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Ammonia Free Cleaning Solution for Post-CMP Cleaning (Chemical Mechanical Polishing)

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ABSTRACT

Ammoniacal nitrogen is one of the new parameters incorporated under the Industrial Effluents Regulation in Environmental Quality Act (EQA) starting from January 1st, 2010. Under this regulation, ammoniacal nitrogen limit for industries is regulated at a maximum limit of 10 ppm and 20 ppm depending on whether the facilities located upstream or downstream of the water catchments area. However, the ammoniacal nitrogen limit for semi-conductor companies that started their operation before 2010 has been increased to twice the initial limit due to concerns raised by some of the affected companies. This temporary limit was loosened until January 1st, 2020. The ammoniacal nitrogen is contributed by the use of ammonium hydroxide solution in the wafer fabrication industry, particularly in Chemical Mechanical Polishing (CMP) process. In CMP, the surface of silicon wafers being polished with slurry causing deposition of debris on the wafers. The cleaning process after polishing is termed as a post-CMP step. This paper focuses on the evaluation of post-CMP cleaning efficiency using a SpeedFam IPEC (SFI) AvantGaard™ 776 polisher tool. There are two stages of post-CMP steps named as buffing and scrubbing process. There were studies conducted by past researchers on post-CMP cleaning but none of these studies can be adopted either because the techniques were not economical for production scale compared to wet cleaning process or the chemicals selected are ammonia-based. The objective of the study is to analyse cleaning efficiency at both buffing and scrubbing steps and formulate an alternative solution that does not contain ammonia without compromising cleaning efficiency. It was discovered that the particles on the wafers were effectively removed with efficiency removal of 99% during the buffing step and the special formulated acid named SilTerra Cleaning Solution (SCS) provides comparable capability with ammonium hydroxide on particles and metallic in which both achieved cations and anions removal efficiency higher than 97%. The unique formulation of SCS contains hydrogen peroxide, sulphuric acid, and an additive. The chemical is proprietary of SilTerra by four inventors including the corresponding author. SCS was selected for this evaluation since it contains necessary ingredients to oxidize and dissolve the contaminants. The attempt to skip the application of chemicals during post CMP scrubbing was not promising as anions removal efficiency yielded lower than 95% removal efficiency.

Keywords: Ammoniacal Nitrogen, Environment and Post-CMP Cleaning.

1. INTRODUCTION

Ammoniacal Nitrogen is a measure for the amount of ammonia in waste products or effluents. Monitoring and reporting of ammoniacal nitrogen in wastewater effluent analysis are made mandatory under the Environmental Quality Act (Industrial Effluent) regulations 2009 [1]. The

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limit in industrial effluent is set at 10 parts per million (ppm) for Standard A and 20 ppm for Standard B. Standard A is meant for users who operate upstream of water catchments area and standard B is for facilities located downstream. However, ammoniacal nitrogen limit for semiconductor companies that started their operation before January 1st, 2010 has been set to twice of initial limit in Table 1 before full implementation of original regulation limit by 1st January 2020.

Table 1 Ammoniacal Nitrogen Limit in effluents [1]

Trade/ Industry	Industrial Effluent Limit Environmental Quality Act		
	Parameter	Standard A	Standard B
Existing & new companies (1st Jan 2020)	Ammoniacal Nitrogen	10ppm	20ppm

The use of ammonium hydroxide-based solution in wafer fabrication particularly Chemical Mechanical Planarization (CMP) has led to high ammoniacal nitrogen in the effluent. This study presents the analysis of an alternative solution that is non-ammonia based known as SilTerra Cleaning Solution (SCS) during post CMP wafers cleaning process.

2. BACKGROUND

2.1 Wafer Fabrications

An integrated circuit (ICs) is a set of electronic circuits build on this silicon wafer which can function as a microprocessor, an oscillator, an amplifier, a computer memory, a timer, and many others [2]. The process of wafer fabrication starts with a raw material called raw wafers. These wafers are divided into many identical square areas which will produce silicon chips. A wafer is a thin slice of crystalline silicon used in the wafer fabrication. A raw wafer made of silicon being used as a raw material because of its ability to form a high-quality silicon dioxide. Raw wafers shall undergo a multi-step process by adding layers for the formation of electronic circuits in a repeated and identical structure called dies. The individual microcircuits are diced from a patterned wafer and packaged for use in optical computer components, radio frequency amplifiers, components of light-emitting diode (LED) and many other electronic devices. A photo of raw silicon wafers is shown in Figure 1.

Figure 1. Raw Silicon Wafers.

As the size of ICs decreases, cleaning efficiency needs to be improved as smaller particles and contaminants may now cover part of ICs causing yield loss and potential scrap due to out of specifications. This can be translated into more stringent contamination control and better particles removal. The electronic circuits on wafers are produced after undergoing multiple microfabrication process steps such as implant, metallization, dielectric deposition, CMP, photolithography and etching. A simplified process step is shown in Figure 2.

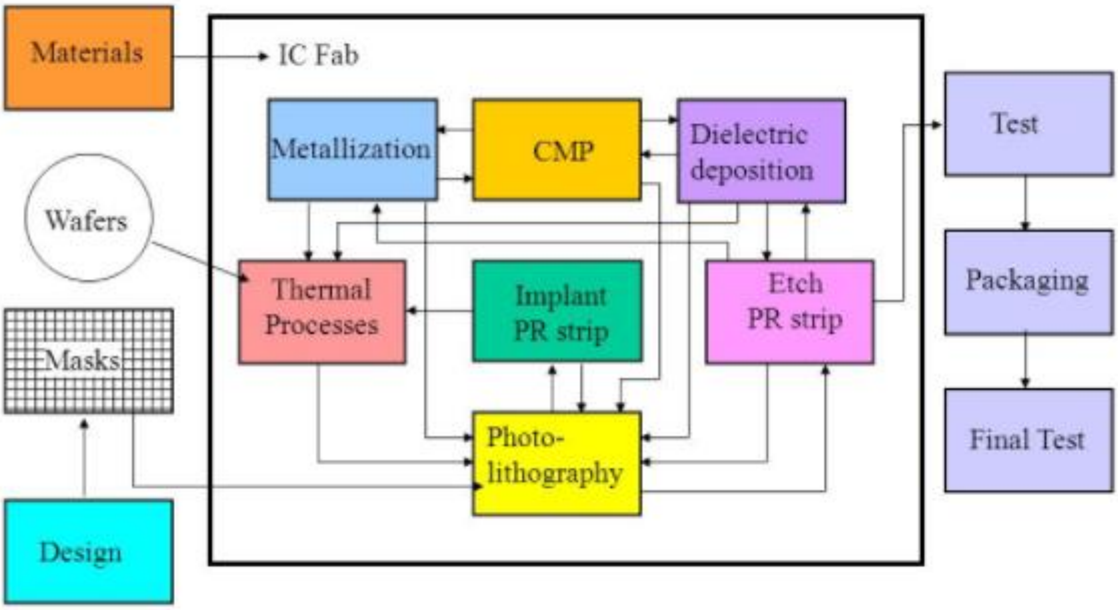


Figure 2. Process Flow in Wafer Fabrication [3].

2.2 CMP Polishing Process

Chemical Mechanical Polishing or Chemical Mechanical Planarization (CMP) is a polishing process which combines a chemical and mechanical polishing to polish and remove the dielectric layer and unwanted materials on silicon wafers. CMP has emerged as a critical step in integrated circuit fabrication in front end and back end processing [4]. In a CMP machine, a wafer is mounted on a wafer carrier and is scrubbed against a polishing pad under a load with a rotary motion in which a downward force is applied and pushing it against the pad as shown in Figure 3. Slurry, a liquid contains abrasive particles is introduced to the polishing pad made of polymeric material with a porous surface to facilitate the chemical reaction between the wafer and the slurry. Both the plate and the carrier are then rotated under the oscillation on the carrier to remove material and even out any irregular topography.

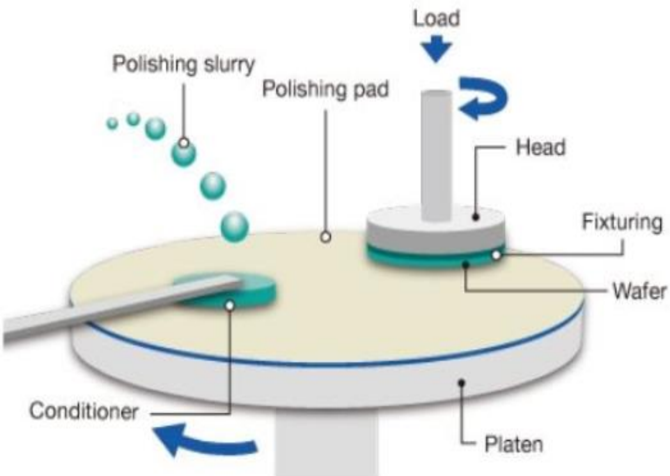


Figure 3. CMP Polishing processes schematic diagram [5].

This process involves close contact between the wafer surface and the pad with slurry. The polishing pad presses these abrasive particles to polish the wafer surface and the wafer surface is uniformly polished. The wafer is held by vacuum at the carrier to prevent unwanted particles from being deposited on the wafer surface while unloading and loading the wafer into the machine. The chemical reaction between the wafer surface and the slurry is tuned to achieve certain removal rate per the desired planarization. It is known that the material removal is caused by mechanical removal such as lateral crack, direct indentation, or micro-scratching by abrasives [6]. The reduction of structure dimensions in the ICs increases the number of transistors, hence, achieving a multilevel interconnection, thus, requires a higher degree of planarization [6]. The slurry chemistry is used to change the surface property to a softer layer in order to increase material removal rate [7]. The chemical effect was defined as the effect of changing the mechanical properties of the wafer surface through the presence of the polishing media. CMP slurry made of abrasive particles and chemical components such as polymeric additives, oxidizer, pH adjuster, passivation agent and dispersant to suit the polishing purpose of desired surface planarization [7]. Despite multiple CMP hardware and consumables, CMP slurry is considered the most influencing parameter [7]. The reaction mechanism is not clear because the slurry chemistry information is considered proprietary and not declared by the manufacturers. CMP oxide mechanism is illustrated in Figure 4.

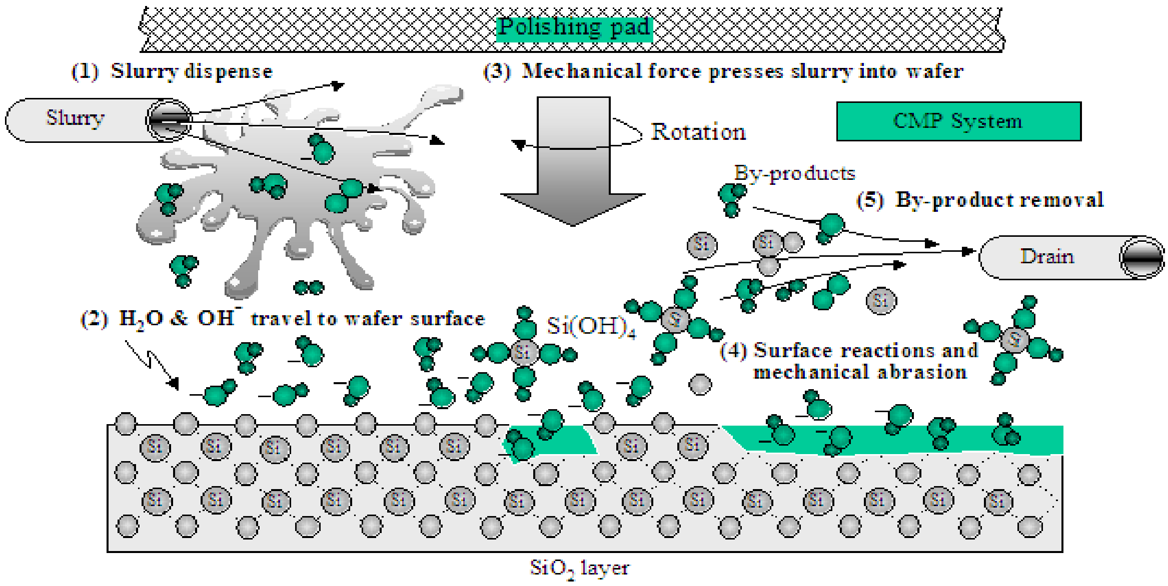


Figure 4. CMP Oxide Reaction Mechanism [8].

2.3 Post CMP Cleaning

The CMP process is a dirty step which leaves a considerable amount of residues from the removed materials. Oxide slurry is rich in metallic ions and particles; thus, post CMP steps need to be designed to remove these residues. During the CMP step, the residual particles mostly from the slurries and from the polished material. The particles either get adsorbed at the surface or embedded in the top layer due to the pressure applied by the pad. Removal of residues on wafers after CMP polishing process is crucial as these residues can cause various defects to the wafers. Kern and Puotinen developed ammonia-based chemical solutions known as Standard Clean (SC) solutions in 1965 [9]. These solutions are used as cleaning agents on silicon-based surfaces because of their volatility and low reactivity with silicon compounds [10]. There are two types of SC solutions used known as is Standard Clean 1 (SC1) and Standard Clean 2 (SC2). SC1 requires mixing of water, hydrogen peroxide and ammonium hydroxide and SC2 are prepared by combining water, hydrogen peroxide and hydrochloric acid. Ammonium hydroxide and SC solutions are commonly used in wafer fabrication for removing by creating charge repulsion [11]**Error! Reference source not found.**. The removal process of particles, organic residues and other residuals after polishing is termed as post CMP cleaning. Post-CMP cleaning used to be a simple process of rinsing with ultrapure water to remove slurry particles [12]. The post CMP cleaning is crucial in wafer technology as one of its objectives is to manufacture high-quality surfaces of fine dimensions and the cleaning methods are aggressively explored as the number of published papers increases significantly as shown in Figure 5 [8,13].

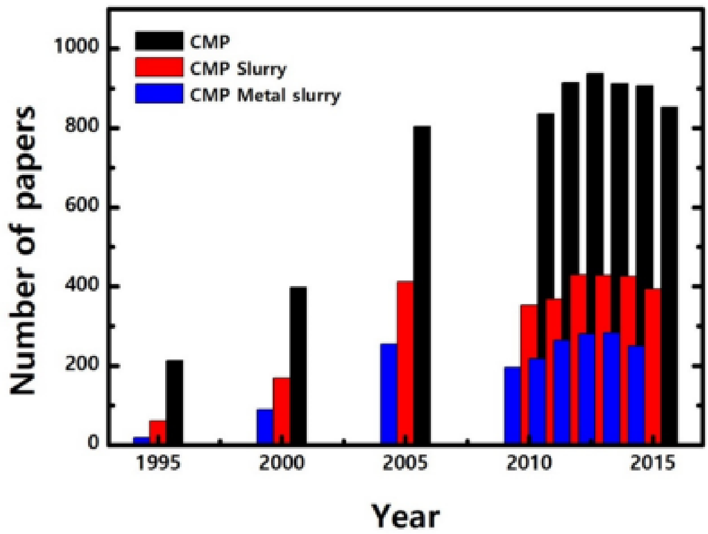


Figure 5. CMP Abrasive Market Trend in Semiconductor Industry [13].

Table 2 Comparison of few Post CMP Techniques [14]

Post CMP Cleaning	Cleaning Media	Particle removal concept	Advantages	Disadvantages	Ref
Scrubbing	Polyvinyl alcohol brush	Hydrodynamic & mechanical force	Good cleaning efficiency	▪ Particles redeposited causing contamination ▪ Scratches	[15]
Hydrodynamic jets	Pressure jets	Hydrodynamic drag force	Low cost & easy maintenance	▪ High pressure may cause structure damage	[16]
Mega-sonic acoustic	Frequency pressure wave	Megasonic power	Good cleaning efficiency	▪ High operational cost ▪ Risk of structural damage	[17]
Cryogenic cleaning	High-pressure liquid CO_2	Hydrodynamic drag force	Good cleaning efficiency	▪ High operational cost ▪ Risk of structural damage	[18]
Buffing	Buff pad	Hydrodynamic & mechanical force	Good cleaning efficiency	▪ Scratches	[19]

Apart from the listed post CMP cleaning techniques shown in Table 2, there are other new cleaning processes such as laser cleaning, hydrogen plasma and ultraviolet being developed but these processes have limitations on operating costs and throughput as these processes cannot clean a batch of wafers at once [20]. Moreover, the economic studies state that wet cleaning processes are preferable due to the low-cost operation [16]. In the recent development, the gas-phase cleaning process such as fluoride, ozone and chlorine have enabled the cleaning of certain niche applications, but this method has not displaced liquid cleaning for various reasons [21]. Buffing is one of the most commonly used cleaning processes and this direct contact cleaning is proven more effective in particles removal and produced a substantially cleaner surface [22-23]. Besides the hydrodynamic forces exerting on particles, there are other forces arising due to the direct contact of the pad leading to the removal of particles. Although high pressure is more effective for particle removal, very high pressure on the buff can damage the surface.

3. EXPERIMENTAL

3.1 CMP Oxide Polishing

SpeedFam IPEC (SFI) AvantGaard™ 776 polisher tool was used to polish wafers in this study. SFI polishers as shown in Figure 6 have a complete dry-in and dry-out CMP wafer processing by integrating the multi-platen CMP with Buff station using Politex Prima Hi pad and Ontrak DSS200 Synergy. The experiments to evaluate particle removal efficiency were carried out using twenty-five prime-grade eight-inch diameter silicon wafers (Si P-type Epi). Wafers were polished using oxide slurry with the commercial name of SS25E by the polisher and these wafers were sent for buffing process. There are two stages of post CMP cleaning process flows adopted by SpeedFam IPEC (SFI) tools called Buffing and Scrubbing. Buffing is performed at a Buff Station while the Scrubbing step is taken place at OnTrack Cleaner. Both steps are integrated together to ensure contaminants are removed effectively from polished wafers. The three steps of the CMP process using the SFI tools are shown in Figure 7.

Figure 6. SpeedFam IPEC (SFI) AvantGaard 776

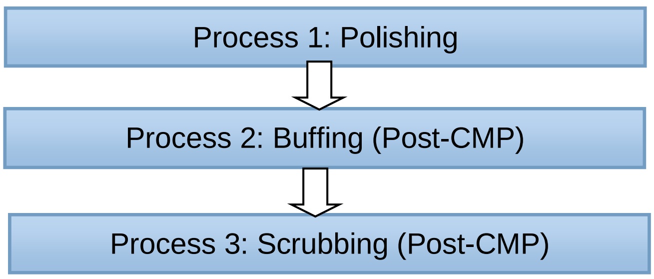


Figure 7. CMP Oxide Process Steps at the SFI Tools.

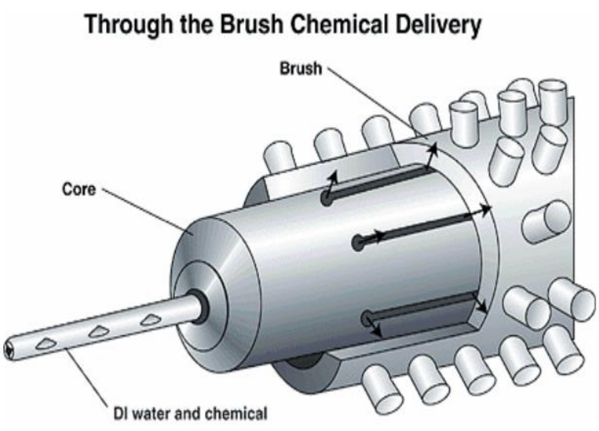
3.2 Post CMP - Buffing

In CMP Oxide process, wafers were polished with Oxide slurry before undergoing the first stage of post-CMP cleaning named Buffing in which the wafers' surfaces come in direct contact with a soft PVA (Polyvinyl Alcohol) pad and Tungsten slurry to remove contaminants by rotational torque when spinning takes place. Tungsten slurry was pre-blended with hydrogen peroxide at a fixed concentration for the buffing process. Photo of the Buff station is shown in Figure 8.

Figure 8. Schematic of Two Buffing Station.

3.3 Post-CMP - Scrubbing

In SFI tools, the wafers moved to Scrubbing step using On-Track Cleaner upon completion of Buffing step. Scrubbing has a pair of roll brushes positioned on bottom and top of the wafer that rotates with a continuous flow of ultrapure water and diluted ammonium hydroxide solution at a concentration of between 1% to 2% used for rinsing [24]. Figure 9 shows the solutions flow path through the brush during Scrubbing stage. In evaluating cleaning efficiency by the Scrubbing step, wafers after polishing step were brought to the Scrubbing process using the OnTrack Cleaner without Buffing process.



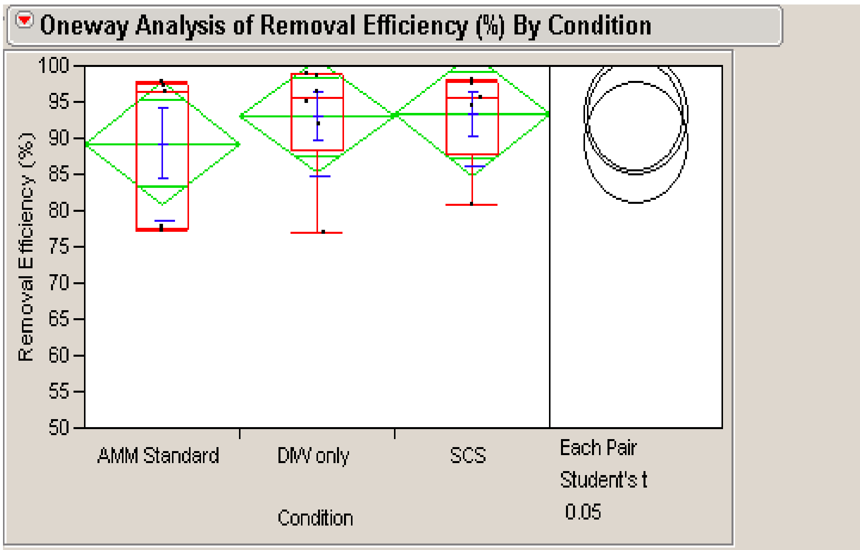
leaner.

A double-sided brushes scrubber made by DSTec were used to scrub the wafers at a prescribed speed and pressure. During Scrubbing, wafers were scrubbed by rinsing using Ultra Pure Water (UPW) without any chemicals in order to understand the contribution of chemical in the post-CMP cleaning process. Next, the effectiveness of Scrubbing being evaluated with SCS and ammonium hydroxide along with UPW rinse. Ammonium hydroxide solution was considered as the standard recipe used during post-CMP cleaning. The number of particles on wafers was then counted using Tencor Surfscan SP1 Tbi. Rigaku TXRF3750 X-ray spectrometer was used to analyse impurities removal efficiency.

4. RESULTS AND DISCUSSION

4.1 Particle Removal Efficiency for Buffing & Cleaning Steps

Particles cleaning efficiency was investigated by measuring particles before and after buffing termed as adders. In normal qualification practice, the acceptable number of adders is less than 40 and the lower adders quantity is preferable. The number of particles on wafers was measured before the experiments with all the wafers recorded >100 counts. Evaluations of particle removal efficiency at Cleaning step were carried out using three different media solution: UPW, diluted ammonium hydroxide solution and diluted SCS. The one-way ANOVA test was carried out and the test result is shown in Figure 10.

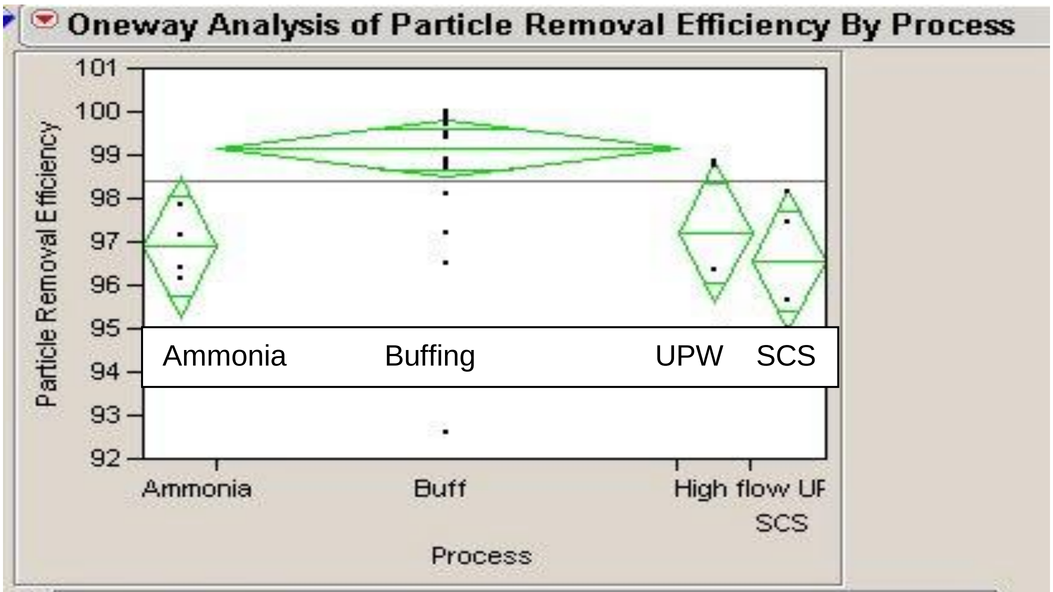


Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
AMM Standard	5	89.2865	10.7525	4.8087	75.935	102.64
DIW only	6	92.9825	8.2045	3.3495	84.372	101.59
SCS	5	93.3196	7.0682	3.1610	84.543	102.10

Figure 10. ANOVA - Particles Removal by Scrubbing (3 Solutions).

One-way ANOVA with 95% confidence intervals being chosen while comparing the individual means obtained from the experiment. Injection of diluted ammonia was replaced with De-ionized Water (DIW) or also known as Ultra Pure Water (UPW) and Silterra Cleaning Solution (SCS), one solution at a time during scrubbing. Even though SCS recorded highest average particles removal efficiency of 93.3%, followed by UPW/DIW at 93% and lowest of 89.3% by diluted ammonia, the t-test circles generated by the one-way analysis of variance (ANOVA) indicated that particles removal efficiency for all these solutions were comparable. The means for all the three conditions were non-distinctive since the circles overlap with one another. In short, all three solutions delivered a comparable particle removal efficiency during the scrubbing process. Particle removal efficiency for all these solutions was comparable at an average removal rate between 96% to 97%. Experiments of particle removal efficiency at Scrubbing step were then performed and compared using the one-way ANOVA test with 95% confidence intervals per Figure 11.



Oneway Anova

Summary of Fit

Rsquare	0.330333
Adj Rsquare	0.269454
Root Mean Square Error	1.597472
Mean of Response	98.40541
Observations (or Sum Wgts)	37

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Process	3	41.54062	13.8469	5.4261	0.0038
Error	33	84.21330	2.5519		
C. Total	36	125.75392			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Ammonia	4	96.8850	0.79874	95.260	98.510
Buff	25	99.1320	0.31949	98.482	99.782
High flow UPW	4	97.2200	0.79874	95.595	98.845
SCS	4	96.5700	0.79874	94.945	98.195

Figure 11. Comparison of Buffing & Scrubbing Particle Removal Efficiency.

It was found that 99% of particles were removed based on the difference between particles count before and after the process. The superiority of Buffing technique in particles removal can be seen from the ANOVA test in Figure 11. A p-value was calculated for an F-distribution and a p-value of 0.0036 was obtained for the above experiment. The p-value of lower than the 0.05 indicating that Buffing was more superior in the particle removal with an efficiency of 99% and distinctively higher compared to scrubbing with any of the solutions. Based on the test, buffing technique has a distinctive higher particle removal capability compared to the Scrubbing step. Nevertheless, all four experiments delivered particles adders of less than 45, meeting minimum process requirement.

4.2 Metallic and Ions Removal Efficiency at Cleaning Step

Metallic residuals on wafer surface present as adsorbed ions, oxides, hydroxides and salts from polishing activity, consumables, and environment itself [25-27]. Experiments on impurities removal efficiency were analysed on wafers' surfaces by X-ray spectrometer, TXRF3750 and results are shown in Table 3, Table 4 and Table 5.

Table 3 Pre and Post Cations Test Results after Scrubbing with UPW

Elements	Wafer 1		Delta	Removal	Wafer 2		Delta	Removal
(ppm)	Pre	Post		%	Pre	Post		%
Iron (Fe)	343	4	339	98.9	617	2	615	99.8
Potassium (K)	274	5	339	98.3	290	0	290	100
Calcium (Ca)	1614	3	1611	99.8	524	2	522	99.6
Titanium (Ti)	1854	87	1767	95.3	866	0	866	100

Table 4 Pre and Post Cations Test Results after Scrubbing with SCS

Elements	Wafer 1		Delta	Removal	Wafer 2		Delta	Removal
(ppm)	Pre	Post		%	Pre	Post		%
Iron (Fe)	221	2	219	99.1	185	3	182	98.6
Potassium (K)	314	0	314	100	275	3	272	98.9
Calcium (Ca)	3986	2	3984	99.9	1376	41	1335	97.0
Titanium (Ti)	1404	0	1404	100	631	11	620	98.2

Table 5 Pre and Post Anions Test Results after Scrubbing with Ammonia

Elements	Wafer 1		Delta	Removal	Wafer 2		Delta	Removal
(ppm)	Pre	Post		%	Pre	Post		%
Iron (Fe)	221	2	219	99.1	185	3	182	98.6
Potassium (K)	314	0	314	100	275	3	272	98.9
Calcium (Ca)	3986	2	3984	99.9	1376	41	1335	97.0
Titanium (Ti)	1404	0	1404	100	631	11	620	98.2

Wafers were poured with oxide slurry and kept until the wafer surfaces became dry. Only two wafers were tested for Scrubber cleaning using each of the three solutions. Results of cations residual from TXRF indicated no significant difference in Potassium, Iron, Calcium and Titanium ions removal when ammonia, SCS and UPW were used as a media during the scrubbing cleaning. However, ammonium hydroxide and SCS solution yielded better removals of Chlorine and Sulphur anions as shown in Figure 12. The removal percentage are tabulated in Table 6, Table 7 and Table 8.

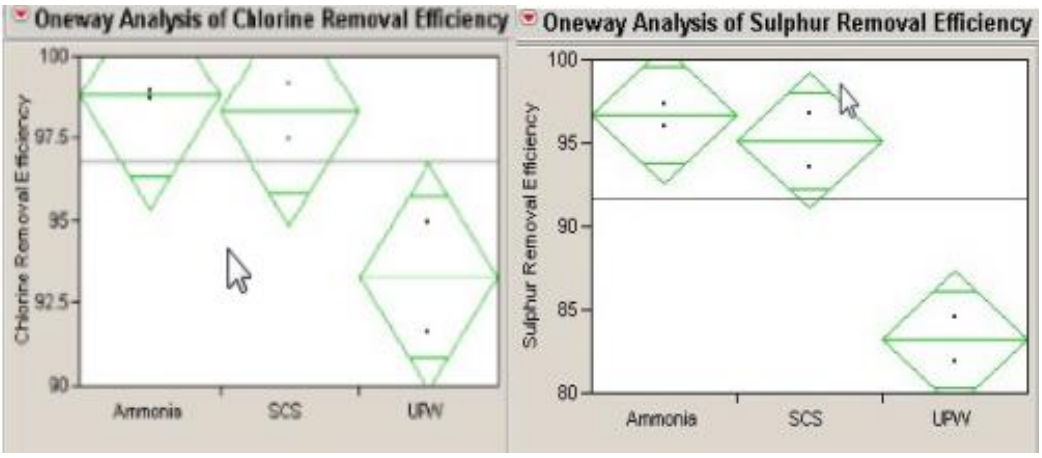


Figure 12. ANOVA Anions Test Results after Scrubbing.

Table 6 Pre and Post Anions Test Results after Scrubbing with UPW

Elements (ppm)	Wafer 1 Pre	Wafer 1 Post	Delta	Removal (%)	Wafer 2 Pre	Wafer 2 Post	Delta	Removal (%)
Chlorine (Cl)	306	26	280	91.6	576	29	546	94.9
Sulphur (S)	279	51	228	81.8	279	43	236	84.5

Table 7 Pre and Post Anions Test Results after Scrubbing with SCS

Elements (ppm)	Wafer 1 Pre	Wafer 1 Post	Delta	Removal (%)	Wafer 2 Pre	Wafer 2 Post	Delta	Removal (%)
Chlorine (Cl)	1231	10	1221	99.1	1207	31	1177	97.4
Sulphur (S)	849	55	794	93.5	1508	48	1460	96.8

Table 8 Pre and Post Anions Test Results after Scrubbing with Ammonia

Elements (ppm)	Wafer 1 Pre	Wafer 1 Post	Delta	Removal %	Wafer 2 Pre	Wafer 2 Post	Delta	Removal (%)
Chlorine (Cl)	348	6	342	98.3	266	4	262	98.5
Sulphur (S)	631	20	611	96.8	375	10	365	97.3

The solution formulated by SilTerra named as SCS is acidic in nature and contains a combination of sulphuric acid (H_2SO_4), hydrogen peroxide (H_2O_2) and an additive. The chemical was selected for this evaluation since it contains necessary ingredients to oxidize and dissolve the contaminants per working principles shown in Figure 13 under the oxidation and dissolution mechanism.

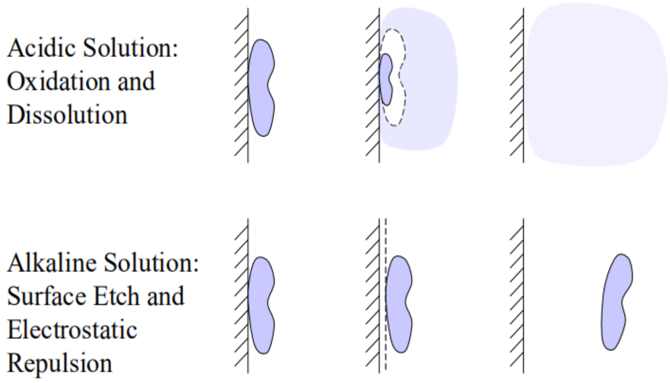


Figure 13. Illustration of Contaminants Removal by Acidic and Alkaline Solution [8].

The creation of electrostatic repulsion by diluted ammonium hydroxide creates similar charges surrounding the contaminants and wafer surfaces resulting in these two surfaces repel from one another. Some studies confirmed that ingredients in SCS; sulphuric acid and hydrogen peroxide are excellent agents for removing surfactants, particles, metallic contaminants from wafer surface [28-30]. However, the combination of these three chemicals for application at post CMP cleaning is unique and new. Based on these findings, the formulated SCS mixture can deliver comparable cleaning efficiency with ammonium hydroxide. Overall, SCS is consistently comparable with ammonium hydroxide in the removal of all the mobile ions. The findings on ions removal are consistent with the suggestion made in the literature that the removal of mobile ions is effectively done by means of chemicals. Even though it is generally known that mobile ions residuals may cause damage to the devices but the allowable tolerance and impact from these residual anions are not clear for post CMP stage. This requires further study and risk analysis since chemicals free processing at post CMP scrubbing process has many other benefits associated with the environment, handling, and operational cost. At the moment, the alternative cleaning utilizing Megasonic energy is becoming a common method, but this technique still employs chemistry during the cleaning process [31].

5. CONCLUSION

The application of ammonium hydroxide during Scrubbing is a subject of concern upon the incorporation of ammoniacal nitrogen into Environmental Quality (Industrial) Regulations 2009. Various post-CMP cleaning chemistries employed elsewhere but solutions used are mostly ammonia-based mixtures. The buffing process delivers an excellent particle removal whereas Scrubbing act as a back up to rinse any leftover particles particularly metallic contaminants on the wafer. Combination of existing Buffing step and Cleaning using the ammonia-free solution, SCS, able to meet both particles and metallic removal efficiency. This will lead to free ammoniacal nitrogen effluent from CMP Oxide tools process. The capability of SCS in cleaning and its potentials in replacing ammonium hydroxide is undeniable. An effort to eliminate the use of the chemical by relying on ultra-pure water (UPW) has resulted in incomplete removal of residues. Even though it is generally known that mobile ions especially metallic residuals or cations may cause damage to the circuits but the allowable limit of mobile anions after post-CMP processed wafers are not discussed in detailed. This requires further evaluation since chemicals free processing at post CMP scrubbing process is highly desirable due to other benefits associated with the environment, handling, and operational cost.

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