RUTHENIUM CAPPING LAYER PRESERVATION FOR 100X CLEAN THROUGH PH DRIVEN EFFECTS

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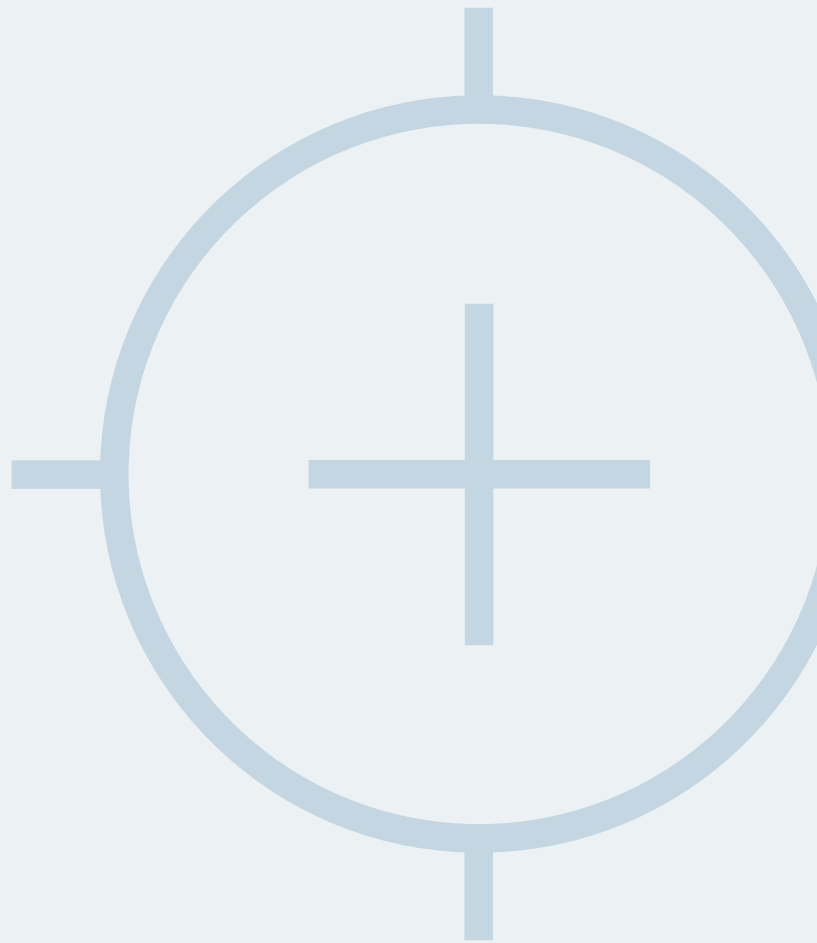
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In the absence of pellicle a EUVL reticle is expected to withstand up to 100x cleaning cycles. Surface damage upon wet and dry cleaning methods has been investigated and reported in recent years [1]. Thermal stress, direct photochemical oxidation and underlying Silicon layer oxidation are reported as the most relevant root-causes for metal damage and peeling off [2,3]. An investigation of final clean performance is here reported as a function of operating pH; the results show increased Ruthenium durability in moderately alkaline environment. The electrochemical rationale and the dependency of the reducing strength of the media with the pH will be presented as possible explanations for reduced damage.
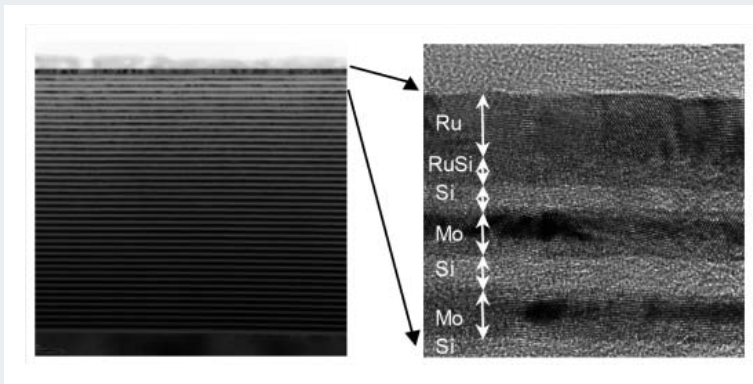
Figure 1 *EUV reticle schematic: Molybdenum and Silicon layers alternate to form the reflector. Ideal reflectivity is found at 40 pairs of Mo/Si layers. 2-3 nm layer of Ruthenium is required to protect Silicon from oxidation and damage*

INTRODUCTION

EUV technology uses light reflected from the photomask surface rather than light transmitted through the substrate, which changes the photomask nature for the imaging process onto the wafer-level from transmissive to reflective. So far, the best EUV reflector design known for lithographic purposes at 13.5 nm is based on the stacking multilayer concept. Schematic representation of the EUV reflector scheme and the TEM image are shown in Figure 1 [4].

Silicon is easily oxidized into silicon dioxide, thus, the EUV reticle required a protective capping layer. This is currently accomplished by a 2-3 nm layer of metallic Ruthenium; this is chosen because of its high transmission in the extreme ultra violet wavelength range and quite high resistance toward corrosive conditions [5,6].

Presently, no pellicles are available for EUV masks to protect the pattern side from contamination during storage, use or transport. This implies that EUV masks are more exposed to contamination than optical masks and thus it is expected that EUVL masks need to undergo more cleaning cycles during their useful life in order to maintain high device production yields. With increasing cleaning cycles mask defectivity remains one of the obstacles to commercial viability [7,8]. Key to overcome this is the development of a mask clean process that is effective for defect removal and preserves the integrity of the mask surface. Damage to the Ru capping surface degrades EUV reflectivity which can lead to critical dimension (CD) shift and non-uniformity [9].

Therefore, it is very crucial to understand the effect of cleaning and exposure processes on EUV mask quality and printing performance.

A full cleaning cycle includes a surface preparation step followed by photoresist removal, particle and ion removal, and final clean. It has been shown that Ruthenium damage occurs mostly during the final clean step, where the metal layer is directly exposed to cleaning chemistry [10].

In this paper we report a new chemical approach for final clean which preserves the Ruthenium durability up to more than 100 cleaning cycles. Possible chemical explanations for metal preservation will be proposed.

BACKGROUND

Previously, SUSS MicroTec demonstrated techniques for organic removal, surface preparation, residual ion removal and final clean without surface damage on the 193i masks [11,12,13]. These new techniques are based on POU UV exposure of the wet cleaning chemistry and the mask surface simultaneously. Recently, the major root-causes for Ruthenium capping layer damage have been extensively reviewed and several mechanisms for metal damage have been proposed; two major root causes for capping layer degradation have been individuated:

1) Direct or in-direct metal oxidation [10]
2) Silicon dioxide formation underneath the Ruthenium layer, leading to metal peeling off the surface [3]

Ruthenium oxidation can occur during exposure of the metal layer to UV-light and oxidizing agents:

Ru (metallic) + UV-light + Oxidizing agent ==> Ru (oxidized)
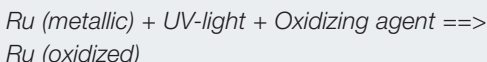
Commonly used UV-light source are the two main mercury emissions at 185 and 254 nm; oxidizing agents are formed in-situ through the photolysis of absorbing media such as molecular Oxygen, Ozone or Hydrogen Peroxide.

Recently, an additional mechanism for Ruthenium peeling off has been proposed; Oxygen, in the form of molecule or atom (as radical) can inter-diffuse into the capping layer, as shown in Figure 2.
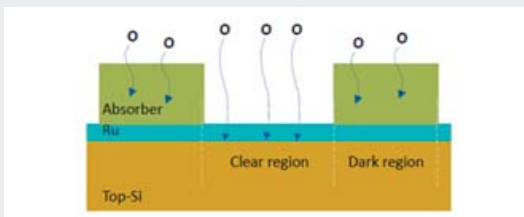

Figure 2 Oxygen inter-diffusion through Ruthenium layer into the under-laying Silicon layer [3]

Under the absorber features Oxygen inter diffusion is quite difficult to occur; however, in clear regions, Oxygen can diffuse through the 2 nm thick metal layer and get in contact with the first Silicon layer. This, in turn, leads to oxidation as this is very favorable thermodynamic process for elemental silicon. Amorphous silicon dioxide has a higher volume than elemental Silicon, thus, the expansion of the underling Silicon layer leads to Ruthenium peeling off (Figure 3).
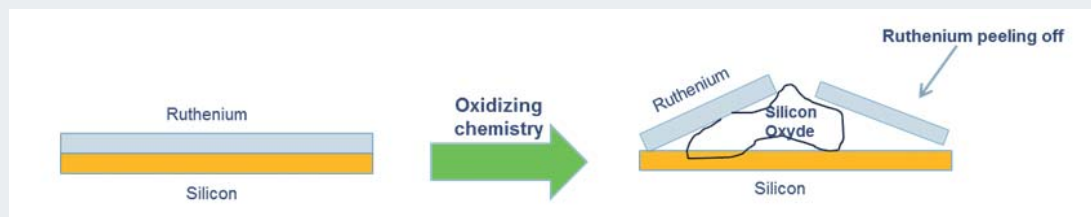

Figure 3 Silicon dioxide formation with resulting volume increase and Ruthenium peeling-off
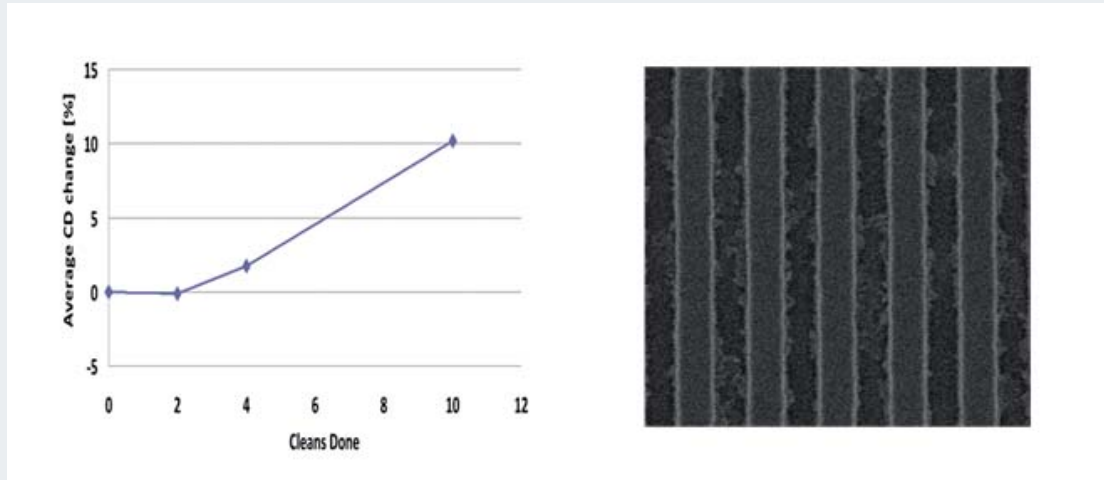
Figure 4 *Average relative CD change in printed CD on wafer-level after cleaning the mask in total for 10 times; a majority of these changes occurs between 4th and 10th cleaning process of the EUV mask (left); evidence of Ruthenium peeling at the interface between absorber and capping layer (right)* [15]

Previous reports showed how EUV reticles can undergo capping layer damage after only a few cleaning cycles; Figure 4 shows a published result from 2011, where SEM surface image documents the Ruthenium damage in the form of massive peeling around the absorber features.

The damage becomes even more evident at the interface between absorber and Ruthenium capping layer. This suggested that thermal stress could also contribute to the damage. High pressure Mercury lamps used during in-situ UV process have residual emission in the infrared spectrum, which could in turn heat the mask materials leading to stress and peeling off [2].
Furthermore, it has been observed that the damage depends on mask vendor origin. This indicates a strong influence of the mask manufacturing process on surface durability.
Especially the absorber etch process has been reported to play a major role in the stability of the Ru layer during cleaning [14].

EXPERIMENTAL

Process parameters
All the tests were performed using the SUSS SMT PE MaskTrack*PRO* (MT*PRO*) mask cleaning tool. The process parameters were automatically monitored and controlled with a standard recipe programmed on the MT*PRO* tool. DI water used for the tests was de-gassed before it was supplied to the cleaning chemical distribution system. Chemicals and gases were added into the de-gassed water to prepare the respective cleaning media. The cleaning media tested are: Chemical A ($4 \leq pH \leq 6$), Chemical B ($6 \leq pH \leq 8$) and Chemical C ($10 \leq pH \leq 12$).
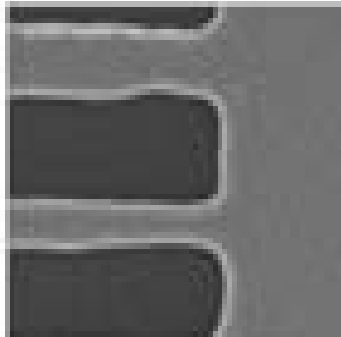
Characterization
To evaluate Ruthenium damage SEM pictures were taken at specific mask locations. TEM was used to asses Ruthenium integrity after 100 cleaning cycles.
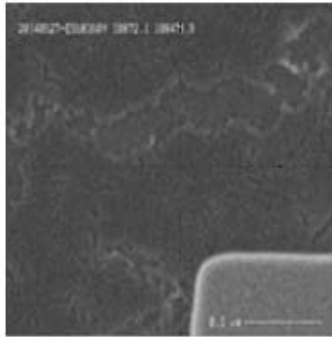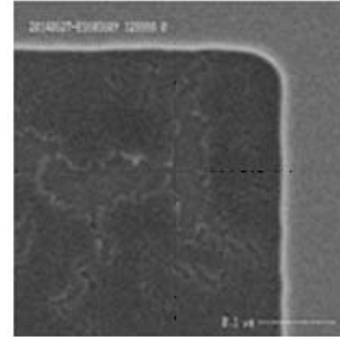
| TOP CORNER | MASK CENTER | BOTTOM CORNER |

Figure 5 *SEM image of Chemical A treated EUV test pattern: Ruthenium capping layer peeling-off was observed in the top and bottom mask corners (left and right images). No damage was observed for the central region of the EUV mask*

EXPERIMENTS PERFORMED, RESULTS AND DISCUSSION

Repeated final clean cycles, up to 100 times, have been performed by using in-situ UV technology. A water solution at pH comprised between 4 and 6 was used as baseline (Chemical A). The organic removal mechanism has been reported as direct Carbon activation through absorption of light followed by reaction with molecular Oxygen leading to consecutive oxidation steps; the final decomposition products of this photochemical reaction are CO_2 and water [2]. Ozone formation through media absorption at 185 nm, followed by photolysis into Hydroxyl radicals, also contributes to hydrocarbon decomposition. Figure 5 shows SEM image at different surface locations of Chemical A ($4 \leq pH \leq 6$) treated EUV test reticle after 100 cleaning cycles.

Massive peeling off was observed for Ruthenium at the top and bottom areas of the test EUV mask. Only the central area of the mask resulted in undamaged capping layer; this observation demonstrates once more that the absorber etching parameters play a major role in the surface integrity upon repetitive cleaning cycles [15].

Electrochemical motivation

As explained, one major root-cause for Ruthenium damage is direct metal oxidation [10]. The 100 X clean experiment was conducted in water solution of Chemical A, which had a measured pH comprised between 4 and 6. A review of the electrochemical behavior of possible Ruthenium states (metallic ruthenium or ruthenium oxides) is required to individuate the best media environment for reduction or elimination of the damage. Figure 6 shows the Pourbaix diagram for Ruthenium.
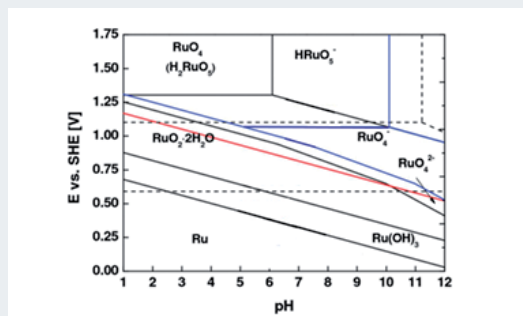

Figure 6 *Pourbaix diagram for Ruthenium. Electrochemical potential for ruthenium and ruthenium oxides is decreasing with increasing pH* [16]

Pourbaix diagrams are a schematic representation of the electrochemical potential (i.e., the amount of energy needed to get oxidation) respect to pH. These diagrams are strictly holding in solution phase, but can provide a fair estimation of the oxidation behavior of the possible involved ruthenium oxides (and metallic ruthenium). At higher electrochemical potentials, i.e., in highly oxidizing conditions, RuO_4 is expected to be formed. This oxide is extremely volatile (boiling point 40°C), thus leading to surface etching upon its formation. The Pourbaix diagram shows how at more alkaline pH values the system is in a reducing environment, with more stable lower oxidation state oxides or hydroxides such as RuO_2 or $Ru(OH)_3$ [16]. Based on this scenario we decided to investigate the Ruthenium damage as a function of operating pH. A shift at more alkaline pH was then decided to check if direct metal oxidation has a major role in the damage mechanism. To further motivate the choice of shifting the pH at more alkaline pH we reviewed the electrochemical behavior of different Ruthenium oxides at alkaline pH values along with electrochemical properties of aqueous alkaline media. Literature reports the ability of Hydroxide ion to be a good one electron reducing agent [17]:

$$OH^- + M ==> OH^\cdot + M^- \quad (I)$$
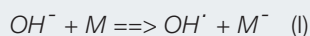
Equation (I) shows the one electron transfer to a generic metal center, with production of reduced M^- and $OH^\cdot$. As confirmation of the reducing power of Hydroxyl ions, the literature reports how RuO_4 is reduced into more stable RuO_4^- and RuO_4^{2-} at alkaline pH [18]:

$$RuO_4 + 2OH^- ==> RuO_4^{--} + 2OH^\cdot \quad (II)$$

Equation (II) shows Ruthenium tetra oxide reduction by means of two mono-electron reducing events.

With emitting light sources at 185 nm Hydroxyl radicals can be formed (by light absorption from water and Oxygen, leading to atomic oxygen which in turn reacts with water to lead to $OH^\cdot$); also, equations (I) and (II) show as the reaction byproduct of metal reduction in alkaline media is Hydroxyl radical; however, literature reports that Hydroxyl radical is partially converted into $O^{\cdot-}$ radicals in alkaline media:

$$OH^\cdot ==> O^{\cdot-} + H^+ \quad (III)$$

This can be written as a simple reaction between $OH^\cdot$ and OH^-:

$$OH^\cdot + OH^- ==> O^{\cdot-} + H_2O \quad (IV)$$
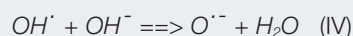
This conversion is quantitative for pH>12 and partial at pH = 10÷12. $O^{\cdot-}$ can be exposed to the same reactions as $OH^\cdot$, however its negative charge reduce its electrophilic character, i.e., in the presence of a metal center the one electron oxidation process is less favorable respect to $OH^\cdot$ [19]. As for organic removal, where the mechanism is mostly by Hydrogen abstraction, their behavior is very similar [20]. This shows how conversion of OH radicals into $O^{\cdot-}$ radicals prevents metal oxidation. In presence of light source emitting below 200 nm, Ozone is likely to be formed, due to UV-light absorption from dissolved molecular Oxygen; Ozone will be readily converted into Hydroxyl radicals, however, if the conversion is not quantitative, some un-decomposed Ozone could still be in solution. Molecular Ozone is reported to be cause of surface oxidation for EUV mask cleaning [21]. In alkaline conditions, however, Ozone is readily converted to Hydroxyl radicals [22]:

$$2O_3 + OH^- ==> OH^\cdot + 3O_2$$
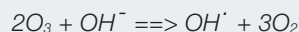
This may further minimize the risk of direct surface oxidation.

Our literature overview of the electrochemical properties leads to the following conclusions:

1) Alkaline pH values represent a reducing condition (lower electrochemical potentials for Ru oxides; Figure 6) [17]

2) OH⁻ as such, can act as reducing agent; RuO_4 that could arise from direct metal oxidation can be efficiently reduced back to RuO_4^{2-} (equations I and II) [18]

3) Hydroxyl radicals (arising from 185 nm exposure or by-product of reduction with OH⁻ shown in equations I and II) are partially converted into less oxidizing $O^{\cdot-}$ species. $O^{\cdot-}$ are less aggressive toward metal centers, and equally efficient toward organic degradation [19,20]

4) Any generated Ozone is readily converted into Hydroxyl radicals and then into less oxidizing $O^{\cdot-}$ ions, thus minimizing risk of surface damage [21]

The above described four conclusions, in combination with experimental results from Chemical A treatment during final clean, which were conducted at acid pH (between 4 and 6), directed us to decide to run extensive (up to 100 cleaning cycles) at increased pH. We planned to run experiments at two different pH values, such as:

a) Chemical B, $6 \leq pH \leq 8$
b) Dissolved strong base at high dilution, Chemical C, $10 \leq pH \leq 12$

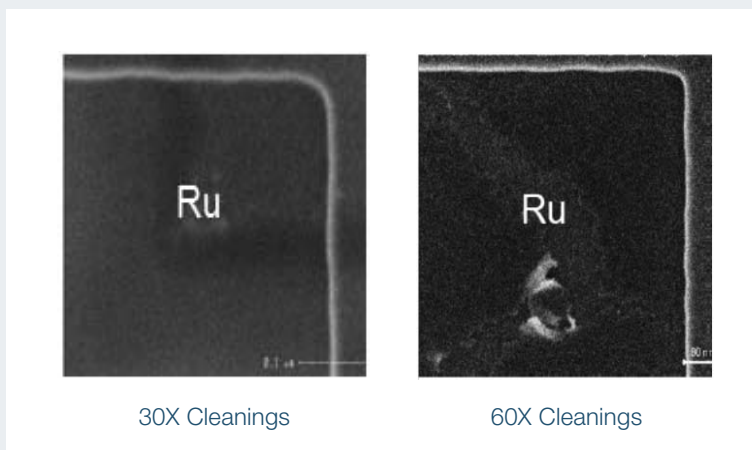| 30X Cleanings | 60X Cleanings |

Figure 7 SEM images taken at different mask locations after cleaning of EUV test reticle at pH comprised between 6 and 7 (Chemical B) Left: after 30X cleaning cycles no Ruthenium damage is found. Right image: after 60X cleaning cycles, peeling off of metal layer was observed

Chemical B, pH comprised between 6 and 8. Results.

Given the results collected by using Chemical A as cleaning media, we thought to use Chemical B at pH comprised between 6 and 8 as intermediate experiment to check if a slight increase in pH would have led to any improvement in surface integrity. A test pattern EUV mask was cleaned multiple times in Chemical B and AFM inspection followed every 30 cleaning cycles. Figure 7 shows SEM images collected after 30 and 60 cleaning cycles:

After 30 cleaning cycles in Chemical B no Ruthenium damage was found, whereas at 60 cleaning cycles peeling off of the metal layer was observed. The experiment was not brought up to 100 cleaning cycles because a visible damage was already present after 60 cycles.

Chemical C; molecular motivation.

In section *"Electrochemical motivation"* we have provided motivations for choosing alkaline chemistry during final clean of EUV mask.

After having performed experiments at pH comprised between 6 and 7 we wanted to bring the pH to more alkaline values, such as between 10 and 12. Higher pH values (>12), although favorable in terms of oxidation chemistry, would have high risk of metal dissolution through the formation of soluble hydroxides. pH driven effects could not be the only ones playing a role in surface preservation. In section *"Background"* we have reported the current root-causes for Ruthenium peeling, and mentioned a direct oxidation and Oxygen inter-diffusion as most relevant root-causes. The alkaline pH could cover the direct oxidation root-cause; however, Oxygen inter-diffusion has little to do with pH. To address this equally relevant root-cause for Ruthenium damage we have made molecular and reactivity considerations on choosing the appropriate base. As a matter of fact, the molecular structure of the used base could play an important role in surface integrity.

Literature reports a strong molecular size dependent effect happening during alkaline silicon and silicon dioxide etching; the bigger is the positive counter-ion used, the slower is the etch rate into the silicon, because bigger positive ions electrostatically bond on the silicon surface and prevent further diffusion into the bulk silicon (blocking effect) [23]. Ammonia is very often used as a base during semiconductor cleaning; however the small size of the NH_4^+ cation represent a high risk for oxygen inter-diffusion through the Ruthenium capping layer. For this reason, Chemical C was chosen to be:

- A strong base
- A base with molecular structure such that the positive counter ion is much bigger comparing to NH_4^+ ion

Both Ammonia and Chemical C bases give in solution Hydroxide ions, thus both bases could be used for pH adjustment. However, the (positive) counter ion dimensions are quite different, with the B^+ ion being substantially bigger than NH_4^+ ion.

For these explained reasons we have chosen to conduct our experiment at pH comprised between 10 and 12 by using as a base such BOH. BOH was also chosen to be a non-coordinating agent. Coordinating agents (i.e., compounds capable to establish a direct bond with metal center through available external electrons) can in fact promote metal dissolution though the formation of soluble complexes; Ammonia is a good coordinating agent, whereas BOH is not.

To avoid possible interaction with cleaning UV mechanism (i.e., direct absorption of Hydrocarbons at 254 nm) we verified that absorption at 254 nm of chemical A, Chemical B and chemical C was very low. Negligible absorption at 254 nm is measured for the three used media, thus direct absorption of Hydrocarbons at 254 nm is not influenced by media absorption. Residual absorption of BOH at 185 nm could be expected, however massive photolysis can be excluded due to the very low BOH concentration in solution.

Chemical C, pH comprised between 10 and 12. Results.

Extremely diluted BOH solutions ($10 \leq pH \leq 12$) were used to run cleaning cycles and evaluate surface integrity afterword. Figure 8 shows SEM images of EUV test pattern after 30, 60 and 100 cleaning cycles.

AFM images revealed that after 30, 60, and 100 cleaning cycles Ruthenium peeling off was not observed; only in a single spot, an increase in surface roughness was observed.

SEM IMAGES AFTER 30 CLEANING CYCLES
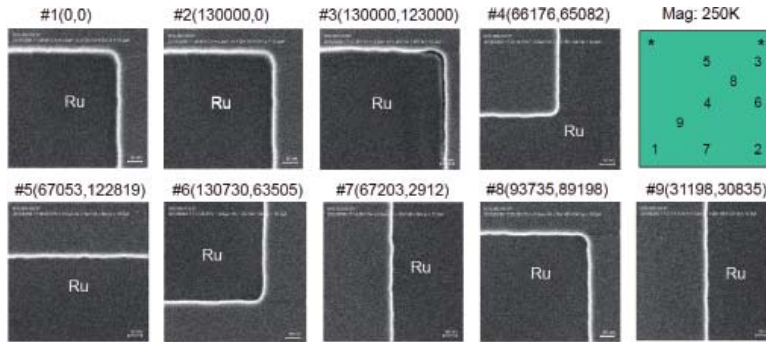
SEM IMAGES AFTER 60 CLEANING CYCLES
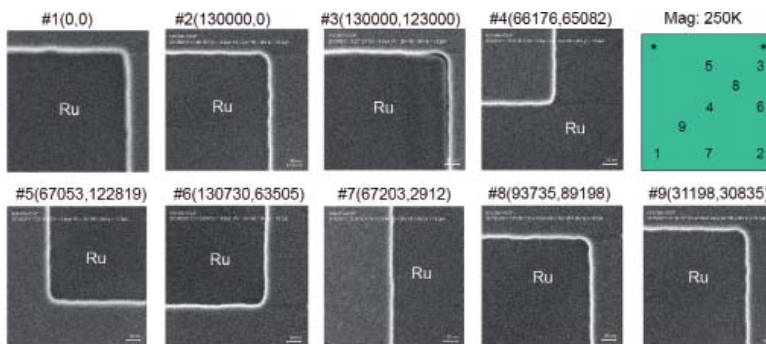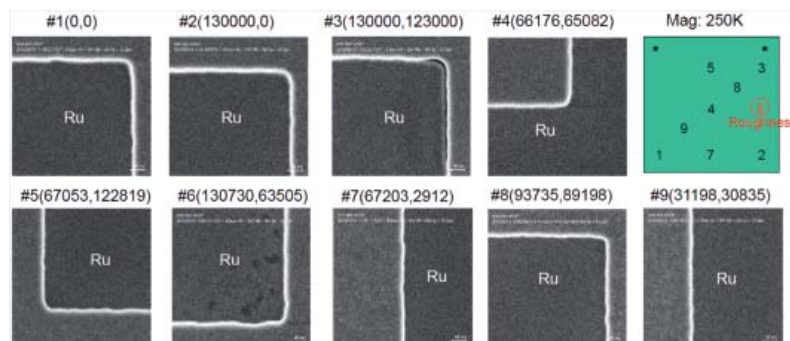
SEM IMAGES AFTER 100 CLEANING CYCLES

To asses Ruthenium integrity, TEM analysis was performed on Ruthenium capping layer; Figure 9 shows TEM results.
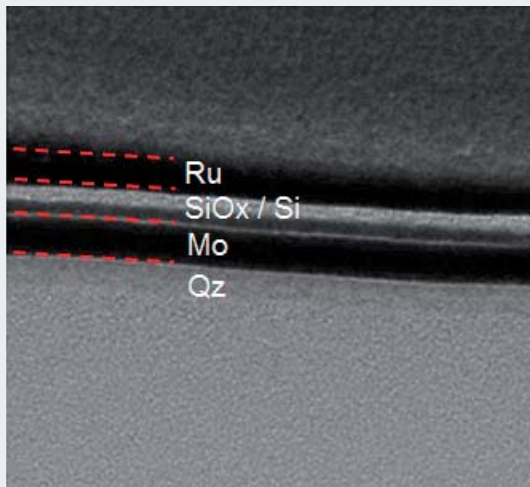

Figure 9 *TEM image of Ruthenium capping layer of EUV test pattern reticle exposed to in-situ UV final clean for 100 cleaning cycles with alkaline (10 ≤ pH ≤ 12) chemistry. The image shows uniform Ruthenium layer without damage; this is on top of a single Si/Mo layer; also the underlying silicon does not increase its volume, ruling out massive Silicon oxidation*

The SEM image shows preserved Ruthenium integrity; the metal layer looks uniform and without damage. A mono-layer of Si/Mo has been used for this test EUV mask; the Silicon layer appears undamaged and uniform through the whole scanned area. Massive Silicon oxidation can be excluded because no increased volume has been observed.

CONCLUSIONS

EUV masks are more likely to be cleaned due to the lack of pellicle respect to traditional 193 nm Mask. Particle deposition and carbon contamination can occur in several points in time of the mask lifetime, like during handling and exposure steps; along with these degrading effects Ruthenium damage is often observed in the form of volatile RuO_4 formation. In this paper, the latest proposed root-causes for Ruthenium damage have been recalled; direct metal layer oxidation, and underlying silicon dioxide formation are presented as major root-causes for capping layer peeling. A review of the electrochemical properties of Ruthenium oxides shows how alkaline pH place the metal layer into reducing conditions, thus optimal to decrease risk of direct Ruthenium oxidation. The ability of Hydroxyl ions to act as reducing agent is also recalled; furthermore, hydroxyl radicals convert into less oxidizing $O^{\cdot-}$ species in alkaline media. Any undissociated molecular Ozone is also rapidly converted into Hydroxyl radicals. These theoretical considerations were tested by running sequential cleaning experiments at increasing pH (Chemical A, 4 ≤ pH ≤ 6; Chemical B, 6 ≤ pH ≤ 8; Chemical C, 10 ≤ pH ≤ 12). Results show decreasing damage with increasing pH, and Ruthenium capping layer preservation after 100 cleaning cycles when operating at pH comprised between 10 and 12. Molecular effects (such as blocking effect) cannot be ruled out; thus, the choice of base for alkaline conditions may be of crucial importance. For an extensive understanding of surface integrity mechanism, further experiments are conducted by varying the base molecular structure, to separate pH from molecular symmetry driven effects. Weak vs strong bases, coordinating vs non-coordinating bases as well as small vs big molecular sized bases are being tested.

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Davide Dattilo receives the third best poster award at SPIE Photomask Technology 2015 conference, held in Monterey (CA) in September 2015. On the left, Mr. Naoya Hayashi, Research Fellow Dai Nippon Printing Co Ltd, Fellow Member. Co-authors are Uwe Dietze and Jhy-Wei Hsu.

Since September 15th 2012 Davide Dattilo is working as Process Scientist in the Surface Technology Innovations department at SUSS MicroTec. He is leading chemistry related projects in Photomask cleaning.Davide graduated in Chemistry in 2000 at the University of Calabria (Italy) where he attended a three years Ph.D. program, with final graduation in 2004, in Molecular Materials. His main focus was non-linear optical materials and their application in photonic systems. He moved to University of Padova for three years getting a post-doctoral position in Surface Chemistry (2007).
Before joining SUSS MicroTec Davide worked for five years as Staff Scientist in a bio-technology company dealing with the developing of MEMS, DNA microarrays and biosensors.