Industrial filtration

Metal reduction from highly viscous microelectronics fluids

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Keywords

Purification, filtration, defect reduction, photoresist, high-purity chemicals

Reprinted with permission: Proceedings Volume 13428, Advances in Patterning Materials and Processes XLII; 134282U (2025)
doi.org/10.1117/12.3057201

Event: SPIE Advanced Lithography + Patterning, 2025, San Jose, California, United States

Abstract

Various high-viscosity chemicals are used in photoresist applications. The metal content of these chemicals is of concern as they may contaminate wafers during processing and post-processing by diffusing into circuit components and affecting their performance over time. The high viscosity of these chemicals results in a high pressure drop across porous structures such as membranes or monoliths. A new monolith microstructure has been developed to allow processing of high-viscosity fluids for metal reduction while maintaining high flow rates. Experiments were performed to compare the flow rates and the metal reduction of the monoliths. In addition, the metal reduction capacities were compared. The results of metal reduction will be presented using the new monolith.

Introduction

High-purity fluids and gels are needed for various microelectronics applications [1]. These fluids include high-polymer solutions, epoxies, amines, photoresists, masks, and barrier layers. The low metal content of these fluids is of prime importance because the metals interfere with main functions such as conductivity, resistance, capacitance, and dielectrics [2]. Purification methods such as distillation cannot be employed for these high-viscosity solutions because they lead to phase separation, but ion exchange technology can be used. However, the packed bed of the ion exchange beads is not very efficient. A large volume of beads is needed, making it difficult to reach a purity of low parts per trillion (ppt). There are also membranes with grafted ion exchange properties that can be used for removing metals. These membranes have pore sizes that are smaller than one micron. Such structures are associated with high pressure drops when used for high-viscosity fluids. Nevertheless, the flow of high-viscosity fluids through thick porous structures has been studied [3]. Despite these studies, the combined effects of viscous flow in porous structures and ion exchange properties are still not well known. The objective of this study was to develop structures that can operate with high-viscosity fluids at high flow rates while efficiently removing metal ions.

Experimental procedures

Two ion exchange chemistries were used in the design of different monoliths. Sulfonic acid (strong cation exchange) chemistry is denoted as SCP, while amino phosphate (chelating) chemistry is denoted as APP. Both use H ions for ion exchange. The two chemistries were used in monoliths with two different densities. One monolith was denser with smaller mean pore size, known as MIP-UHP SCP or MIP-UHP APP, and the other was less dense with larger mean pore size, known as MIP-HV SCP or MIP-HV APP. These structures were subjected to fluids at various viscosities and upstream pressures, and flow rates were measured at each viscosity and pressure. In addition, propylene glycol methyl ether acetate (PGMEA) was spiked with common metals of interest in microelectronics manufacturing and passed through the monolith to measure the metal removal capability of the two chemistries. The metal content of the spiked and the purified samples were analyzed using inductively coupled plasma mass spectrometry (ICP-MS).

Results and discussion

The results of flow rate measurements at various viscosities and pressure settings are shown in Figure 1. The samples listed as UHP are denser media with a typical ion exchange capacity of 450 mEq per 10 in. cartridge, while the HV samples have more open structures with a typical ion exchange capacity of 250 mEq per 10 in. cartridge.

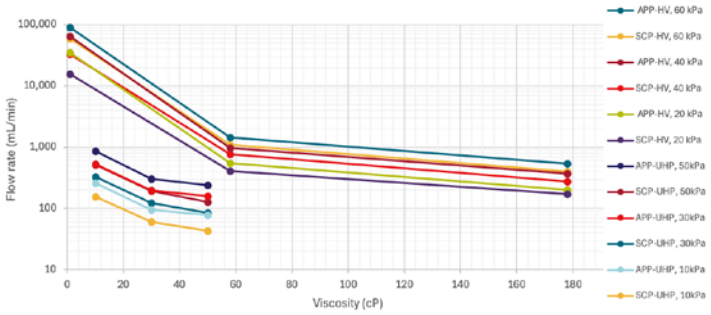


Figure 1. Flow rate versus pressure at various viscosities.

The effect of increasing viscosity on flow rate is significant. The relationship between these two factors is a negative exponential, meaning that the flow rate will be reduced exponentially by increasing the viscosity. For the UHP grade, the flow rate at viscosities larger than 50 cP is not practical as the flow rate drops to less than 100 mL/min per 10 in. cartridge. In comparison, the HV grades can operate at 10 times faster flow rate at comparable upstream pressures. For fluids with viscosities higher than 50 cP, both the SCP and APP in the UHP format did not result in meaningful flow rates for a 10 in. cartridge.

Higher flow rates are seen when the pressure across the monoliths is increased across any monolith grade. However, the increase in flow rate because of increase in differential pressure is not as pronounced as lowering the viscosity. There is a linear relationship between the increase in differential pressure and the flow rate. This behavior is shown in Figure 2.

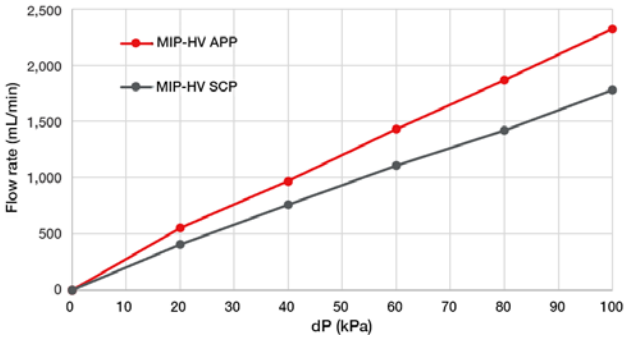


Figure 2. Flow rate versus differential pressure (dP) across MIP-HV SCP and APP monoliths using a glycerin solution at 58 cP.

The metal removal efficiency of both structures with either chemistry was assessed. The results are summarized in Figures 3–6. Both MIP-HV and MIP-UHP demonstrated a high removal efficiency of >90% for total metal ion contamination in PGMEA. The results of metal removal show that the low-density MIP-HV with the two chemistries of SCP and APP exhibit varying affinities toward different metals. The APP chemistry is more effective in removing heavier metals with higher valence (e.g., Mg, Ca) while the SCP chemistry is more effective at removal of lighter metals that are monovalent. The denser structure of MIP-UHP with both chemistries shows very effective removal of all metals.

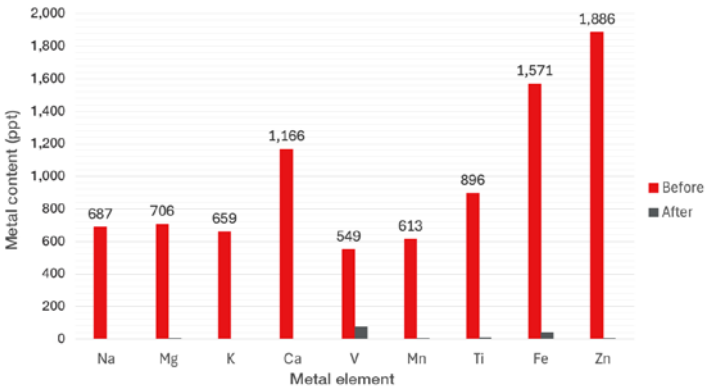


Figure 3. Metal removal from PGMEA by single pass using MIP-HV SCP grade (2.0 L/min).

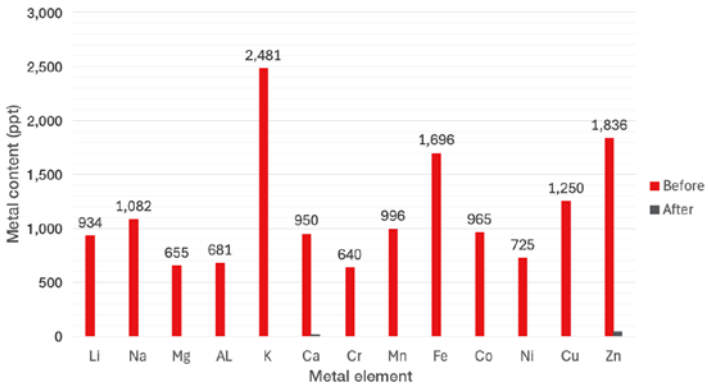


Figure 4. Metal removal from PGMEA by single pass using MIP-UHP SCP grade (0.7 L/min).

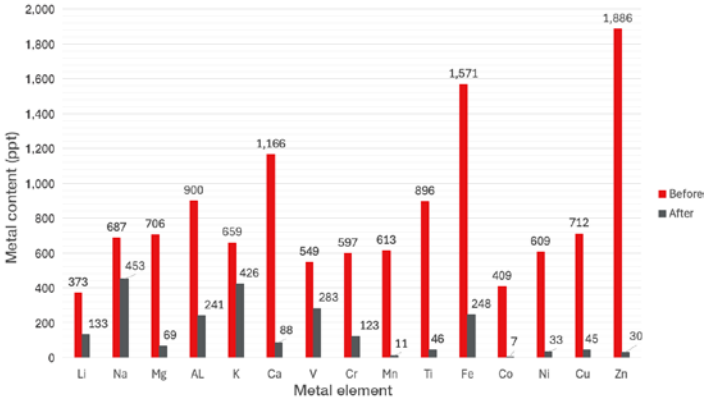


Figure 5. Metal removal from PGMEA by single pass using MIP-HV APP grade (2.0 L/min).

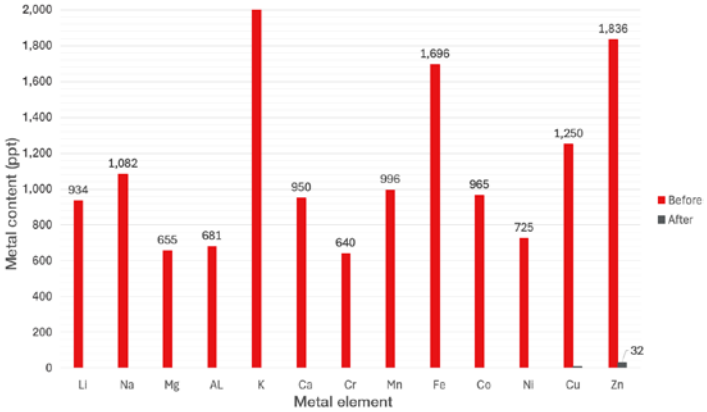


Figure 6. Metal removal from PGMEA by single pass using MIP-UHP APP grade (0.7 L/min).

Conclusions

High-viscosity fluids are used in the manufacturing process of integrated circuits. These fluids have metal impurities that if not removed could cause functional and reliability issues. There are very limited options for removing metals from these fluids. Two ion exchange chemistries were used to design monoliths with porous structures that could accommodate reasonable flow and reduce metal content. Both ion exchange chemistries of SCP and APP were effective at removing metals from PGMEA. The SCP chemistry showed the capability for removal of most metals while the APP chemistry was more efficient at removing heavier metals that are multivalent.

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doi.org/10.1117/12.3057201

Event: SPIE Advanced Lithography + Patterning, 2025,
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