# Novel Method to Characterize the Retention of Polydisperse Particles by Advanced Membranes

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## INTRODUCTION

Chemicals, movable and immovable parts in the fluid path, and auxiliary equipment all introduce contamination into semiconductor processes. Particulate contamination in semiconductor manufacturing processes is polydisperse in nature with the typical distribution skewed toward particle sizes <20 nm. Filters made from various polymer membranes are used by semiconductor fabs to reduce incoming contamination.

Historically, membrane retention efficiency has been characterized using Gold Nanoparticles (NP), Silica NP and Quantum dots, all of which are monodisperse in nature. Though a significant amount of information about membrane retention performance has been acquired using monodisperse, 2 nm to 1 µm particles, there is a substantial gap in understanding the outcome of a polydisperse particle challenge.

In this study, we present a novel method using atomic force microscopy (AFM) to characterize retention performance of advanced membranes when challenged with polydisperse particles whose size distribution better reflects contamination in process chemicals. Two modified polytetrafluoroethylene (PTFE) membranes with different retention ratings were tested using the new method. Results from the polydisperse retention study are compared between two filters to highlight the differences and illustrate the utility of this method to better understand retention in process chemistries.

## METHODS

- **Instruments:** Bruker FastScan AFM; Hitachi High-Technologies F-7000 Fluorescence Spectrophotometer
- **Samples:** Two modified polytetrafluoroethylene (PTFE) filters (Filter 1 and Filter 2)
- **Test particles:** Thermo Fisher Scientific Fluorescent polystyrene latex (G25 PSL) beads
- **Experiments:** 32 ppb G25 were used to challenge the filters, the filtrates and feed were collected and measured with spectrometer and AFM, respectively. Each sample was scanned as 300 images on a wafer surface, and the height of particles was collected as the particle size.

## RESULTS AND DISCUSSIONS

### G25 particle and its size distribution

G25 particles were chosen for testing because:

G25 PSL beads are usually measured by dynamic light scattering (DLS) methods, which indicate an average particle size of 25 nm. Figure 1, comparing different measurement methods for the same beads, suggests that the PSL beads are polydisperse and have an average size of 12 nm, as demonstrated by the number weighted distribution.

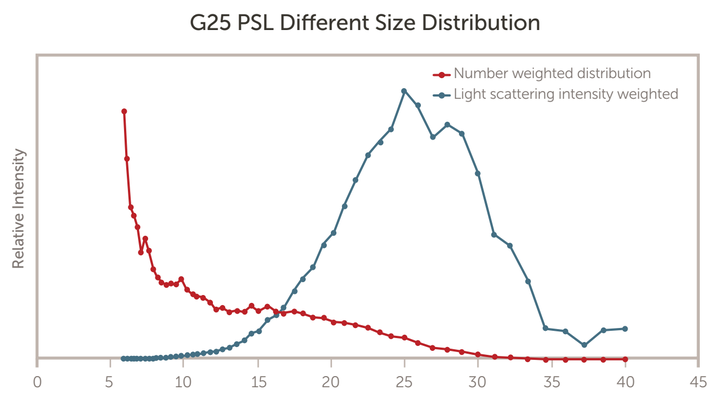


*Figure 1. G25 particle size distribution collected using Liquid Nanosizer as a number weighted size distribution (in red), the average size is about 12 nm. However, when converted to light intensity size distribution, the average size is about 25 nm which agrees with manufacturer claimed DLS test results.*

The PSL beads are spherical and are therefore easily measured by the AFM technique. G25 particle size distribution measured by AFM matches well with Cryo-EM (electron microscope) analysis results for the particle larger than 8 nm. Figure 2 shows the particle size distribution measured by both AFM and Cryo-EM.

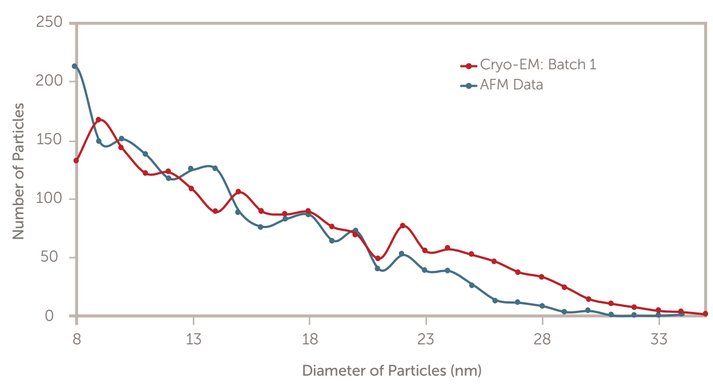


*Figure 2. AFM test results match well with Cryo-EM test results on G25 analysis when particle size is larger than 8 nm.*

### Particle size distribution analysis after filtration

Two modified PTFE membranes with different retention ratings were challenged by using 32 ppb G25 feed. Fluorescence intensity was used to calculate the retention of two filters at 1% Monolayer particle loading. The retention results of Filter 1 and Filter 2 are 75.6% and 68.5%, respectively. 300 AFM images from each sample were scanned. The particles ≥5 nm in the feed and filtrate were further analyzed and compared. Figure 3 shows the particle size distribution of the feed and filtrate. Clearly, after filtration, the particle counts in all size ranges decrease when compared to the feed. Also, in all size ranges, Filter 1 shows fewer particles than Filter 2, which aligns with retention ratings and fluorescence retention test results.

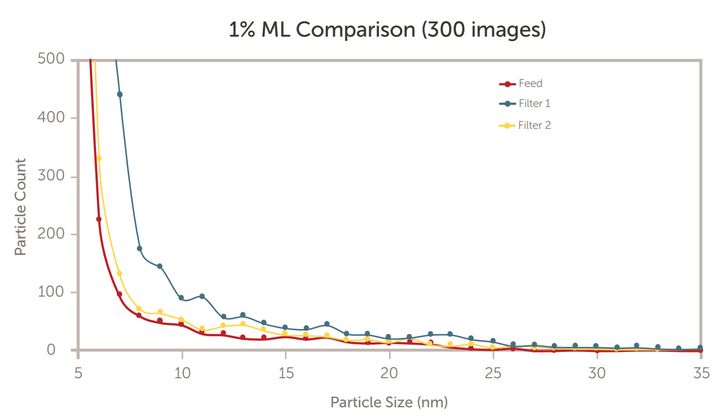


*Figure 3. Particle size distribution comparison before and after filtration.*

### An estimation of particle retention by different size bins

Two significant reasons limit this method as a quantitative method:

- AFM detects all particles and non-volatile residues on the wafer surface, which includes the mixture of G25 beads and any defects on the wafer surface.
- The uniformity of particles dispersed on the wafer.

Considering these two reasons, the test system was flushed for >1 hour to avoid possible shedding of particles. We compared the particle size distribution (5 nm to 35 nm) of the two filters before and after filtration. In this case, we could estimate the retention performance in each single size bin (see Figure 4). Filter 1 has a better retention performance at almost every size bin from 5 nm to 35 nm. The results agree with our regular fluorescence retention results and the filter retention ratings.

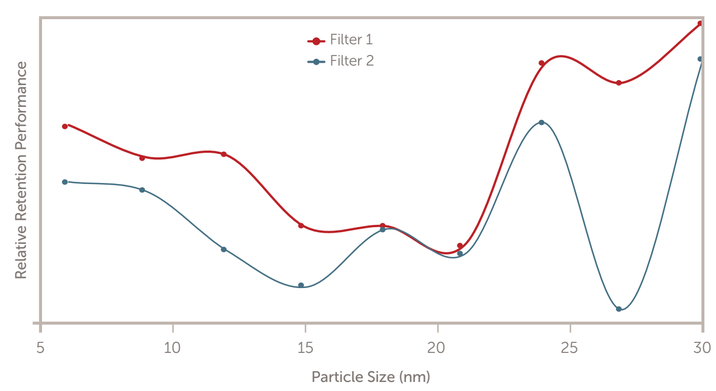


*Figure 4. Filter performance comparison: Filter 1 has a better performance than Filter 2 at almost every size bin from 5 nm to 35 nm.*

## SUMMARY

- Polydisperse, spherical G25 particles, ranging in size from 3 to 35 nm, are useful for evaluating relative filter performance
- G25 size distribution collected with AFM matches the results collected using other characterization techniques. The AFM method can provide particle information at particle sizes as low as 5 nm particles
- Filter retention performance can be observed by comparing the changes in the G25 size distribution post filtration
- While not quantitative, the method provides retention information in each size bin (from 5 to 35 nm)
- Filter 1 has a better performance than Filter 2 in almost all size ranges which aligns with fluorescence test results

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