# IntelliGen<sup>®</sup> High-Viscosity Dispense Systems Enables Faster Start-up Times in High Viscosity Fluid Operations

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## INTRODUCTION

Priming a filter is one of the more time-consuming tasks occurring during dispense system startup when a filter is installed into a wafer coating machine. The coating machine can be down for a relatively long time especially when the process fluid has high viscosity. As expected, high viscosity flow characteristics differ greatly from low viscosity. High viscosity flow characteristics cause a resistance in flow which extends the time it takes for the liquid to fully fill the flow path. It is even more problematic if the flow path is moderately porous because the liquid flow is inhibited by the presence of air trapped inside the pores. The trapped air, caused by the delayed pore filling process, generates small air bubbles that form and gradually get released when the fluid passes through the porous flow path. Eliminating small air bubbles also takes a very long time.

Entegris' IntelliGen® HV (IG-HV) dispense system is designed specifically for high viscosity fluid operations. With IG-HV users can easily set the delta pressure of the filter upstream and down-stream during the filtration segment up to 40 psi which helps to pressurize the filter upstream. Using the piston in its fill motor, the IG-HV enables the highly viscous liquid to thoroughly penetrate the membrane pores. This allows the air occupying the membrane pores to be displaced by liquid and then to be effectively eliminated afterwards. The IG-HV can also dispense a viscous liquid with a high dispense rate of up to 3.00 mL/s. This capability helps flush away air bubbles adhering to the tube wall through the nozzle.

This application note demonstrates the capabilities of IG-HV with high viscosity fluids and explains in detail how a new filter can be quickly wetted during a start-up procedure using the IG-HV.

# BACKGROUND

This section presents an example of a recipe used to prime Impact<sup>®</sup> HV 1.0  $\mu$ m with 1,000 cP process fluid. The priming needed to be repeated twice to obtain an acceptable on-wafer particle level. The approximate 5-hour start-up time used for priming was too long, and so it became necessary to configure a new priming recipe that would greatly reduce the start-up time.

Figure 1a and Figure 1b display incorrect parameter settings that ultimately resulted in incorrect filter priming. Figure 1a shows:

- Vent cycle set to step 1
- Outlet cycle set to step 2
- Purge to vent cycle set to step 3
- The number of outlet cycles in step 8 set to 10 cycles

An analysis of the original recipe revealed three factors that likely caused the inefficient priming:

# 1. Order of Vent Cycle, Outlet Cycle and Purge to Vent Cycle in Steps 1 through 3 during filter priming.

During the filter priming phase, it is effective to first replace the bulk air inside the pump (another name for the dispense system) and filter cartridge. The Vent cycle replaces the bulk air in the upstream of the filter, and the Purge to Vent cycle replaces the bulk air in the downstream of the filter. The Vent cycle should be used first, followed by the Purge to Vent cycle so that the bulk air can be completely removed from the fluid flow path inside the pump body and filter cartridge. The Outlet cycle is used instead of the Purge to Vent cycle in step 2. The Outlet cycle in the filter priming cycle pushes the liquid forward to the nozzle through the filter. If this cycle is used while the bulk air still remains in the filter downstream, it is possible that air will move backwards to the filter upstream during the Outlet cycle operation. Consequently, air will pass into the membrane pores and get trapped there, and then air will gradually get released after the liquid flows through the filter. This might explain why the original recipe had to be repeated twice to reach an acceptable on-wafer particle level.

Eile				Chemical Volumes					
					To Vent Across Filter To Nozzle To Rec.				
1	IGHV dry pump (	prime				540 ml	1820 ml	320 ml	0 ml
	Cycle Type		Cycles						
1	Vent	•	3	÷					
2	Outlet	•	6	÷					
3	Purge to Vent	•	10	÷					
4	Purge to Inlet	•	60	÷					
5	Vent	•	2	÷					
6	Purge to Inlet	•	5	÷					
7	Vent	•	2	÷					
8	Outlet	•	10	÷					
9	Vent	•	1	÷					
10	Stop	-	1	÷					

*Figure 1a. Setting the filter priming cycle sequence in the original recipe.* 

## Figure 1b shows Delta P set to 5.0 psi.

rstem Process Recharge Priming P	echarge Priming Recharge Continue	d	
Fill Rate and Pressure Control			
Mode: Automatic	Rate: 2.000 (mL/s)	Pressure: -10.0 psi	
Vent Rate and Pressure Control			
Mode: Manual	Rate: 1.000 (mL/s)	Pressure: 10.0 psi	

*Figure 1b. Setting of the recharge parameter during priming in the original recipe.* 

## 2. Number of Outlet Cycles in Step 8.

Regarding high viscosity liquids, air bubbles that easily adhere to the tube wall are very difficult to remove if the amount of liquid flow is low. Ten cycles seem insufficient to completely flush air bubbles that adhere to the tube wall.

## 3. Delta P used in Filtration Segment.

The Delta P setting shown in Figure 1b facilitates the flow of liquid through the filter. If the delta P is set too low, it is difficult for the liquid to flow upstream to downstream through the membrane pores.

# RECOMMENDED PRIMING RECIPE MODIFICATION

To improve the performance of the original recipe, the following priming recipe modifications are recommended:

- 1. Replace the bulk air inside the upstream of the pump and filter cartridge.
- 2. Pressurize the liquid to fill up small membrane pores.
- 3. Replace the bulk air inside the downstream of the pump and filter cartridge.
- 4. Eliminate air trapped inside the membrane pores and air bubbles adhering to the tube wall through the nozzle.

Figure 2a and Figure 2b show the parameter settings for the modified recipe. Figure 2a shows:

- Vent cycle set to step 1
- Filter prime cycle set to step 2
- Purge to vent cycle set to step 4
- Number of Outlet cycles in step 8 set to 100 cycles

🏶 Edit Primine Sequence	
Eile	Chemical Volumes
	To Vent Across Filter To Nozzle To Rec.
1 IGHV dry pump prime	300 ml 2720 ml 2000 ml 0 ml
Cycle Type Cycles	
1 Vent 💌 5 📩	
2 Filter Prime 🗾 1	
3 Vent 2	
4 Purge to Vent 💌 5 🔆	
5 Purge to Inlet  30 .	
6 Vent 2	
7 Outlet 100 :	
8 Stop • 1 •	
	Cancel Cancel

*Figure 2a. Setting the filter priming cycle sequence in the modified recipe.* 

## In Figure 2b, Delta P is set to 40.0 psi.

late: 1.000 (mL/s)	Pressure: -10.0 psi	
late: 1.000 (mL/s)	Pressure: 20.0 psi	
	Rate: 1.500 (mL/s)	

*Figure 2b. Setting the recharge parameter during filter priming in the modified recipe.* 

This modified recipe replaces the Outlet cycle in step 2 referenced in the original recipe's filter priming cycle. The filter priming cycle pressurizes a liquid by using the piston in the dispense system's fill chamber. As mentioned in the introduction, displacement of air caused by liquid and effective elimination of air is expected when using this priming cycle. In addition, notice that the number of Outlet cycles in step 8 is set to 100 cycles. Setting a high number for Outlet cycles helps to ensure that air either trapped inside the membrane pores or air that adheres to the tube wall can be completely eliminated.

## EXPERIMENTAL TESTING CONDUCTED AT ENTEGRIS

Experimental testing conducted at Entegris included an IG-HV dispense system installed with an Impact<sup>®</sup> HV OF UPE 1.0  $\mu$ m filter. Pure glycerol (viscosity at approximately 1,000 cP at 20°C) was used as the dispensed fluid. Each new dry Impact HV filter was used to test the performance of each priming recipe. The tubing of the testing rig was first filled with glycerol. Note that glycerol itself is filtered by another completely wetted Impact HV 1.0  $\mu$ m to remove dissolved air bubbles. A high-speed camera was used to detect and capture instances of air bubbles in the outlet tube.

# TESTING RIG

The configuration details of the testing rig are shown in Figure 3, and the in-house laboratory results are presented in Table 1.

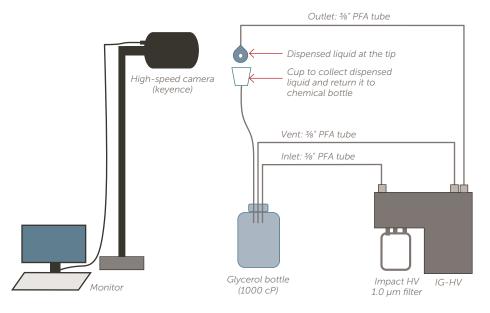


Figure 3. Configuration details of the testing rig.

The data in Table 1 reveals the extent of air bubbles in the outlet tube observed by a high-speed camera, plus the priming time and chemical amount consumed during priming of each recipe.

The testing results show that the modified priming recipe performed more favorably than the original recipe. For example, in the modified recipe, the amount of air bubbles observed in the outlet tube at the first dispense recipe is 3 compared to 60 in the original priming. At the 50th dispense, the amount of air bubbles in the modified recipe is 2 compared to 20 in the original priming.

Recipe	ORIGINAL M	ICRON RECIPE	MODIFIED MICRON RECIPE		
Dispense number	Bubble amount	Average bubble size	Bubble amount	Average bubble size	
1	60	1 mm	3	2 mm	
5	13	1 mm	7	4 mm	
10	8	1 mm	8	2 mm	
15	9	1 mm	1	1 mm	
20	8	0.05 mm	N/A	N/A	
25	6	0.05 mm	N/A	N/A	
30	5	0.05 mm	N/A	N/A	
40	7	0.02 mm	N/A	N/A	
50	20	0.02 mm	2	0.02 mm	
Priming time (min)	145	5	125		
Chemical consumed (mL)	860	)	23	00	

## Table 1. Data comparing original recipe and modified recipe results

Note: N/A refers to the situation where no bubble appears on camera monitor.

## ACTUAL PROCESS TEST RESULTS

The next step was to compare performance between the original recipe and the modified recipe as conducted on an actual wafer coating machine. Even in the actual process, the test results of the modified recipe demonstrated better performance than that of the original recipe. Figure 4 shows the data shared by Micron with Entegris.

# PIX Filter Prime Recipe Modification and Gain

- Average downtime before the prime recipe modification was approximately 15 hours
- After prime recipe modification, filter priming can be performed with 1x priming
- Prime recipe modification was very accommodating; filter startup could be performed in approximately 10 hours
- No product or defectivity issues occurred after implementing the new priming recipe

**PIX Workstation Filter Change Hours** 

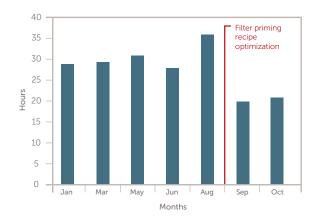


Figure 4. Real process test result obtained from the testing at Micron.

## CONCLUSION

Based on Entegris' in-house test results and Micron's actual process findings, IntelliGen High Viscosity dispense system, IG-HV, is proven to provide a workable solution for helping advanced semiconductor manufacturers greatly reduce start-up time in high viscosity filter priming operations.

## ACKNOWLEDGEMENTS

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