Particle removal efficiency evaluation of filters in high temperature IPA

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INTRODUCTION

The need of isopropyl alcohol (IPA) for wafer drying has been increasing in the semiconductor industry, because the structure of semiconductor devices is getting more complicated and IPA drying is more preferable to avoid pattern disruption of complicated device structure. Also, as the feature size of the semiconductor devices continuously decreasing, the cleanliness level of IPA needs to be further improved. Filtration is an indispensable technology to control the cleanliness, and more and more filters with finer removal rating are now being used to achieve the required cleanliness level.

Filter's removal rating is determined by particle removal efficiency (PRE) under a standard condition, which is typically room temperature (RT) water [1]. In general, however, the actual PRE varies depending on the chemical and temperature to be used. Therefore, understanding the PRE in the actual chemicals is important as well as selecting an appropriate removal rating for effective usage of filters. For this reason, we have developed PRE evaluation methods in several chemicals which are commonly used in the semiconductor manufacturing processes [2~6]. Regarding PRE in IPA, we evaluated Pall 10-nm-rated surface-modified polytetrafluoroethylene (SM-PTFE) membrane filter using three kinds of test particles with their size around 10 nm at RT, and found that the filter shows sufficiently high PRE in the chemical. On the other hand, IPA is often used at elevated temperature when used in the actual processes. Therefore, in this study, we developed a method that can evaluate PRE in high temperature IPA in order to investigate the influence of temperature on filter's PRE. The test method was applied to evaluate two kinds of Pall SM-PTFE filters with each different removal rating, and the difference in actual PRE between the filters in RT and high temperature IPA was discussed.

EXPERIMENTAL

Particle size distribution measurement in IPA (before and after heating)

The heating temperature of IPA was decided to be 70 °C. In the actual manufacturing process, the temperature of IPA varies with each user's condition. However, since the boiling point of IPA is 82 °C, temperature of 70 °C or lower is adopted in most cases.

For filter's PRE evaluation, we need to choose test particle to be challenged to test filters. In the previous PRE evaluation in RT IPA [6], we used the following three kinds of test particles: Platinum particle covered with polyethylenimine (Pt PEI), zirconia particle (ZrO_2), and gold particle covered with polyvinylpyrrolidone (Au PVP). These particles have each different charged state; Pt PEI is positively charged, Au PVP is negatively charged, and ZrO_2 is nearly neutral. Jin-Goo Park *et al.* [7] conducted zeta potential measurement of silica and silicon particles in water and water/IPA mixture, and reported that zeta potential of the particles approached neutral values as the concentration of IPA reached 50 volume%. In view of these facts, we assumed that ZrO_2, silica, and silicon particles have similar charged stated in 100% IPA. Since silica and silicon particles are presumed to be ones of actual contaminants during wafer cleaning process, we decided to focus on the test using ZrO_2 that shows similar behavior with silica and silicon particles.

Previously, the particle size distribution in IPA was measured by means of dynamic light scattering (DLS, *Zetasizer** Nano ZS, *Malvern**) at RT. In this time, the particle needs to be dispersed in 70 °C IPA as well. In order to confirm the fact, the ZrO_2 added to IPA (the suspending solution) in a PFA bottle was heated up to 70 °C and kept for 20 minutes; then, the solution was cooled down to RT and measured by DLS. We have assumed that if the particle is dispersed at RT after heating, it was dispersed during the heating as well. Another solution without heating was also prepared and measured for comparison. Although the measurement tool (DLS) is capable of measuring the particle size at 70 °C, we adopted the procedure described above, because measurement at high temperature needs to consider the temperature dependence of other parameters such as refractive index of the ZrO_2 and IPA, which makes direct data comparison between at RT and at high temperature somewhat difficult.

Particle challenge test of filters in RT and 70 °C IPA

In order to evaluate PRE in RT and 70 °C IPA, particle challenge tests of filters were performed using the test system shown in Figure 1. First, the test particle (ZrO_2) was added to electronic grade IPA to make suspending solution and it was poured in the reservoir (i.e. at the upstream of the disk holder). For the test in 70 °C IPA, the reservoir was placed in the water bath at 70 °C for 10 minutes to stabilize the designated temperature. For the test in RT IPA, the same filtration system was employed except for not using the water bath. Next, the suspending solution (influent) was filtered by each filter at the flow rate of 5 ml/min., and the effluent was collected in a sampling bottle. Finally, the zirconium (Zr) concentration in the influent (= C_0) and the effluent (= C) was quantified with ICP-MS (7700s, Agilent) to calculate PRE (= $[1-C/C_0] \times 100$).

Test filters were Pall 10-nm-rated and 5-nm-rated SM-PTFE membranes, both of which are filters used in the leading-edge semiconductor device manufacturing processes. Table 1 summarizes the overall conditions of this evaluation. Test was repeated five times. Note that the test condition No.1 in the table 1 is the same condition as in the previous study [6].

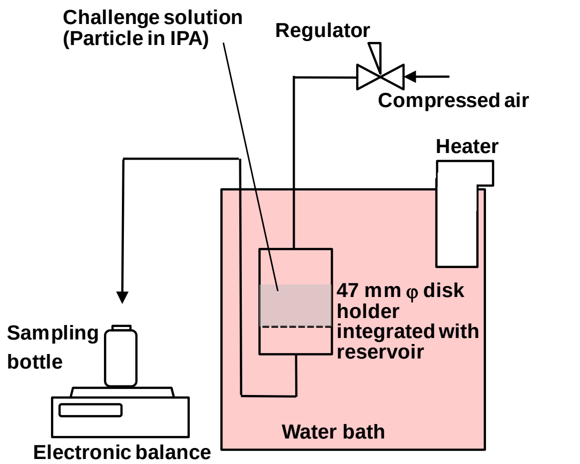

Figure 1. Schematic of particle challenge test system for PRE evaluation of filters in RT and 70 °C IPA. Test filters were 47 mm φ disk format. The water bath was only used for the tests at 70 °C to keep the elevated temperature. Constant flow rate (5 ml/min.) was implemented by adjusting the pressure regulator.

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

Test condition No.	Filter rating (nm)	Temperature	Chemical
1	10	RT	IPA
2	10	70 °C	
3	5	RT	
4	5	70 °C	

Results and Discussion

Particle size distribution measurement in IPA (before and after heating)

As shown in Figure 2, the ZrO_2 particle used for the challenge test showed almost no change in the size distribution even after heating up to 70 °C in IPA, and thus the particle in 70 °C IPA was presumed to be dispersed same as in RT IPA. These data ensure that we can directly compare the PRE at RT and 70° C in IPA.

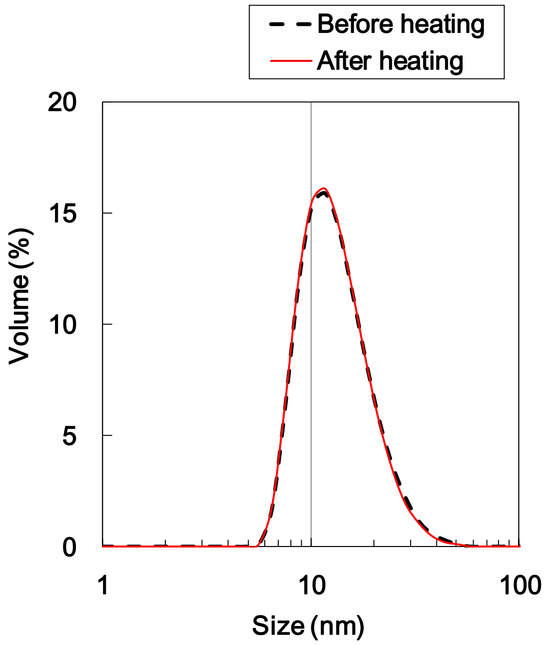

Figure 2. Particle size distribution (in volume) of ZrO_2 in IPA. This particle was used for the filter's challenge test in IPA. The dotted black line indicates the distribution before heating, and the red line indicates the distribution after heating up to 70 °C.

Particle challenge test of filters in RT and 70 ℃ IPA

Figure 3 shows results of the challenge tests in IPA. In RT IPA, both of the 10-nm-rated and 5-nm-rated filters showed sufficiently high PRE (> 99%). In contrast, PRE of the 10-nm-rated filter slightly deteriorated at 70 °C compared to that at RT, whereas the 5-nm-rated filter maintained high efficiency at 70 °C. These results indicate the filter removal rating determined based on the test in RT water is reflected on the PRE in 70 °C IPA.

In the past study, we have reported that PRE of a 12 nm-rated SM-PTFE filter decreased in 90 °C 96% sulfuric acid (H_2SO_4) [3], whereas PRE of a 10 nm-rated SM-PTFE didn't decrease in 70 °C diluted (pH1) hydrochloric acid (HCl) [5]. Considering these results including the current study, filters' PRE tends to deteriorate in high temperature chemicals, though the degree of decrease depends on the chemical and temperature employed.

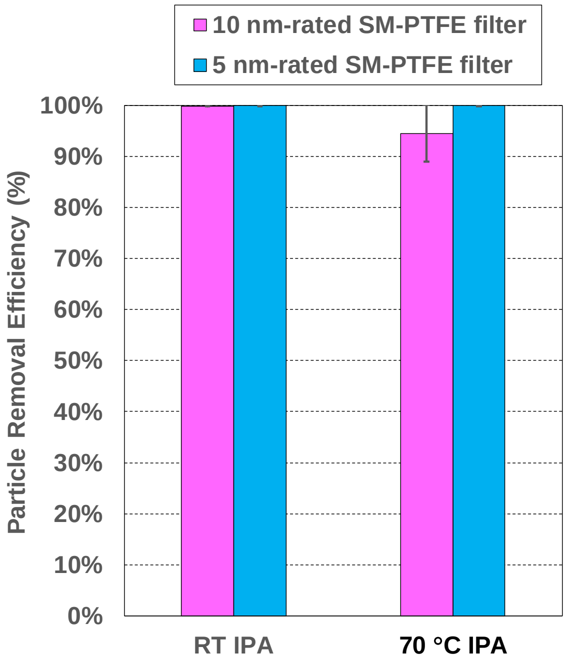

Figure 3. Results of the particle (ZrO_2) challenge tests in RT and 70 °C IPA. PRE of each test condition is shown. For each condition, tests were repeated five times and the average is shown with the error bar (standard deviation).

CONCLUSION

A PRE evaluation method of filters in high temperature IPA was developed. The evaluation was performed for Pall 10-nm-rated and 5-nm-rated SM-PTFE membrane filters with ZrO_2 particles, the size of which is around 10 nm both in RT and 70 °C IPA. In RT IPA, both of the 10-nm-rated and 5-nm-rated filters showed sufficiently high PRE (> 99%). In contrast, PRE of the 10-nm-rated filter slightly deteriorated at 70 °C compared to that at RT, whereas the 5-nm-rated filter maintained high efficiency at 70 °C. These results indicate the filter removal rating determined based on the test in RT water is reflected on the PRE in 70 °C IPA. Considering the PRE evaluation results in the past [3, 5] and the current study, filters' PRE tends to deteriorate in high temperature chemicals, though the degree of decrease depends on the chemical and temperature employed.

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