UC-006

Particle removal efficiency evaluation of filters in IPA

Tomoyuki Takakura and Shuichi Tsuzuki
Nihon Pall Ltd.
46, Kasuminosato, Ami-machi, Inashiki-gun, Ibaraki 300-0315 Japan
tomoyuki_takakura@ap.pall.com

Abstract – As part of the study to investigate filter performance in the actual conditions, particle removal efficiency evaluation method of filters in isopropyl alcohol was developed. Three kinds of particles which exhibit each different charged state in the chemical, and the sizes of which are around 10 nm, were selected as test particles for the evaluation. Pall 10-nm-rated surface-modified PTFE membrane filter was evaluated with the particles. The result was compared with the removal efficiency in deionized water. Also, the effect of adsorption on the removal efficiency in the system was discussed.

INTRODUCTION

Isopropyl alcohol (IPA) is a chemical commonly used for the wafer drying process in the semiconductor industry. The control level of contaminant in IPA is getting severer, as the feature size of the semiconductor devices decreases. Filtration plays an important role to reduce the contaminant. Particle size removed by filter for IPA is required to be smaller in accordance with the control level of the contaminant. Another important requirement of filters used in IPA is to minimize extractables from the filters themselves. From this perspective, polytetrafluoroethylene (PTFE) membrane filters are being used in more cases, whereas high-density polyethylene (HDPE) membrane filters had been most commonly used in IPA.

For filter removal rating, gold particle covered with mercaptosuccinic acid (Au MSA) is used as a test particle (i.e. challenge particle) in deionized water (DIW); MSA minimizes adsorption effect of gold particles to membranes [1]. It is the worst case condition for filter, since sieving effect almost exclusively works for particle removal. In general, however, particle removal efficiency (PRE) of filters is affected by the chemical and the temperature employed. Therefore, to know the PRE in the actual chemicals is of great importance for the appropriate filter usage. Based upon this idea, we have developed PRE evaluation methods in H_2SO_4 [2, 3] and diluted HCl [4, 5]. In this article, as part of these studies in the actual chemicals, we report a PRE evaluation method in IPA.

EXPERIMENTAL

We evaluated Pall 10-nm-rated surface-modified PTFE (SM-PTFE) membrane, which is one of filters used in the leading-edge semiconductor device manufacturing process.

Regarding test particle, Au MSA is not stably dispersed in IPA [6]. Alternatively, we chose the following three particles with the size around the filter rating (10 nm) and each different charged state because electrostatic interaction is a factor that affects PRE: Platinum particle covered with Polyethylenimine (Tanaka Kikinzoku Kogyo, Pt PEI), Zirconia particle (Nissan Chemical Industries, ZrO_2), and gold particle covered with Polyvinylpyrrolidone (Tanaka Kikinzoku Kogyo, Au PVP). We evaluated size distribution and zeta potential of these particles in IPA by dynamic light scattering (DLS, Zetasizer Nano ZS, Malvern).

Particle challenge tests of filters were performed with these particles. The test system described in Figure 1 was employed as follows: Each particle was added to electronic grade IPA in the PFA reservoir, and then the suspending solution (influent) was filtered by each membrane at the flow rate of 5 ml/min; the effluent was collected in a sampling bottle. Each Au, Pt, and Zr concentration in the influent (=C_0) and the effluent (=C) was quantified with ICP-MS (7700s, Agilent) to calculate removal efficiency (=[1-C/C_0] $\times$ 100). For reference, tests with 10-nm-sized Au MSA in DIW were performed. Also, 10-nm-rated HDPE filter was tested with Pt PEI in IPA for data comparison.

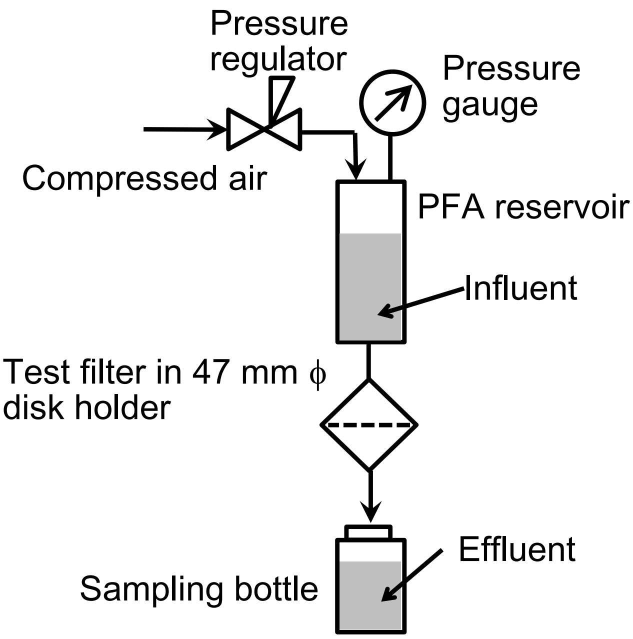

Figure 1. Schematic of particle challenge test system for particle removal efficiency evaluation of filter membranes. Constant flow rate (5 ml/min.) was implemented by adjusting the pressure regulator.

RESULTS AND DISCUSSION

As shown in Figure 2, the peak particle sizes of the three different kinds of particles were around 10 nm in IPA with a small variation in size; Pt PEI was slightly smaller than 10 nm, and ZrO_2 was slightly larger than 10 nm. These results indicate that the particles are not agglomerated in IPA, therefore can be used for PRE evaluation. The size distribution data of the Au particle in DIW, which was used for PRE evaluation in DIW, has a peak at 10 nm as reported in the previous study [1].

As shown in Figure 3, in IPA, Pt PEI was positively charged, Au PVP was negatively charged, and ZrO_2 was nearly neutral.

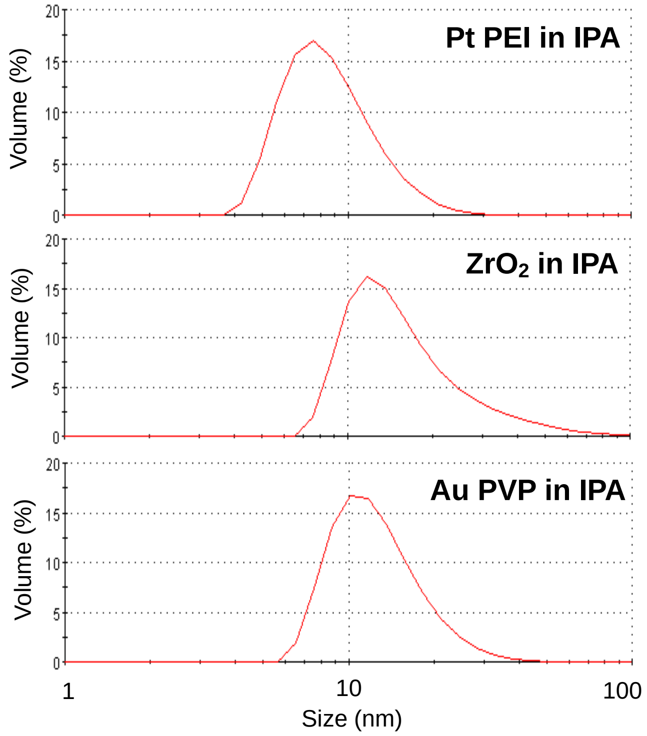

Figure 2. Particle size distribution in IPA measured by DLS. These particles were used for the challenge tests in IPA. The three particles' sizes were around 10 nm with a small variation in size; Pt PEI was slightly smaller than 10 nm, and ZrO_2 was slightly larger than 10 nm.

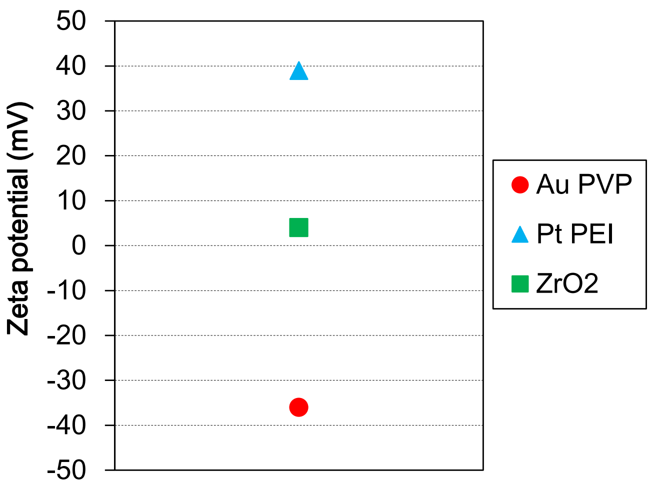

Figure 3. Zeta potential of the three kinds of particles in IPA measured by DLS. These particles were used for the challenge tests in IPA.

The size distribution of ZrO_2 remained unchanged for 24 hours after making the suspension, though it agglomerated after 1 week presumably due to the neutral charge; the ZrO_2 was used within 5 hours after preparation. The result that zeta potential of ZrO_2 shows neutral value in IPA is similar to that of silica and silicon particles [7] which are presumed to be ones of actual contaminants during wafer cleaning process.

Figure 4 shows PREs of 10-nm-rated SM-PTFE membranes challenged with three kinds of particles in IPA. PREs in IPA showed sufficiently high values for all the three kinds of particles, though we need to note each particle has some difference in size. Overall, since the SM-PTFE membrane removed three kinds of particles with each different charged states (i.e. negative, positive, and neutral) effectively, it can be concluded that this filter is capable of removing various particles that may exist in IPA.

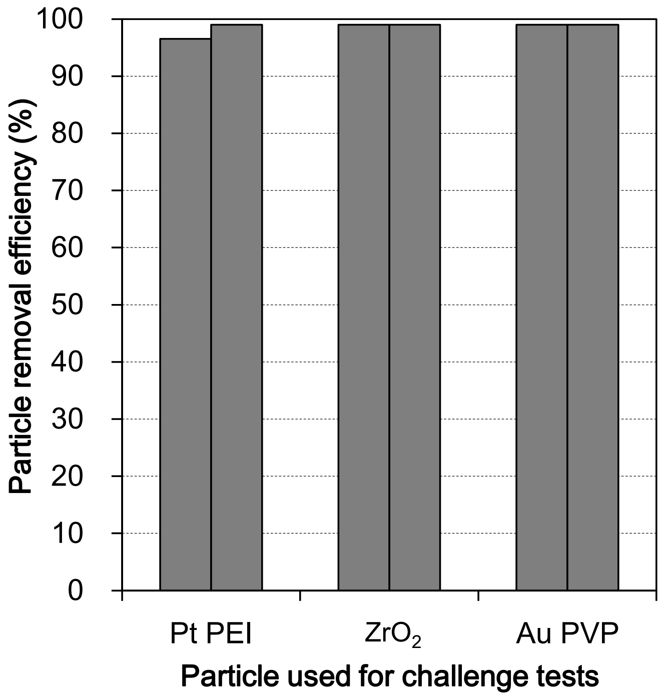

Figure 4. Challenge test results of Pall 10-nm-rated SM-PTFE membranes with three kinds of particles in IPA. Each test was repeated twice.

Figure 5 shows PRE comparison between 10-nm-rated SM-PTFE and HDPE membranes in DIW and in IPA; significant difference in PRE between DIW and IPA is shown. In DIW, both membranes showed PREs of > 95% for 10-nm-sized Au particle. In IPA, SM-PTFE membranes showed PREs of > 95%, whereas HDPE membranes were around 50%. Since the size of Pt PEI is smaller than 10 nm as shown in Figure 2, the results of HDPE membranes are reasonable if we consider only sieving effect. Thus, the result that PREs of SM-PTFE with Pt PEI in IPA were greater than that in DIW indicates adsorbing effect worked for the particle removal as well as sieving effect.

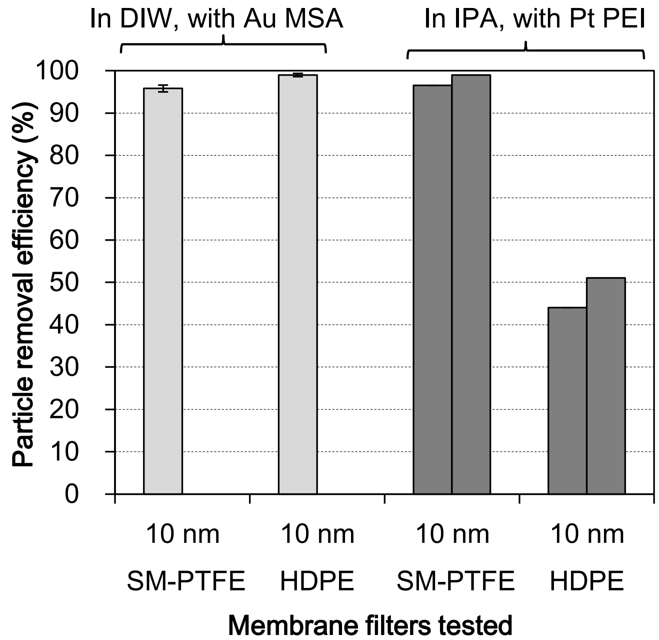

Figure 5. Challenge test results of Pall 10-nm-rated SM-PTFE and HDPE membranes in DIW (light gray) and IPA (dark gray). The challenge particles used were Au MSA in DIW and Pt PEI in IPA. The data in DIW are average of several tests with error bars. The same data in Figure 4 are shown for the results of SM-PTFE in IPA. Note that the size of Pt PEI is smaller than 10 nm as shown in Figure 2.

CONCLUSION

A PRE evaluation method of filters in IPA was developed. The evaluation was performed for Pall 10-nm-rated SM-PTFE membrane filters with three kinds of particles, the sizes of which are around 10 nm. The SM-PTFE filters showed PREs of > 95% with all the three kinds of particles. Since the SM-PTFE membrane removed three kinds of particles with each different charged states (i.e. negative, positive, and neutral) effectively, it can be concluded that this filter is capable of removing various particles that may exist in IPA. Comparison data between PREs of SM-PTFE and HDPE, and PREs in DIW and in IPA indicate that the SM-PTFE filter removed particles in IPA by adsorbing effect as well as sieving effect. Further investigations will explore the detailed mechanism of adsorption in this system.

REFERENCES

[1] T. Mizuno, et al., "A Novel Filter Rating Method Using Less Than 30-nm Gold Nanoparticle and Protective Ligand," *IEEE transactions on semiconductor manufacturing*, Vol. 22, No. 4, pp. 452-461, 2009.

[2] T. Takakura and S. Tsuzuki, "Particle removal efficiency evaluation of filters in 150 °C sulfuric acid," presented at *the 75th JSAP Autumn Meeting*, 17P-A14-6, 2014.

[3] T. Takakura and S. Tsuzuki, “Evaluation of particle removal efficiency of filters in high temperature sulfuric

acid using 30 nm liquid particle counter,” in *Proceedings of Joint eMDC & 2015 ISSM*, 2015.

[4] M. Hashimoto and S. Tsuzuki, "Removal efficiency with actual chemical for wafer cleaning process," presented at *the 60th JSAP Spring Meeting*, 28P-G8-14, 2013.

[5] T. Takakura and S. Tsuzuki, "Particle removal efficiency evaluation of filters in 70 °C diluted hydrochloric acid," presented at *the 63rd JSAP Spring Meeting*, 19p-S223-10, 2016.

[6] T. Mizuno, et al., "Dispersion stability of standard nanoparticles for filter performance evaluation," presented at *the 57th JSAP Spring Meeting*, 20P-C-7, 2010.

[7] Jin-Goo Park, et al., "Interfacial and Electrokinetic Characterization of IPA Solutions Related to Semiconductor Wafer Drying and Cleaning," *J. Electrochem. Soc.*, vol. 153, pp G811-G814, 2006.