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Cleaner Filters

for Cleaner Wafers

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AT A GLANCE

The cleanliness of fluoropolymer filters has a direct impact on the extractable metal contamination levels found on wafer surfaces. The data shows that new wafer cleaning specifications require filters to undergo post-extraction cleaning processes.

Table 1. Metallic Contamination After Installation of Standard Filter A (10^8 atoms/cm^2)

	K	Ca	Cr	Fe	Zn
Detection limit	5	5	4	8	4
Prior to filter change	-	-	5	-	4
First SC2+SC1	-	6	7	240	90
Second SC1	13	13	19	198	40
Second SC2+SC1	9	-	7	256	17
Second SC2+second SC1	-	10	10	58	15

For critical cleaning applications, the cleanliness specifications for metal contaminants have shifted from the process chemicals to actual measurements of surface metals on the wafer. Therefore, the cleanliness of all materials that impact metal contamination must be considered. Filtration can have a significant impact on the metal contamination level in an SC1 clean chemistry. Filters that contribute total metals of <1 ppb extracted are typically necessary to achieve consistent purity levels within the SC1 chemistry. However, use of high-purity filter assemblies may be insufficient; a new proprietary extraction process is shown to be necessary to achieve the cleanliness specifications for today's fab processes.

Table 2. Metallic Contamination After Installation of Filter B (10^8 atoms/cm^2)

	K	Ca	Cr	Fe	Zn
Detection limit	5	5	4	8	4
Prior to filter change	-	11	-	-	7
First SC2+SC1	-	7	-	29	22

Table 3. Metal Levels After Installation and Cold SC2 Clean, Filter B (10^8 atoms/cm^2)

	K	Ca	Cr	Fe	Zn
Detection limit	5	5	4	8	4
First SC2+SC1	-	41	5	152	83

Metallic contamination

The sources of metallic contamination in wet processing include the chemicals; contaminated wafers processed in the chemicals; and components of the processing tools, such as pumps, valves and filters. Manufacturers of parts and the end users recognize these sources and have taken steps to clean the parts to make certain that the contamination does not reach the wafer surface.

Equipment manufacturers that supply the semiconductor industry recognize that metallic contamination is a primary concern to semiconductor manufacturers, and strive to produce products that meet the requirements of their customers. However, the suppliers are also faced with increasingly tighter controls as the geometries shrink and as the detection methods for metals on wafer surfaces improve. Thus, materials manufacturers need to improve their cleaning processes to keep up with tightening controls.

Semiconductor manufacturers operate all their processes with control strategies to avoid issues that exposure to contamination can cause. As detection methods improve, the control limits of critical processes are tightened to assure that control is maintained on impurity levels. For example, the move from total reflection X-ray fluorescence (TXRF) to vapor phase decomposition/inductively coupled plasma mass spectrometry (VPD/ICPMS) for monitoring metal contamination on surfaces allows for a ~100× improvement in detection and allows control limits to be established on contaminants that were previously undetectable.

Our contamination control processes operate within control limits that are established by statistical methods based on the operation of the tool during normal semiconductor manufacturing. During normal operations — or whenever an event occurs that we suspect can introduce contamination — we check the operation to make sure levels are within control limits.

Equipment installation

In a high-volume manufacturing facility, the IC manufacturer must plan preventive maintenance (PM) operations to minimize the impact on wafer throughput, and also train the maintenance personnel to work in such a way as to minimize the possibility of contamination of the processing baths. In addition, after completion of any PM procedures, which intrude on the wafer processing area, cleaning of the baths is essential to remove any metallic contamination that may have been introduced to the bath. The bath is then requalified to make certain the wafers will not be exposed to unacceptable levels of contamination.

With the use of more sensitive methods of metallic contamination detection on wafer surfaces, such as VPD/ICPMS, we have been able to set control limits for metallic contamination on surfaces and react more quickly when contamination levels increase in the baths as a result of equipment degradation. We also established the required levels that the bath must reach after a PM procedure, before the tool can be returned to manufacturing use. So when we replace filters, valves or pumps in a manufacturing process, we must be certain that the bath has returned to the same cleanliness level that we saw before the PM.

Our standard method for installing a filter in an SC1 (DI:NH_4OH:H_2O_2) bath would be to install the filter in the housing, being careful to never touch the filter, rinse the bath three times with water, fill the SC1 bath with the chemicals for an SC2 process (5:1:1 DI:HCl:H_2O_2), circulate at 60°C for one hour, rinse the bath three times with water, and then set up the bath to be qualified for standard performance including metallics on wafers processed through the bath. The results we present here are from a 0.05 µm pore size non-dewetting PTFE membrane filter constructed with specific cleaning procedures. All our baths — including the SC1 — operate with two filters in parallel in the recirculation system, and both filters are replaced at the same time.

With the use of VPD/ICPMS to detect metal contaminants, we have seen that filter changes, especially in an SC1 bath, followed by chemical clean of that bath were not returning the bath to the levels we define as being in control. The iron concentration, in particular, was high. A single clean of the bath would not repeatedly produce a clean bath based on our specifications, and a second clean would sometimes also fail. We tried filters from different suppliers and obtained similar results.

Because of these uncertainties and the sensitivity of the SC1 process to metallic contamination of surfaces, we would keep the bath out of production until it passed the tests. Multiple cleaning steps were necessary to qualify the SC1 process. Thus, this uncertainty would cost us extra chemicals and analysis time to clean the bath and, more importantly, production time lost in waiting for the tests to confirm the acceptability of the process.

Results — SC1 process cleanliness

The first tests (Table 1) involved a typical installation of a new standard filter (Filter A) in an SC1 bath. This type of scenario was representative of filters from multiple manufacturers. The test run before the filter change indicates that the bath metallic level was within our control limits for all metals tested. After filter installation and first clean, the results, especially for iron, were above the control limits. A second SC1 bath set up after the first was also tested and found to be above the control limit. A second clean was performed using SC2, and this test also failed. Only after the second SC1 bath was set up after this second clean did the results come back within the control limits.

These results are typical of those seen with the standard filters we had purchased, and were repeated a number of times. This testing, cleaning and retesting would typically consume 12 hours of production time after the filters were installed. The filters never passed on the first clean test.

As a result of this type of analysis, we contacted a filter supplier and asked if they could provide a cleaner filter. The data in Table 2 shows the results for the installation of a filter supplied that uses a proprietary cleaning process (Filter B), and are typical of all installations since we began using this filter.

A separate test was run during a filter installation in another bath (Filter B). In this test, we used the same installation pro-

Table 4. Critical Metals in Semiconductor Processing

Element	Detection level (ppb)
Aluminum (Al)	0.07
Calcium (Ca)	0.02
Chromium (Cr)	0.027
Copper (Cu)	0.014
Iron (Fe)	0.014
Lead (Pb)	0.007
Lithium (Li)	0.00009
Magnesium (Mg)	0.012
Manganese (Mn)	0.011
Nickel (Ni)	0.014
Potassium (K)	0.028
Sodium (Na)	0.0006
Zinc (Zn)	0.03

Table 5. Commercial Fluoropolymer Filter Data (µg/cartridge)

	Filter #1	Filter #2	Filter #3
Aluminum	12.8	34.2	14.3
Barium	0.5	0.9	0.3
Beryllium	<0.05	<0.05	<0.05
Boron	0.8	1.0	1.4
Cadmium	0.3	0.1	0.06
Calcium	551.8	672.5	370.9
Chromium	17.3	18.3	22.0
Copper	4.1	6.9	5.0
Gold	<0.5	<0.5	<0.5
Iron	98.7	129.7	155.7
Lead	0.5	0.5	0.6
Lithium	<0.05	<0.05	<0.05
Magnesium	4.0	5.7	6.0
Manganese	1.2	1.8	2.1
Nickel	8.9	12.5	17.0
Potassium	3.0	3.5	3.7
Sodium	6.8	10	10.1
Tin	0.1	0.2	0.3
Titanium	0.2	0.3	0.4
Zinc	5.2	5.7	13.6
Total	716.3	903.9	623.6

Table 6. Commercial High Purity/Low Extractable Filters (µg/cartridge)

	Filter C	Filter D	Filter E	Filter F
Aluminum	0.1	0.8	9.7	9.8
Barium	0.03	0.05	0.1	<0.01
Beryllium	<0.5	<0.5	<0.5	<0.5
Boron	<0.1	5.1	1.5	<0.1
Cadmium	<0.01	<0.01	<0.01	<0.01
Calcium	1.1	3.2	16.0	14.4
Chromium	<0.05	0.3	0.8	1.0
Copper	0.07	0.3	0.6	0.66
Gold	<0.5	<0.5	<0.5	<0.5
Iron	0.4	4.9	18	11.6
Lead	<0.05	<0.05	0.1	<0.05
Lithium	<0.05	<0.05	<0.05	<0.05
Magnesium	<0.05	0.5	1.0	0.5
Manganese	<0.05	<0.05	0.2	0.2
Nickel	<0.05	0.7	0.7	1.1
Potassium	<0.1	0.3	3.3	0.1
Sodium	<0.1	1.5	6.9	1.2
Tin	<0.05	<0.05	0.05	0.1
Titanium	<0.05	<0.05	<0.05	0.1
Zinc	0.4	1.1	1.4	1.2
Total	2.1	18.6	60.3	41.8

cedure, except the SC2 clean was carried out at ambient temperature without heating the SC2 clean solution (Table 3). The iron levels were within our control limit (1.6 × 10 atoms/cm^2), even without the heated bath.

Filter cleanliness

A proprietary filter cleaning method has been developed to reduce the metal extractables from fluoropolymer filters used in semiconductor process chemicals. Fluoropolymer filters typically consist of a PTFE (polytetrafluoroethylene) membrane with fluoropolymer support and hardware (PTFE, perfluoroalkoxy [PFA] and fluoroethylenepropylene [FEP]). These are considered clean materials; however, both the materials themselves and the filter manufacturing processes can be sources of metallic contamination that can be released into the process chemicals they are filtering.

The 13 metals that are of greatest concern are given in Table 4. This proprietary cleaning process has achieved consistent total metals extractables levels from 10 in. cartridge filters of <1.5 µg/10 in. cartridge with blank correction (the average level for these filters is >1.0 µg/10 in. cartridge). Lot-to-lot quality control data is gathered from fluoropolymer filters treated by this process to monitor consistency in the metals levels from the processed filters.

Not all fluoropolymer filters are equal with respect to total metals extracted from the filter. Standard fluoropolymer filters that have not undergone any post-assembly cleaning processes

Table 7. Filter C After Filter Assembly Extraction Process

Element	µg/cartridge	Blank — µg	Sample minus blank
Lithium	0.013	0.005	0.008
Sodium	0.201	0.070	0.131
Magnesium	0.059	0.024	0.035
Aluminum	0.416	0.055	0.361
Potassium	0.348	0.297	0.051
Calcium	0.287	0.062	0.225
Chromium	0.300	0.246	0.054
Manganese	0.060	0.050	0.010
Iron	0.448	0.077	0.371
Nickel	0.088	0.041	0.047
Copper	0.071	0.056	0.015
Zinc	0.139	0.034	0.105
Lead	0.034	0.015	0.019
Total	2.464	1.032	1.432
Std. Dev.	0.153	0.086	0.129

(Table 5) would be unacceptable in SC1 process chemistry without extensive post-installation cleaning. Also, not all post-assembly cleaning processes will produce filters with equal levels of cleanliness. Table 6 gives data on metals extractables from filters that have post-assembly cleaning to reduce metals contamination. These filters are considered "high purity/low extractables."

Extraction data from a specific fluoropolymer filter that is used by Texas Instruments (Dallas) for the SC1 process chemistry (Table 6) using a standard extraction process (Filter D) was not meeting the surface metals contamination levels required by the specification. Instead, a new proprietary extraction process for Filter C was able to consistently meet the surface metals specification in the SC1 chemistry. The data in Table 7 represents the average extraction level of 20 filters.

Conclusions

High-volume manufacturing operations depend on reliable sources of materials and parts that make it unnecessary to check contamination levels after installation. The suppliers must understand how the level of cleanliness of their products can affect the operation of the processes that use the products.

The data presented here demonstrates that the cleanliness of fluoropolymer materials and the filters made from them are not enough to assure contamination-free parts for critical applications. The manufacturing operations used to assemble the filtration products can also have a significant impact on the performance of that product. The data demonstrates that there is a direct relationship between the extractable metal contaminants measurement performed on filters and the contamination levels on wafer surfaces.

Filters produced using a proprietary cleaning process that assures consistent low-level cleanliness can improve throughput in a high-volume production line by assuring purity of the process and consistently meeting the wafer surface metals specification. •

Reference

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