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Characterization of Nylon Membranes for Nano-Particle Filtration

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ABSTRACT

The purity of fluids exposed to a wafer is critical as the node sizes are approaching low single digit nanometers. The potential impurities could be solid or semi-solid particles, soluble metal cations, anions, organic species, or gases. All these need to be removed for optimum yield and minimal defects. The technical challenge associated with reducing the node sizes also relates to membranes production as membranes with low single digit pore sizes are needed to remove nanometer impurities. The interest in nylon membrane is twofold as it can mechanically remove fine particles as well as by its adsorptive capabilities. In this study, nylon membranes with pore sizes of 2, 5, 10, and 20 nm were characterized for microstructure, flow rate versus pressure drop, bubble point, and metal extractables in PGMEA.

Keywords: Purification, Filtration, Defect reduction, Photoresist, High Purity Chemicals

1. INTRODUCTION

Improvements in lithographic resolution process in semiconductor industry uses multiple lithography steps to produce multi-level circuits, which brings challenges of contamination control in all aspects of the lithography process [1]. Due to multiple steps involved in the lithographic process, particulate contamination, microbubbles, and metallic contamination are detrimental to the wafer processing. As the node sizes shrink, the removal of particles that are smaller than the node size becomes critical. As reported by the International Technology Roadmap for Semiconductors 2015 (ITRS2015), for features at or smaller than 14 nm, it is critical to remove particles up to 7 nm [2]. The reduction of feature sizes to 7 nm or smaller requires a tighter membrane filtration for particle removal and yield improvement. In this regard, the endless need for higher purity of fluids that encounter wafer demands better reduction of the nanoparticles which, if not removed, will affect the performance and the yield of the product. In addition, trace metals in these fluids can deposit and diffuse into the circuit components and have damaging impact. Both the nanoparticles and the metals need to be removed to the lowest level possible. Nylon membrane has both amine and carboxyl functional groups on the surface which can absorb metals. The removal of the nanoparticles and metals [3] can be achieved using a nylon membrane rated to single digit pore size.

2. EXPERIMENTAL PROCEDURES

Bubble Point

The 47 mm filter membrane discs were installed in housings and wetted with isopropyl alcohol (IPA). Air was then introduced into each housing upstream of the filter membrane to remove any remaining IPA. Tubing was attached downstream of the filter membrane and the free end was placed into a beaker of water. The pressure was steadily increased to a value 10 psi less than the anticipated bubble point and then in 1 psi increments, allowing 5-10 seconds of stabilization in between each incremental increase until a steady stream of bubbles was observed exiting the tube. The terminal pressure was recorded as the bubble point.

Flow vs Pressure Drop

The 10-inch cartridges were housed and flushed with De-ionized (DI) water. Cartridge differential pressure was then measured at flow rates of 0.5, 1.0 and 1.5 gallons per minute.

SEM Imaging

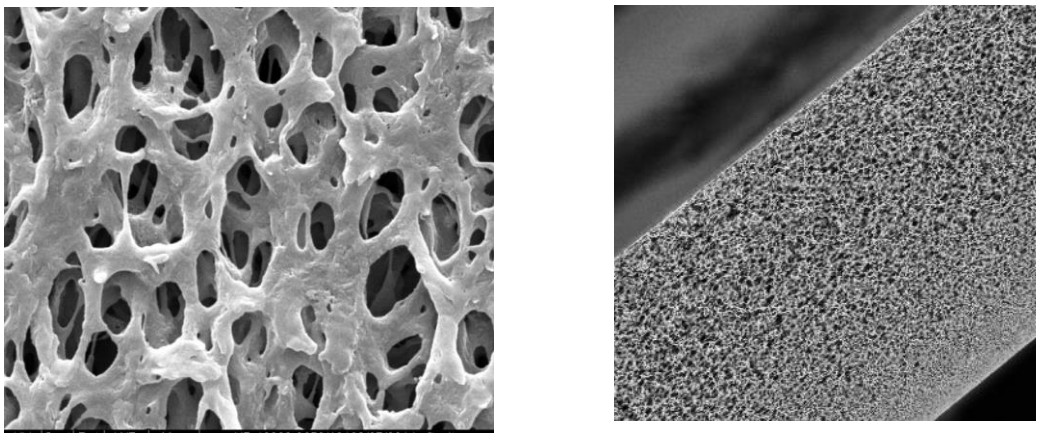
Filter membrane cross sections were prepared by a freeze-fracture technique employing liquid nitrogen. The cross sections and upstream and downstream membrane surfaces were mounted with carbon tape onto aluminum stubs. The mounted samples were sputter coated with gold for SEM examination under high vacuum.

Metals Extraction in PGMEA

The 10-inch filter cartridges were soaked in PGMEA overnight and the solution was analyzed with ICP-MS.

3. RESULTS AND DISCUSSION

Microstructural analysis of the membrane cross-section and surfaces showed high pore volume with an asymmetric structure of larger pores on the upstream that reduce in size moving directionally downstream. This structure should allow for high contaminant holding capacity while operating at lower pressure drop that is critical for nano-filtration processes.



(a) (b)

Figure 1. Microstructure of the (a) pore structure of the nylon membrane, and (b) cross-section of the membrane showing the gradient structure of the pore sizes from upstream to downstream of the membrane.

Flow Rate versus pressure drop for nylon membranes rated 1, 5, 10, and 20 nm are shown in Figure 2. The tighter the membrane, the higher the associated pressure drop for the same flow rate.

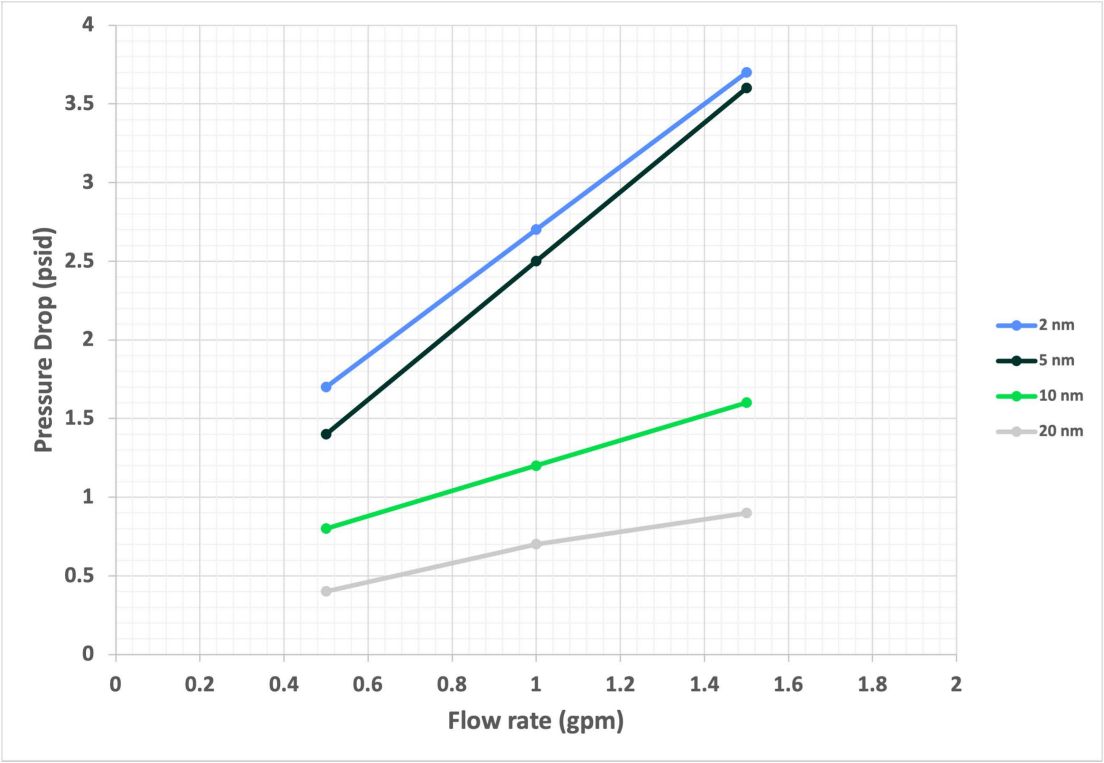


Figure 2. Flow rate versus pressure drop for various nylon nano-filter membranes.

Bubble point is indicative of the largest pore size in a membrane. It is expected that by making a tighter membrane the largest pore size also decreases. The results of the bubble point with isopropanol alcohol are shown in figure 3. The bubble point of the membranes increases with reduction of membrane pore size from 20 nm to 2 nm.

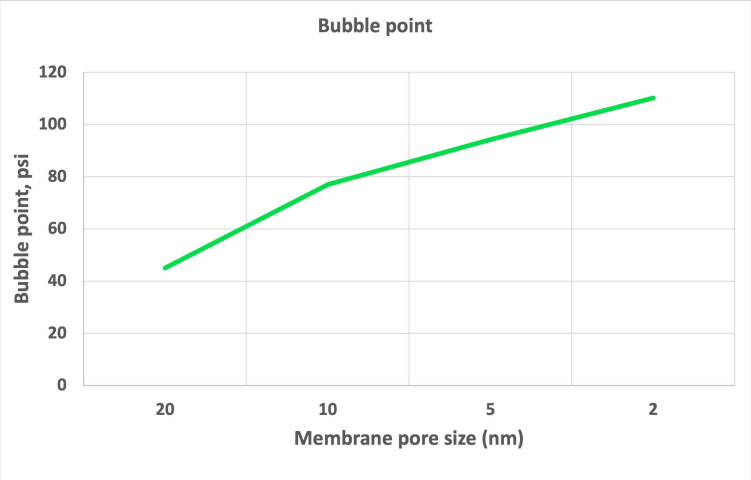


Figure 3. The bubble points of 20, 10, 5, and 2 nm nylon membranes.

Extractables from a membrane are a big concern when used with microelectronic fluids that contact the wafer. The extractables could be metallic, organic, or particulates. The results of 24 hours soak of the nylon membrane in propylene glycol methyl ether acetate (PGMEA) is shown in Table 1. The control is the PGMEA that was used for the soak test. For some metals, the concentration is reduced which can be attributed to the nylon capability for adsorption of ionic species as reported before.[3]

Table 1. The metal concentration in ppb after soaking 24 hours in PGMEA.

Sample	B	Ca	Fe	Mg	K	Na
Control	0.4	1.2	0.4		0.3	12
Sample 1	<0.1	1.5	<0.1	<0.1	0.2	0.2
Sample 2	<0.1	0.7	0.1	<0.1	0.1	0.1

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