CONTAMINATION-FREE LIQUID FLOW CONTROLLER

Abstract

The accurate control of liquid flow is critical for attaining high process yields in wet etch, CMP delivery, and on-demand chemical blending applications. To maintain high purity, a flow control technology must minimize particle addition to the fluid stream.

This paper will present laboratory data to demonstrate the low levels of particle generation associated with the use of a Differential Pressure (DP)-based flow controller. The flow controller performs closed loop control using direct flow and pressure measurement, and is used for in-situ monitoring and control of a process liquid flow rate. Differential Pressure flow measurement technology allows this device to measure liquid flow rate without the use of moving parts.

The flow controller's valve technology provides control with low particle addition to the fluid stream during actuation. The valve seat and diaphragm are designed to minimize dead volume and fluid shear, reducing the possibility of process contamination. Also, to avoid excessive movement, the valve diaphragm actuation stroke is minimized.

Testing was completed under three different conditions that represent probable use cases of the device. Test results indicate low levels of particle generation during flushing and normal operation of the device confirming that semiconductor equipment users can use the flow controller for high purity applications sensitive to particle generation. This provides users with another tool for managing materials integrity.

Executive Summary

This paper will present laboratory data to demonstrate the low levels of particle generation associated with the use of a Differential Pressure (DP)-based flow controller. The flow controller performs closed loop control using direct flow and pressure measurement, and is used for in-situ monitoring and control of a process liquid flow rate. A differential pressure (DP) flow measurement technology allows this device to measure liquid flow without the use of moving parts.

The accurate control of liquid flow is critical for attaining high process yields in wet etch, CMP delivery, and on-demand chemical blending applications. To maintain high purity of the process fluid, a flow control technology must minimize particle addition to the fluid stream.

The particle testing was performed using ultrapure water (UPW) and a PMS Liquistat liquid-borne particle counter. The initial flushing of the flow controller showed that after flushing with 80 liters of UPW, each controller contributed about 0.1 particles per mL for greater than 0.1µm particles. Cycling the flow controllers between 20% and 80% of flow capacity showed particle counts decreased rapidly and reached about 50 particles/controller/cycle for greater than 0.1 µm particles within 500 cycles and about 10 particles/controller/cycle within 10,000 cycles. Continuing cycling of the controllers from 0% to100% of flow capacity showed that after about 300 cycles, the particle counts reached the background level, indicating this operation did not release significantly more particles than the previous cycling pattern. During the test, another particle counter, PMS M65, was also used in parallel to the Liquistat particle counter to ensure the accuracy of the test results. Both counters agreed well. The results by M65 are not shown in this paper.

The low level of particle generation of the flow controller confirms that semiconductor equipment users can use the flow controller for high purity applications sensitive to particle generation. This provides users with another tool for managing materials integrity.

Introduction

The accurate control of liquid flow is critical for attaining high process yields in wet etch, CMP delivery, and on-demand chemical blending applications. To maintain high purity of the process fluid, a flow control technology must minimize particle addition to the fluid stream.

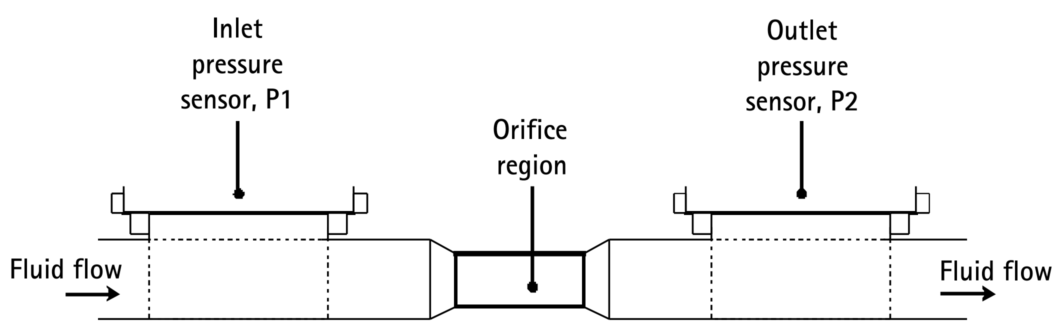
Particles may cause defects for wet etch applications, where the control of hydrofluoric acid (HF) flow is critical. Wet cleaning and on-demand chemical blending applications requiring accurate control must also use devices that generate as few particles as possible. As the sophistication of CMP slurry dispense applications increases, a flow control technology that minimizes particle generation will be key.

To meet the needs of a "particle-free" flow control device, some fundamental design considerations are:

- The flow measurement technology should contain no moving parts
- The control valve technology should minimize the movement of wetted valve parts
- The "dead-volume" should be minimized

To meet the needs of a flow measurement technology with no moving parts, a Differential Pressure (DP) based flow measurement technology is used. Since DP technology calculates flow by measuring pressure drop across an orifice (Figure 1), there are no moving parts in the fluid stream.

The unique design of the valve seat and valve diaphragm (Figure 2) enables the flow controller to control fluid flow rate with minimal deflection of the wetted diaphragms. Microprocessor based electronics link the measurement and control elements; the user's set-point is compared to the measured flow rate, and using standard electronic control techniques, the valve position is adjusted accordingly. A block diagram of the device is shown in Figure 3.



$$\text{Flow} \propto k\sqrt{(P1-P2)}$$

Figure 1: Differential Pressure (DP) flow measurement technology.

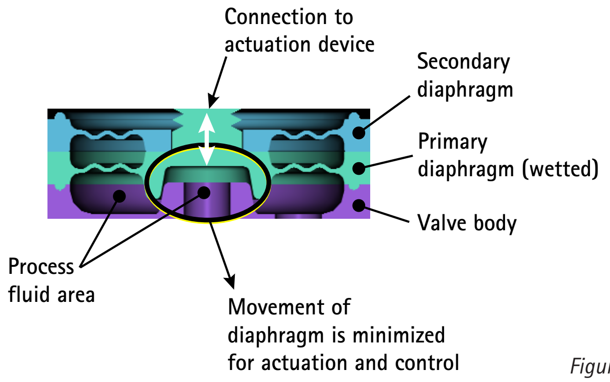


Figure 2: Control valve technology.

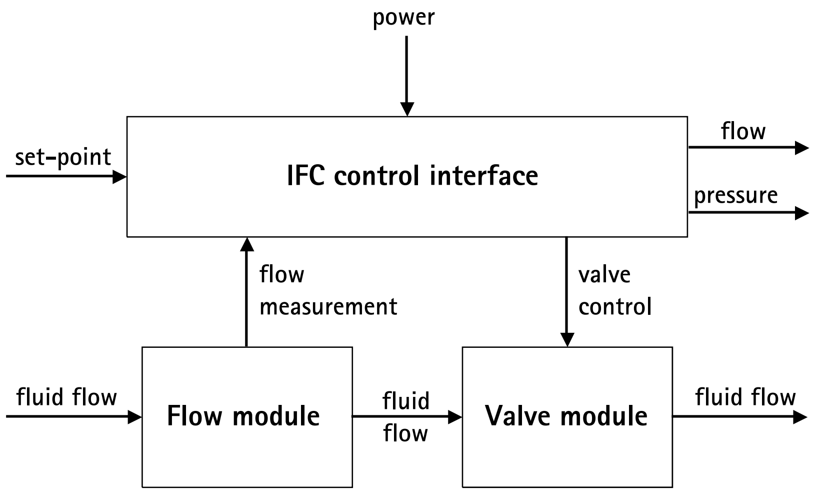


Figure 3: Block diagram of flow controller.

Test Overview

Particle testing was performed using ultrapure water and a PMS Liquistat liquid-borne particle counter. The testing was done at the Entegris, Inc. Technology Characterization Laboratory located in Chaska, Minnesota, USA. The testing was complete in December 2001.

Three different tests were conducted.

- A test to determine particle count during initial flushing of the device.
- A test to determine particle count when the device is cycled between 20% and 80% of full scale flow. This simulates a continuous control application.
- A test to determine particle count when the device is cycled between 0% and 100% of full scale flow. This simulates a batch control application.

Products Tested

The testing was performed on two (2) model 6500-T4-F02-D06-K-P1-U1 flow controllers manufactured by NT International, Minneapolis, Minnesota, USA. The rated flow control capacity of each unit was 1250 ml/minute (maximum) and 125 ml/minute (minimum). Each flow controller was equipped with ¼˝ Flaretek® flared tube connections from Entegris.

Test Results

The results shown in Figure 4, 5, and 6 have not had the background subtracted. This is done so that the background particle level of the system can be included in the graphs for the convenience of the readers who may be interested in this information. In order to obtain the net particle contribution by the flow controllers, one should subtract the background average from the readings in the graphs.

The initial flushing results are shown in Figure 4. The particle counts of the initial flushing of the flow controllers decreased rapidly to 0.1 particles/ml/controller (reading of 0.2 minus background average of 0.1) of ≥0.1µm after 80 liters of UPW had flowed through each flow controller.

The flow controllers were then cycled between 20% and 80% of the rated flow capacity. As shown in Figure 5, particle counts decreased to about 50 particles/controller/cycle of ≥0.1 µm after approximately 500 cycles and about 10 particles/controller/cycle after 10,000 cycles (both with background subtracted). Over 30,000 cycles were completed on the units at the termination of this test.

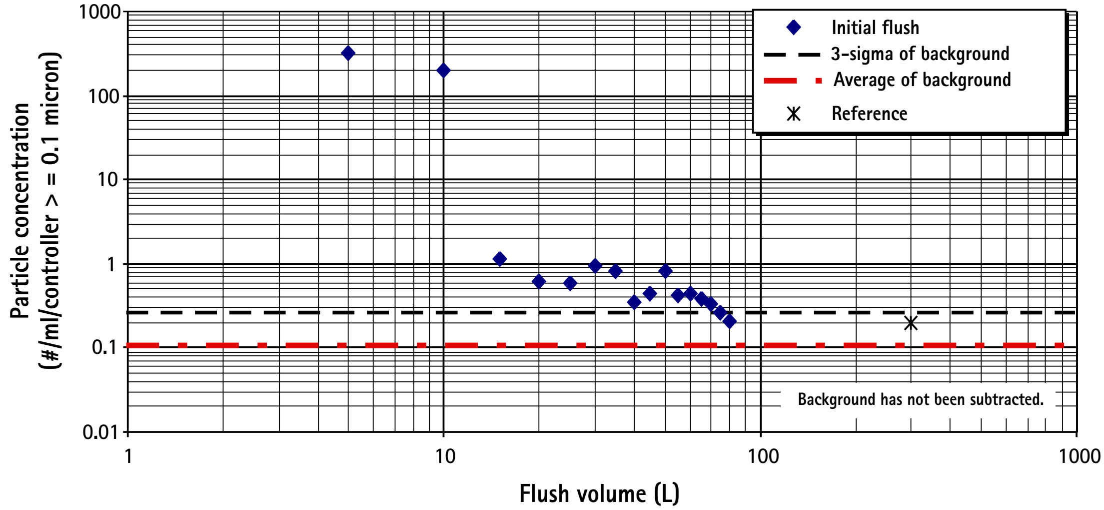


Figure 4: Particle counts during the initial installation and flush mode with a total flow rate of 2000ml/min (1000ml/min each).

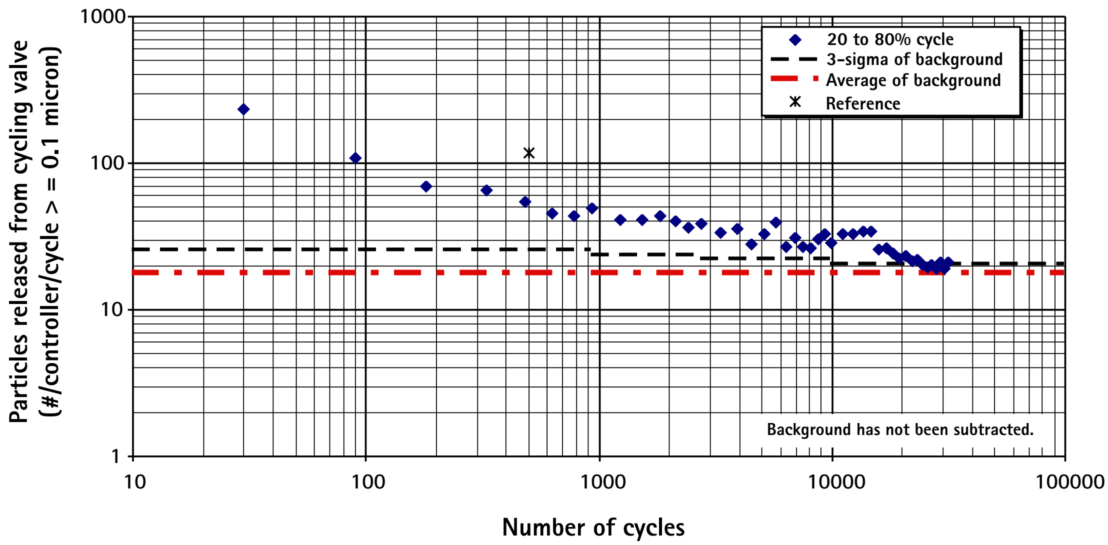


Figure 5: Particle counts during cycling from 250 to 1000ml/min flow rate (20 to 80%). Cycle rate of 10 seconds per cycle.

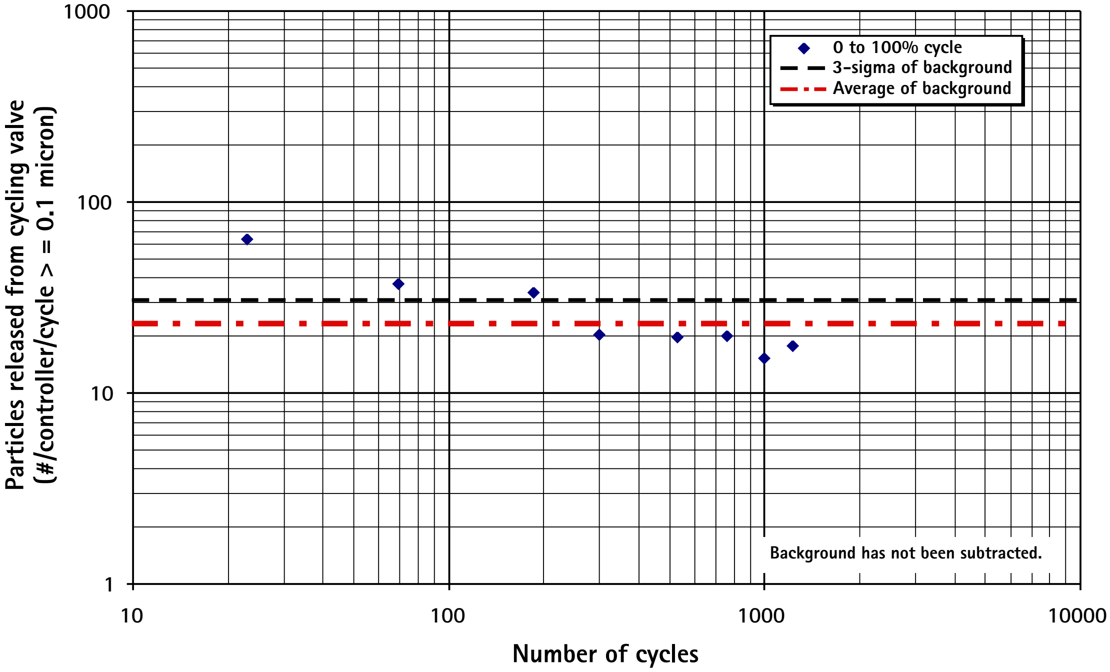


Figure 6: Particle counts during cycling from 0 to 1250ml/min flow rate (0 to 100%), after about 30,000 cycles with the 20-80% pattern. Cycle rate of 13 seconds per cycle.

The flow controllers were then cycled between 0% and 100% of the rated flow capacity. As Figure 6 shows, particle counts reached the background after about 300 cycles, indicating this operation did not release significantly more particles than the previous cycling pattern.

In Figure 4 and 5, a reference point was also plotted. The reference point represents the proposed specifications in the industry. Clearly, this technology significantly reduced the particle generation compared to the reference.

During the test, another particle counter, PMS M65, was also used in parallel to the Liquistat to ensure the accuracy of the test results. Both counters agreed well. The results by M65 are not shown in this paper.

Figure 7 and Figure 8 plot the inlet pressure and outlet flow of each flow controller at approximately 20% and 80% of rated flow capacity and at 0% to 100% of rated flow capacity, respectively. As a note, the flow controller outputs a flow signal (4-20mA) and an inlet pressure signal (4-20mA).

Test Method

The test manifold for the particle testing of the two flow controllers is illustrated in Figure 9. Two flow controllers were tested in parallel and the valves of the flow controllers were cycled alternatively during the cycle test. This was necessary to minimize the abrupt pressure fluctuation in the system due to opening and closing of the valves.

Initial Flushing Sequence

The flow controllers were adjusted to 80% of the rated flow capacity (16.8mA output signal) for a total flow rate of 2000 ml/min (1000ml/min/controller). Particle count measurements were started after water flowed through the flow controllers for five minutes.

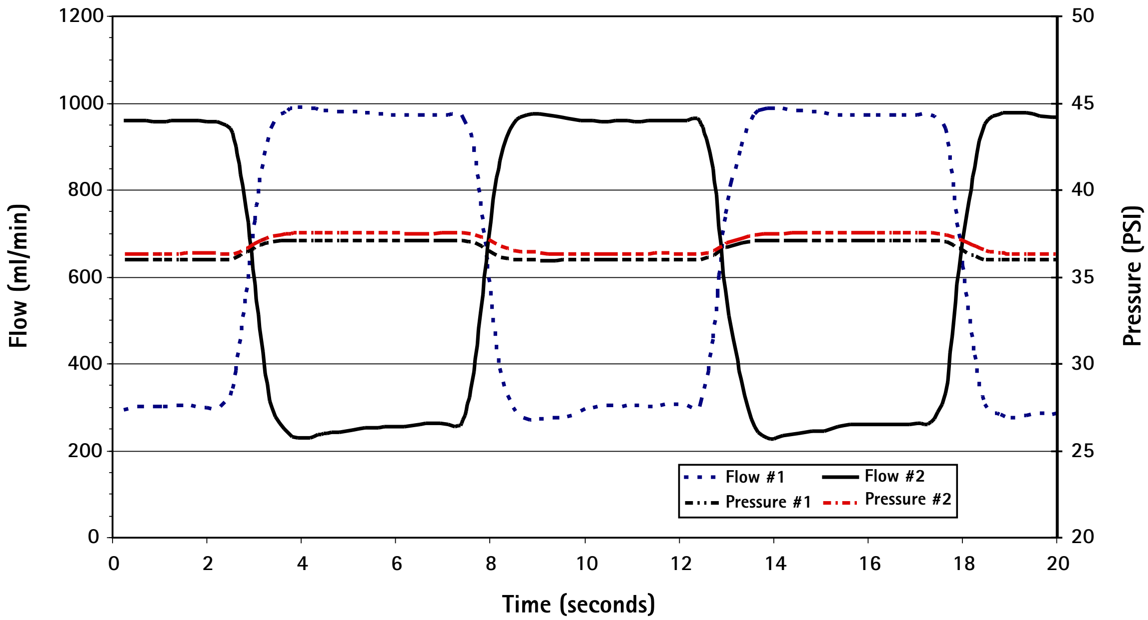


Figure 7: Outlet flow and inlet pressure vs. time during cycling from 250 to 1000ml/min flow rate (20 to 80%). Cycle rate of 10 seconds per cycle.

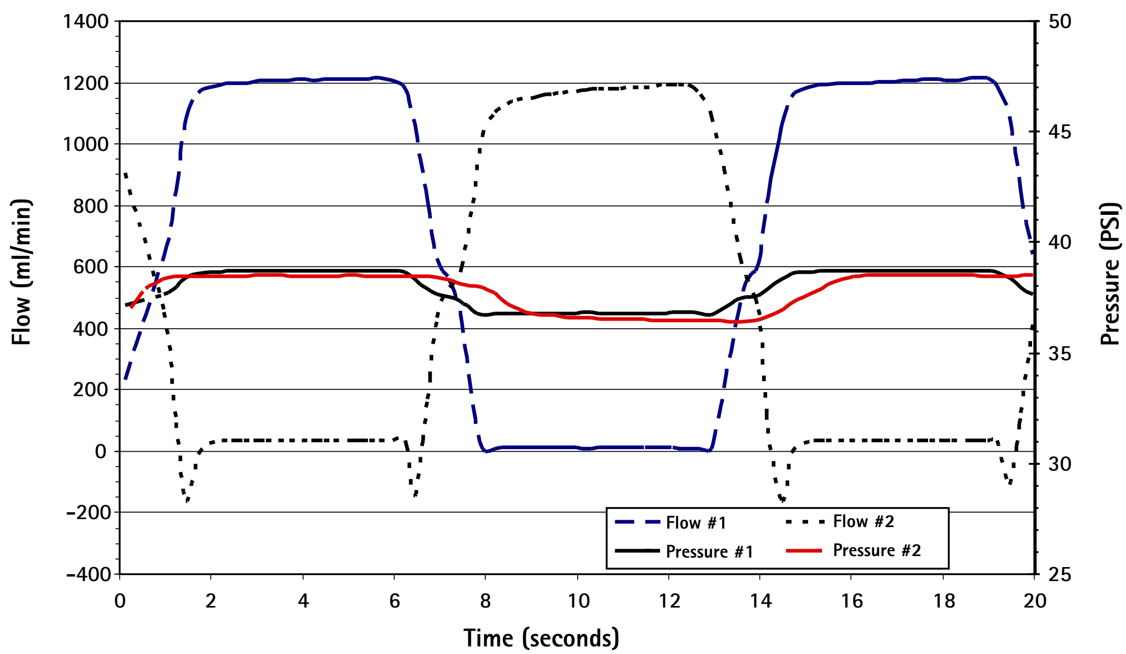


Figure 8: Outlet flow and inlet pressure vs. time during cycling from 0 to 1250ml/min flow rate (0 to 100%). Cycle rate of 13 seconds per cycle.

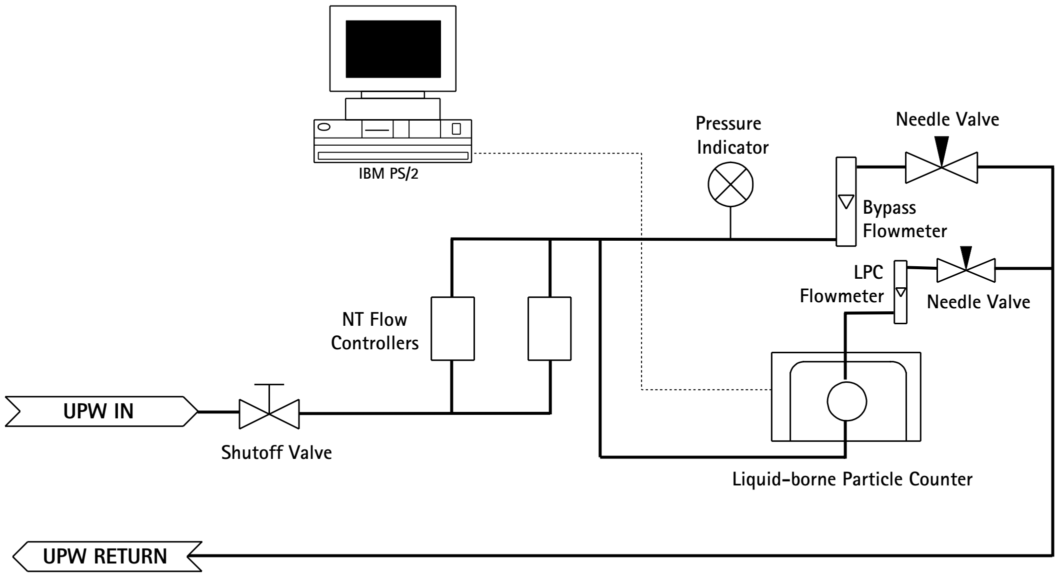


Figure 9: Schematic of the liquid-borne particle testing manifold.

Cycling at 20 and 80% of Flow Capacity

After the initial flushing sequence, the flow controllers were cycled from approximately 80% of flow capacity (1000ml/min) to approximately 20% of flow capacity (250ml/min). The cycling occurred such that flow controller #1 flowed at 20% of rated flow, while flow controller #2 flowed at 80%, and vice versa so a near constant 1250ml/min total flow rate was maintained through the test manifold. This method of cycling was implemented to reduce sudden pressure fluctuations in the system. The flow controllers were cycled at a rate of 10 seconds per cycle (six cycles/min). The flow and pressure was also recorded for one minute out of every 60 minutes of run time.

Cycling at Maximum/Minimum Flow Capacity

After the 20/80% cycling was completed, the testing then cycled the flow controllers from 100% (maximum) of flow capacity (1250ml/min) to 0% of flow capacity (0 ml/min). The cycling occurred such that flow controller #1 flowed at 0% when flow controller #2 flowed at 100% and vice versa such that 1250 ml/min flow was maintained through the test manifold. The flow controllers were cycled at a rate of 13 seconds per cycle until particle counts reached the UPW background. The flow and pressure was also recorded for one minute out of every 60 minutes of run time. Note that the flow controllers are calibrated from 10% to 100% of their full scale flow; therefore the erratic flow rate results near zero, as shown in Figure 8, are due to the flow being out of the calibration range.

Sample Calculations

Using the Liquistat Particle Counter, Calculation of particles per controller per cycle

The viewing volume of the Liquistat particle counter is 50 ml/min with a flow rate of 50 ml/min. The sampling time was five minutes. Therefore, a raw count of six particles in a five minute reading will give a concentration of 6/(50x5)= 0.024 particles/ml. The total flow rate through the controllers was 1250ml/min. There were two controllers cycling at six cycles per minute. Thus, a 0.024 particles/ml count will give 0.024x1250/2/6= 2.5 particles/controller/cycle.

Using the Liquistat Particle Counter, Calculation of 3 sigma of background level

Assume the background level was 0.1 particles/ml for ≥0.1 µm particles, with a standard deviation of 0.06 particles/ml, then the 3-sigma level is 0.1+(3x0.06)= 0.28 particles/ml. For 1250 ml/min flow, two controllers cycling at six cycles per minute, this background level contributes 0.28x1250/2/6= 29.2 particles/controller/cycle.

Conclusion

Testing was completed under three different conditions that represent probable use cases of the Differential Pressure based flow controller. Test results indicate low levels of particle generation during flushing and normal operation of the device, which confirms that semiconductor equipment users can use the flow controller for high purity applications sensitive to particle generation.

Figure 10: Photo of two (2) flow controllers used for particle generation testing.

Authors

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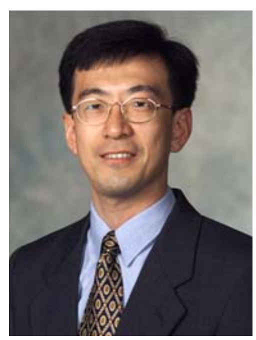
Chuck Gould is the Product Marketing Manager for NT International's High Purity Measurement Products. He holds a bachelor's degree in chemical engineering from the University of Minnesota and is currently completing a master's degree. Gould has over six years experience serving the semiconductor industry, including experience with a leading semiconductor capital equipment manufacturer.



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Huaping Wang is a test engineer in the Technology Characterization Laboratory of Entegris, Inc. He is responsible for particle related product testing. Wang received a M.S.M.E. degree in particle technology from the University of Minnesota, Twin Cities, MN in 2001, and a M.E. and a B.S. degree in heating, ventilation and air conditioning from Tongji University, Shanghai, China in 1996 and 1993 respectively.

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