Solvent pre-wetting as an effective start-up method for point-of-use filter

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ABSTRACT

An effective filter start-up method has been required by device manufacturers, mainly in order to reduce waste volume of lithography process chemicals, which become more expensive as lithography technology advances. Remaining air was monitored during static-pressure-driven filter start-up. As a result, 3500 ml of the resist was needed to eliminate remaining air. For improvement, cyclohexanone pre-wetting was applied prior to the resist introduction. As a result, the resist volume needed for the solvent displacement was 1900 ml, approximately half the volume required for static-pressure-driven start-up. Other solvents were evaluated for the pre-wetting start-up method. Results, in descending order of performance were PGME (best) < PGMEA = IPA < cyclohexanone (worst). Moreover, air displacement performance strongly correlated with Hansen solubility parameter distance between each solvent and nylon 6,6 material.

Keywords: Filter start-up, Pre-wetting, Microbridge, Hansen solubility parameter, Deaeration

1. INTRODUCTION

An effective filter start-up method has been required by device manufacturers, mainly in order to reduce waste volume of lithography process chemicals, which become more expensive as lithography technology advances. Pall Corporation has quantitatively demonstrated the effectiveness of filter start-up methods, such as static-pressure-driven method, application of back pressure, and deaeration of the fluid, using a proprietary developed method for dynamically measuring the remaining air within a filter.[1,2] In this paper, the effectiveness of pre-wetting a Pall Photokleen™ EZD-2 point-of-use (POU) filter with asymmetric nylon 6,6 membrane rated at 20 nm (P/N: PHD11ANMEH11) with solvent, prior to the introduction of Fujifilm topcoat-less resist (FAiRS-PA01), and a study of solvent optimization for pre-wetting and effects on litho process defectivity are reported. Effects of deaeration applied to pre-wetting solvent are also reported.

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DOI: 10.1117/12.916026

2. EXPERIMENTAL

2.1 Method of measuring remaining air

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 additional pressure due to tubephragm squeezing. Thus, the remaining air in the capsule filter can be measured indirectly by measuring the weight of dispensed fluid. Actual remaining air volume was calculated using volume difference of each data point from that of start up completion and inlet and outlet pressures at each data point and shown in equation (1).

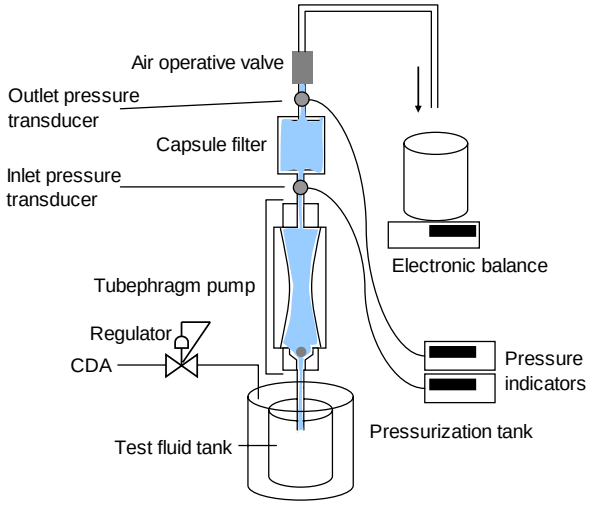

Figure 1. Test stand employed for remaining air measurement in capsule filter.

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

where: $V_{\text{by dispense}}$ = 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 filter inlet and outlet during pump suction (kPag).

c = Averaged pressure of filter inlet and outlet during pump dispensing (kPag).

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2.2 Static-pressure-driven filter start-up method

After filter installation into the start-up test stand, shown in Figure 1, a static air pressure was applied to test fluid within a test fluid tank on the upstream side of the test filter. Then, an air-operated valve, located downstream of the test filter, was opened in order to start fluid flow. Remaining air measurements, described in section 2.1, were collected at discrete throughput intervals, such as 50 ml, 100 ml, ···. The pressure on the test fluid was released to atmosphere during each measurement. In a previous study, the static-pressure-driven method was found to be more effective for eliminating remaining air than a pump-driven method (70% reduction in fluid consumed).[2]

2.3 Solvent pre-wetting

A trend of the remaining air was monitored during static-pressure-driven filter start-up, which is a method commonly implemented among device manufactures. Fujifilm topcoat-less immersion resist (FAiRS-PA01) and Pall Photokleen EZD2 filter with asymmetric nylon 6,6 membrane rated at 20 nm (PHD11ANMEH11) were tested for the experiment. The applied static pressure was 50 kPag. In an attempt to improve start-up methodology further, the test filter was pre-wetted with a compatible prior to resist introduction. In this case, the total resist consumption was equal to an amount needed to sufficiently displace the pre-wetting solvent from the filter capsule volume, as evaluated via spin thickness recovery. Cyclohexanone, which was confirmed not to affect the solubility of the resist polymer, was used for initial testing.

After-development inspection (ADI), focusing on microbridge defects, was also performed using 45 nm half pitch line and space pattern immersion lithography, to compare practical effect of the solvent pre-wetting. Cyclohexanone was used as pre-wetting solvent. Fujifilm FAiRS-PA01 topcoat-less immersion resist and Pall Photokleen EZD2 filter with asymmetric nylon 6,6 membrane rated at 20 nm (PHD11ANMEH11) were used in the generation of ADI results.

2.4 Study for appropriate solvent

Other solvents were evaluated for the pre-wetting start-up method. A pre-wetting solvent that can easily displace air within the filter capsule is preferable. Performances of cyclohexanone, propylene glycol monomethyl ether acetate (PGMEA), propylene glycol monomethyl ether (PGME), and isopropanol (IPA) were compared for the same test filter (PHD11ANMEH11) and resist (FAiRS-PA01). As with cyclohexanone, the static-pressure-driven filter start-up method at 50 kPag was used for testing. Then, comparisons were made between start-up performance and chemical properties of each solvent, in order to identify a direction for further start-up method improvement.

2.5 Hansen solubility parameters[3]

Hansen solubility parameters (HSP) describe affinity between materials using a multidimensional vector that quantifies non-polar atomic interactions, permanent dipole-permanent dipole molecular interactions, and hydrogen bonding molecular interactions.[3] The affinity between two materials is described as HSP distance which is defined in equation (2).

$$(Ra)^2 = 4(\delta_{d2} - \delta_{d1})^2 + (\delta_{p2} - \delta_{p1})^2 + (\delta_{h2} - \delta_{h1})^2 \qquad (2)$$

where

Ra = Distance in HSP space between material 1 and material 2 ($MPa^{1/2}$)

δ_{x1} = Hansen parameter for material 1 ($MPa^{1/2}$)

δ_{x2} = Hansen parameter for material 2 ($MPa^{1/2}$)

subscripts, x: d = dispersion component, p = polar component, h = hydrogen bonding component

Hansen solubility parameters for materials used in the various tests and HSP distance between each solvent and nylon 6,6, calculated using equation (2), are shown in Table 1.

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Table 1. Hansen solubility parameters and HSP distance (Ra) between each solvent and nylon 6,6

Material	δ / $MPa^{1/2}$			Ra / $MPa^{1/2}$
	δ_d	δ_p	δ_h	
Cyclohexanone	17.8	6.3	5.1	7.4
PGMEA	15.6	5.6	9.8	6.3
PGME	15.9	4.6	11.5	5.3
IPA	16.1	5.5	16.4	6.3
Nylon 6,6	18.5	5.1	12.3	0.0

2.6 Solvent deaeration

Filter start-up improvement using deaerated IPA was found in previous study.[2] In the current study, deaeration was applied to a practical pre-wetting solvent. Prior to testing, the pre-wetting solvent was deaerated in a stainless-steel tank at -50 kPag using a vacuum pump for 60 minutes. Remaining air measurements during static-pressure-driven filter start-up were collected using the method described in sections 2.1 and 2.2. Applied static pressure was 50 kPag. Cyclohexanone, a common practical solvent used in lithography process, and a Pall Photokleen EZD2 filter with asymmetric nylon 6,6 membrane rated at 20 nm (PHD11ANMEH11) were employed for the testing. The testing was performed twice to check reproducibility.

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3. RESULTS AND DISCUSSIONS

3.1 Solvent pre-wetting

Test result for filter start-up using Fujifilm FAiRS-PA01 topcoat-less immersion resist is shown in Figure 2. As a result, 3500 ml of the resist was needed to eliminating remaining air for the commonly-used static-pressure-driven method.

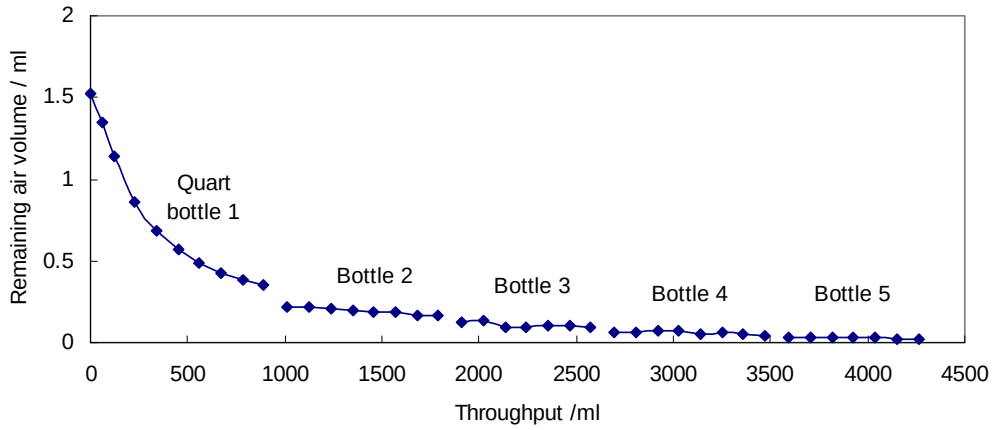

Figure 2. Remaining air in capsule filter vs. resist throughput during static-pressure-driven start-up method. Static pressure applied was 50 kPag. Test fluid is Fujifilm FAiRS-PA01 topcoat-less immersion resist and test filter is Pall PHD11ANMEH11 asymmetric nylon 6,6 20 nm rated filter.

For improvement, solvent pre-wetting was applied prior to resist installation. In this case, the total resist consumption was equal to an amount needed to sufficiently displace the pre-wetting solvent from the filter capsule volume, as evaluated via spin thickness recovery, because film thickness is reduced using a resist diluted by remaining pre-wetting fluid in the filter while not by remaining air in no pre-wetting, shown in Figure 3. As a result, the resist volume needed for the solvent displacement was 1900 ml, approximately half the volume required for static-pressure-driven start-up in Figure 2. The comparison is also summarized in Table 2.

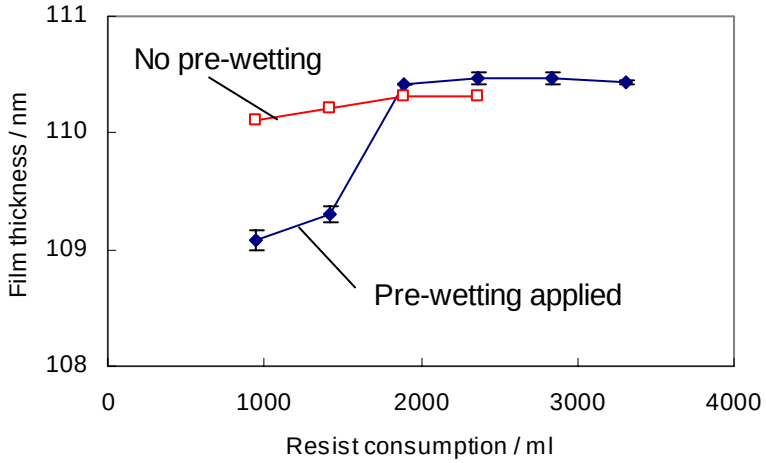

Figure 3. Resist spin film thickness vs. resist purge volume after solvent pre-wetting. Data for the standard process (no pre-wetting) are also plotted to confirm recovery of film thickness. Error bars indicate the range of data values for two sample runs. Cyclohexanone was used for pre-wetting solvent. Resist was Fujifilm FAiRS-PA01 topcoat-less immersion resist. Test filter was Pall PHD11ANMEH11 asymmetric nylon 6,6 20 nm rated filter.

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Table 2. Summary of pre-wetting effectiveness in filter start-up

Start-up method	Resist consumption	Notes
No pre-wetting applied	3500 ml	Resist displaces air
Solvent pre-wetting applied	1900 ml	Resist displaces pre-wetting solvent

ADI was also performed to compare practical effect of filter start-up improvement. Figure 4 shows the results of microbridge defect counts on wafer. As a result, solvent pre-wetting caused a reduction in defect levels that were observed during the standard start-up method (i.e., no pre-wetting), especially 2 to 3 days after filter installation. A mechanism to explain the difference in results is hypothesized, whereby a population of nearly-insoluble component fractions aggregated around remaining air in the filter and formed gel-like particles, which manifest as microbridge defects on wafer. This component was essentially added to migrate toward the surface driven by its lower surface energy to achieve repelling surface for subsequent immersion water[4]. But in filter start up, the component fraction may be aggregated around bubbles which can be regarded as a small surface. During subsequent normal equipment operation (i.e., dummy dispense, wafer processing), remaining air and defect-causing gels are displaced over the next 48 hours. Also, the fact that excursion started on day 2—not on day 1, immediately after filter installation—suggests that gel formation occurred over a period of about 24 hours.

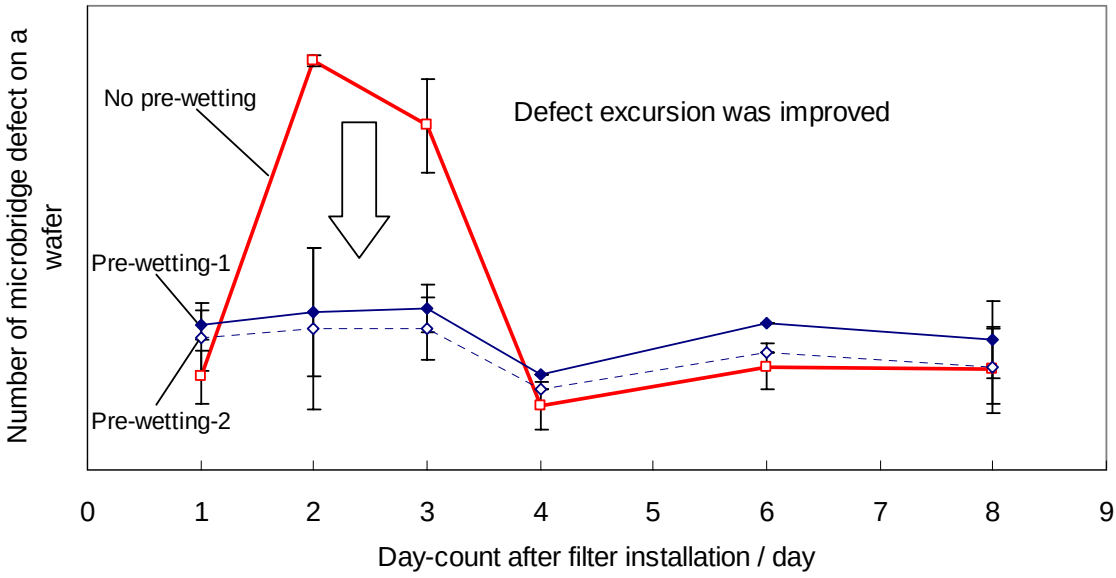

Figure 4. Trend plots of microbridge defect count for 45 nm half pitch line and space pattern, after filter installation, for various extents of solvent pre-wetting. Error bars indicate standard deviations of multiple inspections collected each day. Resist was Fujifilm FAiRS-PA01 topcoat-less immersion resist. Test filter was Pall PHD11ANMEH11 asymmetric nylon 6,6 20 nm rated filter.

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3.2 Study for appropriate solvent

Other solvents were evaluated for the pre-wetting start-up method. A pre-wetting solvent that can easily displace air within the filter capsule is preferable. Results, in descending order of performance (remaining air after 700 ml throughput) were PGME (best) < PGMEA = IPA < cyclohexanone (worst), as shown in Figure 5. Moreover, air displacement performance strongly correlated with Hansen solubility parameter (HSP) distance between each solvent and nylon 6,6 material. Based on the results, a solvent that has a smaller HSP distance to nylon 6,6 is recommended as a pre-wetting fluid, if the solvent satisfies other requirements for the process.

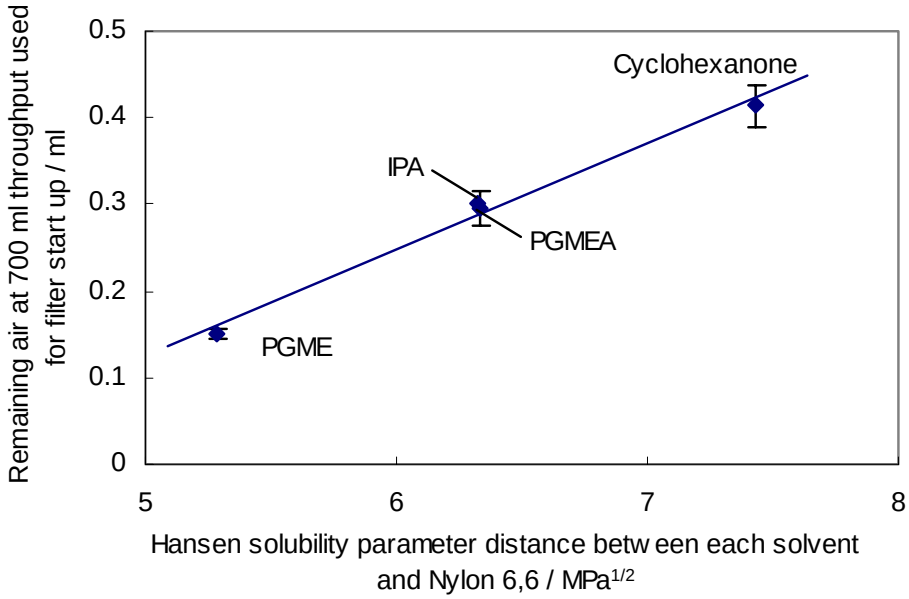

Figure 5. Correlation of remaining air volume after 700 ml throughput during filter start-up to Hansen solubility parameter distance between solvent and filter membrane, Ra. Start-up method: 50 kPag static-pressure-driven. Error bars indicate the range of data values for two sample runs.

3.3 Solvent deaeration

Figure 6 shows a comparison of remaining air in static-pressure-driven filter start-up method for both untreated and deaerated pre-wetting solvent. Using deaerated cyclohexanone, a significant reduction of remaining air was observed at each measurement point. Thus, the effectiveness of using deaerated fluid for filter start-up was confirmed in a practical solvent, in addition to previous work, which used a simulated solvent of IPA.

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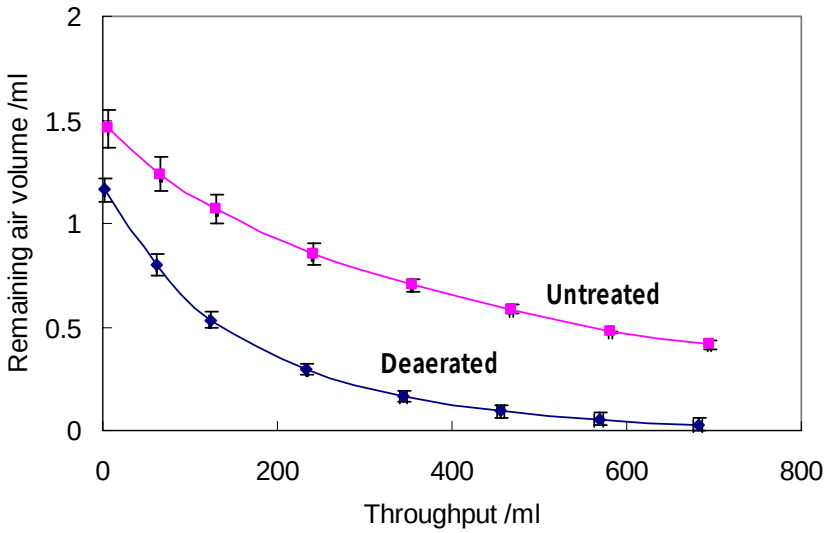

Figure 6. Remaining air in capsule filter vs. fluid throughput during static-pressure-driven start-up method. Static pressure applied was 50 kPag. Test fluids are deaerated and untreated cyclohexanone. Test filter is Pall PHD11ANMEH11 asymmetric nylon 6,6 20 nm rated filter. Error bars indicate the range of data values for two sample runs.

4. CONCLUSION

Based on results of the current study, a 50% reduction in resist consumption during filter start-up was realized after pre-wetting the filter with a practical, compatible solvent. Approximately 1900 ml of resist were required to displace pre-wetting solvent and recover spin film thickness, compared to approximately 3500 ml that were consumed during direct displacement of air. Also, it is suspected that air remaining within the filter may play a role in microbridge defect generation, and that solvent pre-wetting can be used to eliminate transient microbridge defect excursion, which is observed within 48 hours of filter change-out. Various solvents were studied for nylon 6,6 filter pre-wetting, and a strong correlation was found between air displacement performance of a solvent and its Hansen distance to nylon 6,6. Further, deaeration, which was previously found to enhance filter start-up performance of an ideal solvent (IPA) was found to be similarly effective in an actual resist solvent, cyclohexanone.

In this study, defect mode attributed to filter remaining air was microbridge excursion. The mode may vary with fluid chemistry, filter membrane material, and other process conditions. Solvent pre-wetting prior to resist installation is recommended for reduction of defects that are attributable to remaining air. The appropriate solvent for pre-wetting should have a small Hansen distance to the filter membrane material. Further, deaeration of pre-wetting solvent is recommended to reduce both maintenance time and pre-wetting solvent consumption.

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