Filter Priming Recipe for IntelliGen® ULV Dispense System with Oktolex™ Membrane Technology

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Priming a filter in the start-up step of chip production is very sophisticated. Engineers must understand the characteristics of both the filter membrane and the liquid, then be able to apply a suitable priming method to the dispense system that helps reduce or eliminate wafer defects. An optimal filter priming procedure can lead to a quick start-up.[1]

Entegris research has resulted in a priming recipe that can effectively eliminate potential sources of bubble defects from filters. This research is detailed in the application note titled, *The Study of Effectiveness of Priming Cycle in IntelliGen® ULV*.[2] This application note briefly reviews key concepts of filter priming and introduces the optimum priming recipe for the Impact® 8G filter with Oktolex™ membrane technology when used with the IntelliGen ULV dispense system.[3]

Oktolex membrane technology is a cleaner, faster, and more effective way to remove the most challenging contaminants with a tailored approach to the specific contamination control needs of Krypton Fluoride Laser (KrF), Argon Fluoride laser (ArF), and Extreme Ultraviolet (EUV) lithography for Logic, DRAM, and 3D NAND devices.[4] The ultra-high molecular weight polyethylene (UPE) membrane removes critical photochemical contaminants, improving defect reduction.

Due to its good solubility, OK73 thinner, a mixture of Propylene Glycol Monomethyl Ether (PGME) and Propylene Glycol Monomethyl Ether Acetate (PGMEA), is commonly used as a standard solvent for both negative and positive resists in the three types of lithography processes mentioned above.[5] Cyclohexanone (CHN) is a thinner that is sometimes used in the resists for ArF Spin-on Hard Mask (SOH) lithography, a lithography method developed to prevent the pattern collapse.

To achieve the best performance, the filter priming recipe needs to be adjusted with each slight change in thinner composition. OK73 thinner is highly compatible with, and can spontaneously wet, the UPE membrane. However, it is slightly more difficult for CHN to do so. This application note provides two priming recipes for using Oktolex membrane technology when the process liquid is OK73 thinner, and when the thinner is CHN, respectively.

KEY CONCEPTS OF A FILTER PRIMING RECIPE

A good filter priming recipe must be able to effectively remove bulk air, microbubbles, and nanobubbles from spaces inside the filter cartridge and membrane pores. Figure 1 illustrates the priming cycle sequence of the IG-ULV dispense system studied in the previous work.[2] In that study, the Selectable step (6) was varied by changing key priming technologies. Results show that the best performance was achieved when the Backflush to Vent cycle was selected, followed by the Flush Bubbles cycle as second best.

RECOMMENDED PRIMING SEQUENCE

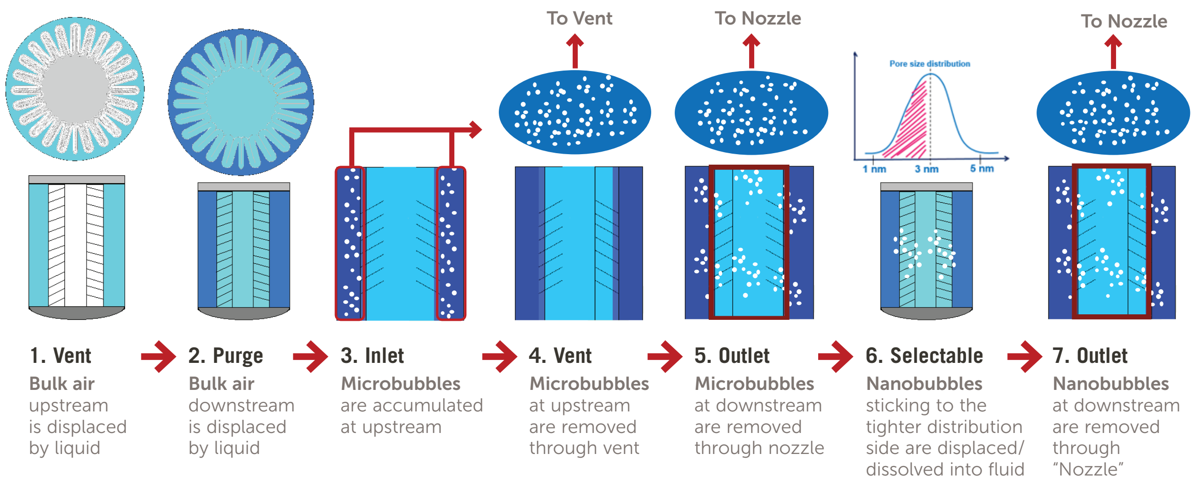


Figure 1. Sequence of seven priming cycles.

PRIMING RECIPES

The two priming recipes shown in Figures 2 and 3 were the focus of this study. Figure 2 shows the best recipe is the Backflush to Vent cycle at step 6, and Figure 3 shows the second-best recipe is the Flush Bubbles cycle at step 6.

Most of the two priming recipes shown at right are the same as those tested in the previous study, except for the number of outlet cycles at steps 5 and 7. In the previous study, 10 outlet cycles were used in both steps, but in this study 40 outlet cycles were used at step 5, and 60 outlet cycles were used at step 7. The increase in outlet cycle counts was for conditioning the Oktolex membrane surface. Users should adjust the number of outlet cycles at steps 5 and 7 based on the pattern quality on the wafer.

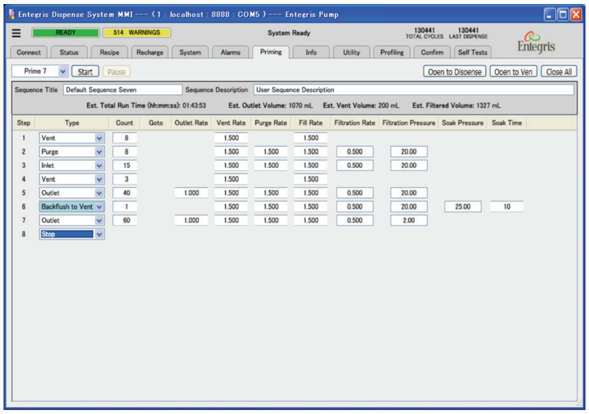


Figure 2. Priming sequence of the best recipe, Backflush to Vent cycle at step 6.

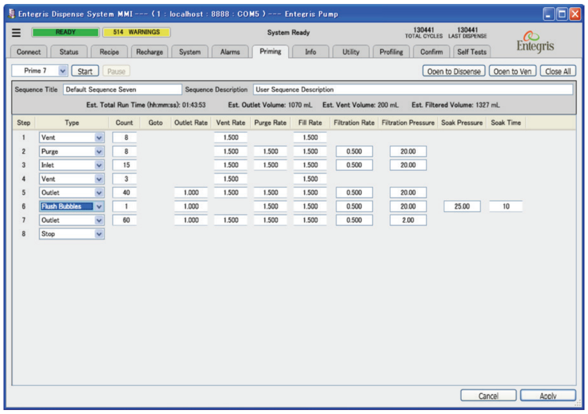


Figure 3. Priming sequence of the second-best recipe, Flush Bubbles cycle at step 6.

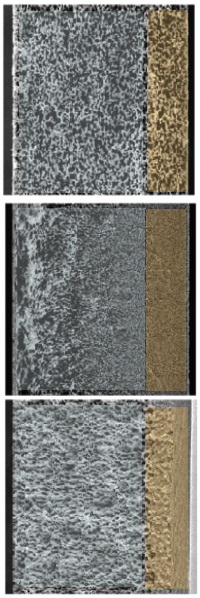
BACK FLUSH TO VENT CYCLE REVIEW

There are three segments in the Backflush to Vent cycle. Soaking Backwards, Filtration Backwards to Vent, and Normal Filtration. Liquid is pressurized into the membrane pores from downstream. Tight pores downstream can be filled by liquid, and microbubbles inside tight pores can be immediately eliminated through the the vent during the Filtration Backwards to Vent segment.

Sym

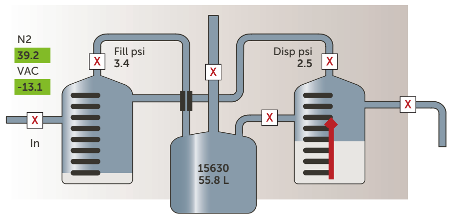
Asy

Duo

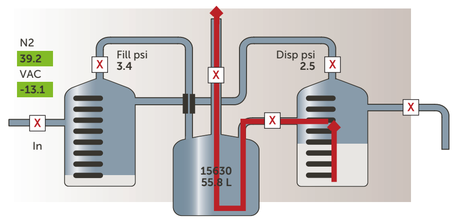


Refers to area where pores can be effectively wetted

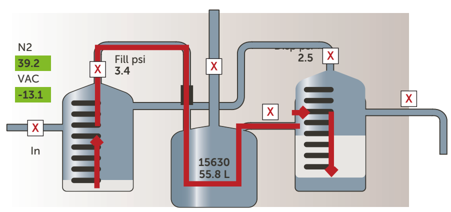
Refers to area where pores can be moderately wetted



1. Soaking backwards
Pressurize from downstream to enable liquid to penetrate membranes' pores and dissolves nucleation site on membrane surface into liquid.



2. Filtration backwards to vent
Eliminate the liquid with dissolved nucleation site through vent.

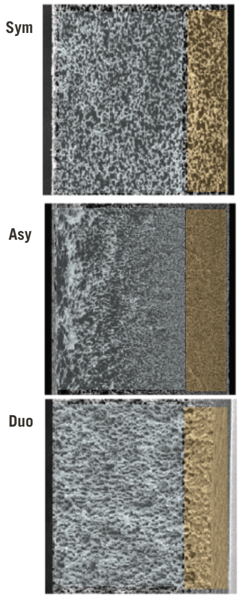


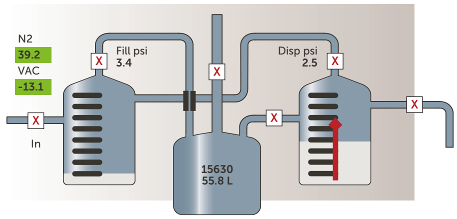
3. Normal filtration
Push air bubbles to dispense chamber.

Figure 4. Backflush to Vent cycle.

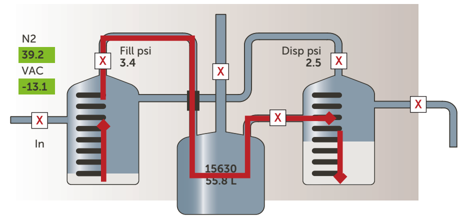
FLUSH BUBBLES CYCLE REVIEW

There are four segments in the Flush Bubbles cycle. Soaking Backwards, Filtration to Outlet, 10 mL Dispense, and Normal Filtration. Liquid is pressurized into membrane pores from downstream. Tight pores downstream can be filled by liquid, and microbubbles inside tight pores can be immediately eliminated through the outlet during the 10 mL Dispense segment.

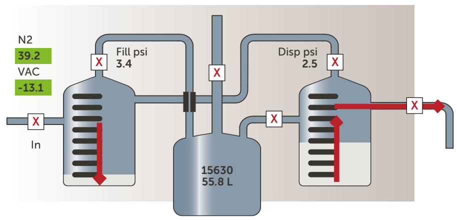




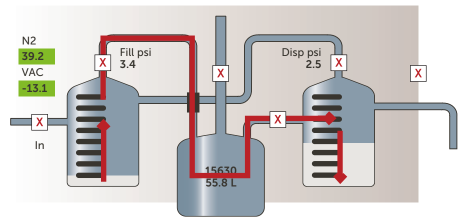
1. Soaking backwards
Pressurize from downstream to enable liquid to penetrate membranes' pores.



2. Filtration to outlet
Outlet valve opens and liquid is pushed through membrane pores and bubbles are brought to dispense chamber.



3. 10 mL dispense
Bubbles come with liquid during filtration are removed to nozzle.



4. Normal filtration
Pump starts a recharge for next dispense by starting normal filtration segment.

Refers to area where pores can be effectively wetted

Refers to area where pores can be moderately wetted

Figure 5. Flush Bubbles cycle.

EXPERIMENTATION

In this test, repetition of the best (Backflush to Vent) and second-best (Flush Bubbles) recipes were conducted on the Impact 8G filter with Oktolex membrane technology using two types of process fluids: 1. mixture of 30% PGME and 70% PGMEA and, 2. mixture of 30 % CHN and 70% PGMEA. Mixture 1 is the composition of OK73 thinner and will be called "OK73 thinner" throughout this document. Mixture 2 is the composition of resist solvents for SOH lithography and will be called "SOH-based solvent" throughout this document.

An IntelliGen ULV dispense system with firmware V1005_987 installed and an Impact 8G filter with Oktolex membrane technology was used. Figure 6 illustrates the test setup. 400 mL of process fluid was used for each condition. An Impact 8G UPE UC 3 nm filter was used in the baseline establishment step.

After a low and stable baseline was achieved, the Impact 8G UPE UC 3 nm filter was replaced by a dry, new Impact 8G filter with Oktolex membrane technology and the priming recipe of interest was tested. The low and stable baseline is defined by a moving average of 5 data points where 0.15 µm microbubbles is lower than 2.0 for 100 continuous dispense cycles.

After priming, liquid was dispensed continuously through the liquid particle counter, monitoring for microbubbles. The dispensed liquid in continuous dispense mode was stored in the 30 mL cylindrical collection vessel before being drawn into the particle counter by the syringe sampler. After passing through the particle counter, the liquid was returned to the bottle. Particle counter models Rion® KS41-A measuring 0.15 µm was used. A sample flow of 2.0 mL at 1.0 mL/s was measured every 60 seconds and recorded over 500 cycles after completion of priming.

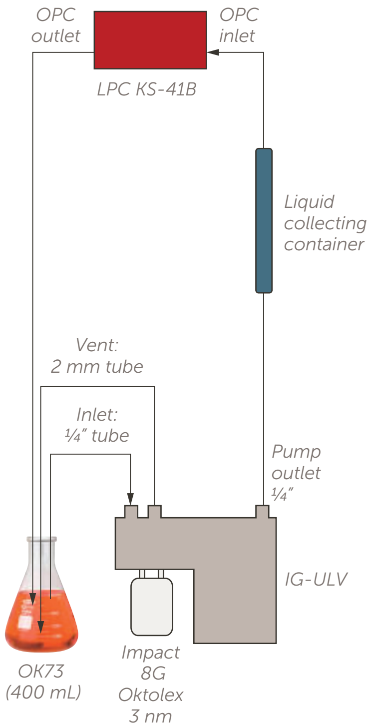


Figure 6. Test setup.

RESULTS

Test results of OK73 thinner obtained from the previous study are brought into account for the comparison with the results obtained from this study. The results of SOH-based solvent are from this study only.

In the case of OK73 thinner, results obtained from the previous study and this study are corresponding, the Backflush to Vent recipe shows slightly better performance than the Flush Bubbles recipe. Figures 7a through 7c show particle count averages calculated from the 151st cycle to the 200th cycle with Impact 8G Oktolex, Impact 8G UPE 3 nm, and Impact 8G UPE DUO 3 nm filters, respectively. A lower average value indicates a better priming performance.

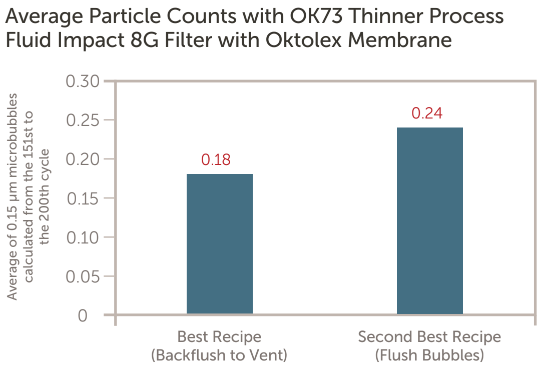


Figure 7a.

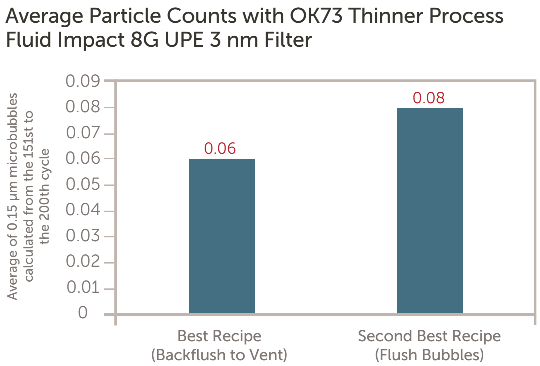


Figure 7b.

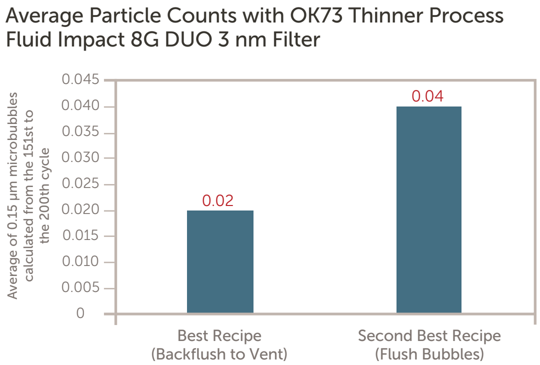


Figure 7c.

Figures 7a-c. Priming performance comparison of the Backflush to Vent recipe and Flush Bubbles recipe with various Impact 8G filters.

Results of SOH-based solvent testing are reversed in that the Flush Bubbles recipe shows better performance than the Backflush to Vent recipe. Figure 8 compares priming performance of the Backflush to Vent and Flush Bubbles recipes when SOH-based solvent is the process fluid.

Average Particle Counts with SOH-based Solvent Process Fluid Impact 8G Filter with Oktolex Membrane

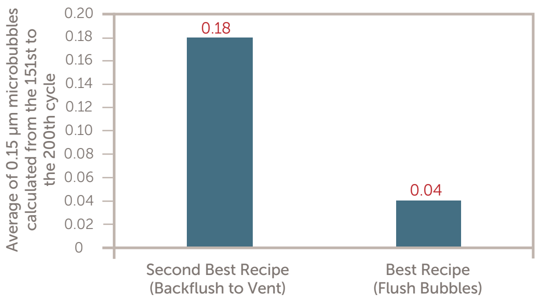


Figure 8. Priming performance comparison of the Backflush to Vent and Flush Bubbles recipes.

DISCUSSION

Figures 9a and 9b show raw data particle counts obtained after priming the Impact 8G filter with Oktolex membrane using the Backflush to Vent and Flush Bubbles recipes, respectively. Both tests used OK73 thinner as the process fluid. Figures 10a and 10b show raw data particle counts obtained for the same priming recipes using SOH-based solvent as the process fluid.

Backflush to Vent Recipe with OK73 Thinner Process Fluid Impact 8G Filter with Oktolex Membrane

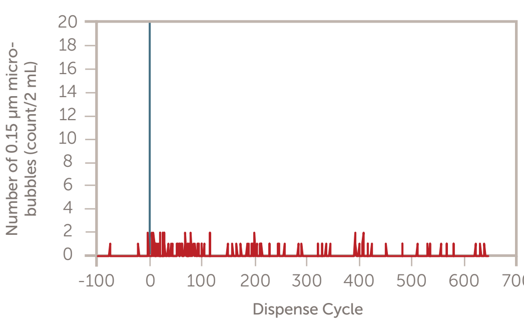


Figure 9a.

Flush Bubbles Recipe with OK73 Thinner Process Fluid Impact 8G Filter with Oktolex Membrane

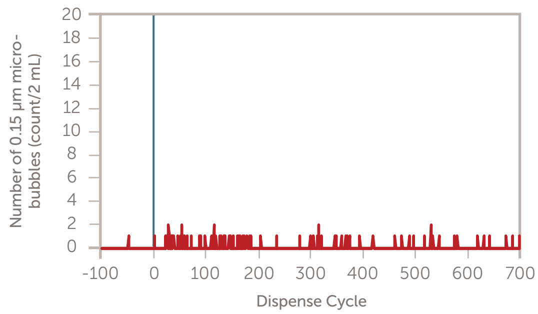


Figure 9b.

Figures 9a-b. Raw particle data from testing Backflush to Vent and Flush Bubbles priming recipes using OK73 thinner process fluid.

Backflush to Vent Recipe with SOH-based Solvent Process Fluid (PM: CHN = 7.3) Impact 8G Filter with Oktolex Membrane

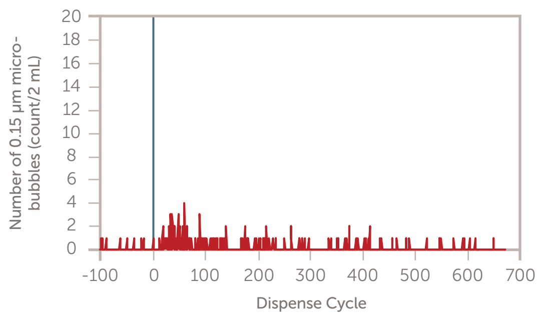


Figure 10a.

Flush Bubbles Recipe with SOH-based Solvent Process Fluid (PM: CHN = 7.3) Impact 8G Filter with Oktolex Membrane

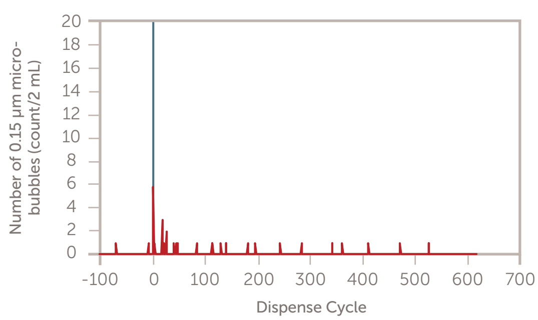


Figure 10b.

Figures 10a-b. Raw particle data from testing Backflush to Vent and Flush Bubbles priming recipes using SOH-based solvent process fluid.

When the process liquid is OK73 thinner, raw data shows the Backflush to Vent and Flush Bubbles recipes are not significantly different, but when the process liquid is SOH-based solvent, raw data for the Flush Bubbles recipe is better than that of the Backflush to Vent recipe.

OK73 thinner comprises two low-surface-tension liquids, PGMEA (28 mN/m) and PGME (27 mN/m). Liquids like OK73 thinner with low-surface tension can spontaneously wet the UPE surface. Therefore, small bubbles adhering to the UPE surface (nucleation site) can easily detach from the UPE surface and be dissolved into the liquid once pressure is applied. On the other hand, SOH-based solvent comprises CHN (35 mN/m) and PGMEA. CHN has slightly higher surface tension compared to PGMEA and PGME, which makes it harder for small bubbles to detach. This slightly higher surface tension is the cause of the difference in raw data.

The Backflush to Vent and Flush Bubbles cycles differ in the way they apply pressure onto the liquid in the soaking step. In the Backflush to Vent cycle, liquid with dissolved nucleation sites moves backward through the filter and is eliminated through the vent port. While in the Flush Bubbles cycle, liquid with dissolved nucleation sites moves downward before being eliminated through the outlet port. The dispense piston used for applying pressure on liquid moves back downward to its home position before advancing again to push the liquid away from dispense chamber through the outlet port.

When the process liquid is OK73 thinner and nucleation sites can easily detach from the UPE surface and become smaller in size or be completely dissolved into the liquid once pressure is applied, it does not matter if the liquid with dissolved nucleation sites is eliminated through the vent port in the Backflush to Vent cycle or through the outlet port in the Flush Bubbles cycle. The nucleation sites can be effectively eliminated by both methods. This explains why there is no significant difference in raw data when OK73 is used as process liquid.

When the process liquid contains CHN, it has less ability for liquid to wet the UPE surface. This causes the nucleation site to remain on the membrane surface even after applying pressure. When the Backflush to Vent cycle is used, the dissolved nucleation site cannot pass through the filter and cannot be eliminated through the vent port because they are not small enough. Thus, they remain in the dispense chamber and can be released once the continuous dispense starts, which is why we see larger numbers of microbubbles in the raw data. When the Flush Bubbles cycle is used, the liquid with the nucleation site can be eliminated through the outlet port because there is no filter as a barrier. Moreover, in the Flush Bubbles cycle, there is a decrease in pressure inside the dispense chamber when it moves downward to the home position of the dispense piston. This enlarges the nucleation site and facilitates the detachment of the nucleation site from the UPE surface, which then can be effectively eliminated through the outlet port. Figures 11 and 12 summarize the concepts explained on page 8.

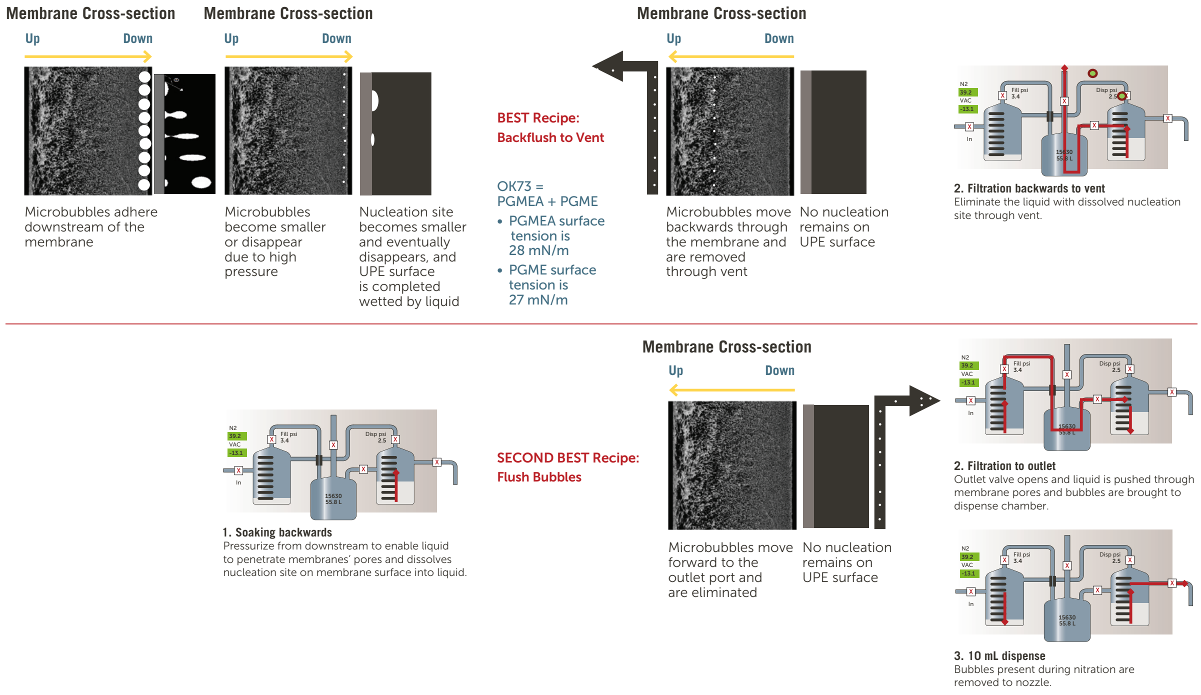


Figure 11. Illustration explaining why test results for OK73 thinner are not significantly different

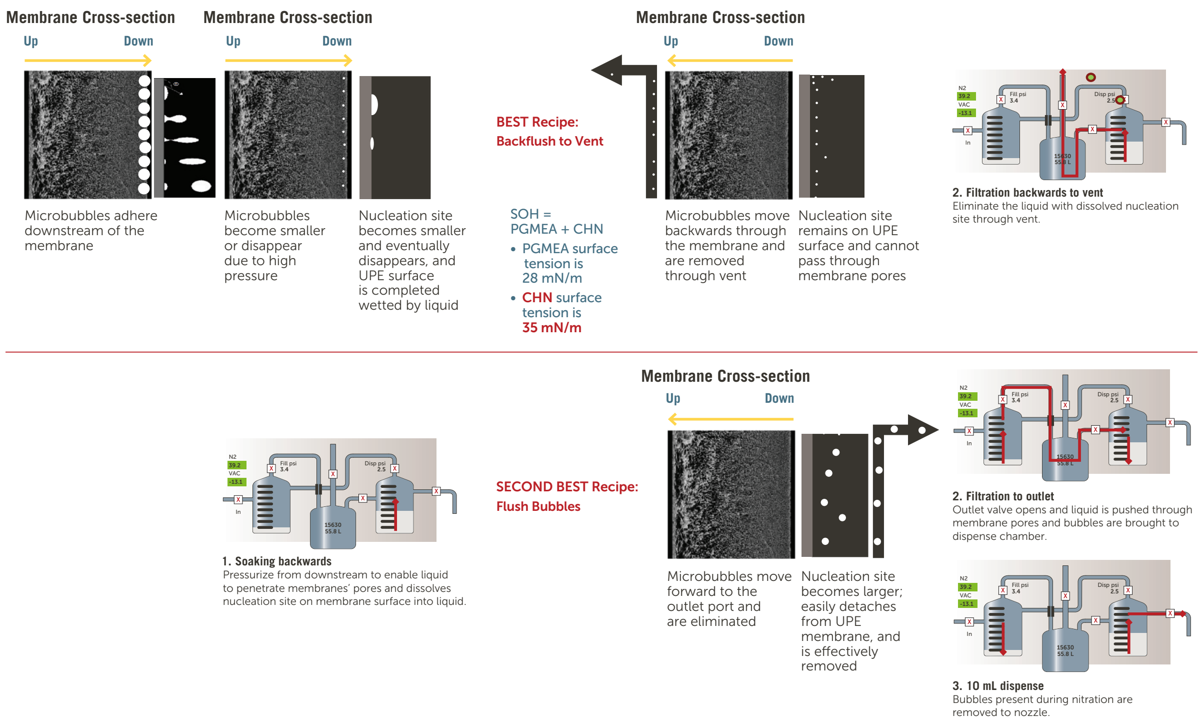


Figure 12. Illustration explaining why Flush Bubbles recipe shows better performance than Backflush to Vent recipe.

SUMMARY

Table 1. Summary of the optimal priming recipes for each process fluid and filter type

Based solvent of process fluid	Lithography application	Filter type	Suitable recipe
OK73 thinner	KrF, Arf, EUV	Impact 8G Oktolex Impact 8G UPE 3 nm Impact 8G DUO 3 nm	Backflush to vent (best)
Mixture of 20 – 30 % CHN and 70 – 80% PGMEA	ArF spin-on hard mask	Impact 8G Oktolex	Flush bubbles (second best)

CONCLUSION

Various types of solvents are used in photoresists for KrF, ArF, and EUV lithography. Even though solvents with low-surface tension are preferable for use with UPE membranes, sometimes lithography processes require photoresists with solvents containing a high-surface-tension liquid component. The IntelliGen ULV dispense system, with robust priming technologies, can effectively prime point-of-use filters with both low- and high-surface tension liquids. In-house test results confirm, with the right application of the dispense system, microbubbles can be effectively removed from filters, reducing wafer defects and increasing yield. The Backflush to Vent cycle shows good priming performance for low-surface tension liquids, while the Flush Bubbles cycle shows good priming performance for high-surface tension liquids. By following the guidelines in this application note and incorporating an IntelliGen ULV dispense system into the process, users can be more confident that fewer microbubbles would be released during chip production.

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