Cleaning of step-and-flash imprint masks with damage-free nonacid technology

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Abstract. Step-and-flash imprint lithography (S-FIL®) is a promising lithography strategy for semiconductor manufacturing at device nodes below 32 nm. The S-FIL 1:1 pattern transfer technology utilizes a field-by-field ink jet dispense of a low-viscosity liquid resist to fill the relief pattern of the device layer etched into the glass mask. Compared to other sub-40-nm critical dimension (CD) lithography methods, the resulting high resolution, high throughput through clustering, 3-D patterning capability, low process complexity, and low cost of ownership of S-FIL makes it a widely accepted technology for patterned media as well as a promising mainstream option for future CMOS applications. Preservation of mask cleanliness is essential to avoid the risk of repeated printing of defects. The development of mask cleaning processes capable of removing particles adhered to the mask surface without damaging the mask is critical to meet high-volume manufacturing requirements. We present various methods of residual (cross-linked) resist removal and final imprint mask cleaning. Conventional and nonconventional (acid-free) methods of particle removal are compared and the effect of mask cleaning on pattern damage and CD integrity is also studied. [DOI: 10.1117/1.3462815]

Subject terms: step-and-flash imprint lithography; nanoimprint; template; imprint mask; cleaning; critical dimension change.

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1 Introduction

Recently, step-and-flash imprint lithography (S-FIL) has drawn much attention and been added to the International Technology Roadmap for Semiconductors (ITRS) lithography roadmap for the 32-nm node and beyond as an alternative to optical lithography. Nanoimprint lithography has also established strong positions in other areas including discrete track and bit pattern media as well as high-brightness light emitting diodes. In the S-FIL process, drops of low-viscosity, UV-curable resist are deposited between the substrate and the imprint mask (template) before bringing them into close contact. On contact, the imprint mask is illuminated through its backside, thereby cross-linking the UV sensitive resist. The imprint mask is separated, leaving behind an inverse replica of the imprint mask pattern onto the substrate. A more detailed description of the process can be found elsewhere.[1,2]

While traditional optical lithography has either struggled to produce reliable sub-45-nm features or is haunted by its prohibitedly high cost-of-ownership (CoO), S-FIL has repeatedly demonstrated[3–5] its resolution capability of 20 nm and below with low CoO. Looking at the ITRS lithography roadmap latest edition,[6] nevertheless, there are several open issues need to be addressed before S-FIL can advance to the production phase, among which are defect size, defect density, and critical dimension (CD) uniformity, etc. Table 1 lists selected ITRS imprint mask criteria that are affected by imprint mask cleaning treatment. To overcome the challenges, it is imperative to establish a cleaning procedure involving effective chemistries and a well-controlled process environment. Use of improper chemistries or processes may not only lead to poor particle and residual polymer removal efficiency but may also damage the imprint mask. Aggressive cleaning mechanisms may roughen the surface and alter CDs. Nonautomated cleaning processes result in contamination from human intervention. Haze formation results from residual sulfate or ammonium ions posttraditional piranha and SC1 (standard clean solution number 1) cleans. Pattern damage can be attributed to inadequate MegaSonic power control etc.[7] Therefore, the development of a proper cleaning approach that is capable of minimizing physical changes to the imprint mask while effectively achieving particle and residual polymer removal, particularly removal of cured polymer stuck in between nanoscale features, is essential and requires more attention.

The importance of imprint mask cleaning should not be underestimated; however, relatively few reports dedicated to imprint mask cleaning can be found.[8–10] Figure 1 depicts a typical imprint mask process flow throughout its lifetime as well as stages where cleaning plays a critical role. A precleaning step follows resist strip, prior to the quartz dry etch step. After finishing the quartz patterning process, a 6025 substrate is diced and polished into four templates,

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Table 1 Selected ITRS imprint mask requirements (near-term years).

Year of Production	2008	2009	2010	2011	2012	2013	2014	2015
Flash $\frac{1}{2}$ pitch (nm)	45	40	36	32	28	25	23	20
CD mean to target (nm)	1.1	1.0	0.9	0.8	0.7	0.6	0.6	0.5
Defect size impacting CD (nm) x, y	4.5	4.0	3.6	3.2	2.8	2.5	2.3	2.0
Defect size impacting CD (nm) z	9.0	8.0	7.1	6.4	5.7	5.1	4.5	4.0
Trench width roughness (nm, 3 sigma)	3.4	3.0	2.7	2.4	2.1	1.9	1.7	1.5
Trench bottom surface roughness (nm, 3 sigma)	7.6	6.7	6	5.4	4.8	4.2	3.8	3.4
Imprint mask absorption (%)	<2	<2	<2	<2	<2	<2	<2	<2
Near surface defect (nm)	51	45	41	36	32	29	26	23
Defect size, patterned imprint mask (nm)	35	30	30	20	20	20	20	10
Defect density (number/cm^2)	0.03	0.03	0.03	0.01	0.01	0.01	0.01	0.01

which require cleaning before the actual imprinting process. Finally, cleaning is done on an as-needed basis in between imprint product runs. Traditional piranha and SC1 techniques have shown their value in effectively cleaning wafers, photomasks, and imprint masks. However, they are considered major contributors to the production of sulfate compounds that cause haze, adversely affecting the transparency of the imprint mask. Also, an acid process is considered a "not-so-clean" chemistry; it contributes particle adders. A more environmentally benign alternative is to utilize an ultraclean acid-free process that significantly reduces the risk of sulfate haze formation. Moreover, an acid-free process being cleaner and greener, reduces the typical costs associated with acid usage such as specialized handling, waste disposal, etc., and therefore, further reducing CoO of S-FIL technology. Aqueous ozone (DIO_3) is generally considered a promising alternative for organic resist removal; however, the widespread use is hindered by poor removal rates and inability to effectively remove highly cross-linked materials. In this study, we utilized photolyzed O_3 water as an alternative to conventional DIO_3. DIO_3 photolysis is carried out in a three-step process: (1) light-induced homolysis of O_3 in water, (2) an oxygen atom is produced that reacts with water to form hydrogen peroxide, (3) hydrogen peroxide photolyzes further to produce hydroxyl radicals.[11]

Hydroxyl radicals have higher oxidation potential (2.8 V) compared to ozone (2.07 V); therefore, they are much more potent oxidizing agents. When such hydroxyl radicals are exposed to photoresist materials or other organic compounds (C–H), they react primarily by hydrogen abstraction (deprotonation) to produce an organic radical (R') [Eq. (4)], which reacts quickly with dissolved oxygen

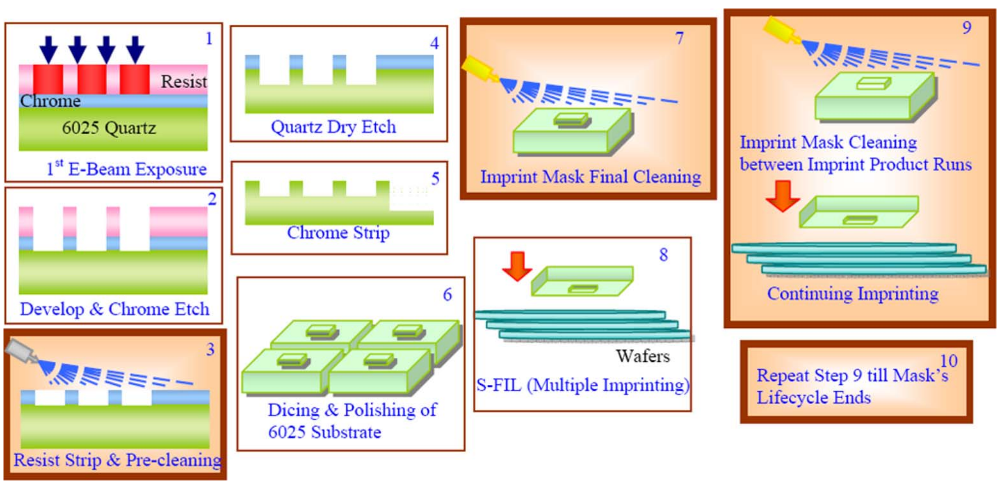


Fig. 1 Cleaning steps (highlighted) required during S-FIL imprint mask fabrication and its service life cycle (not drawn to scale).

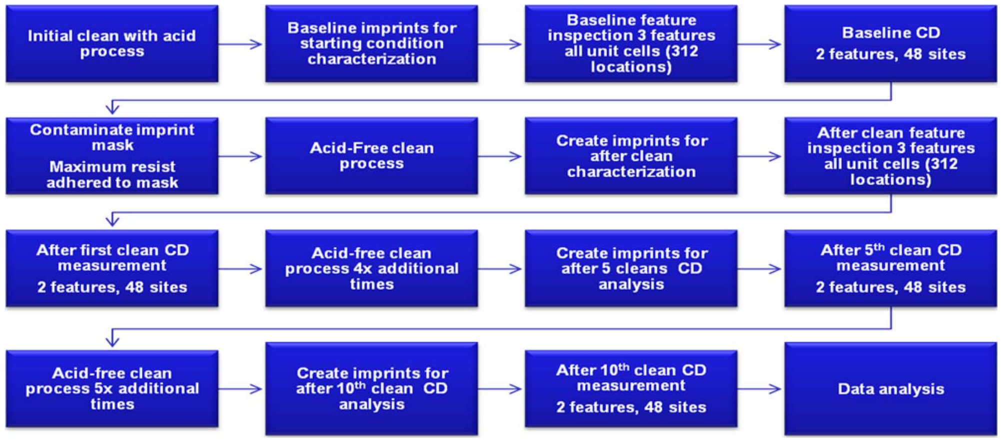


Fig. 2 Flowchart showing details of a step-by-step process sequence used to characterize the cleaning process.

to yield an intermediate organic peroxyl (RO_2') [Eq. (5)]. These intermediates initiate thermal (chain) reactions of oxidative degradation, leading finally to carbon dioxide, water, and inorganic salts.[12] We utilized this innovative method[13] to remove highly cross-linked imprint resist materials and organic residues from nanoimprint lithography (NIL) templates. This process is implemented in an automated, flexible, and multifunctional cleaning system that is well suited for exploration of S-FIL imprint mask cleaning.

$$O_3 + h\nu \rightarrow O_2 + O('D), \quad (1)$$

$$O('D) + H_2O \rightarrow H_2O_2, \quad (2)$$

$$H_2O_2 + h\nu \rightarrow HO' + HO', \quad (3)$$

$$HO' + RH \rightarrow R' + H_2O, \quad (4)$$

$$R' + O_2 \rightarrow RO_2^-. \quad (5)$$

2 Experimental

The experimental process flow used for studying cleaning efficacy, feature damage, and CD change is shown in Fig. 2. The flowchart explains step-by-step tasks as performed for imprinting, cleaning, feature inspections, and CD measurements to thoroughly evaluate the technology. The experiments were designed to verify the cleaning process efficacy of imprint masks during use in a front-end manufacturing environment. Some steps in the manufacture of the imprint masks such as dicing and polishing can result in hard-to-remove particles, and thus an initial clean is performed to ensure a known good starting point. This initial clean is a one-time process and thus has minimum impact on the imprint mask quality.

Although direct scanning electron microscopy (SEM) inspection of the imprint mask has been demonstrated, access to these tools is limited. An indirect method of characterizing imprints made from the mask was chosen to expedite the cycles of this experiment. The copy-exact nature of imprinting makes this type of analysis possible. The baseline imprints and all subsequent imprints of the mask were performed using the Drop-on-Demand™ SFIL technology developed by Molecular Imprints with an Imprio™ i100 tool. After the baseline characterization imprints were made, the imprint mask was purposely contaminated with the entire resist volume used in an imprint. This step is performed by altering the imprint process so that the imprint releases from the wafer and adheres to the mask. This type of contamination establishes a worst-case condition to ensure that the cleaning process is designed to handle all possibilities. Typically, residual organics may remain on the mask as a result of many cycles of imprints. This excessive contamination step is used to ensure that a single cleaning cycle can successfully remove the maximum resist load possible, and thus prove sufficient cleaning efficacy.

The cleaning was performed on HamaTech APE's MaskTrack series tool, which is specially customized and designed for automated imprint mask handling. HamaTech APE has developed photolyzed DIO_3 process for removing UV-cured polymer residues that are left on the imprint mask after a step-and-flash imprint cycle. The cleaning process used for these experiments starts with surface conditioning of the imprint mask followed by acid-free organic removal using photolyzed DIO_3 process. Surface conditioning is an important first step in wet cleaning, which ensures proper wetting of of the surface to be cleaned with cleaning media. Conventionally, such surface conditioning is done by dry methods such as 172-nm vacuum (VUV), which requires a separate chamber and controlled environment. On contrary, we used UV photon energy and deionized (DI) water to perform surface conditioning under atmospheric conditions. The final particle removal was done by MegaSonic-assisted SC1 solution cleaning, further concluded with an ultrapure DI water rinse and spin dry (Fig. 3). Since conventionally imprint mask cleaning is done in piranha baths, automated SPM cleaning was also performed on other imprint masks using the same cleaning tool and it was compared with the acid-free cleaning performance. The same process sequence as acid-free cleaning was used except that organic removal was done with dynamic concentration controlled SPM ($H_2SO_4 + H_2O_2$).

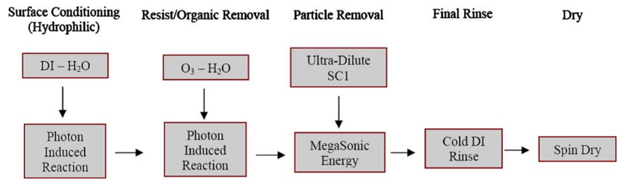


Fig. 3 Flowchart showing process flow used during cleaning of S-FIL imprint masks.

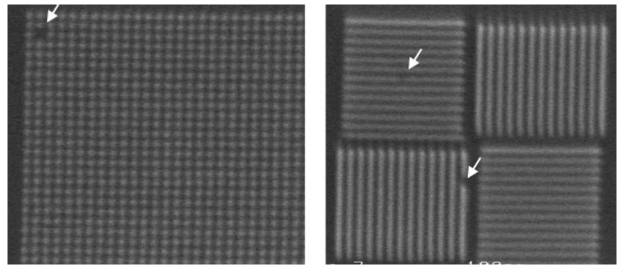


Fig. 5 Typical defect examples that can be identified with the SEM review inspection.

Complete removal of the contamination and restoration of the mask surface is determined by top-down SEM imaging of the first imprint after the clean and comparing feature fidelity to the baseline imprints. This evaluation is performed with a JEOL JWS7505 review SEM. The pattern used for this analysis is shown in Figs. 4(a) and 4(b). The 26- $\times$32-mm field contains an 8×13 array of unit cells. Each unit cell contains 32-nm hole inspection cells and 28- and 32-nm line space inspection cells, as shown in Fig. 4(c). Inspection cells were imaged in all unit cells for both the baseline and the imprint after a single clean. Three hundred twelve pairs of images at a minimum of 60k$\times$ magnification were compared. From experience, contaminated fine features in an imprint masks will not fully print on a wafer. Typical failures (shown in Fig. 5) are examples of the type of defects that can be identified with this method. The particle defects or broken features on the template would imprint as a broken line or missing pattern on the printed substrates. Since S-FIL is a 1:1 technology, such an inspection methodology holds valid for verifying cleaning efficacy.

In addition to evaluating the resist removal efficacy of the cleaning process, the effects of many cleaning cycles must be established. Multiple cleaning cycles during the life cycle of an imprint mask could result in surface damage in which the first indications will appear as changes in the CDs of very fine features. For this analysis, 28- and 32-nm line space features were measured in 24 unit cells identified by the red circles in Fig. 4(a). (Color online only.) Imprints of the mask taken for the baseline, after the first clean, after the fifth clean, and after the 10th clean, were used for this study. Imprint masks used in a manufacturing environment will most likely experience less than 10 cleans before being replaced, and thus this experiment encompasses the useful imprint mask life. Top-down SEM imaging of imprinted fields was performed at Cerium Labs with a Hitachi 4800 analytical SEM at a magnification of 200k$\times$. The CD measurements were determined using SIMAGIS analysis software. A thin metal coating was applied to each sample to reduce charging effects that deteriorate the image stability. Special attention to sample orientation in the coater as well as deposition conditions were taken to ensure the coating has a minimal impact on the result.

As mentioned, a 65-mm imprint mask is generally fabricated after dicing and polishing a 6025 photomask sub-

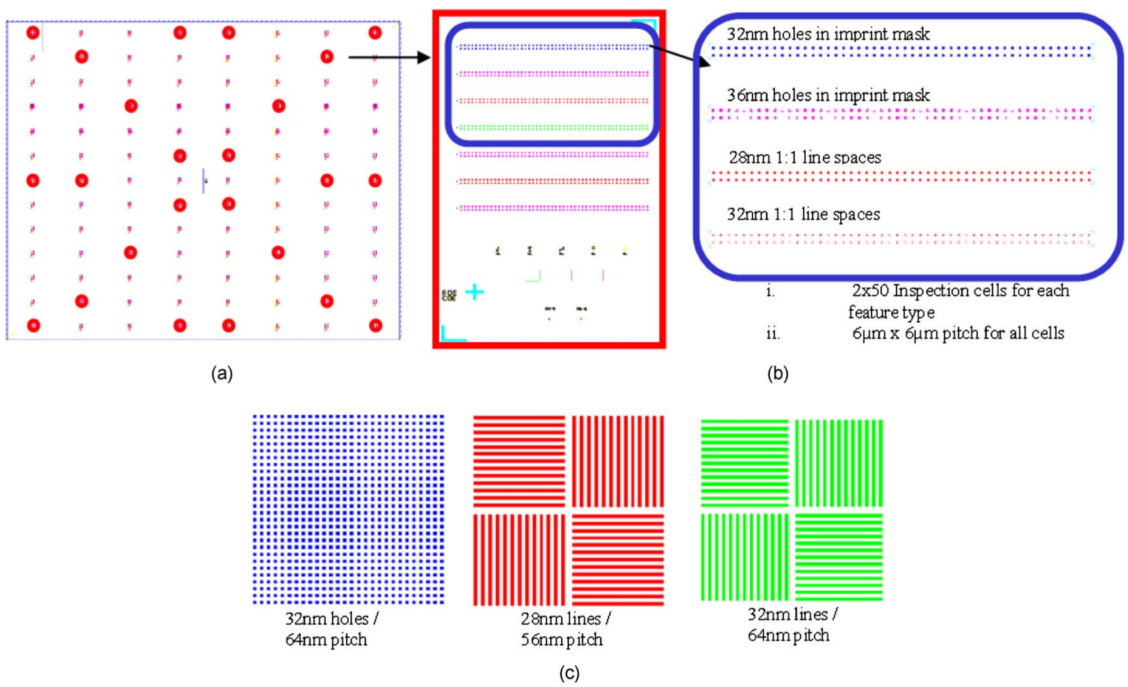


Fig. 4 (a) A 26- $\times$32-mm field layout with 104 unit cells in an 8×13 array (b) detail of single unit cell describing feature type and size, and (c) detail of the specific inspection cells.

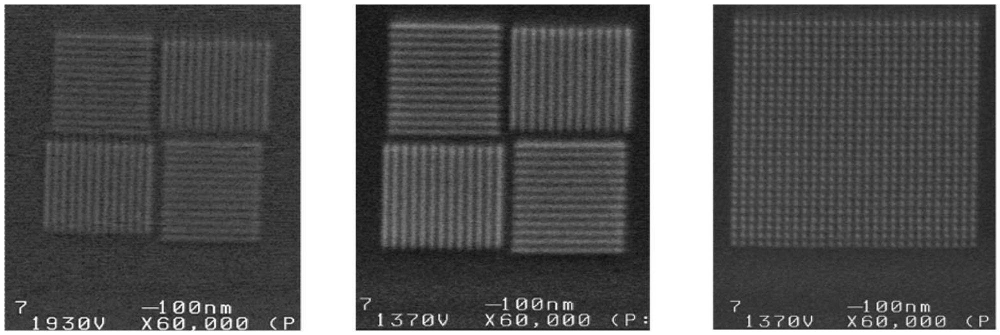


Fig. 6 Examples of images after the removal of the contamination imprint by the acid-free cleaning process.

strate and can contain hard to remove residual contaminants (e.g., SiO_2). To verify the particle removal efficiency (PRE) achievable through acid and acid-free cleaning, we used five 65-mm imprint masks without any pattern and three imprint masks with 1-μm lines/spaces pattern, which were diced and polished with a protection layer on the imprint mask surface. The protection layer was removed after dicing and polishing and the masks were cleaned multiple times using manual piranha cleaning systems. After this initial cleaning, the masks were inspected on Lasertec Magics2351 inspection system to acquire premeasurement data. The inspection area used was a 25- $\times$25-mm square mesa on a 65-mm mask. Particle sizes down to 70 nm could be detected. The imprint masks were then cleaned on HamaTech APE's MaskTrack automated cleaning tool. The postcleaning data on number and location of particles was obtained. The PRE was calculated from precleaning and postcleaning inspection data maps and particle counts. The masks with and without patterns were cleaned using both acid and acid-free processes for comparison. Several process conditions in acid-free cleaning were varied to observe their effects on PRE.

3 Results and Discussion

3.1 *Cleaning Efficacy and CD*

The comparison of the 312 sets of images to determine cleaning efficacy concluded that all images were free of visible defects. Although the images from the review SEM have low resolution, the tool has the capability to verify that all features were present and fully populated. A single cleaning cycle performed using the customized mask cleaning process on the HamaTech APE's MaskTrack series, was able to effectively remove the entire resist load from an imprint, including all organic contaminants within the relief patterns of the mask. After being cleaned, the mask reproduced imprints identical to the baseline. Examples of the clean images are shown in Fig. 6.

With the cleaning efficacy established, the next step was to ensure that the cleaning cycle has a minimal impact on the imprint mask patterns features. This result was obtained by measuring the CD of 28- and 32-nm line space resist patterns at the same locations within an imprint field made after the mask was cleaned 1, 5, and 10 times. Because SC1 cleaning solution (NH_4OH/H_2O_2) is part of the process, it is suspected that some CD loss would be observed. An SC1 process at elevated temperatures and higher concentrations is known to etch SiO_2 surfaces. For example a 1:1 solution at 60 °C will have an etch rate[14] of 0.1 nm/min. However, in the imprint mask cleaning process under discussion, a room temperature SC1 solution with dilute concentration was used. Surface etching during the clean can result in widening of the relief features, and thus cause the imprint patterns to have increased resist CDs, as illustrated in the sketch of Fig. 7.

Figure 8 shows the measured CD as a function of cleaning cycles. The black data points identify the mean values for each data set collected between cleans. Two data sets were collected for the 10th cleaning cycle. Linear trend lines show approximately a 0.06-nm increase in CD of the imprinted features per acid-free cleaning process.

The data were further analyzed to reduce the measurement noise and eliminate the systematic CD error resulting from the mask manufacturing process. Differences between the baseline and the first clean are virtually all measurement error; thus, the same field locations for these data sets can be averaged to provide a better comparison baseline. The two data sets after the 10th clean are also combined by same site averaging. Figure 9 illustrates the change in CD across the field as a result of approximately 10 cleans. The average change in CD is 0.56 nm over 10 cleans, also suggesting a change of 0.06 nm per clean. Again, more data are required to reach any definitive conclusions about a trend. A subnanometer change in CD over the life of the mask is manageable.

3.2 *Acid and Acid-Free Cleaning*

Three identically patterned 65-mm rigid imprint masks used in this particular experiment have 45-nm line features at 2:1 pitch.[15] These masks were also intentionally contaminated with imprint resist such that the etched features were completely filled and capped with thin layer of cross-linked polymer. Two masks were cleaned with dynamic concentration controlled SPM process, and third mask was cleaned

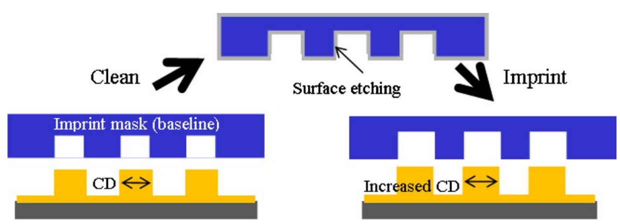


Fig. 7 Change in CD as a result of surface etching during cleaning.

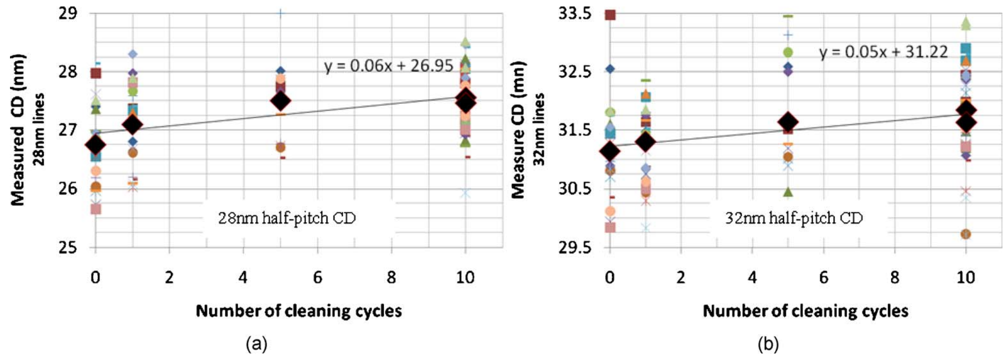


Fig. 8 Measured CD for (a) 28- and (b) 32-nm imprint resist lines as a function of acid-free cleaning cycles.

with a nonacid process using the automated tool. All the 252 imprinted sites inspected using wafer review SEM showed excellent removal of cross-linked resist from the 45-nm lines using acid as well as the acid-free process. All the 45-nm lines and large fin structures were completely resolved. Figure 10(a) shows the device structure layout that was cleaned, Fig. 10(b) shows the imprint from a nonacid cleaned mask, and Fig. 10(c) shows an imprint from an acid cleaned mask.

The main difference between acid and nonacid cleaning process is in the mechanism of cross-linked resist removal. Since UV-cured resists are highly cross-linked and very difficult to remove, piranha (SPM) solutions have been favored traditionally to dissolve such hard organics. The effectiveness of SPM ($H_2SO_4+H_2O_2$) solution in removing organic contaminants is due to two distinct mechanisms. The first mechanism involves dehydration of the hydrocarbon compound by the concentrated sulfuric acid. The second process is the sulfuric-acid-assisted conversion of hydrogen peroxide into hydronium ion and O radical which are very aggressive oxidants: $H_2SO_4+H_2O_2 \rightarrow H_3O^+ + HSO_4^- + O$. Both of these processes lead to oxidative degradation of the cross-linked polymer at very fast rates. The photolyzed DIO_3 process (nonacid), as explained in Sec. 1, produces hydroxyl radical species, which are much stronger oxidizing agents than a SPM mixture, thus giving similar process results without any disadvantages of an acid process. These results demonstrate that this nonacid process is fully qualified for imprint mask cleaning.

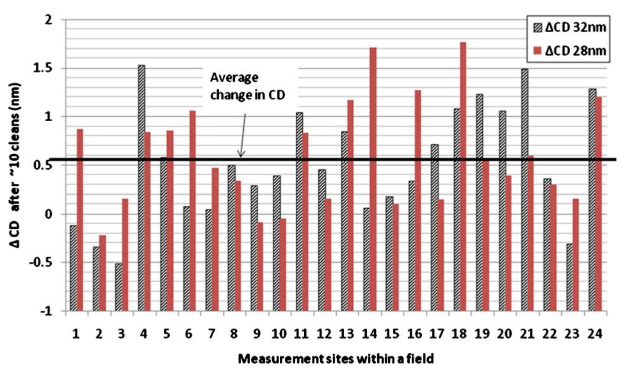


Fig. 9 Change in CD across the field as a result of approximately 10 cleaning processes.

3.3 *PRE*

The PRE fraction was calculated by dividing total number of particles removed (prepost) with initial number of particles (pre). The particles added by the cleaning process were also considered as part of the post cleaning particle count. Figure 11 shows the PRE plot for patterned and unpatterned imprint mask surfaces cleaned under different conditions detailed in the table below the plot. Both of the tests done on an imprint mask (without pattern) using acid cleaning gave a maximum PRE of 82%, whereas 99% PRE was achieved using the nonacid process. Although there is no doubt that the acid process is capable of removing organic contamination, as demonstrated in the previous section, the acid is known to add impurities and particles. These impurities are difficult to remove with a subsequent SC1 MegaSonic treatment. Therefore numbers of post particles after an acid cleaning are still higher than an acid-free cleaning. After establishing the optimized process condition for nonacid cleaning of imprint masks without a pattern, the same conditions (optimized and unoptimized) were repeated on a mask with 1-μm L/S pattern. Similar results were obtained on a patterned imprint mask. A PRE closer to 100% was obtained, with only a single particle remaining on the cleaned surface.

4 Conlusions and Future Work

An acidfree cleaning process was identified for removing any potential contamination resulting from imprint processing. CD change as a function of cleaning was within the measurement error of our analyses, but did show a trend of a 0.06-nm average increase in CD per cleaning cycle. The

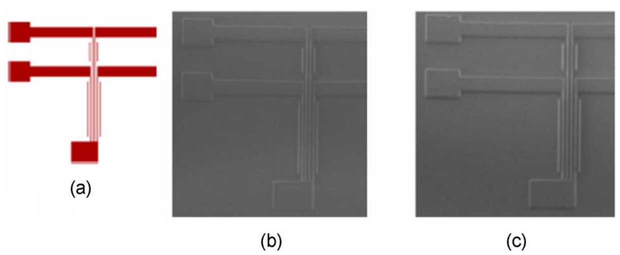
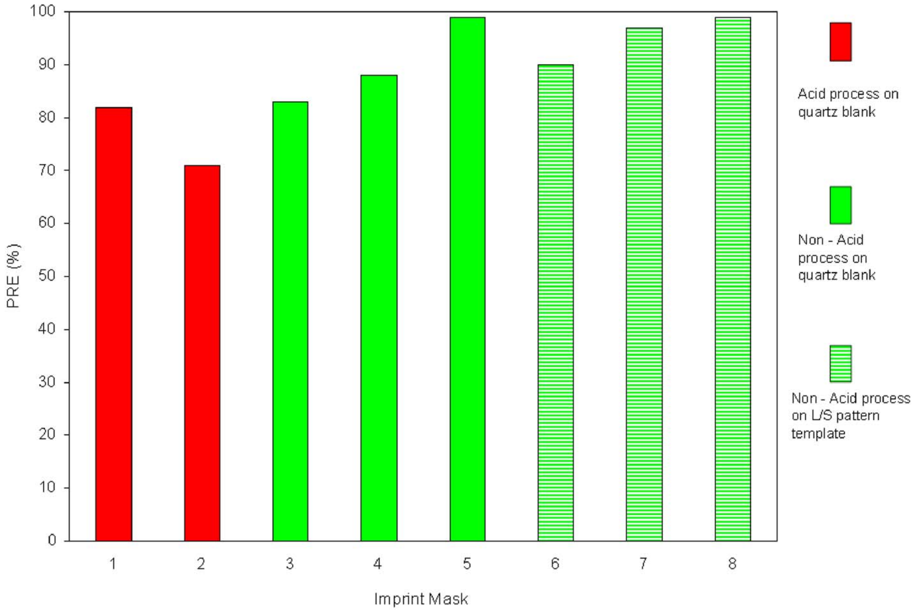


Fig. 10 (a) Device structure layout with 45 nm lines,[12] (b) SEM image of an imprint from a nonacid process cleaned imprint mask, and (c) SEM image of imprint from an acid-process-cleaned imprint mask.



Imprint mask #	1	2	3	4	5	6	7	8
Cleaning Process	Acid	Acid	Non-Acid	Non-Acid	Non-Acid	Non-Acid	Non-Acid	Non-Acid
Imprint mask Type	Blank	Blank	Blank	Blank	Blank	Pattern	Pattern	Pattern
Variables	Test1	Test 2	Test 1	Test 1	Test 2	Test 1	Test 1	Test 2
PRE	82%	71%	83%	88%	99%	90%	97%	99%

Fig. 11 Plot showing PRE values achieved on blank and patterned imprint masks using different cleaning conditions.

acid-free process developed on the automated tool showed a PRE of 99% on patterned as well as blank imprint masks. With advanced technology nodes, the defect density requirements for imprint masks are becoming increasingly stringent; therefore, it is imperative that the cleaning process does not add any particles and masks have zero surface particles (e.g., above 23 nm by 2015, ITRS) after cleaning. This is only possible when cleaner chemistries and automated process control and handling are utilized during imprint mask cleaning; therefore, a completely acid-free process with 100% PRE is much desired. Additional experiments are required to further optimize the process with ultradilute SC1 solutions and ionized H_2 water to further reduce the influence of the cleaning cycle on the CD uniformity of the features on the imprint mask while maintaining the particle removal capability.

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