Bubbles, which can translate to wafer defects, are critical in the lithography process. While bubbles smaller than 100 µm can be completely dissolved into the resist, larger diameter bubbles (typically >300 µm diameter), cannot be fully adsorbed by the resist and the bubbles can remain even after one hour. Sensing bubbles of this size has become a critical challenge within lithography processes.

To address this challenge, Entegris has developed an integrated flowmeter and dispense system with robust air-monitoring software. The software utilizes the principle of heat conduction in different media combined with an exact copy theory to detect the existence of air bubbles. This software is called “Flow Profile Compare.”

In-house testing has proven that the Flow Profile Compare monitoring software is sensitive to air bubbles as small as ~ 800 µm in diameter. Combining the flowmeter with this software into your production line would prevent the adversities caused from air bubbles in photochemicals.

**Principle of Flowmeter and Flow Profile Compare**

The flowmeter has a sensor that measures the temperature gradient at two points. Due to a correspondence of liquid flow and temperature gradient, the flowmeter can convert the observed temperature gradient into liquid flow rate. When bubbles pass through the temperature sensor during a measuring time, the sensor can sense the distorted temperature gradient and output the flow rate profile signal that is different from one where there are no bubbles. Afterwards, the dispense system compares the difference in the two flow rate profiles. If the difference exceeds the alarm setting limit, the dispense system will alert the alarm.

There are two types of alarms, a warning alarm and an error alarm. A warning alarm is alerted when the measured value exceeds the set warning limit, and an error alarm alerts when the measured value exceeds the set error limit. A warning alarm tells users that the measured value is close to an unacceptable level, while the error alarm tells that the measured value has reached the unacceptable level. Both warning and error alarms can send the signal to the coater developer and halt the process and prevent defective wafers. Users can select what type of alarm they want to use to halt the process. For details please see Entegris application note Confirmation tools – Advanced Monitoring of Photolithography Dispense Operation (IG-LV, IG-ULV and IG-MV).
Figure 1 shows how Flow Profile Compare works in case the error alarm is selected to halt the production process.

Figure 1. Diagram demonstrating how flowmeter works as a confirmation tool.

**EXPERIMENT**

Testing was performed in the Entegris facility using an IntelliGen® LV pump with firmware “Released V1005_987” installed with Impact® 2 OF UPE 3 nm filter. Isopropyl Alcohol (IPA) and 193 nm BARC were used as the process fluids.

Air bubble events were created in order to evaluate the sensitivity of the flowmeter and its software. A flow profile was compared for both IPA and 193 nm BARC.

Air bubbles of the following sizes, 0.001 mL to 0.009 mL in 0.001 mL incremental, 0.01 mL to 0.09 mL in 0.01 mL incremental and 0.1 mL were injected into the outlet tubing via the injection port made of a T-Connector. The bubble is controlled to flow through the flow sensor at the same time the sensor measures the liquid flow rate. Flow Profile compare value is observed and recorded.

**Tubing configuration**

<table>
<thead>
<tr>
<th>Location</th>
<th>ID × length</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bottle to pump inlet</td>
<td>¼” 500 mm</td>
</tr>
<tr>
<td>Pump outlet to air injection port</td>
<td>¼” 50 mm</td>
</tr>
<tr>
<td>Pump outlet to flow sensor</td>
<td>¼” 500 mm</td>
</tr>
<tr>
<td>Flow sensor to air operating valve</td>
<td>¼” 700 mm</td>
</tr>
<tr>
<td>Air operating valve to nozzle</td>
<td>2 mm × 1000 cm</td>
</tr>
<tr>
<td>Vent line</td>
<td>2 mm × 50 cm</td>
</tr>
</tbody>
</table>
RESULT AND RESULT DISCUSSION

Table 1 summarizes the Flow Profile Compare data percentages. The lower the percentage, the more sensitive it is to air bubbles. Table 1 shows that with an appropriate setting of dispense volume and dispense rate, the flow profile compare can give a percentage value lower than 70%. In general, the alarm limit is set at 80% for Warning and at 70% for Error. The test results prove that a dispense pump can alert an Error alarm in the presence of the smallest bubble, 0.001 mL or diameter of 0.8 mm (800 µm) in size. This data confirms that adding a flow meter into the production line helps prevent air bubbles from being dispensed onto wafers.

Table 1. Summary of percentage of flow profile compare

<table>
<thead>
<tr>
<th>Bubble size</th>
<th>Flow compare</th>
<th>Dispense volume (DV) and dispense rate (DR)</th>
<th>Flow compare</th>
<th>Dispense volume (DV) and dispense rate (DR)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.001 mL</td>
<td>62% DV 1.7 mL DR 0.35 mL/sec</td>
<td>96% DV 0.7 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.002 mL</td>
<td>31% DV 0.5 mL DR 0.15 mL/sec</td>
<td>32% DV 0.5 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.003 mL</td>
<td>14% DV 0.3 mL DR 0.15 mL/sec</td>
<td>26% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.004 mL</td>
<td>27% DV 0.3 mL DR 0.15 mL/sec</td>
<td>53% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.005 mL</td>
<td>27% DV 0.3 mL DR 0.15 mL/sec</td>
<td>38% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.006 mL</td>
<td>58% DV 0.3 mL DR 0.15 mL/sec</td>
<td>17% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.007 mL</td>
<td>34% DV 0.3 mL DR 0.15 mL/sec</td>
<td>35% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.008 mL</td>
<td>38% DV 0.3 mL DR 0.15 mL/sec</td>
<td>33% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.009 mL</td>
<td>52% DV 0.3 mL DR 0.15 mL/sec</td>
<td>33% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.01 mL</td>
<td>55% DV 0.3 mL DR 0.15 mL/sec</td>
<td>33% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.02 mL</td>
<td>34% DV 0.3 mL DR 0.15 mL/sec</td>
<td>36% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.03 mL</td>
<td>34% DV 0.3 mL DR 0.15 mL/sec</td>
<td>42% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.04 mL</td>
<td>38% DV 0.3 mL DR 0.15 mL/sec</td>
<td>41% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.05 mL</td>
<td>39% DV 0.3 mL DR 0.15 mL/sec</td>
<td>44% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.06 mL</td>
<td>44% DV 0.3 mL DR 0.15 mL/sec</td>
<td>37% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.07 mL</td>
<td>37% DV 0.3 mL DR 0.15 mL/sec</td>
<td>38% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.08 mL</td>
<td>44% DV 0.3 mL DR 0.15 mL/sec</td>
<td>46% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.09 mL</td>
<td>42% DV 0.3 mL DR 0.15 mL/sec</td>
<td>44% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.1 mL</td>
<td>44% DV 0.3 mL DR 0.15 mL/sec</td>
<td>43% DV 0.3 mL DR 0.15 mL/sec</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Note: The lower the percentage, the more sensitive to air bubbles.
Figures 3 through 21 summarize the differences between reference flow rate profile (dispense without air bubble) and the flow rate profile of the dispense with air.

Reference of 0.001 mL, 193 nm BARC
Dispense Volume = 1.7 mL Dispense Rate = 0.35 mL/s

Reference of 0.001 mL, IPA
Dispense Volume = 0.7 mL Dispense Rate = 0.15 mL/s

0.001 mL Air, 193 nm BARC, 62%
Dispense Volume = 1.7 mL Dispense Rate = 0.35 mL/s

0.001 mL Air, IPA, 96%
Dispense Volume = 0.7 mL Dispense Rate = 0.15 mL/s

Top: Reference flow rate profile for 0.001 mL in 193 nm BARC
Bottom: Reference flow rate profile for 0.001 mL air in IPA

Figure 3.

Top: Flow rate profile of the dispense with 0.001 mL air in 193 nm BARC resulting in 62% flow profile compare
Bottom: Flow rate profile of the dispense with 0.001 mL air in IPA resulting in 96% flow profile compare
Reference of 0.002 mL, 193 nm BARC
Dispense Volume = 0.5 mL Dispense Rate = 0.15 mL/s

Top: Reference flow rate profile for 0.002 mL in 193 nm BARC
Bottom: Reference flow rate profile for 0.002 mL air in IPA

Figure 4.

Reference of 0.003 mL, 193 nm BARC
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Reference flow rate profile for 0.003 mL in 193 nm BARC
Bottom: Reference flow rate profile for 0.003 mL air in IPA

Figure 5.
Reference of 0.004 mL, 193 nm BARC
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Reference flow rate profile for 0.004 mL in 193 nm BARC
Bottom: Reference flow rate profile for 0.004 mL air in IPA

Figure 6.

Reference of 0.005 mL, 193 nm BARC
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Reference flow rate profile for 0.005 mL in 193 nm BARC
Bottom: Reference flow rate profile for 0.005 mL air in IPA

Figure 7.
Reference of 0.006 mL, 193 nm BARC
Dispense Volume = 0.3 mL Dispen Rate = 0.15 mL/s

0.006 mL, 193 nm BARC, 58%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Reference flow rate profile for 0.006 mL in 193 nm BARC
Bottom: Reference flow rate profile for 0.006 mL air in IPA

Figure 8.

Reference of 0.006 mL, IPA
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

0.006 mL Air, IPA, 17%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Flow rate profile of the dispense with 0.006 mL air in 193 nm BARC resulting in 58% flow profile compare
Bottom: Flow rate profile of the dispense with 0.006 mL air in IPA resulting in 17% flow profile compare

Reference of 0.007 mL, 193 nm BARC
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

0.007 mL, 193 nm BARC, 34%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Reference flow rate profile for 0.007 mL in 193 nm BARC
Bottom: Reference flow rate profile for 0.007 mL air in IPA

Figure 9.

Reference of 0.007 mL, IPA
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

0.007 mL Air, IPA, 35%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Flow rate profile of the dispense with 0.007 mL air in 193 nm BARC resulting in 34% flow profile compare
Bottom: Flow rate profile of the dispense with 0.007 mL air in IPA resulting in 35% flow profile compare
0.008 mL Air, 193 nm BARC, 38%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

0.008 mL Air, IPA, 33%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

0.009 mL Air, 193 nm BARC, 52%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

0.009 mL Air, IPA, 33%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Flow rate profile of the dispense with 0.008 mL air in 193 nm BARC resulting in 38% flow profile compare
Bottom: Flow rate profile of the dispense with 0.008 mL in IPA resulting in 33% flow profile compare

Figure 10.

Top: Flow rate profile of the dispense with 0.009 mL air in 193 nm BARC resulting in 52% flow profile compare
Bottom: Flow rate profile of the dispense with 0.009 mL air in IPA resulting in 33% flow profile compare

Figure 11.
Reference of 0.01 mL, 193 nm BARC
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Reference of 0.01 mL, IPA
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Reference flow rate profile for 0.01 mL in 193 nm BARC
Bottom: Reference flow rate profile for 0.01 mL air in IPA

Figure 12.

0.01 mL Air, 193 nm BARC, 55%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

0.01 mL Air, IPA, 33%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Flow rate profile of the dispense with 0.01 mL air in 193 nm BARC resulting in 55% flow profile compare
Bottom: Flow rate profile of the dispense with 0.01 mL air in IPA resulting in 33% flow profile compare

Figure 13.

Reference of 0.02 mL, 193 nm BARC
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Reference of 0.02 mL, IPA
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Reference flow rate profile for 0.02 mL in 193 nm BARC
Bottom: Reference flow rate profile for 0.02 mL air in IPA

Figure 13.

0.02 mL Air, 193 nm BARC, 34%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

0.02 mL Air, IPA, 36%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Flow rate profile of the dispense with 0.02 mL air in 193 nm BARC resulting in 34% flow profile compare
Bottom: Flow rate profile of the dispense with 0.02 mL air in IPA resulting in 36% flow profile compare
Figure 14.

- Top: Reference flow rate profile for 0.03 mL in 193 nm BARC
- Bottom: Reference flow rate profile for 0.03 mL air in IPA

Figure 15.

- Top: Reference flow rate profile for 0.04 mL in 193 nm BARC
- Bottom: Reference flow rate profile for 0.04 mL air in IPA
Reference of 0.05 mL, 193 nm BARC
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Reference of 0.05 mL, IPA
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Reference flow rate profile for 0.05 mL in 193 nm BARC
Bottom: Reference flow rate profile for 0.05 mL air in IPA

Figure 16.

0.05 mL, 193 nm BARC, 38%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

0.05 mL, IPA, 44%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Flow rate profile of the dispense with 0.05 mL air in 193 nm BARC resulting in 38% flow profile compare
Bottom: Flow rate profile of the dispense with 0.05 mL air in IPA resulting in 44% flow profile compare

Figure 17.

Reference of 0.06 mL, 193 nm BARC
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Reference of 0.06 mL, IPA
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Reference flow rate profile for 0.06 mL in 193 nm BARC
Bottom: Reference flow rate profile for 0.06 mL air in IPA

Figure 17.

0.06 mL Air, 193 nm BARC, 44%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

0.06 mL Air, IPA, 37%
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Top: Flow rate profile of the dispense with 0.06 mL air in 193 nm BARC resulting in 44% flow profile compare
Bottom: Flow rate profile of the dispense with 0.06 mL air in IPA resulting in 37% flow profile compare
Reference of 0.07 mL, 193 nm BARC
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Reference of 0.08 mL, 193 nm BARC
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Reference of 0.07 mL, IPA
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Reference of 0.08 mL, IPA
Dispense Volume = 0.3 mL Dispense Rate = 0.15 mL/s

Fig 18. Top: Reference flow rate profile for 0.07 mL in 193 nm BARC
Bottom: Reference flow rate profile for 0.07 mL air in IPA

Fig 19. Top: Reference flow rate profile for 0.08 mL in 193 nm BARC
Bottom: Reference flow rate profile for 0.08 mL air in IPA

Top: Flow rate profile of the dispense with 0.07 mL air in 193 nm BARC resulting in 38% flow profile compare
Bottom: Flow rate profile of the dispense with 0.07 mL air in IPA resulting in 38% flow profile compare

Top: Flow rate profile of the dispense with 0.08 mL air in 193 nm BARC resulting in 44% flow profile compare
Bottom: Flow rate profile of the dispense with 0.08 mL air in IPA resulting in 46% flow profile compare
**Figure 20.**

Top: Reference flow rate profile for 0.09 mL in 193 nm BARC resulting in 42% flow profile compare
Bottom: Reference flow rate profile for 0.09 mL air in IPA resulting in 43% flow profile compare

**Figure 21.**

Top: Reference flow rate profile for 0.10 mL in 193 nm BARC resulting in 44% flow profile compare
Bottom: Reference flow rate profile for 0.10 mL air in IPA resulting in 43% flow profile compare
Figure 22 compares the sizes of bubbles that can be detected by a liquid particle counter (left), Flow Profile Compare Confirmation tool (middle), and Maximum Pressure Confirmation tool (right). It was confirmed in an Entegris laboratory that with the integration of flowmeter technology, bubble detection improves 20 times compared to pressure sensor based technology. We can see that the smallest bubble detected by Flow Profile Compare is 0.001 mL in volume, while the smallest bubble detected by Maximum Pressure is 0.02 mL. However, users must be aware that the air bubble must pass through the flowmeter to be detected by Flow Profile Compare. On the other hand, the air bubble does not need to pass through the pressure sensor to be detected by Maximum Pressure, it just needs to exist in the outlet line. This functionality differentiates the Flow Profile Compare Confirmation tool from the Maximum Pressure Confirmation tool.

Size of Detectable Bubble by Various Confirmation Tools

- **Minimum detectable bubble size**
  - **Volume < 0.001**
    - Ø < 0.8 mm (0.03”)
  - **Volume = 0.001**
    - Ø ≈ 0.8 mm (0.03”)
  - **Volume = <0.02 mL**
    - Ø ≈ 2.5 mm (0.1”)

- **Metrology**
  - Liquid particle counter
    - High-speed camera
  - Flow profile compare
    - Drop to ≈ 60% or below
    - Need to pass through flow sensor at the same timing as dispense starts
  - Maximum pressure
    - Decrease 1 psi
  - Average pressure
    - Decrease 0.5 psi
  - Air detect volume
    - Increase 0.018 mL

- **Confidence range of detection**
  - Volume = 0.005 mL
    - Ø ≈ 1.8 mm (0.07”)
  - Volume = 0.04 mL
    - Ø ≈ 3 mm (0.12”)

*Figure 22: Diagram comparing size of bubbles that are detected by a liquid particle counter (left); Flow Profile Compare Confirmation tool (middle); Maximum Pressure Confirmation tool (right).*
WINDWOS OF OPERATION

To successfully detect bubbles using the flowmeter, correctly setting up the dispense volume and dispense rate are critical. An improper setting will lead to failed bubble detection. Based on in-house testing, the optimal setting for detecting an air bubble with a diameter of 1.3 mm or larger has a dispense volume = 0.3 mL and a dispense rate of 0.15 mL/sec. Successfully detecting an air bubble with a diameter of 1.2 mm has a dispense volume = 0.5 mL and a dispense rate of 0.15 mL/sec. Successfully detecting an air bubble with a diameter of 0.8 mm has a dispense volume = 1.7 mL and a dispense rate of 0.35 mL/sec. The smaller the air bubble is, a higher dispense volume and dispense rate are needed to increase the time necessary for the flowmeter to detect the existence of air. It is necessary that the bubble pass through the sensor installed inside the flowmeter at the same time the flow sensor measures the liquid flow rate. If the timing is off, bubble detection cannot be successful.

1. When building the dispense line, do not use a fitting for the tubing connection, especially between the pump outlet port and the flowmeter. If there is a fitting, bubbles coming from the dispense pump will get stuck inside the fitting and cannot pass through the flowmeter.

2. About 50 cm before the flowmeter location, incline the tubing at a 10–15 degree angle. Too steep an angle makes air bubbles move too quickly and they cannot be detected by the flowmeter. Conversely, too flat an angle causes bubbles to move too slowly to reach the flowmeter and be detected.

3. About 5 cm before the flowmeter location, ensure the tubing is perfectly horizontal. To achieve successful air detection, the air bubble needs to pass through the sensor installed inside the flowmeter at the exact same time the flow sensor measures the liquid flow rate. Perfectly horizontal tubing ensures that air bubbles move slowly enough to be detected by the flowmeter.

4. When building the dispense line, make sure that the air bubble stops right before the flowmeter before the next dispense starts. If the air bubble stops in front of the flowmeter, it is more likely to be detected than if it does not stop and pass through the flow sensor before the next dispense begins.

Figure 23. Bubble diagram of dispense volume and dispense rate for a successful bubble detection.

SETUP FOR A SUCCESSFUL BUBBLE DETECTION BY FLOW SENSOR

Besides using proper dispense volume and dispense rate, the dispense line should be set up properly. Follow these four tips to achieve successful bubble detection.

1. Do not use a fitting for tubing connection
2. Tubing about 50 cm before flowmeter should be inclined at an angle of 10–15°
3. Tubing about 5 cm before flow sensor should be horizontal
4. Bubble should stop right before flowmeter and flow through the sensor inside flowmeter at the same time the sensor measures flow rate during dispense segment

Figure 24. Setup of production line for successful bubble detection.
CONCLUSION

With an appropriate window of operation and dispense line, the experiment on both 193 nm BARC and IPA showed successful bubble detection. This means that Flow Profile Compare can be applied to various photochemicals without any serious concerns. Because this bubble-sensing software uses copy exact theory for the confirmation, the last dispense is the same as the reference. As long as the liquid measured for the reference and for the last cycle are the same, this bubble sensing system, Flow Profile Compare, can be applied to all types of chemicals in lithography process to detect bubbles and prevent their translation to costly wafer defects.

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