Characterization of Non-Amine-based Post-Copper Chemical Mechanical Planarization Cleaning Solution

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Abstract

Copper surfaces can become contaminated by slurry particles and organic residues during chemical mechanical planarization (CMP). Silica particles are widely used as abrasives, while benzotriazole (BTA) is widely used as corrosion inhibitor, in copper CMP slurries. The contaminated copper surface needs to be cleaned by using an effective cleaning solution. These material contaminate the copper surface during CMP and need to be removed by using an effective cleaning solution. The objectives of this work were to develop a non-amine-based alkaline cleaning solution and characterize the solution based on the benzotriazole (organic residue) removal and particle removal efficiency. Cesium hydroxide and potassium hydroxide were used as cleaning agents and ethylene glycol was used as a corrosion inhibitor. Ethylene glycol acts as a chelating agent as well in the cleaning composition. BTA removal was characterized using contact angle measurements, X-ray photoelectron spectroscopy and electrochemical impedance spectroscopy (EIS) techniques. The corrosion protection ability of the cleaning solutions was quantified by potentiodynamic polarization studies. Both potassium hydroxide- and cesium hydroxide-based solutions exhibited high BTA and silica particle removal. When compared to a potassium hydroxide based cleaning solution, cesium hydroxide based cleaning solutions were found to be more effective in terms of low surface roughness and low etch rate.

Keywords: Post-Cu-CMP, Cesium hydroxide, Ethylene glycol, Non-amine cleaning solution, Benzotriazole
1. Introduction

Copper is widely used as an interconnecting material to replace aluminum in integrated circuits due to its useful properties, such as low resistivity and high electromigration resistance [1,2]. Copper is deposited by means of an electrochemical deposition process. Excess copper in the dual damascene process has to be removed by means of chemical mechanical planarization (CMP). In general, a Cu CMP slurry consists of abrasive particles, an oxidizing agent, a chelating agent and a corrosion inhibitor. Benzotriazole (BTA) is the most common corrosion inhibitor used in Cu CMP slurries [3]. However, the CMP process leaves lot of residual contaminants such as organic residues, abrasive particles and metallic contaminants on the copper surface. The organic residue is mostly BTA, while abrasive particles are generally silica [4]. BTA makes the surface highly hydrophobic in nature, which causes severe drying issues and also poor adhesion of the stacking layers. In addition to these contaminants, the CMP process also creates process induced defects such as scratches. Hence post-Cu CMP cleaning of the wafer using an efficient cleaning solution is necessary. BTA can exist in one of three forms, depending on the pH of the slurry [5]. BTA can exist in a protonated form (BTAH+) in strongly acidic media, while in weakly acidic, neutral or weakly alkaline media it exists in the form of BTA; in strongly alkaline media it exists as BTA− [5]. Tromans constructed a potential pH diagram for the Cu-BTA complex and indicated the type of species for a given set of conditions [6]. Copper forms a stable Cu-BTA complex in the pH range 4 to 10 [6-9]. In other words, the Cu-BTA complex is highly insoluble in this pH range [10]. Copper forms two different types of oxides, cuprous oxide and cupric oxide, and the acidic-based post-Cu CMP cleaning solution dissolves both types of oxides from the copper surface, while an alkaline-based post-Cu CMP cleaning solution selectively dissolves the cupric oxide leaving the cuprous oxide to passivate the
surface [4]. Hence, alkaline-based cleaning chemistry is preferred to an acidic-based cleaning solution, as the copper surface is less damaged, according to the Cu Pourbaix diagram [11].

Ammonium hydroxide and tetra methyl ammonium hydroxide (TMAH) have been examined for use of alkaline-based post-Cu CMP cleaning solutions. Severe material loss was encountered for the ammonium hydroxide-based cleaning solution, as ammonia etches the copper surface at higher rates [12]. Thus, a TMAH-based cleaning solution has been used widely for post Cu CMP applications [13,14]. Both the ammonium hydroxide and TMAH are classified as amine-based cleaning agents.

In addition to either one of these compounds, the cleaning solution also consists of a chelating / complexing agent, and a corrosion inhibitor. The corrosion inhibitor should be hydrophilic to overcome the previously mentioned problems associated with hydrophobic surfaces. As amine-based compounds possess high vapor pressures, they can easily evaporate to the environment. Furthermore, amine species can deteriorate the lithography process in semiconductor fabrication units [15, 16]. As such, expensive filters are needed to remove these species from clean room air in industrial applications, leading to higher cost and increased maintenance. In addition, TMAH may cause severe health issues if inhaled or absorbed through skin [17,18]. Hence, the semiconductor industry prefers to use non-amine-based chemistries for all applications. Recently, Barnes et al. proposed a non-amine alkaline cleaning solution, which is devoid of amine and ammonium-containing compounds. The cleaning solution consists of cesium hydroxide, glycerine and iminodiacetic acid [19]. However, the exact role of each compound was not reported. Though amine-based alkaline chemistry has been extensively studied, non-amine-based alkaline chemistry has not been comprehensively studied yet. In this
work, cesium hydroxide and potassium hydroxide are evaluated as candidates in Cu CMP cleaning solution. Ethylene glycol is commonly used as a corrosion inhibitor in CMP slurries as well as in cleaning chemistry [20, 21]. Ethylene glycol can also act as a chelating agent in cleaning chemistry [22]. Contact angle measurements, X-ray photoelectron spectroscopy (XPS) and electrochemical impedance spectroscopy (EIS) techniques were used to confirm BTA removal. Potentiodynamic polarization studies were performed to evaluate the corrosion protection ability of the copper surface cleaned with the proposed cleaning solution. Atomic force microscopy (AFM) was used to monitor the surface quality of copper and field-emission scanning electron microscope (FESEM) was used to quantify the number of particles.

2. Experimental

Copper wafer (Buysemi, Korea) coupons of 2 x 2 cm² were cut from 8 inch blanket copper wafers and were used for all the experiments. The Cu wafers were pre-cleaned in isopropyl alcohol for 1 min. Benzotriazole (BTA) from Aldrich, ethylene glycol (EG) and sodium perchlorate (NaClO₄) from Sigma Aldrich, potassium hydroxide from Samchun, and cesium hydroxide from Alfa Aesar were used for the experiments. Organic contaminant removal from copper coupons was characterized using a static contact angle analyzer (Phoenix 300, SEO Korea). Prior to the contact angle measurements, copper coupons were treated with a 0.1 wt% BTA solution. BTA-treated copper coupons were treated with 200 ml of an alkaline solution of various concentrations. Unless mentioned otherwise, the duration of the cleaning experiment was 1 min. Finally, the copper coupons were rinsed with de-ionized water (DIW) followed by drying using nitrogen gas.
The electrochemical experiments were conducted in a standard three-electrode quartz cell, and the data were acquired using a potentiostat (VersaSTAT 3, Princeton Applied Research, USA). Ag/AgCl (saturated KCl) was used as the reference electrode, a treated copper coupon was used as the working electrode, and a platinum mesh was used as the counter electrode; 0.1 M NaClO₄ was used as the supporting electrolyte. Electrochemical experiments were also conducted with untreated copper coupons in 0.1 M NaClO₄. Potentiodynamic polarization curves were obtained in the range of -250 to +500 mV with respect to the open circuit potential (OCP), at a scan rate of 1 mV/s. EIS measurements of treated copper coupons were conducted at an open circuit potential in a solution containing only 0.1 M NaClO₄. EIS spectra were acquired in the frequency range of 10 kHz - 0.1 Hz with a potential amplitude of 10 mV rms.

XPS (Sigma Probe, Thermo VG, UK) was operated at a base pressure of 3 x10⁻⁹ mbar. The photoelectron spectra were excited by an Al-Kα (1486.6 eV) anode operating at a constant power of 100W. During the spectra acquisition, a constant analyzer energy (CAE) mode was employed at a pass energy of 20 eV and a step of 0.1 eV. XPS spectra were obtained using copper coupons treated with various solutions.

A commercial colloidal silica-based slurry with a solid concentration of 6 wt% was used to contaminate the wafer for the polishing experiments. The primary particle size of the abrasive was 60 nm, and the pH value of the slurry was 9.5. A constant slurry flow rate of 200 ml/min was maintained for the polishing experiments. The wafers were polished with a down pressure of 0.85 psi. The platen and carrier head were rotated at a speed of 60 rpm, and a Politex polishing pad was used for the polishing experiments. The duration of the polishing run was 1 min. The
cleaning experiments were performed in a Class-10 clean room. Contaminated coupons were cleaned using the developed cleaning solution for a fixed cleaning time of 1 min. Coupons contaminated by silica particles were captured by FE-SEM (Mira3, TESCAN, Czech Republic). Particle removal efficiency, that is, the ratio of the average number of particles removed during cleaning to the total number of particles before cleaning, was calculated based on the particle counts obtained from the FESEM image using a software package (Image Pro).

The zeta potentials of silica particles dispersed in DIW and various solutions were measured using a zeta analyzer (ELS-Z, Otsuka Electronics, Japan). For static etch rate experiments, the copper coupons were treated with a cleaning solution for 10 min. The pre- and post- cleaning of copper film thicknesses were measured using a four-point probe (Advanced Technology Instruments, CMT SR5000, Korea). An AFM (Park Systems, Korea) was used to measure the surface roughness of the wafer coupons.

3. Results and Discussion

Figure 1 shows the contact angles of DIW on the BTAtreated copper coupons dipped in various concentrations of CsOH and KOH solutions for 1 min. Hydrophobicity and hydrophilicity of a surface can be measured using contact angle measurements [23, 24]. The contact angle of water on a fresh copper surface was ~30°, which shows it is hydrophilic, and it was taken as reference for this study. The contact angle on a BTA-treated copper coupon was found to be ~ 64°. Copper coupons became hydrophobic after treatment with 0.1 wt% of BTA for 1 min [25]. The stability of the Cu-BTA complex strongly depends on the pH of the slurry used for polishing. The Cu-BTA complex is unstable in the higher alkaline ranges. Both the CsOH and KOH solutions are capable of removing the BTA layer from the copper coupons. The
addition of 0.25 wt% of alkali reduces the contact angle to about 25° and further addition of 
CsOH decreases the contact angle even further while in case of KOH, it decreases only slightly. 
A solution of 1 wt% of alkali corresponds to 0.067 M CsOH and 0.2 M KOH. CsOH is a 
stronger base compared to KOH and this may explain the observed results. The contact angle of 
Cu surface treated with BTA and then 1 wt% alkali is significantly lower than that of fresh Cu 
surface. This is probably because the fresh surface may have oxides of Cu which are removed by 
the cleaning solution. Similar trends were reported in literature [26]. A comparison of the results 
with the two solution shows that KOH solution of higher molarity is needed to reduce the contact 
angle to the same extent as CsOH solution of lower molarity. An alkali concentration of 1 wt% 
was chosen for further experiments. The contact angle was found to be 12° and 19° for 1 wt% 
CsOH and 1 wt% KOH solutions, respectively, and the results indicate that CsOH is more 
effective in making the surface hydrophilic. The addition of 1 wt% EG to 1 wt% CsOH and 1 
wt% KOH increased the contact angle of copper coupons slightly (27° and 30°, respectively), but 
the surface was still hydrophilic.

The data points in Figs. 2(a-b) show the electrochemical impedance spectra of copper 
coupons treated with various solutions, which include BTA and BTA-treated copper cleaned 
with CsOH and KOH solutions. EIS was also performed for the bare copper, which can be taken 
as a reference. The spectra show a semicircular pattern irrespective of the type of surface 
treatment. Even though BTA-treated copper coupons shows a similar pattern to bare copper, the 
magnitude of the impedance at lower frequencies is several times higher. Prasanna et al. reported 
that an increase in polarization resistance for the BTA-treated copper coupons can be attributed 
to the adsorption of BTA on the copper surface [27]. As the impedance spectra for the other three
cases are several times lower than the BTA-treated samples, an expanded graph of the spectra is shown in Fig. 2b. The low frequency impedance magnitude of the spectrum corresponding to the KOH-treated sample is comparable to that of fresh Cu, while that of the CsOH-treated sample is even lower than that of fresh Cu. Simulated impedance values are superimposed with the experimental data points and are shown in the form of a line in Fig. 2(a-b). The electrical equivalent circuit (EEC), which accounts for the presence of the film is shown in Fig. 2c, and it was used to model the experimental data. In the circuit model, $R_s$ is the solution resistance, $Q_1$ and $Q_2$ are the constant phase elements (CPE) associated with the passive film and electrical double layer (EDL) at the metal/electrolyte solution interface, respectively. $R_1$ and $R_2$ denote the resistance associated with the passive film and EDL at the metal/electrolyte solution interface, respectively [28]. In ideal cases, capacitances would be employed to represent the double layer and the passive film but in most real cases CPEs simulate the trends more accurately. The impedance of the CPE is given by $Y(j\omega)^n$ where the deviation of the exponent ‘n’ from 1 indicates the deviation from ideal capacitance behavior. On the other hand, if the values are close to 0.5, it indicates that diffusion limited processes are dominant. Summation of $R_1$ and $R_2$ yields the polarization resistance which can be used to calculate the surface coverage [29-31]. The simulated and experimental impedance values are shown in Fig. 2(a-b) as points and a smooth line, respectively. BTA removal efficiency was calculated using the formula given below [27]:

$$\text{BTA Removal (\%)} = \left[ \frac{R_{p,\text{Max}} - R_{p,x}}{R_{p,\text{Max}} - R_{p,0}} \right] \times 100$$
where $R_{p,x}$ is the polarization resistance of BTA-adsorbed Cu treated with a cleaning solution, $R_{p,0}$ is the polarization resistance of untreated Cu and $R_{p,\text{Max}}$ is the polarization resistance of Cu treated with a BTA solution. The best fit parameters for the simulated value of impedance data are shown in Table 1. On fresh Cu surface, and on surfaces treated with CsOH or KOH, a passivating film is not expected to be present and while on the BTA treated surface, the passivated film is expected to be present. The exponent ‘$n_1$’ is in the range of 0.9 to 0.95 for all the cases except KOH treated surface. For the exponent ‘$n_2$’ the values are close to 0.5 in most cases and this indicate that diffusion limited processes such as movement of dissolved ions from the surface to the bulk or the movement of oxidizing agents to the surface are important. Beyond this, there is no clear trend that can be interpreted directly. The key trend that is observable is based on the polarization resistance values and based on them, BTA removal efficiency was calculated to be 102% for a 1 wt% CsOH solution and 96% for a 1 wt% KOH solution. CsOH was found to be more efficient in removing the Cu-BTA complex when compared to a KOH-based cleaning solution. This result is consistent with the contact angle measurements. A BTA removal efficiency of slightly more than 100% for CsOH may be considered to be 100% within experimental error. It is equally likely that the oxides of copper present on the surface could have been partially removed by CsOH, which would result in a slightly lower $R_p$ value.

Potentiodynamic polarization studies on the copper coupons treated with various solutions were performed using 0.1 M NaClO$_4$ as a supporting electrolyte, and the results are shown in Fig. 3. This experiment was also conducted for the fresh Cu coupon, which was taken as the reference. Anodic and cathodic branches of the polarization plots were extrapolated, and the corrosion current density ($I_{corr}$) was obtained from the intersection of the tangent lines. The
I_{corr} values are tabulated in Table 1. Potentiodynamic polarization studies were conducted in the potential range of -0.25 to 0.50 V vs. OCP, but only the linear region of the Tafel curve was considered for I_{corr} estimation. Fresh copper was found to have an I_{corr} value of 0.7 µA/cm². The I_{corr} value of the BTA-treated copper coupon was found to be 0.1 µA/cm², which shows the corrosion inhibition action of the BTA. Fresh copper coupons treated only with 1 wt% CsOH and 1 wt% KOH solution showed higher values of corrosion current density, i.e., 2.6 µA/cm² and 3.5 µA/cm², respectively. This shows that these alkaline solutions promote copper corrosion and need to be used with a corrosion inhibiting agent for cleaning purposes. The I_{corr} value of the bare copper treated with 1 wt% ethylene glycol was found to be 0.7 µA/cm², which is comparable to that of fresh Cu. The corrosion current of the copper coupon treated with 1% BTA after treating with a cleaning solution containing 1 wt% CsOH and 1 wt% EG showed an I_{corr} of 0.6 µA/cm². This shows that the cleaning solution containing CsOH+EG effectively removes the BTA while protecting the copper surface from corrosion. However, the addition of EG to KOH did not decrease the corrosion current significantly, and the high corrosion current density value of 1.8 µA/cm² suggests that the KOH+EG solution was not effective in controlling the copper corrosion. The molarity of the alkali in the CsOH solution is lower than that in the KOH based solution and this may result in a better surface coverage of EG on the Cu surface, resulting in better corrosion resistance. Etch rate experiments of copper coupons were conducted using CsOH +EG- and KOH +EG-based cleaning solutions for 10 min. The etch rates were found to be 7 Å/min and 11 Å/min for the copper coupons treated with CsOH- and KOH- based solutions, respectively, which corroborates the results of potentiodynamic polarization experiments.
The Cu coupons treated with various solutions were analyzed by XPS to confirm the removal of BTA from the surface. The fresh wafer and the wafers treated with 0.1 wt% BTA, 0.1 wt% BTA followed by 1 wt% CsOH + 1 wt% EG and 0.1 wt% BTA followed by 1 wt% KOH + 1 wt% EG were used for the XPS analysis. The Cu2p3, N1s and O1s spectra are presented in Figs. 4(a-c). Copper coupons treated only with BTA showed a strong peak near 400 eV in the N1s spectrum. The N1s spectrum peak is attributed to the nitrogen atom of benzotriazole. This peak is absent in all the other Cu coupons, which supports the results of electrochemical experiments and shows that both cleaning solutions removed the Cu-BTA complex. The Cu spectra showed two stronger peaks at 932 and 952 eV, which correspond to Cu 2p3/2 and Cu 2p1/2, respectively, and two satellite peaks of weaker intensity at 943 and 962 eV. The presence of the satellite peaks at 943 and 962 eV indicates that Cu is present in cupric form on the surface [32]. However, the presence of Cu2O cannot be ruled out based on the XPS spectra. It is reported that Cu2O would show a peak at 932 eV while CuO would show a peak at 934 eV. Similarly, Cu2O would show a peak at 952 eV while CuO would show a peak at 954 eV. The close proximity of these peaks suggests that it would be difficult to identify the presence of Cu2O in presence of CuO based on the XPS data near those binding energies. On the other hand, the satellite peaks at 943 and 962 eV are exhibited only by CuO and hence the results confirm the presence of CuO on the Cu surface. For the samples treated with CsOH and KOH, the intensity of the satellite peaks is lower, which indicates a partial removal of the copper oxides by these solutions. The O1s spectrum showed a peak at ~ 531 eV for all the copper coupons, but it is less in intense for CsOH- and KOH-treated surfaces, also suggesting that some oxide is removed from the surface [27]. These results support the conclusions from EIS data that the cleaning solutions completely remove BTA and may partially remove copper oxides from the surface.
The copper coupons were contaminated using a CMP polisher and were cleaned using a 1 wt% CsOH and 1 wt% KOH solution. Both solutions removed 75% of the contaminated particles. Contaminated copper coupons were also cleaned with a 1 wt% CsOH+1 wt% EG solution and a 1 wt% KOH+1 wt% EG solution. Typical FESEM images of copper coupons are shown in Fig. 5(a-c). FESEM images were taken on 3 different locations on copper coupons and were repeated for 3 different samples. Particles were mostly removed when 1 wt% EG was used with 1 wt% CsOH solution. However, few particles still remained in the case of Cu coupons cleaned with a 1 wt% KOH + 1 wt% EG solution. In the case of copper coupons cleaned using a CsOH-based cleaning solution, most of the particles were removed from the surface, except for a few particles in the SEM images taken near the corner region; particle removal efficiency (PRE) was found to be greater than 99%. However, copper coupons cleaned with KOH-based cleaning solution showed relatively more particles when compared to a CsOH-based cleaning solution and the PRE was found to be ~ 98%. While these numbers are practically the same, they indicate that the proposed solutions are viable candidates for post Cu CMP cleaning. Particle density per unit area was found to relatively higher for the coupons cleaned with KOH-based solution, in comparison to the CsOH-based cleaning solution. Particle density per unit area can be further reduced by introducing physical forces in the form of megasonic energy along with the cleaning solution, which can be considered in a future study.

The zeta potential (ZP) results for the silica particles dispersed in DIW and various solutions are shown in Fig. 6. The ZP of the silica particle dispersed in DIW was found to be -38 mV. The ZP of the silica particle dispersed in a CsOH-based cleaning solution was found to be -41 mV, while the silica particle dispersed in a KOH-based cleaning solution was -29 mV. Here,
the pH of the cleaning solution was ~13. Several reports suggest that the copper surface will exhibit a highly negative charge in the alkaline pH region [33-35]. Thus, the particles are dislodged easily from the copper surface in the case of CsOH-based solution due to higher electrostatic repulsive force, which also prevents re-deposition. This result may also be one of the reasons for increased removal of particles when using the CsOH-based cleaning solution when compared to KOH-based cleaning solution.

AFM images of the fresh copper surface and those cleaned with the two cleaning solutions are shown in Figs. 7 (a-c). An effective cleaning solution should result in low surface roughness after the cleaning process. The surface roughness of a fresh copper coupon was found to be 2.3 nm, which can be taken as a reference in this study. Surface roughness values of copper coupons cleaned with CsOH and KOH-based cleaning solutions were 0.9 nm and 1.7 nm, respectively. In conjunction with the other experimental results, this shows that a CsOH solution with EG is suitable for BTA and particle removal in post Cu CMP cleaning.

4. Conclusion

Cleaning solutions consisting of CsOH and KOH as cleaning agents and ethylene glycol as a corrosion inhibitor were evaluated. Based on contact angle measurements, a minimum concentration of 1 wt% alkali is required for the removal of BTA. XPS analysis confirmed the removal of BTA from a copper surface for both solutions. EIS measurements also showed complete removal of BTA with CsOH and KOH solutions. CsOH and KOH are capable of breaking the Cu-BTA complex and making the surface hydrophilic. Ethylene glycol acts as a good corrosion inhibitor to the CsOH-based cleaning solution but not in a KOH-based cleaning
solution. In addition, a CsOH +EG solution offered better particle removal due to the larger zeta potential of the particles, a lower Cu static etch rate and a lower roughness compared to the KOH+EG solution and was found to be suitable in all aspects for post Cu CMP cleaning.

Acknowledgements

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References


Table captions

Table 1.EEC parameters obtained for Cu treated with various solutions, which are presented in Fig. 2.

Table 2.Corrosion current density of copper coupons treated with the various solutions.
Figure Captions

**Figure 1.** Contact angle measurements of BTA-treated copper coupons treated with various concentrations of cesium hydroxide and potassium hydroxide.

**Figure 2.** Simulated impedance data superimposed with experimental raw data for Cu in a solution containing only supporting electrolyte in 0.1 M NaClO₄. Prior to the electrochemical
experiments, copper was treated with various solutions: **a)** (A) The bare copper, (B) 0.1 BTA-treated, (C) 0.1 wt% BTA treatment followed by 1 wt% CsOH solution and (D) 0.1 wt% BTA treatment followed by 1 wt% KOH solution. **b)** Expanded view at lower impedance for clarity and **c)** electrical equivalent circuit model used to fit the impedance data.

**Figure 3.** Potentiodynamic polarization curves of the copper treated with the various solutions: (i) (A) bare, (B) 0.1 wt% BTA, (C) 1 wt% CsOH, (D) 1 wt% EG, (E) 0.1 wt% BTA followed by 1 wt% CsOH and (F) 0.1 wt% BTA followed by 1 wt% CsOH + 1 wt% EG; and (ii) (A) bare, (B) 0.1 wt% BTA, (C) 1 wt% KOH, (D) 1 wt% EG, (E) 0.1 wt% BTA followed by 1 wt% KOH and (F) 0.1 wt% BTA followed by 1 wt% KOH + 1 wt% EG (F). Note: Solid line – fresh copper and dotted line – BTA-treated copper.

**Figure 4.** XPS spectra of (a) Cu2p$_3$, (b) N1s, and (c) O1s for the copper coupons treated with various solutions: (1) BTA-treated, (2) BTA-treated coupon followed by 1 wt% CsOH + 1 wt% EG solution, (3) BTA-treated coupon followed by 1 wt% KOH + 1 wt% EG solution and (4) bare sample.

**Figure 5.** FESEM images of the copper coupons: (a) after contamination (before cleaning), (b) after cleaning using 1 wt% CsOH + 1 wt% EG solution and (c) after cleaning using 1 wt% KOH + 1 wt% EG solution.

**Figure 6.** Zeta potential of silica particles in various cleaning solutions.

**Figure 7.** AFM images of the copper wafers: (a) fresh wafer (before cleaning), (b) after cleaning using 1 wt% CsOH + 1 wt% EG solution and (c) after cleaning using 1 wt% KOH + 1 wt% EG solution.
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### Table 2.

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</table>
Figure 1.

![Bar chart showing contact angle (degree) as a function of alkali concentration (wt%) with CsOH and KOH as alkali sources. The x-axis represents alkali concentration (wt%) from 0 to 1% with increments of 0.25%, and the y-axis represents contact angle from 0 to 80 degrees. The chart includes error bars indicating variability.](image-url)
Figure 2.
c.
Figure 3a.

- Bare Copper
- 0.1 wt% BTA
- 1 wt% CsOH
- 1 wt% EG
- -1 wt% CsOH
- -1 wt% CsOH + 1 wt% EG

Figure 3b.

- Bare Copper
- 0.1 wt% BTA
- 1 wt% KOH
- 1 wt% EG
- -1 wt% KOH
- -1 wt% KOH + 1 wt% EG
Figure 4.
Intensity (a.u) vs. Binding energy (eV) for O1s peak.

- Curve 1
- Curve 2
- Curve 3
- Curve 4

The peak is centered around 532 eV, indicating the characteristic energy of the O1s electron transition.
b.
c.
Figure 6.

[Bar chart showing zeta potential (mV) for different solutions: DIW, 1 wt% KOH, 1 wt% KOH + 1% EG, 1 wt% CsOH, 1 wt% CsOH + 1% EG.]
Figure 7.

\[ R_a = 2.3 \text{ nm} \]
$R_a = 0.9$ nm
$R_a = 1.7 \text{ nm}$