and charge transfer resistance (R_{ct}). Also, two constant phase elements (*CPE*) are present to counter the heterogeneity and surface roughness present in the working electrode[26-28].

The best-fit parameters for both the metals in various electrolytes at *p*H 11 are shown in Table 2. The value of n tends to 1 which shows the homogeneity of the electrode surface[29]. The resistance associated with oxide film varies with the addition of both urea and citric acid for Ta and Cu respectively. And finally, shows minimum and maximum film resistance values for the Ta and Cu respectively. This behaviour of variation in the resistance can be ascribed to the formation of metal oxide films at different electrolytic formulations which ultimately controls the dissolution.

The obtained EIS data is checked for causality, stability, and linearity using KramersKronig Transformations. The complex function constitutes the real (Z_{re}) and imaginary (Z_{im}) parts of impedance which are mathematically related using KKT[30-32]. A group of KKT transformed data for both the metals is shown in Figs 5 (a)-(d). The KKT validation plots for the system indicate that the proposed system is stable and linear. Thus, the electrochemical system is found to be stable, causal, and linear, as the experimental and KKT fitted data counterparts match well with each other.

Surface charge and average particle size

Electrophoretic properties of alumina govern the particle stability in the polishing slurry[33, 34]. The effect of pH on the zeta potential of alumina slurry with and without oxidizer and complexing agents are shown in Fig. 6 (a). It can be observed that with a change in pH from acidic to alkaline region, the zeta potential for all the slurry varies from positive to negative potential with an isoelectric point (iep) near neutral for the first 3 cases and an isoelectric point near 3 for the last 2 formulations. At *p*H 11, a negative potential is observed for all the slurry formulations. An absolute zeta potential value of higher than 30 mV indicates a highly stable slurry.

The extent of aggregation or dispersion is influenced greatly by the surface charge possessed by the particles dispersed in the polishing slurry. Subjected to the kind of surface charge and polarity acquired by the particles, it undergoes either aggregation or dispersion as the *p*H is varied [35]. As shown in Fig. 6(b), the slurry formulation I and II show no particular trend of change in the particle size with an increase in *p*H. The particle size decreases with an increase in *p*H for slurries III and IV. The particle size is in the range of 500 to 1500 nm for earlier mentioned slurries but varies significantly for slurry V. For slurry V, the particle size decreases to 460 nm at *p*H 11 from 2673 nm at *p*H 3. The Ta removal rate increases with a decrease in particle size[36] whereas Cu *RR* decreases. It has been observed that the presence of larger agglomerates causes critical CMP defects, surface damage, and alteration in the polishing mechanism[37,38].

The plausible material removal mechanism

Material removal in CMP is governed by chemical and mechanical phenomena taking place at the metal surface. The chemical phenomena comprise of oxidation of the metal surface which results from the interaction of the oxidizer and the metal. The addition of additives also takes part in the chemical action as per their synergy with the metal surface and polishing slurry. After oxidation, the joined friction of abrasive particles and the polishing pad with the metal surface leads to mechanical abrasion. The polishing slurry is

Table 2 — EEC fit parameters of Ta and Cu immersed in various electrolytes

	Ta				Cu			
	A	B	C	D	A	B	C	D
R_{sol} (Ω cm^2)	33.7	30.8	32.7	0.3	65.7	80.6	74.0	66.4
Y_{film}x 10^5 ($\Omega^{-1}s^n$ cm^{-2})	3.6	1.3	2.9	0.1	0.3	0.4	0.8	0.4
n_{film}	0.8	1	0.9	0.7	0.8	1	0.8	0.8
R_{film}(Ω cm^2)	70920	129	34670	29.4	73.5	32.5	1199	70.7
Y_{dl}x 10^5 ($\Omega^{-1}s^n$ cm^{-2})	3.2	2.5	4.7	2.5	4.5	9.9	7.6	5.733
n_{dl}	0.8	0.8	1	0.9	0.8	0.4	0.8	0.8
R_{ct}(Ω cm^2)	144.1	75570	13370	8600	13050	46260	24680	9667

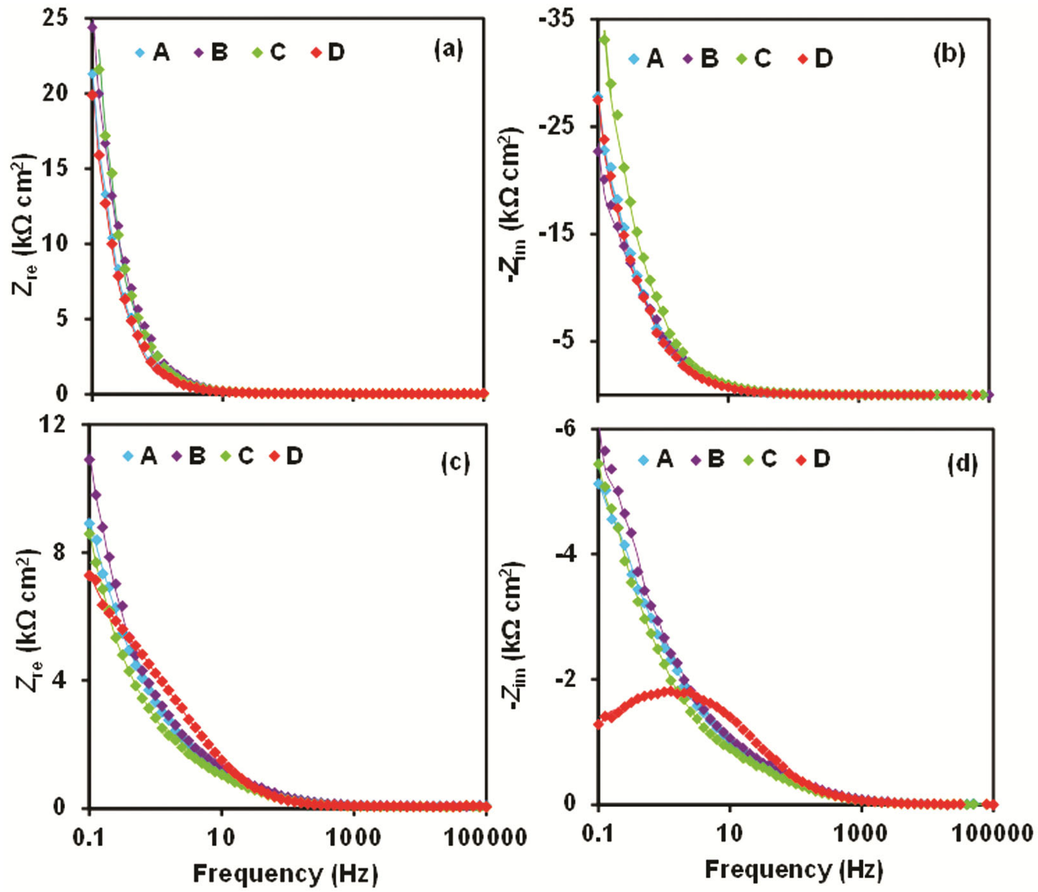


Fig. 5 —KKT fits for Ta (a) Z_{re} vs. frequency, (b) $-Z_{im}$ vs. frequency, and for Cu (c) Z_{re} vs. frequency and (d) Z_{im} vs. frequency, in different electrolytes

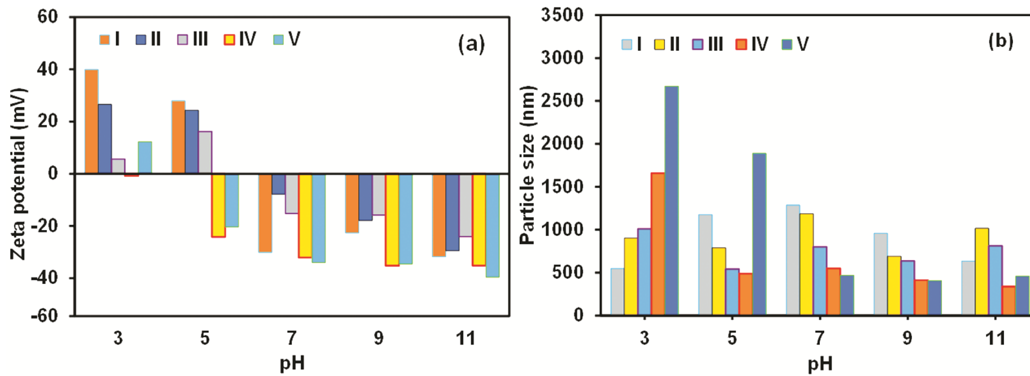
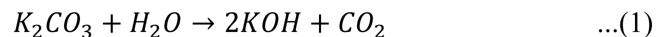


Fig. 6 — (a) Zeta potential *vs p*H, and (b) particle size *vs p*H for various formulated slurries

made to flow between the metal and the polishing pad. The oxidizer present in the slurry oxidizes the Ta surface, and the additives also contribute to the chemical action. The additives act as complexing agents and further form a less stable complex with the oxide of the metal.

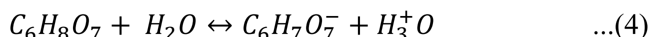
The following chemical reaction is proposed to elucidate the chemical reaction associated with the material removal mechanism for bothmetals. Initially, the oxidizer dissociates in potassium hydroxide when added to the slurry, and the resulted reaction is shown in eq. 1. The next three equations show the break-down of additives into their conjugate ions. Both the metals undergo primary oxidation when kept open to the atmosphere (eq 5[39] and eq 7[40]).



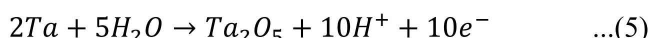
$$K_2CO_3 + H_2O \rightarrow 2KOH + CO_2 \quad ...(1)$$

$$NH_2CONH_2 + H_2O \rightarrow 2NH_3 + CO_2 \quad ...(2)$$

$$2NH_3 \leftrightarrow NH_2^- + NH_4^+ \quad ...(3)$$

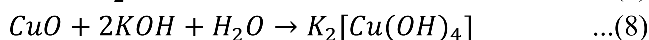

$$C_6H_8O_7 + H_2O \leftrightarrow C_6H_7O_7^- + H_3^+O \quad ...(4)$$

Further, the oxide layer again undergoes oxidation when comes in contact with the slurry shown in eq 6 and eq 8. The ions formed from the dissociation of chemical additives do not take part directly in the reaction, but form complex with the oxidized material which enhances or suppresses the material removal rate.


$$2Ta + 5H_2O \rightarrow Ta_2O_5 + 10H^+ + 10e^- \quad ...(5)$$

$$3Ta_2O_5 + 8KOH \rightarrow K_8Ta_6O_{19} + 4H_2O \quad ...(6)$$

$$2Cu + O_2 \rightarrow 2CuO \quad ...(7)$$


$$CuO + 2KOH + H_2O \rightarrow K_2[Cu(OH)_4] \quad ...(8)$$

Conclusion

Organic additives-based alkaline alumina polishing slurry results in selective removal of Ta and Cu in chemical mechanical planarization. The CMP experiments signify the presence of complexing and inhibiting nature of the additives towards Ta and Cu respectively. As a result, both the chemical action taking place at the same time leads to enhancement of Ta *RR* and suppression of Cu *RR* and finally resulting in a Ta to Cu*RR* selectivity of 1:1.02. Change in particle size does play an important part in material removal. The corrosion density and the resistance values shadow the trend obtained from the CMP experiment result. EIS data was validated using KKT software and fitting shows the linearity of the electrochemical system. The applied electrical equivalent circuit provides insights into the electrochemical reaction occurring. Zeta potential measurements confirm the stability of the polishing slurry. Lastly, both chemical and mechanical material removal mechanism is reported which sums up all the observations drawn from all the mentioned experiments.

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