# Dead-end 式薄膜微過濾處理化學機械研磨廢水: 前處理方式之評估及其影響

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## 摘 要

化學機械研磨(chemical mechanical polishing，CMP)技術因其平坦化效果佳，故近年來被廣泛應用於半導體製程，然而該製程需使用大量超純水以洗淨晶圓表面污染物，因此對於水資源匱乏的台灣來說，CMP 廢水的處理及再利用將越顯重要。而於氧化層研磨製程中因使用含高濃度二氧化矽砥粒的研磨液，導致氧化層 CMP 廢水含有大量微細顆粒，使得此股廢水因不易固液分離而難以回收；目前半導體廠多以傳統混凝沉澱方式處理此股廢水，而為使細小顆粒可被有效去除皆採過量加藥以確保處理水質，但卻產生大量污泥造成後續處理負擔。因此，本研究以混凝/膠凝前處理配合薄膜微過濾處理氧化層 CMP 廢水，以降低混凝劑使用量並改善出流水水質，且可放大廢水中懸浮顆粒大小以減輕薄膜阻塞現象。

本研究首先以常見之混凝劑如多元氯化鋁、硫酸鋁及硫酸鐵進行混凝試驗，以探討總矽移除之最適操作pH值區間、加藥量及混凝劑種類；並添加膠凝劑如陽離子性、中性、陰離子性聚丙烯醯胺及聚氯化己二烯二甲基胺、聚氯甲基一氧三環二甲基胺兩種陽離子性聚合物以放大膠羽顆粒，減輕薄膜之不可逆阻力。於微過濾程序則採低壓(0.65 kg/cm$^2$)過濾，以材質為聚四氟乙烯、孔徑為0.5 μm之薄層複合膜(thin-film composite membrane)進行試驗，紀錄累積濾液體積隨時間之變化，用以評估濾液通量改善情形。於濾程結束後以濾液進行反沖洗，求得反洗後之清水通量以評估薄膜之清水通量回復率並計算薄膜之不可回復阻力。

實驗結果顯示，多元氯化鋁混凝效果較佳，其最適處理 pH 值區間落於偏酸性範圍(pH 值為 5 ~ 7)且劑量為 30 mg/L 時即可達不錯之效果，進行薄膜微過濾時發現，混凝前處理可減緩薄膜通量下降趨勢；而添加微量(0.1 mg/L)陽離子性聚丙烯醯胺則可有效放大膠羽顆粒，且使薄膜通量上升至未添加膠凝劑時的兩倍，亦可減輕薄膜積垢現象。此外，於膠凝程序後添加次氯酸鈉作為氧化劑可降低薄膜之不可回復阻力，提升薄膜清水通量回復率。而經長期性薄膜過濾試驗發現，若單純以混凝程序作為薄膜前處理程序，則薄膜之不可回復阻力上升情形嚴重，因此，有必要於前處理程序添加膠凝劑以減少薄膜阻塞現象。

關鍵字:化學機械研磨廢水、微過濾、積垢、矽、廢水回收

# Treatment of Chemical Mechanical Polishing Wastewater by Dead-end Microfiltration : Evaluation of Pretreatment Process

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## Abstract

Chemical mechanical polishing process is a common practice for wafer planarization of the IC manufacturing. In the process, colloidal silica is used to planarize the oxide wafer surface. In the mean time, a large amount of ultrapure water is used to clean the surface of the wafer. The CMP wastewater, therefore, contains large quantity of fine particles. In Taiwan, most fabs treat the CMP wastewater with the traditional coagulation/sedimentation process, resulting in large quantity of sludge required to be disposed. A more logical solution is to reclaim the water resource from the immense amount of wastewater. This study proposed an innovative CMP wastewater treatment process with microfiltration (MF) coupled with a pretreatment of coagulation/flocculation.

In the experiment, the wastewater was first filtered through a polytetrafluoroethylene (PTFE) membrane of pore size 0.5 μm at a low vacuum pressure (0.65 kg/cm$^2$). The permeate flux and the irreversible fouling of the membrane were monitored. The SEM image of the membrane confirmed the serious fouling by the CMP wastewater. The cake layer was dense and non-porous in appearance. Different coagulants and flocculants were applied in the pre-coagulation process, and their effects on permeate flux and irreversible fouling of the membrane were determined. Results showed that polyaluminum chloride was the most efficient coagulant, which had a wider pH range for total silica removal. In addition, solid silica removal favored acidic condition while alkaline condition enhanced dissolved silica removal. Addition of trace amount of cationic polyacrylamide after the coagulation increased the size of the floc and improved the permeate flux. The addition of oxidant, namely, sodium hypochlorous, after flocculation reduced the irreversible fouling and promoted the pure water flux recovery of the membrane.

Key words: CMP wastewater, wastewater recovery, MF, fouling, silica removal