Effective Start up Study and Factor Analysis for Lithography Process Filter

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Toru Umeda, Shuichi Tsuzuki, Toru Numaguchi
Nihon Pall Ltd.
Ami-machi, Ibaraki, Japan,
toru_umeda@ap.pall.com

Abstract – In order to drive reduced chemical consumption, effective tool operation, effective utilization of next-generation filtration products, and environmental benefits, greater demands will be placed on the development of effective start up procedures for point-of-use filters in advanced lithography resist coating processes. The current study evaluates the effectiveness of two procedural methods, static pressure driven fluid delivery and fluid deaeration, on filter start up improvement. Further, factor analysis revealed inlet pressure to have a significant impact on filter start up quality.

INTRODUCTION

In order to reduce waste volume of lithography process chemicals, which become more expensive as lithography technology advances, and also to reduce exchange time for point-of-use filters, which can impact process cost-of-ownership, an effective filter start up method has been required by device manufacturers. The effective method is, additionally, useful to nylon 6,6 membrane filtration, which is well known to adsorb effectively microbridge precursors in photoresist[1-5], and is designed to extend contact time of fluid and filter membrane[6]. Increased filtration area or increased membrane thickness, both of which enable increased fluid contact time, also tend to increase the initial volume of air within a new filter, thereby causing the need for improved start up methods. Further, waste organic solvents are generally processed by incineration; thus, a reduction of the waste volume of photoresists used for filter start up will contribute to a reduction in environmentally discharged CO_2.

Pall Corporation has quantitatively demonstrated the effectiveness of a filter start up method, which utilizes high-flow pump dispense and application of back pressure, using a proprietary developed method for dynamically measuring the remaining air within a filter. The method has been proved to be valid in commercial wet particle measurements[7].

In this paper, new filter start up methods, such as high flow dispensing method using static gas pressure and using deaerated test fluid, are introduced. The relative significance of factors that are found to influence quick start up is quantified using the remaining air measurement method.

TEST METHOD

Method of measuring remaining air[7]

Principle. Figure 1 illustrates the test stand employed for the remaining air measurement. Normally, a tubephragm dispense pump contains check valves at the inlet and outlet. For our study, the outlet check valve was removed. During the pump suction sequence, the space between an air operative valve, downstream of the filter, and the pump cavity experiences negative pressure, due to expansion of the pump tubephragm, which also draws test fluid from an upstream tank into the pump cavity. Any air that remains in the capsule filter can be drawn into the pump cavity, thereby proportionately decreasing the volume of suctioned test fluid. Consequently, the dispense volume decreases. Also, during the pump dispense sequence, remaining air volume in the capsule filter is reduced by the positive pressure due to tubephragm squeezing. Thus, the remaining air in the capsule filter can be measured indirectly by measuring the weight of dispensed fluid.

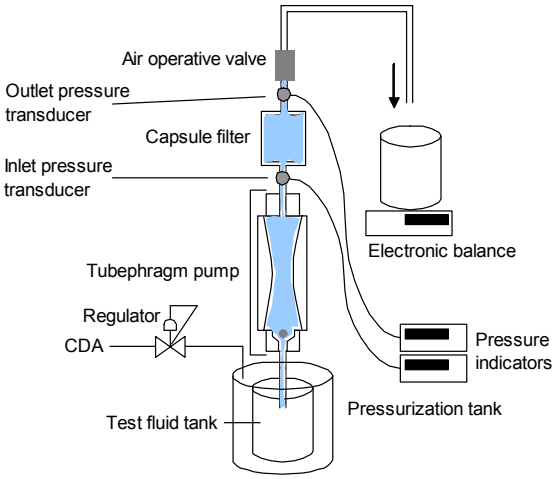


Figure 1 Test stand employed for remaining air measurement in capsule filter.

Method verification. To verify the above method, filter start up using 20 kPaG of static pressure (not using pump) was conducted. Following each 50 ml or 100 ml of pressurized test fluid delivery, pressure for the test fluid tank was reduced to atmospheric, which triggered tubephragm pump dispensing (Iwaki PDS series dispense system) at a rate of 1 ml/(2 sec). Filter inlet and outlet pressures and dispensed fluid weight were measured for each of 10 dispense cycles, and remaining air was calculated using equation (1). Test fluid employed was isopropyl alcohol (TOKUSO IPA) due to its similar surface tension to that of common photoresists. The test capsule filter was a Pall Photokleen™ EZD-2 Asymmetric P-Nylon filter with a 20 nm removal rating.

$$V_{\text{by dispense}} = -\frac{a(100+c)(100+b)}{100(b-c)} \qquad (1)$$

where: $V_{by\ dispense}$ = Remaining air volume in the capsule filter at each measurement point, calculated by dispense weight (ml).

a = Dispense volume difference between each measurement point and start up completion (ml).

b = Averaged pressure of inlet/outlet during pump suction (kPaG).

c = Averaged pressure of inlet/outlet during pump dispensing (kPaG).

Following pressure and dispensed fluid weight measurements, the capsule filter was carefully removed and its weight was measured in order to perform a complementary measurement of remaining air, as calculated in equation (2).

$$V_{by\ weight} = (B - A)/0.79 \qquad (2)$$

where: $V_{by\ weight}$ = Remaining air volume in the capsule filter at each measurement point, calculated by filter weight (ml).

A = Filter mass at each measurement point (g).

B = Filter mass at start up completion (g).

0.79 is the density of isopropyl alcohol (g/ml).

New start up procedure

Static pressure driven method. Using the test stand described in Figure 1, test fluid was delivered through the test filter using 50 kPaG of static pressure, which was applied on the test fluid tank. Again, isopropyl alcohol and a Pall 20 nm rated Asymmetric P-Nylon filter were used as test fluid and test filter, respectively. During filter start up, the remaining air in the test filter capsule was measured following each 50 ml or 100 ml of dispensed volume by means of the method described in the previous section. After the measurement, the test condition was restored to continue the filter start up procedure under static pressure. The test was continued for a total throughput of 1000 ml. The test was then repeated at a static pressure of 100 kPaG. For comparison fluid dispense was conducted using a similarly modified tubephragm pump (i.e., No check valve at pump outlet), but with an air operative valve at the filter outlet, which functioned as check valve.

Method using deaerated fluid. A start up procedure using deaerated isopropyl alcohol as a test fluid was also studied. The test fluid was deaerated in -50 kPaG for 60 minutes before the testing. The same filter, with 20 nm rated asymmetric nylon 6,6 membrane, was employed as test filter. The remaining air reduction was evaluated during static pressure driven start up method utilizing 20 kPaG of inlet pressure. Dissolved gas volumes were estimated[8] as 0.19 and 0.39 cm^3 gas/cm^3 liquid, in the deaerated and non-deaerated fluids, respectively. The start up completion point was defined as the throughput volume for which 95% of maximum dispense rate was recovered *.

*Filter start up completion: Following the start up evaluation, an additional 1000 ml of test fluid was delivered via 100 kPaG static pressure to ensure complete air displacement from the test filter. Fluid dispense weight is measured more than two times during this delivery to confirm the absence of further air purge from within the test filter.

Factor analysis for static pressure driven method.

A detailed study was conducted for factor analysis in static pressure driven methods. Table 1 shows test conditions. Each test was repeated twice to confirm reproducibility.

Table 1 Test conditions for factor analysis

	unit	1	2	3	4
Inlet pressure	kPaG	20	70	70	107
Outlet pressure	kPaG	0	50	0	13
Flow rate	ml/sec	0.5	0.5	1.75	2.4

RESULTS AND DISCUSSIONS

Method verification for measuring method for remaining air.

The relationship between the two remaining air measurement methods is shown in Figure 2. The results for "calculated by dispensing weight" method, which is employed in this paper, agreed with that for "calculated by measured filter weight" method, which intrinsically expresses remaining air volume. Based on the results, it was verified that measurement of the dispensing weight in the former method accurately represents remaining air in the capsule filter.

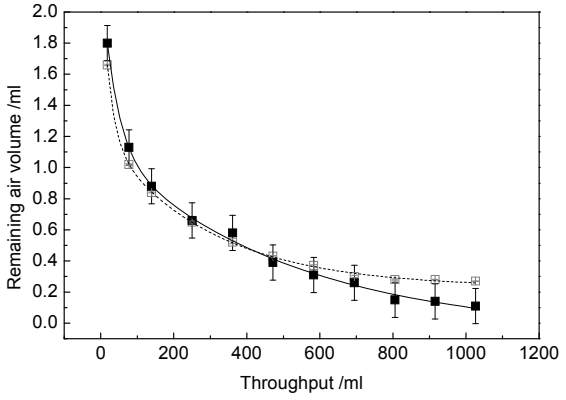


Figure 2 Verification of remaining air measuring method. At 20kPaG static pressure, using IPA, with pump dispense rate of 1ml / 2sec. □: Calculated by dispensing liquid weight, standard deviation is expressed by size of data point marker (open box). ■ : Calculated by measured filter weight, error bars express standard deviation.

New start up procedure

Static pressure driven method. Figure 3 shows results of the static pressure driven start up method. The difference in actual dispense volume from the dispense pump set value (= 1 ml) expresses remaining air in test filter, as described in previous section. In general, the pressure driven methods effected a more rapid filter start up than the tubephragm pump driven method. Moreover, increasing pressure was found to increase the effectiveness of the static pressure driven method.

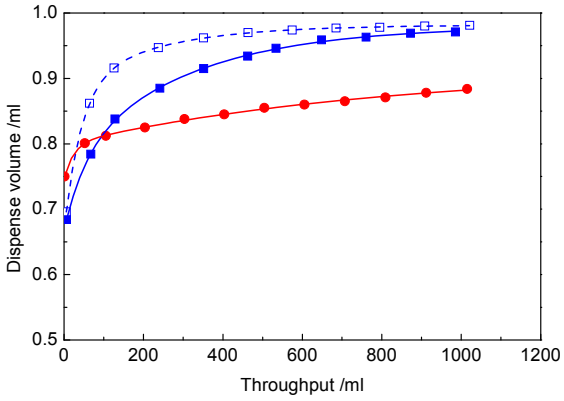


Figure 3 Dispense volume comparison among filter start up methods: ● Tubephragm pump driven method, ■ Static pressure driven method-1 (50 kPaG), □ Static pressure driven method-2 (100 kPaG).

In the pump driven method, negative pressure is observed at the pump outlet during the suction operation, as shown in Figure 4. Under this condition, gas dissolved within the test fluid under atmosphere pressure precipitates, which increases gas phase volume, and ultimately, decreases the fluid dispense volume.

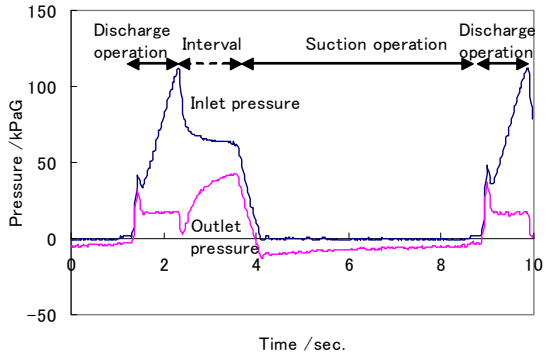


Figure 4 Pressure change during tubephragm pump operation.

Method using deaerated fluid. Table 2 lists throughput volumes required for start up completion (i.e., 95% recovery of commanded dispense rate). Fluid consumption for filter start up using deaerated test fluid was approximately ⅓ that of non-deaerated test fluid.

Table 2 Effect of deaerated fluid for filter start up

Test fluid condition	Estimated dissolved gas volume	Throughput for 95% recovery of dispense rate
	cm^3 gas (STPD)/cm^3 liquid	ml
No deaeration	0.39	612 ± 109
Deaeration	0.19	190 ± 21

Factor analysis of static pressure driven method.

High dispense flow rate and application of back pressure were found to be effective to improve filter start up in previous work[1]. A detailed study was conducted for factor analysis. Figure 5b shows the flow rate of each test run. It is well-known that flow rate of a fluid passing through a filter is proportional to the pressure gradient, according to the Kozeny-Carman equation, expressed as equation (3). (Here, right-side parameters other than P are constant).

$$q = \left\{\frac{\varepsilon^3}{(1-\varepsilon)^2}\right\}\left\{\frac{1}{(S_0^2 \cdot K_c)}\right\}\left\{\frac{P}{(\mu \cdot L)}\right\} \qquad (3)$$

where: q = flow rate

ε = porosity

S_0 = specific surface area

K_c = Kozeny constant

P = pressure gradient

μ = fluid viscosity

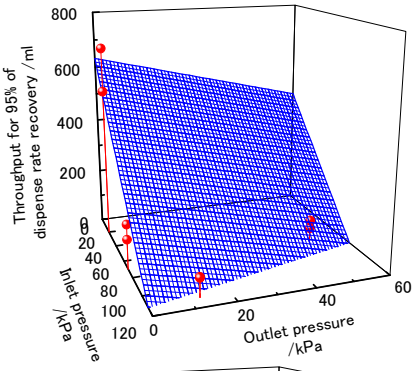
L = thickness of porous layer

In response, a fitted regression equation of the flow rate plane (Figure 5b) indicates the flow rate is proportional to the difference between inlet pressure and outlet pressure (pressure gradient), consistent with the Kozeny-Carman equation. The relation confirms that flow rate, inlet pressure, and outlet pressure are not independent of each other. Figure 5a shows throughput for the filter start up (until dispense rate recovers to 95% of air removal completion) in the condition described in Figure 5b. The required throughput plane fitted expresses, according to the regression equation indicated in the Figure 5, that an increase of both inlet and outlet pressures will cause throughput required for start up completion to decrease. Moreover, the effect of inlet pressure is almost 2× the effect of outlet pressure.

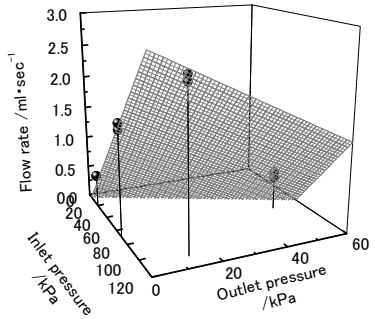
$$\text{Throughput for 95\% of dispense rate recovery} = 680 - 6.2 \times \text{Inlet pressure} - 3.2 \times \text{Outlet pressure}$$

$$R^2 = 0.86$$

(a)



(b)



$$\text{Flow rate} = 0.025 \times \text{Inlet pressure} - 0.025 \times \text{Outlet pressure}$$

Figure 5 (a) Throughput for 95% of dispense rate recovery against inlet pressure and outlet pressure, (b) Flow rate against inlet pressure and outlet pressure in the static driven pressure method.

CONCLUSION

A static pressure driven fluid delivery method was found to be more effective for rapid filter start up than a pump dispensing method. Further, within the static pressure driven methodology, increase of both inlet and outlet pressures causes a decrease in required throughput, with inlet pressure having a greater effect than outlet pressure. Moreover, deaeration of test fluid was found to reduce further the total throughput volume required to achieve complete filter start up.

Many factors, such as process fluid chemical properties, dispense system, process conditions, process defect tolerance, and filter membrane material will influence the rate and quality of process filter start up. Thus, the filter start up procedure should be optimized according to each specific process. Effective start up procedures that consider contributing factors, including those identified in this study, will help to provide direction for procedure optimization efforts.

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