Wet particle source identification and reduction using a new filter cleaning process

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ABSTRACT

Wet particle reduction during filter installation and start-up aligns closely with initiatives to reduce both chemical consumption and preventative maintenance time. The present study focuses on the effects of filter materials cleanliness on wet particle defectivity through evaluation of filters that have been treated with a new enhanced cleaning process focused on organic compounds reduction. Little difference in filter performance is observed between the two filter types at a size detection threshold of 60 nm, while clear differences are observed at that of 26 nm. It can be suggested that organic compounds can be identified as a potential source of wet particles. Pall recommends filters that have been treated with the special cleaning process for applications with a critical defect size of less than 60 nm. Standard filter products are capable to satisfy wet particle defect performance criteria in less critical lithography applications.

Keywords: Wet particle, Filtration, Cleaning, Organic extractables

1. INTRODUCTION

Defect adders on wafer after spin coating, sometimes called "wet particles," is a widely used indicator for the readiness of lithography chemical dispense modules. Wet particle reduction during filter installation and start-up aligns closely with initiatives to reduce both chemical consumption and preventative maintenance time. The incidence of wet particles after filter installation has significantly increased since resolution of the on-wafer particle detection metrology has improved to sub-30 nm detection size. Typically, identification of wet particle sources has been very difficult without a method to directly analyze nanoscale particles. Previous work focused on microbubbles from the filter as a possible source and identified several operational recommendations for improved start-up [1-3].

The present study focuses on the effects of filter materials cleanliness on wet particle defectivity through evaluation of filters that have been treated with a new enhanced cleaning process. Furthermore, we explored the relevance of filter extractable results with regard to the identities and origins of wet particles. Spiked test using suspected compounds was conducted to further identify the wet particle source.

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2. EXPERIMENTAL

2.1 Particle cleanliness and solvent extraction tests

Particle cleanliness and solvent extraction tests were conducted in order to compare the impact on wet particle defectivity for a standard commercial point-of-use filter (Pall PhotoKleen EZD-2X PE-Kleen Filter Assembly) and an analogous product that was treated with a new enhanced cleaning process (Pall Xpress EZD-2X PE-Kleen Filter Assembly). Particle cleanliness in ultrapure water (UPW) of sample filters was measured by sampling downstream of each filter using a RION KS-18FX liquid particle counter, which is capable of particle measurement greater than 40 nm in size. Solvent extractions from sample filters using propyleneglycol monomethyl ether acetate (PGMEA) and methylene chloride were generated via 24 hours soaking and subsequent analysis for various contaminants. Inductively coupled plasma mass spectroscopy (ICP-MS, Agilent 7700s) was used to quantify Li, Na, Mg, Al, K, Ca, Mn, Fe, Ni, Cu, Zn and Pb, and gas chromatography mass spectroscopy (GC-MS, Agilent 5975C) was used to quantify organic compounds from the PGMEA extracts. Methylene chloride extracts were evaporated to dryness and then measured gravimetrically for non-volatile residue (NVR).

2.2 Wet particle defectivity

Evaluations for wet particle defectivity were conducted using a SOKUDO DUO track system to dispense TOK OK73 solvent through sample filters onto bare silicon wafers. Wet particle measurements were collected using a KLA-Tencor Surfscan SP3 inspection system. Two criteria were used. One is an established >60 nm and the other is a state-of-the-art >26 nm. Defect performance trends are resolved by measuring wet particles at 500mL dispense intervals, with flow cessation after 4L.

2.3 Organic compound spike test

To identify what organic compounds best contribute the wet particle, we designedly spiked organic compounds into OK73 solvent then conducted wet particle evaluation. One hydrocarbon (hydrocarbon 1-1), two kinds of additives (additives 1-1, 1-2) and one different type additive (additive 2-1) all of which were commonly detected in the standard (non Xpress) lithography process filter extracts, were added at 0.1 mg/L in OK73 solvents, respectively. To remove unwanted particles possibly contain in the non-electronics grade spike reagents, the spiked solvents were then filtered using 20 nm rated all fluoropolymer filter capsule (Pall Mini Kleen-Change Filter Assembly), which does not extract organic compounds of interest. 10 ml of each spiked solvent was spin-coated on 300 mm bare Si wafer and wet particle measurements above 26 nm threshold were collected using the Surfscan SP3 inspection system. An OK73 solvent processed in the same procedure without spiking was used as a blank.

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3. RESULTS AND DISCUSSIONS

3.1 Particle cleanliness and solvent extraction tests

Results are given in Figures 1-3 and in Table 1. While these data show no significant differences between filter types for >40 nm particle cleanliness and metal extractables, the NVR and organic extractables were far lower for the specially cleaned filters. As the NVR for these HDPE platform filters is believed to be composed of organic compounds, based on FT-IR analyses, the NVR results also indicate that the new enhanced filter cleaning process effectively reduces organic extractables besides those identified by GC-MS results.

Extractables amounts in metals, NVR and GC-MS results shown here should be substantially higher than the values at actual use points, because these are acceleration tests conducted in 24 hours soaking and the specimens are the first droplets which is normally flushed out during the filter start up.

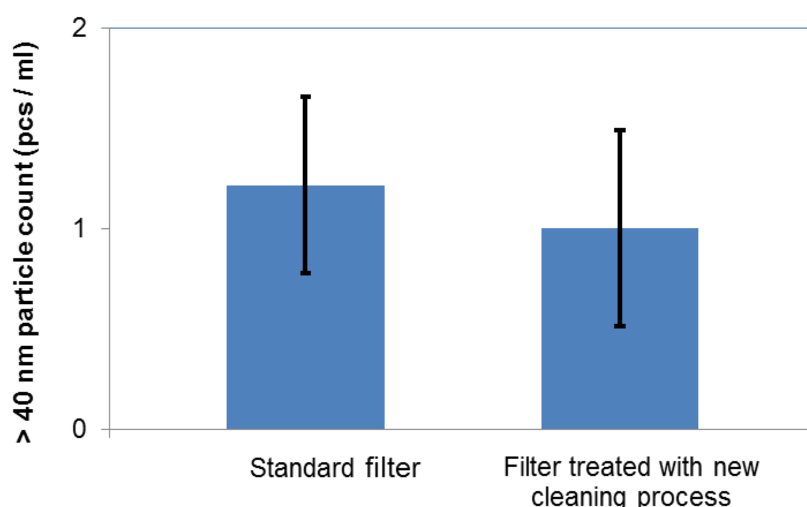

Figure 1. >40 nm particle count in filter outlet in DI water. Error bar is standard deviation during the measurement period.

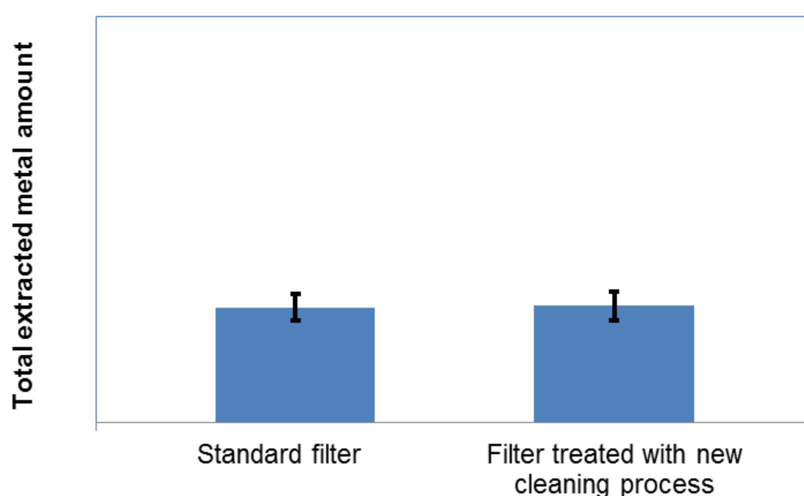

Figure 2. Metal extractables of filters after 24 hours soaking in PGMEA. Total amount of 12 metal elements. Error bar is max and min from 2 samples.

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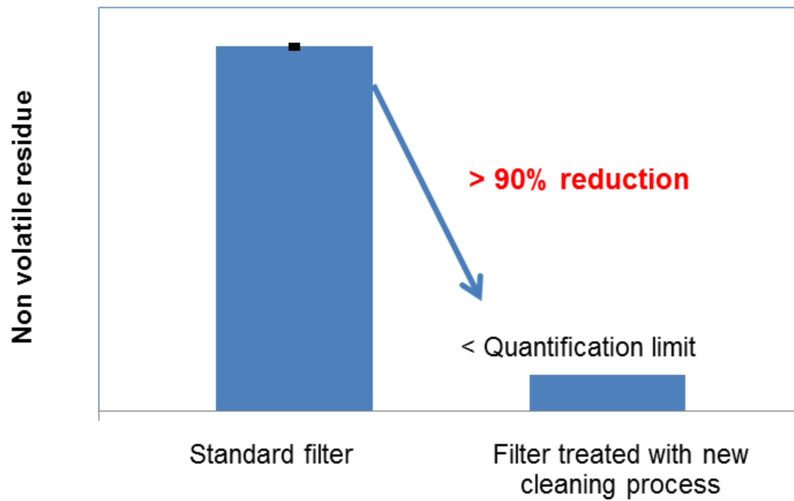

Figure 3. Non-volatile residue from filters after 24 hours soaking in methylene chloride. Error bar is max and min from 2 samples.

Table 1 GC-MS analysis results for organic extractables of point-of-use capsule filters after 24 hours soaking in PGMEA. Semi-quantified using hexadecane calibration. (unit: mg/L), Detection limit=0.15mg/L, Quantification limit=0.5mg/L

Library search results	Standard filter		Filter treated with new cleaning process	
	Sample 1	Sample 2	Sample 1	Sample 2
Hydrocarbon A	> 0.15, < 0.5	> 0.15, < 0.5	> 0.15, < 0.5	> 0.15, < 0.5
Hydrocarbon B	> 0.15, < 0.5	> 0.15, < 0.5	> 0.15, < 0.5	> 0.15, < 0.5
Hydrocarbon C	> 0.15, < 0.5	> 0.15, < 0.5	> 0.15, < 0.5	> 0.15, < 0.5
Hydrocarbon D	> 0.15, < 0.5	> 0.15, < 0.5	Not detected	Not detected
Additive A	> 0.15, < 0.5	> 0.15, < 0.5	Not detected	Not detected
Hydrocarbon E	> 0.15, < 0.5	> 0.15, < 0.5	Not detected	Not detected
Hydrocarbon F	> 0.15, < 0.5	> 0.15, < 0.5	Not detected	Not detected
Additive B	> 0.15, < 0.5	> 0.15, < 0.5	Not detected	Not detected
Additive C	> 0.15, < 0.5	> 0.15, < 0.5	Not detected	Not detected

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3.2 Wet particle defectivity

Wet particle trends throughout the filter start-up is given in Figures 4 and 5. For each filter type the two graphs illustrate a significant difference in defect detection capabilities between established metrology capabilities and a state-of-the-art tool.

3.2.1 Established metrology (>60nm)

With established metrology, as illustrated in Figure 4, there is little difference observed in filter performance between the two filter types at a size detection threshold of 60 nm.

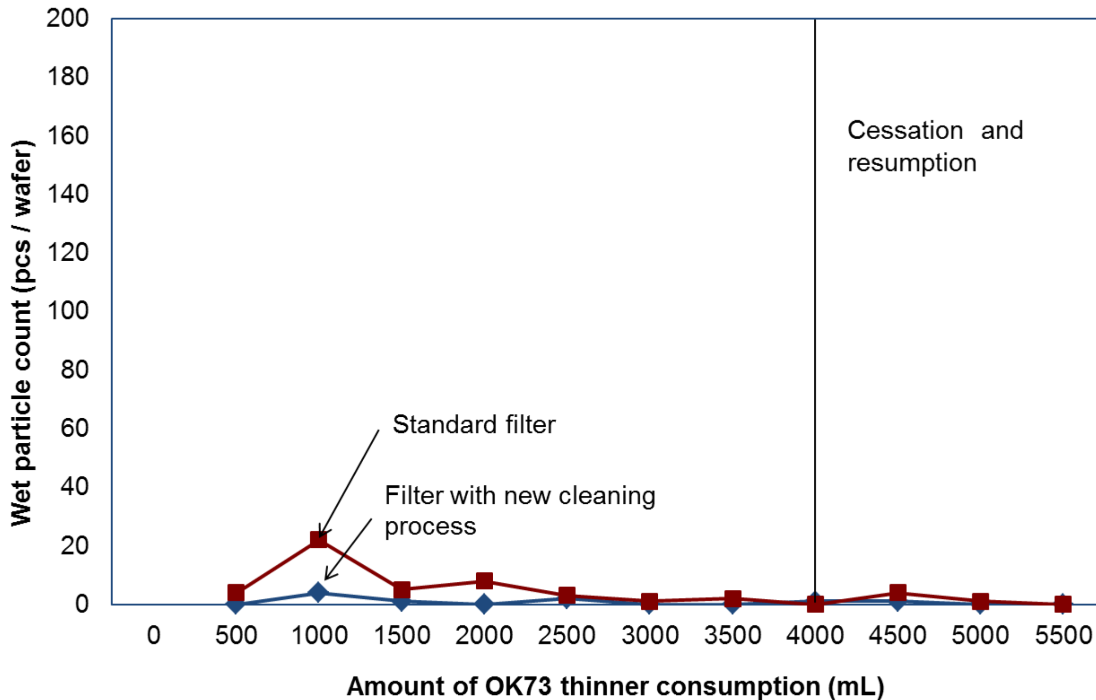

Figure 4. Wet particle count > 60 nm threshold on bare Si wafer vs. fluid consumption after filter installation in SOKUDO DUO. Test fluid was TOK OK73 solvent. Wet particle inspection was conducted using KLA-Tencor Surfscan SP3.

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3.2.2 State-of-the-art tool (>26nm)

Conversely, clear differences in defect performance are observed at a size detection threshold of 26 nm (Figure 5). Additionally, the more sensitive metrology shows the initial wet particle count after 500 mL dispense with the specially cleaned filter was one-tenth (1/10) that of the standard filter. Further, wet particle excursions due to flow cessation were not observed with the specially cleaned filters, while clearly observed in the standard filter upon flow resumption.

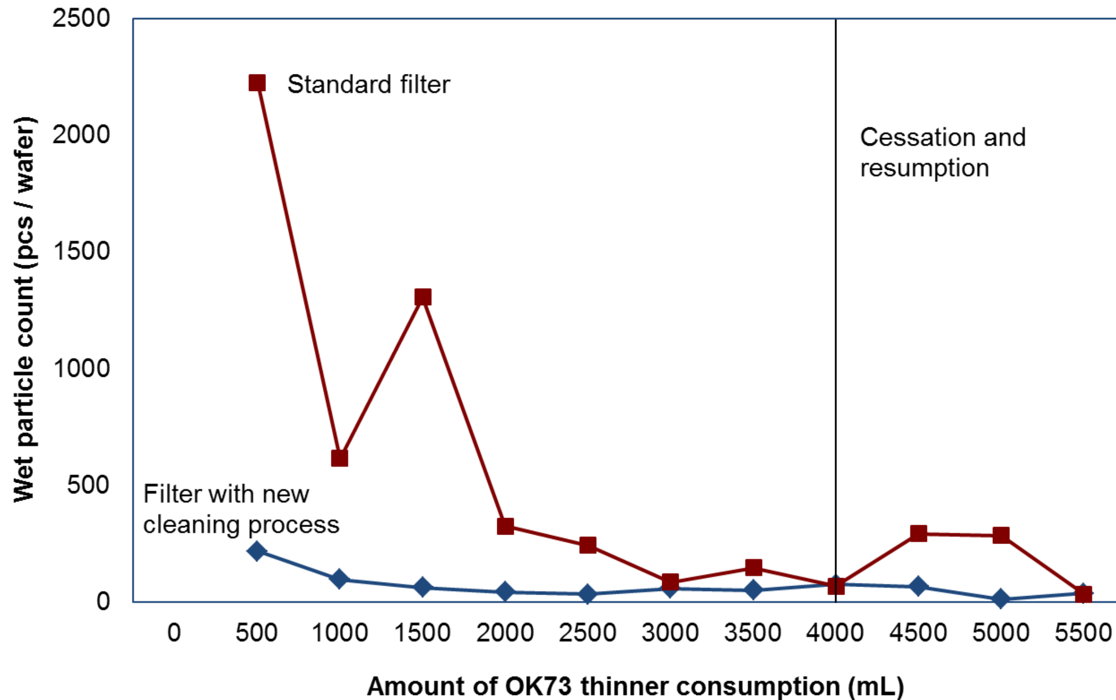

Figure 5. Wet particle count > 26 nm threshold on bare Si wafer vs. fluid consumption after filter installation in SOKUDO DUO. Test fluid was TOK OK73 solvent. Wet particle inspection was conducted using KLA-Tencor Surfscan SP3.

Based on the results, the newly developed filter cleaning process is effective to reduce organic extractables from the standard filter. It can also be suggested that organic compounds can be identified as a potential source of wet particles. The benefits of the specially cleaned filter are best observed with the most advanced sub-30nm inspection capabilities that are currently available.

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3.3 Organic compound spike test

Figure 6 shows the wet particle counts on bare Si wafers spin-coated with the spiked OK73 solvents, in terms of melting points of each spiked compounds. The results were significant though having the rather high blank counts which is assumed to be due to manual operations such as sample bottling, transportation and manual dispensing. As a result, wet particle count appears to be associated with the states of the organic compounds, in other words, results in additive 1-1 and 1-2 whose state at the experimental temperature (23 degree C) were liquid and semi-liquid were blank level, but results in hydrocarbon 1-1 and additive 2-1 whose state were solid were substantially greater. .

The reason why each state determines wet particle count is assumed as follows. After spin-coating, liquid state organic compounds is undetectable because they may remain as thin liquid film or evaporate, while solid state organic compounds is detectable because they may be crystallized.

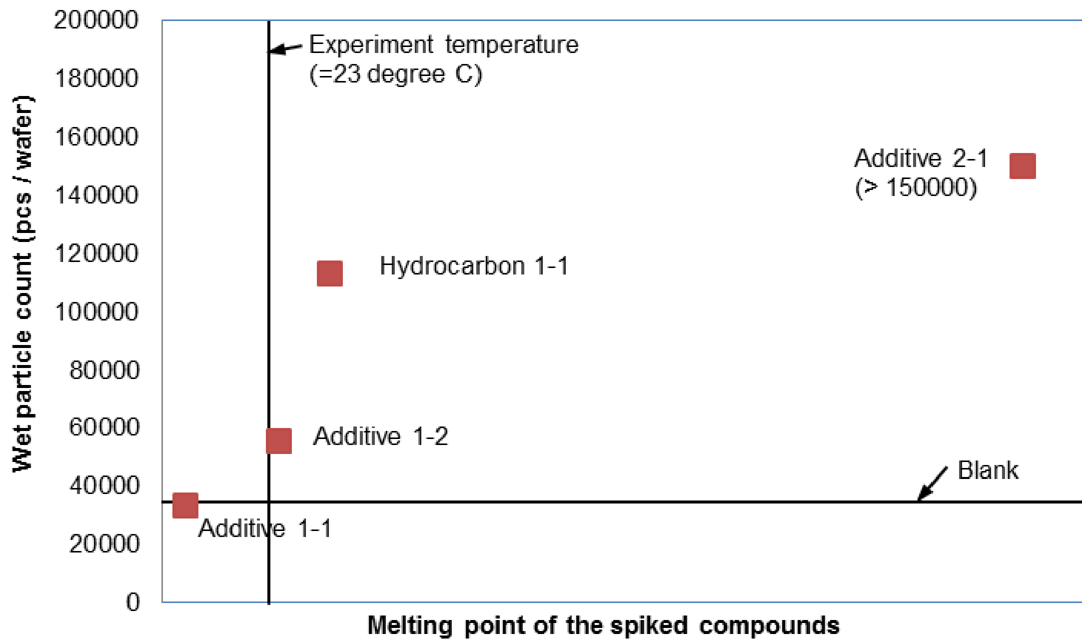

Figure 6 Wet particle count > 26 nm threshold on bare Si wafer after four kinds of organic compounds spiked OK73 spin-coating, in terms of melting points of the spiked compounds. Wet particle inspection was conducted using KLA-Tencor Surfscan SP3.

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4. CONCLUSIONS

Filters treated with a new enhanced cleaning process, which was specifically developed to reduce organic extractables, was effective in reducing wet particle defects during filter start up and mitigated defect excursion after dispense cessation. This suggests that organic compounds extracted from filter materials of construction may be a source of wet particles that are detectable by state-of-the-art defect metrology tools. Further, the spike test directly indicated that the solid state organic compounds are the potential source of the wet particles found in the standard (non Xpress) lithography process filter installation.

Pall recommends filters that have been treated with the special cleaning process (Xpress) for applications with a critical defect size of less than 60 nm. Standard filter products are capable to satisfy wet particle defect performance criteria in less critical lithography applications.

The results should contribute both development for next generation filter products and minimizing chemical consumption and preventive maintenance time in the semiconductor device manufacturers.

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