# Changes in Particle Removal Efficiency of Filters in High Temperature IPA

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The fast pace of innovation at the leading edge of semiconductor device development in the microelectronic industry continues to drive the development of novel filtration solutions. One property of the device manufacturing process that changes is the reduction of devices size (smaller technology nodes) and the narrowing of spaces between structures. These changes in device architecture have resulted in increased use of isopropyl alcohol (IPA) to aid in the drying of wafers after they have been cleaned and rinsed. This occurs after virtually any step where a wet cleaning process occurs. As with any fluid being used during any step in the device manufacturing process, the cleanliness level of IPA needs to be further improved and filtration is an indispensable technology to control the cleanliness. More and more, filters with nanometer size removal rating are being used to achieve the required level of cleanliness.

## METHOD

A filter's liquid removal rating is generally determined by its particle removal efficiency (PRE), which is tested under a standard condition, typically with room temperature (RT) water [1]. However, the actual PRE can vary depending on the chemical and temperature used in a process. Therefore, understanding the PRE in the actual chemicals is important. For this reason, PRE evaluation methods in chemicals, which are commonly used in the semiconductor manufacturing processes, continue to be developed [2~6]. IPA is often used at elevated temperature when used in the actual rinse processes, so a previously unavailable method was developed that can evaluate PRE in high temperature IPA. The test method was used to test two kinds of Pall surface modified (SM) PTFE filters each with different removal ratings. The difference in actual PRE between the filters in RT and high temperature IPA was compared.

To determine the ability of the filters to remove particles from hot IPA two filter types were tested using the settings detailed in Table 1.

As with any PRE test, an appropriate "hard" particle is used to simulate a contaminant during the test. Silica is a common wafer contaminant [7], but it can be difficult to obtain particles small enough (≥ 10 nm) for use in testing filters with pore sizes this small. However, zirconium oxide (ZrO2) is similar enough in behavior to silica and is commercially available in a nanometer- sized particle and can be used for these tests. Figure 1 confirms the actual size of the particles as measured by dynamic light scattering in both RT and hot IPA. The particles were measured to be just slightly larger than 10 nm in diameter.

The tests were performed using the system represented in Figure 2. A disc cut from the membrane that makes up the filter is installed in a sample holder. A suspension containing the test particles (ZrO2) and electronic grade IPA was made and poured into a reservoir on the upstream side of the disc holder. For the test in 70°C IPA, the reservoir was placed in a water bath set at 70°C and allowed to stabilize for 10 minutes in order to reach the necessary temperature. For the test in RT IPA, the same filtration system was employed without the use of the water bath. The ZrO2 suspension was then filtered by each test disc and the effluent was collected in a sampling bottle. The concentration of ZrO2 in the influent and the effluent was determined in order to calculate the PRE for each disc, which is then used to represent the retention capability of a fully integrated filter.

| Test Condition | Filter Rating (nm) | Temperature | Chemical |
|---|---|---|---|
| 1 | 10 | RT | IPA |
| 2 | 10 | 70 °C | |
| 3 | 5 | RT | |
| 4 | 5 | 70 °C | |

*Table 1. Conditions of the particle challenge tests of the SM-PTFE filters*

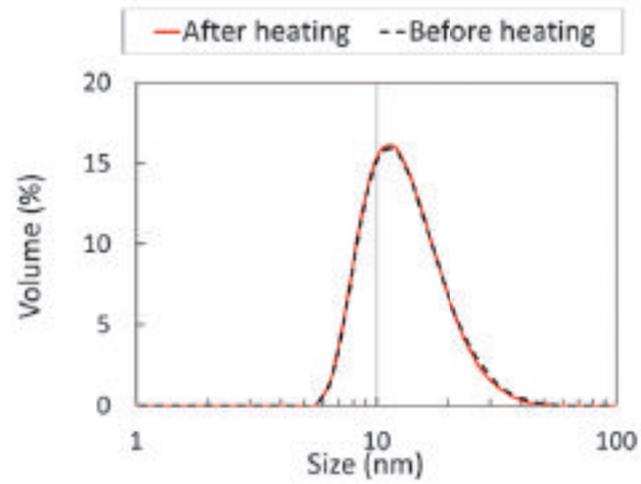


*Figure 1. Particle size distribution (in volume) of ZrO2 in IPA. This particle was used for the filter's challenge test in IPA. The dotted black line indicates the distribution before heating. The red line indicates the distribution after heating up to 70°C.*

Figure 3 shows results of the challenge tests in IPA. In RT IPA, both the 10-nm-rated and 5-nm-rated filters showed sufficiently high PRE (> 99%). In contrast, the PRE of the 10-nm-rated filter slightly deteriorated at 70°C compared to that at RT. The 5-nm-rated filter maintained its high efficiency at 70°C. These results indicate that the filter removal rating determined in RT water is representative of the PRE in 70 C IPA for the 5-nm-rated filter, but not in the case of the 10-nm-rated sample.

In past studies, it was reported that the PRE of a 12 nm-rated SM-PTFE filter decreased in 90 C 96% sulfuric acid (H2SO4) [3] whereas PRE of a 10 nm-rated SM-PTFE did not decrease in 70°C diluted (pH1) hydrochloric acid (HCl) [5]. Considering these additional results, a filter's PRE tends to deteriorate in high temperature chemicals depending on the chemical and temperature used; the farther away you get from pure water at RT, the more deterioration may occur.

## CONCLUSION

The evaluation of 10-nm-rated and 5-nm-rated SM-PTFE membrane filters with 10 nm ZrO2 particles in RT IPA showed sufficiently high PRE (> 99%). In contrast, PRE of the 10-nm-rated filter slightly deteriorated at 70°C compared to that at RT, while the 5-nm-rated filter maintained high efficiency at 70°C. Considering the PRE evaluation results in the past [3,

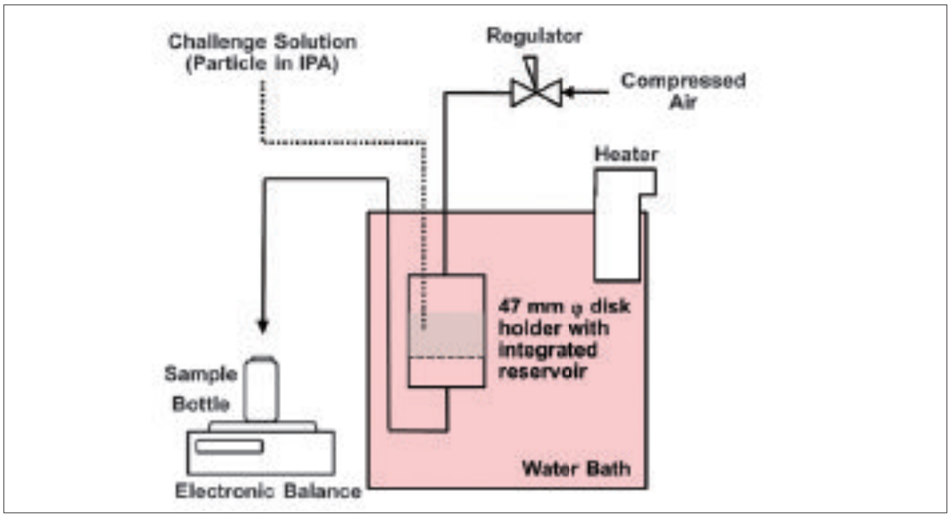


*Figure 2. Schematic of particle challenge test system for PRE evaluation of filters in RT and 70°C IPA. Test filters were 47 mm disc format. The water bath was only used for the tests at 70°C to keep the elevated temperature. Constant flow rate (5 ml/min.) was obtained by adjusting the pressure regulator.*

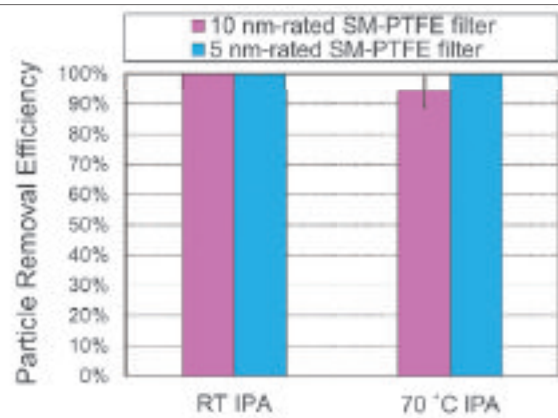


*Figure 3. Results of the particle (ZrO2) challenge tests in RT and 70°C IPA. PRE of each test condition is shown. For each condition, tests were repeated five times and the average is shown.*

5] and the current study, filters' PRE tends to deteriorate in high temperature chemicals. The degree of decrease depends on the chemical, temperature and filter employed. The testing of filters under actual application conditions is a necessary step in determining the appropriate filtration solution for a particular process. As was shown, depending solely on the standard RT data can lead to providing solutions that do not meet the needs of the application.

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