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Use of laminar flow water storage tank (LFWS) to mitigate the membrane fouling for reuse of wastewater from wafer processes

Darren Delai Sun*[1] and You Wu[1,2]

[1]School of Civil and Environmental Engineering, Nanyang Technological University, Singapore 639798
[2]Raffles Institution, 1 Raffles Institution Lane Singapore 575954

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Abstract. This study employed the modified fouling index (MFI) to determine the performance of a two-step recycling system – a membrane filtration integrated laminar flow water storage (LFWS) tank followed by an ion exchange process to reclaim ultrapure water (UPW) from the wastewater generated from semiconductor wafer backgrinding and sawing processes. The first step consisted of the utilization of either ultrafiltration (UF) or nanofiltration (NF) membranes to remove solids in the wastewater where the second step consisted of an ion exchanger to further purify the filtrate. The system was able to produce high purity water in a continuous operating mode. However, higher recycling cost could be incurred due to membrane fouling. The feed wastewater used for this study contained high concentration of fine particles with low organic and ionic contents, hence membrane fouling was mainly attributed to particulate deposition and cake formation. Based on the MFI results, a LFWS tank that was equipped with a turbulence reducer with a pair of auto-valves was developed and found effective in minimizing fouling by discharging concentrated wastewater prior to any membrane filtration. By comparing flux behaviors of the improved system with the conventional system, the former maintained a high flux than the latter at the end of the experiment.

Keywords: modified fouling index; UF/NF membrane; ultrapure water; water reuse

1. Introduction

Semiconductor manufacturers use immense amount of water, of which a large portion is used to produce ultrapure water (UPW). More than 7.5 m^3 of water is required for the manufacturing of one 200-mm wafer. A large facility can consume as much as 1,100 m^3 UPW per day (Klusewitz *et al*. 2002), which produces equivalent amount of wastewater to be treated. Hence, a water conservation program in the semiconductor industry is to serve two aims: to reduce the overall manufacturing cost and to reduce the significance of negative impacts on the environment. For the past few years, a great effort has been made to reduce water consumption in the industry (Allen *et al*. 1999, Veltri *et al*. 2000, You *et al.* 2001, Farmen *et al.* 2002). In most of the cases, membrane systems as a matured technology are widely applied in both wafer processes and non-wafer processes to reclaim the wastewater for reuse purposes.

In the wafer backgrinding and sawing processes, UPW is employed to cool and rinse semiconductor wafers. When different type of wafers made of silicon (Si), gallium arsenide (GaAs) and indium

* Corresponding author, Ph. D., E-mail: ddsun@ntu.edu.sg

phosphide (InP) are being processed, the wastewater normally contains high concentration of fine Si, GaAs, or InP particles (as small as 0.05 μm). It has been proved that ultrafiltration (UF) or nanofiltration (NF) is effective in purifying the wastewater where the effluent is ready for reuse. However, particulate fouling on membrane surface that result in the reduced life span of the membranes implies an increased recycling cost. Currently, the membrane life span is typically from 15 to 18 months in such applications, which accounts for approximately 65% of the recycling cost (Wu 2002a, Knapp *et al.* 2010). Hence, there is an urgent need to look for an alternative cost effective solution to resolve the problems caused by membrane fouling.

Membrane fouling, cake deposition and removal behaviour for fine silicon (Si), gallium arsenide (GaAs) and Indium phosphide (InP) particles are different from those observed with the natural organic matter (NOM) and the bacterial extracellular polymeric substances (EPS). This is due to these fine particles as well as their capacity to physiologically modify their surface in response to environmental conditions. Detailed laboratory studies on the cake formation mechanism and the removal of fine particle in MF/UF (Chen *et al.* 1997) and NF processes (Listiarini *et al.* 2009, 2010, 2011) have been reported which were critical for the understanding of the mechanisms of fouling due to fine particles. These results provided insights to the possibilities of employing an additional measure prior to UF/NF membranes filtration for the mitigation of fouling problem due to fine particles. This new design involves additional measures that are able to improve the current industrial practices and reduce its operation cost significantly.

This paper presents an improved membrane system coupled with a laminar flow wastewater storage (LFWS) tank to recycle the wastewater generated from wafer backgrinding and sawing processes. The LFWS tank was designed with a clarify zone at the bottom which allows suspended solids (SS) to be settled and discharged prior to membrane separation. This research work consists of three parts of study. Part I aims to characterize the wastewater for treatment and recycling; Part II intends to correlate membrane fouling with solid concentration of the wastewater and Part III focuses on membrane fouling reduction by system improvement.

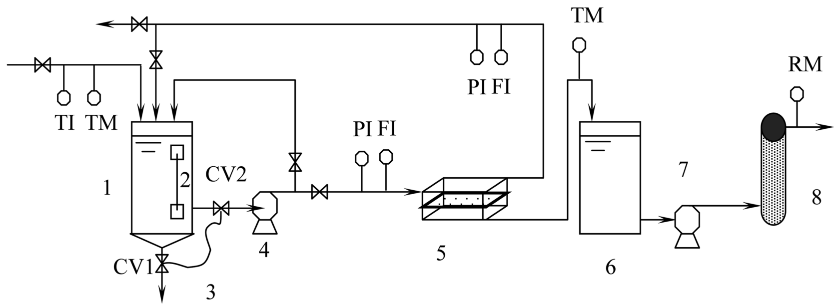
2. Materials and methods

2.1 Wastewater characterization

An InP wafer was grounded on the rear side and then sawed in DGF 841 and DAD 341 machines (Disco Corporation, Japan). The whole process involved a few major steps: rough grinding, fine grinding, cooling and sawing. During the processes, Type E-1 ultrapure water (ASTM, 1999) was continuously injected through a nozzle for flushing and cooling of the wafer. The wastewater collected from these processes was used for characterization and experiment.

Particle size distribution (PSD): Particle size of the waste streams was analyzed using a Mastersizer Microplus Ver 2.18 (Malvern, UK). It measures particle size distribution based on the principle of laser diffraction in the range of 0.05 to 550 mm.

Total suspended solids (TSS) and total organic carbon (TOC): TSS was measured using the method described in Standard Methods (APHA, 1995) Section 2540D. TOC was measured by a Shimatzu 5000 TOC analyzer.



1 LFWS tank	5 Membrane test cell	TI: Temperature gauge
2 Level switch	6 Permeate tank	PI: Pressure gauge
3 Auto-valves	7 IX feed pump	FI: Flowrate gauge
4 Feed pump	8 Ion-exchanger	TM: Turbidity meter
		RM: Resistivity meter

Fig. 1 Schematic diagram of the recycling system

Table 1 Membrane properties and operating conditions

Membrane Type	NTU-2120	NTR-7250
Material	Polyamide	Poly(vinyl alcohol)/polyamide
Molecular weight cut-off (Da)	20,000	600
Effective surface area (cm^2)	60	60
Membrane surface charge (mV)	NA	-5 (at pH=7)
Operating pressure (MPa)	0.1 – 0.4	0.98 – 1.96
Maximum operating pressure (MPa)	0.5	2.9
Feed pH range	2 - 11	2 - 8
Operating temperature (°C)	< 60	< 40

Viscosity and density: The viscosity was measured with Brookfield Viscometer (Brookfield Engineering laboratories, Inc. USA) and the density was measured using a standard SG bottles.

2.2 Membrane and testing unit

Two types of flat sheet membrane: an UF NTU2120 membrane and a NF NTR7250 membrane were used, both were supplied by Nitto Denko Corporation, Japan. Their properties and operating conditions are summarized in Table 1. The membranes were placed in a C10-T test cell for the experiments. All membranes were stabilized with UPW for at least 4 hours prior to any experiment. A laboratory-scale wastewater recycling system was designed and constructed according to the schematic diagram as shown in Fig. 1. The on/off status of the feed pump was controlled by a level switch (High-Low 2 levels) installed in the collection tank. The pump starts at high water level and stops at low water level. An ion-exchanger packed with IR120 and IR420 resins (Rohm & Hass, USA) in the ratio of 2:3 was used as the polishing stage in the system. The system was set up in Singapore Technology Assembly Test Services Ltd. and operated in a continuous operating mode except for backwash. The membranes in a C10-T test cell can be backwashed by swapping the flow inlets and outlets.

2.3 Flux measurement

The permeate flux was defined as the volume of solution passing through the membrane per unit area and time. The flowrate was recorded online by a computer-based apparatus during the experiment and used to compute the flux using Eq. (1). During flowrate measurement, the operating pressure was maintained at 0.2 MPa for the UF membrane and 0.7 MPa for the NF membrane, respectively. Every point collected and computed by the computer was the average of 3 readings so as to avoid possible errors.

$$J = \frac{Q_v}{A_E} \tag{1}$$

where

A_E = Effective membrane area (m^2)
Q_v = Volumetric flowrate (l/hr)
J = Permeate flux ($l/m^2/hr$)

2.4 Laminar flow water storage (LFWS) tank

As shown in Fig. 1, the LFWS tank is designed to reduce the suspended solids in the feed water before being pumped into the membrane test cell. In comparison with a conventional tank, the LFWS tank includes two features to achieve this purpose: a turbulence reducer and a pair of auto-valves. The turbulence reducer is able to minimize the mixing effect between the incoming wastewater and concentrate at the bottom. The pair of control valves (CV1 and CV2) are installed at the membrane feed outlet and pre-reject line. Prior to startup of the feed pump, CV1 will open to discharge the concentrate from the tank for a while. Then, CV1 closes and CV2 opens for the filtration process. Under the laminar flow condition, the feed water containing the total suspended solids (TSS) s was introduced into the tank from the side of the wall at the top of the tank. TSS in water under laminar condition, the aggregate of fouling TSS through the interactions with the wall and growing up which will be settled in the bottom of the tank. This interactions between the wall and TTS will help the growth of the TDS size and therefore to reduce the tank size through the reduction of the hydraulic retention time (HRT) The pre-separated feed water was then introduced into the cell for membrane filtration test. As to be discussed in the following sections, a reduced TSS concentration would reduce the fouling tendency of the membrane by the wastewater.

3. Results and discussion

3.1 Wastewater characteristics

An understanding of the nature of wastewaters is essential to the design and operation of collection, treatment, and recycling facilities and in the engineering management of effluent quality. Ten grab samples were taken from different steps while the InP wafer was being processed. The results are summarized in Table 2. It can be seen from the results that TSS concentrations vary from 50 to 440 ppm. During the cooling step, less suspended solids were washed away. Similar results were obtained at the beginning and end of the grinding and sawing processes. However, very high TSS contents were observed during rough grinding. The turbidity was much correlated to TSS while

Table 2 Summary of wastewater characteristics (sorted by TSS)

Test	TSS (ppm)	Turbidity (NTU)	PSD (μm)	TOC (ppm)	Viscosity (Ns/m^2)	Density (kg/m^3)
Run 1	56	466	0.07-1.44	2.2	0.0025	1.0029
Run 2	74	674	0.05-1.52	1.2	0.0025	1.0043
Run 3	90	871	0.07-1.28	1.3	0.0025	1.0058
Run 4	160	2350	0.05-1.14	1.3	0.0030	1.0081
Run 5	184	2719	0.05-1.82	1.2	0.0030	1.0082
Run 6	190	3323	0.05-1.72	1.3	0.0030	1.0089
Run 7	280	9030	0.06-1.36	1.2	0.0030	1.0096
Run 8	380	20320	0.05-1.42	1.4	0.0035	1.0147
Run 9	425	21445	0.05-1.68	1.4	0.0035	1.0166
Run 10	440	26327	0.05-1.48	1.1	0.0035	1.0232

the viscosity and density slightly increased with increasing TSS. The TOC was very low, implying a low organic content. Other anion and cation concentrations such as chloride, fluoride, calcium, sodium, were also low (Data not shown here). This was due to that the water used for wafer cleaning and cooling was ultrapure and there was no significant change in the composition during wafer grinding and sawing. The results clearly indicate that the wastewater should be able to be reclaimed to ultrapure water by ultrafiltration or nanofiltration membranes and reused for the same process. The membranes achieved >99.99% removal of suspended solids (measured by turbidity). After further polishing by the ion-exchanger, the resistivity of the reclaimed water was constantly greater than 16.0 mega ohms-cm, meeting the Type E-III UPW specifications [11] (ASTM, 1999).

3.2 Fouling tendency

One of the major problems in pressure driven membrane processes is the reduction of the flux far below the theoretical capacity due to membrane fouling. It has been generally agreed that there are five principal fouling mechanisms: a) concentration polarization, b) cake formation, c) inorganic precipitation, d) organic adsorption, and e) biological fouling (Zhou *et al.* 2002). As it can be seen from the wastewater characteristics, only one of the five fouling mechanisms has a direct impact on the membrane in this case, i.e., cake formation resulted from particulate deposition. The wastewater actually contains little ionic contents, organics and microorganisms. However, the wastewater produced from the grinding and sawing processes contains high concentration of InP particles in soluble, colloidal and suspended forms.

A simple fouling index was developed by (Schippers *et al.* 1980) to evaluate the fouling tendency of wastewater and the index has been further refined by other researchers (Rabie *et al.* 2001, Roorda *et al.* 2001, Boerlage *et al.* 2003 and Listiarini *et al.* 2009, 2011), the bigger the value of MFI, the higher the tendency of fouling will occur. The MFI is based on cake filtration theory and especially applicable for the evaluation of particulate and colloidal fouling. The MFI is defined as the gradient of the linear region found in the plot of *t/V* versus *V* from the general cake filtration equation for constant pressure

$$\frac{t}{V} = \frac{\eta R_m}{\Delta PA} + \frac{\eta \alpha C_b}{2\Delta PA^2} * V \tag{2}$$

and

$$MFI = \frac{\eta \alpha C_b}{2 \Delta P A^2} \tag{3}$$

where

V = Filtrate volume (l)
t = Filtration time (s)
α = Specific resistance of cake deposited (-)
C_b = Concentration of particles in the feedwater (mg/l)
R_m = Membrane resistance (kPa)
ΔP = Transmembrane pressure gradient (kPa)
η = Dynamic viscosity (Ns/m^2)
MFI = Modified fouling index (s/l^2)

Eq. (3) indicates that TSS concentration has a direct impact on MFI. Since the wastewater generated from wafer backgrinding and sawing processes varies in TSS contents, it is necessary to determine fouling tendency of the wastewater using MFI at different solid concentrations.

The concentrate wastewater collected from the rough grinding step was diluted with 1x, 2x and 4x ultrapure water to prepare the water samples with different suspended solid concentrations. The TSS concentrations of the diluted water were pre-determined prior to the experiment. The water samples were fed to the membrane test cells at 0.2 MPa for NTU2120 and at 0.7 MPa for NTR7250 membranes, respectively. The total permeate volume at different time intervals were recorded and the MFI was determined by plotting t/V versus V for each membrane test cell. For NTU2120 membrane, the computed MFI stabilized at about 200 – 400 minutes. For NTR7250 membrane, the computed MFI stabilized at about 800 – 1,000 minutes (Fig. 2). The results clearly indicated that the MFI increased with increasing TSS concentration (Fig. 3). According to results presented in

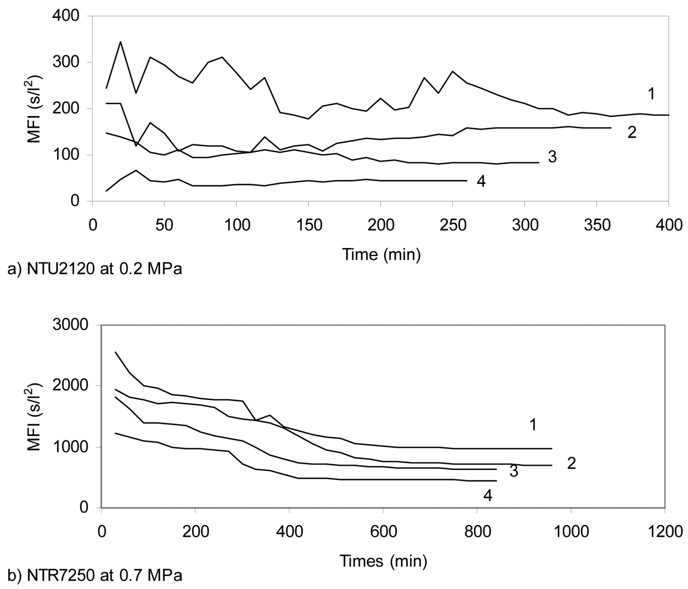


Fig. 2 a. MFI of wastewater for NTU2120 and b. NTR7250 membranes (1 represents 418 mg/l; 2 represents 204 mg/l; 3 represents 94 mg/l; 4 represents 43 mg/l)

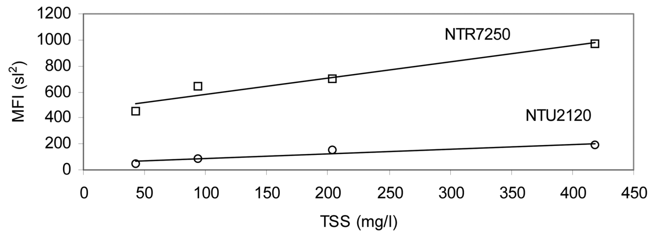


Fig. 3 MFI of wastewater correlated to solid contents

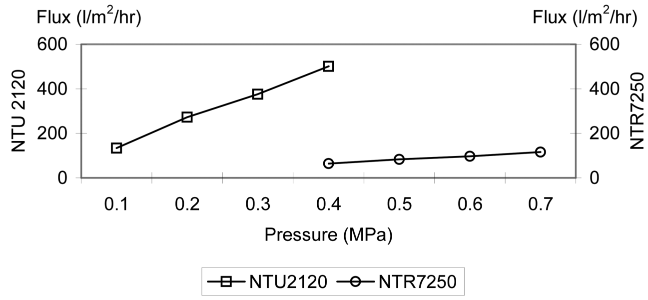


Fig. 4 Flux behavior of clean membrane (UPW as feed water)

Figs. 2 and 3, it was concluded that reduced TSS concentration in the feed water would improve the flux behavior of both UF and NF membranes. The LFSW tank was thus designed according to this observation.

3.3 Flux enhancement

The permeate flux of both clean UF and NF membranes were pre-determined as references (Fig. 4). Two recycling systems were operated in parallel to compare their fouling and flux behaviors over 120 hours. One system was equipped with a conventional water tank while the other was equipped with the LFWS tank. The experiments were conducted for both UF and NF membranes. The wastewater was directly transferred from the wafer manufacturing lines for the experiment, and the solid concentrations were observed to fluctuate within the range as shown in Table 2. There was no temperature control of the incoming wastewater. However, the temperature also fluctuated within a narrow range, 23 – 26°C as the UPW supplied to a wafer backgrinding or sawing processes was controlled between 22±2°C. Backwash was performed at every 12-hour interval and flux readings were recorded before and after the backwash. No chemical cleaning was carried out during the period of the experiment. The results are shown in Fig. 5.

Fig. 5a shows the flux reduction of the recycling system equipped with a conventional wastewater collection tank. During the 120-hour experiment, the flux reduced from 171 to 79 l/m^2/hr for NTU2120 while the flux reduced from 72 to 33 l/m^2/hr for NTR7250. Fig. 5b shows the flux change of the recycling system equipped with the LFSW tank. With the same feed water over the same period of time, the flux reduced from 174 to 104 l/m^2/hr for NTU2120 while the flux reduced from 68 to 39 l/m^2/hr for NTR7250. At the end of the study, the improved system (with LFSW tank) showed higher flux than the conventional system, 24% more for the UF membrane and 15%

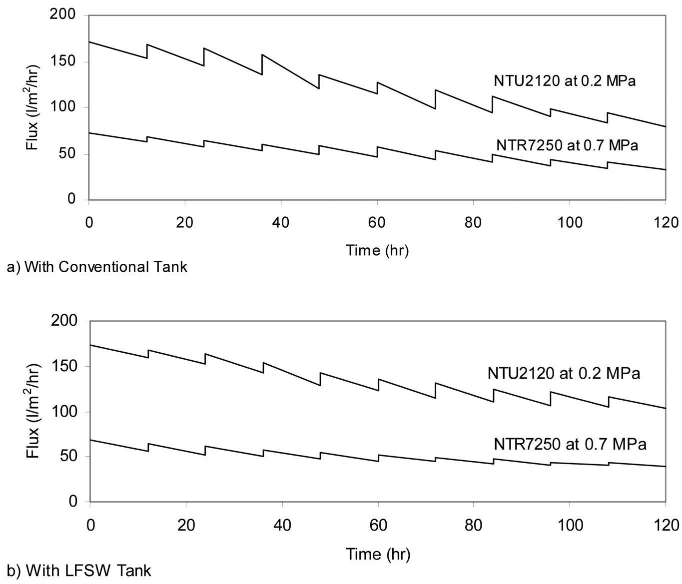


Fig. 5 a. Flux reduction in the conventional tank and b. LFWS

more for the NF membrane, respectively.

The flux enhancement of the improved system could be explained by a reduced "Flux Declining Rate" (FDR), defined as the change in normalized flux between membrane backwashes

$$FDR_i = \frac{J_i - J_{i+1}}{J_0} \times \frac{1}{T} \tag{4}$$

where

FDR_i = Flux declining rate (1/hr)
J_i = Flux after ith backwash ($l/m^2/hr$)
J_{i+1} = Flux before ($i+1$)th backwash ($l/m^2/hr$)
J_0 = Clean membrane flux ($l/m^2/hr$)
T = Time between ith and ($i+1$)th backwash (hr)

In this study, clean membrane flux (J_0) was taken from Fig. 4 while J_i and J_{i+1} were taken from Fig. 5 to compute the normalized flux J_i/J_0 and J_{i+1}/J_0. The individual FDR between two backwashes (12 hours) was determined using Eq. (4). The average flux declining rate (AFDR, over 120 hours) using arithmetic mean of the individual FDRs was computed by

$$AFDR = \frac{1}{n}\left(\sum_{i=1}^{n} FDR_i\right) \tag{5}$$

where

$AFDR$ = Average flux decay rate (1/hr)
n = Number of backwashes (-)

The results are summarized in Table 3. Obviously, the recycling system equipped with the LFWS tank has a lower *AFDR* than the conventional system for both UF and NF membranes.

Table 3 Comparison of FDRs of LFWS and conventional systems

Test Run	LFWS		Conventional	
	NTU2120	NTR7250	NTU2120	NTR7250
FDR 01	0.0279	0.0240	0.0359	0.0200
FDR 02	0.0319	0.0240	0.0459	0.0220
FDR 03	0.0419	0.0240	0.0579	0.0220
FDR 04	0.0499	0.0220	0.0719	0.0200
FDR 05	0.0399	0.0200	0.0399	0.0259
FDR 06	0.0439	0.0140	0.0579	0.0259
FDR 07	0.0419	0.0140	0.0479	0.0259
FDR 08	0.0379	0.0140	0.0439	0.0259
FDR 09	0.0319	0.0080	0.0319	0.0200
FDR 10	0.0240	0.0100	0.0299	0.0160
AFDR	0.0371	0.0174	0.0463	0.0224

It is worth to notice that all the experiments were carried out at a constant pressure: 0.2 MPa for the UF membranes and 0.7 MPa for the NF membranes, respectively. The effect of pressure on membrane fouling was therefore excluded from this report. However, the fouling behavior changed vastly at different operating pressure (data not shown here). This phenomenon was also observed by many other researchers (Lodge *et al.* 2002, Hoek *et al.* 2002, Speth *et al.* 2002). It has been proposed that there might exist a "critical flux" in ultrafiltration and nanofiltration (Madaeni *et al.* 1999, Manttari *et al.* 2000 and Seidel *et al.* 2002). The critical flux is defined as, on start-up, there exists a flux below which a decline of flux with time does not occur (Field *et al.* 1995) and several factors such as cross-flow velocity, electrostatic repulsion and colloid concentration may affect the critical flux. In this work, it seemed that the operating pressure for the UF and NF membranes was above the critical flux. The fouling behavior under the critical flux should be further studied.

4. Conclusions

The fouling potential of wastewater was evaluated using MFI at different TSS concentrations. It was determined that, with increasing TSS concentration, the fouling potential also increased. It implied that a reduced solid content would mitigate membrane fouling in such an application. The laminar flow water storage tank is able to reduce TSS concentration of the feed water by discharging the concentrate from the bottom of the tank prior to filtration. A full-scale recycling system based on the improved design has been implemented in a semiconductor company. It has been confirmed by the operating data that the improved system could reclaim ultrapure water at a lower cost. A patent is to be filed for the LFWS tank developed from this study.

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