

# Resolution of permeation issues in liquid-handling systems

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## **Abstract**

Dissolved gases and bubbles in liquids can be contaminants. As contaminants, they can reduce manufacturing yield or result in failure of chemical-handling systems. They are often the result of permeation of the contaminant species into areas where they are not wanted. This paper addresses two of these situations and shows how the effects can be mitigated.

## **Introduction**

Unwanted gases can cause major problems during semiconductor processing. For example, when HCl and HF penetrate equipment they quickly corrode most metals parts if moisture is present. Some components (e.g. valves) in chemical-handling systems often contain metal parts. The metal parts are isolated from the chemical by polymer parts that are chemically resistant. Unfortunately, these polymers often have relatively low resistance to permeation by gases like HCl and HF. The corrosion caused by the permeating gas can cause premature failure.

Dissolved gases in liquids can also cause problems. For example, if photoresist is transported using pressurized gas, the gas will rapidly dissolve into the surface of the photoresist then diffuse into the bulk of the liquid. Eventually the photoresist will become saturated with the gas at the delivery pressure. When the pressure is released, the photoresist will be supersaturated and gas bubbles may form. If bubbles in photoresist are dispensed onto a wafer, they can cause a defect in the wafer and reduce manufacturing yield.

This paper shows how these problems associated with permeating gases can be alleviated. Although permeation of HCl and HF into valves in chemical handling systems cannot be eliminated, it can be controlled. Valves containing metal springs can be designed with diffusion flow paths that allow corrosive gases to move away from the metal springs. Also, the amount of dissolved gases in pressurized liquids can be greatly reduced by surrounding the liquid with a polymer film.

## **Prevention of spring degradation in valves**

Fluoropolymer diaphragm valves containing metal springs are often used in systems delivering 37% HCl to the points of use in semiconductor process fabs. In simple valve designs, valve

springs are isolated from the HCl by the valve diaphragm. Unfortunately, HCl permeates through fluoropolymer diaphragms fairly quickly and corrodes the springs, thereby decreasing the valve's reliability and time to failure. More complicated designs can reduce the rate of spring corrosion. One design, incorporated in the UPX valve from Saint Gobain Performance Plastics, includes a bellows surrounding the spring and a vent port between the valve diaphragm and the bellows. HCl that permeates through the diaphragm can either permeate through the bellows or exit the valve through the weep port. Since the bellows offers a higher resistance to diffusion than the weep port, the design greatly reduces the rate of spring corrosion.

HCl permeation through the valve components was measured using the test system shown in Figure 1. In this figure, a permeation cell simulating the valve was immersed in a water bath at 30°C. The cell contained two chambers. The bottom chamber was filled with 37% HCl and represented the liquid-filled side of the valve. The upper chamber represented the non-wetted part of the valve in which the spring was located. HCl entering this chamber could potentially corrode the spring.

HCl permeation through the diaphragm was measured in the following manner. Nitrogen was used as a carrier gas to sweep any HCl that permeated into the upper chamber into a scrubber where the HCl was absorbed into a buffer solution. The buffer solution was circulated through a spray nozzle to ensure that the HCl was removed from the carrier gas. The carrier gas was also passed through a second buffer solution used to verify that all of the permeating HCl was removed in the first scrubber. The carrier gas flow rate was controlled and monitored using a mass flow controller.

The chloride concentration of the buffer solution was monitored using a specific ion electrode and used to calculate the HCl permeation rate. The pH 7 buffer solution acted as an ionic strength adjuster to enhance the accuracy of the specific ion electrode and to ensure that there was little change in pH during the course of each experiment. Both ionic strength and pH affect the chloride concentration measurement.

Three valve configurations were tested: a single diaphragm, a diaphragm and a bellows separated by a weep hole, and simply a weep hole. The last configuration was used to estimate the relative resistance of the weep hole and the bellows. All configurations were tested in triplicate and the system background was measured 5 times to allow statistically significant comparisons of the designs.

System background and HCl permeation rates through the three configurations are shown in Figure 2. Data are presented as the total number of  $\mu$ moles of HCl collected in the scrubber chamber as a function of test time. Figure 2 includes least square regressions of the data which represent the rate of change in HCl concentration over time. The replicate runs for each set of conditions were similar. The permeation through the weep port was the fastest, followed by permeation through a single diaphragm. Permeation through the diaphragm-bellow-weep port combination was similar to the background.

**Figure 1: Test system schematic for comparing HCl permeation in valve designs**

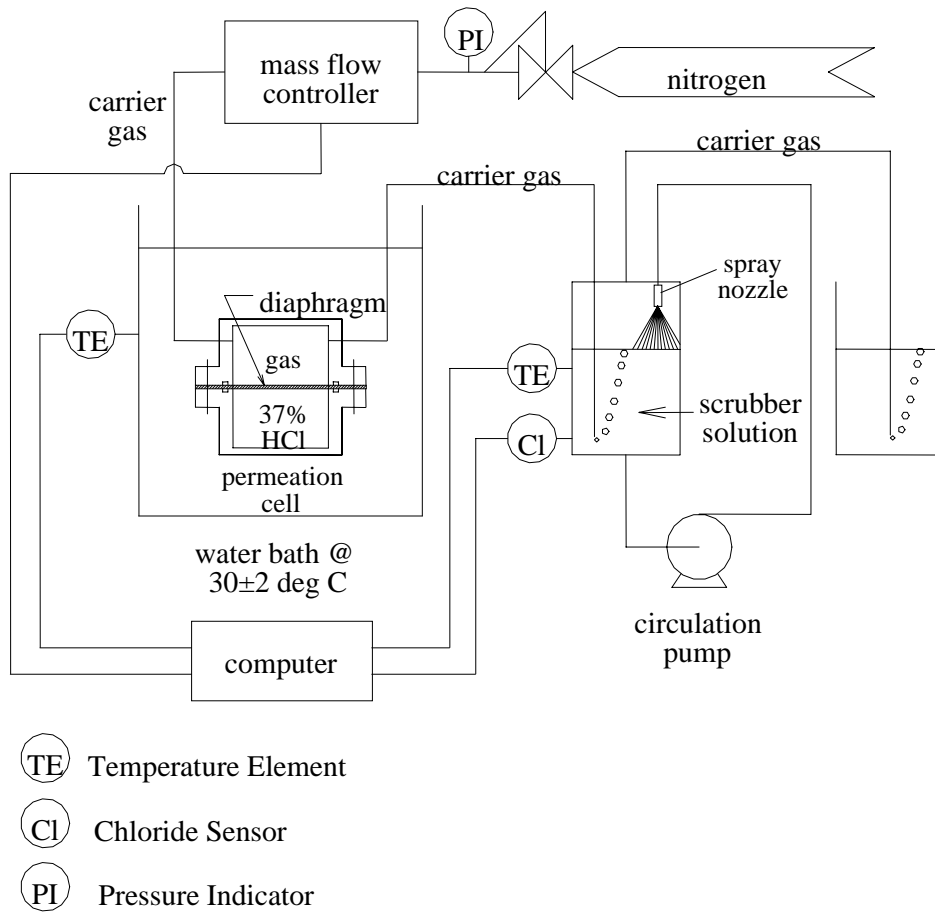
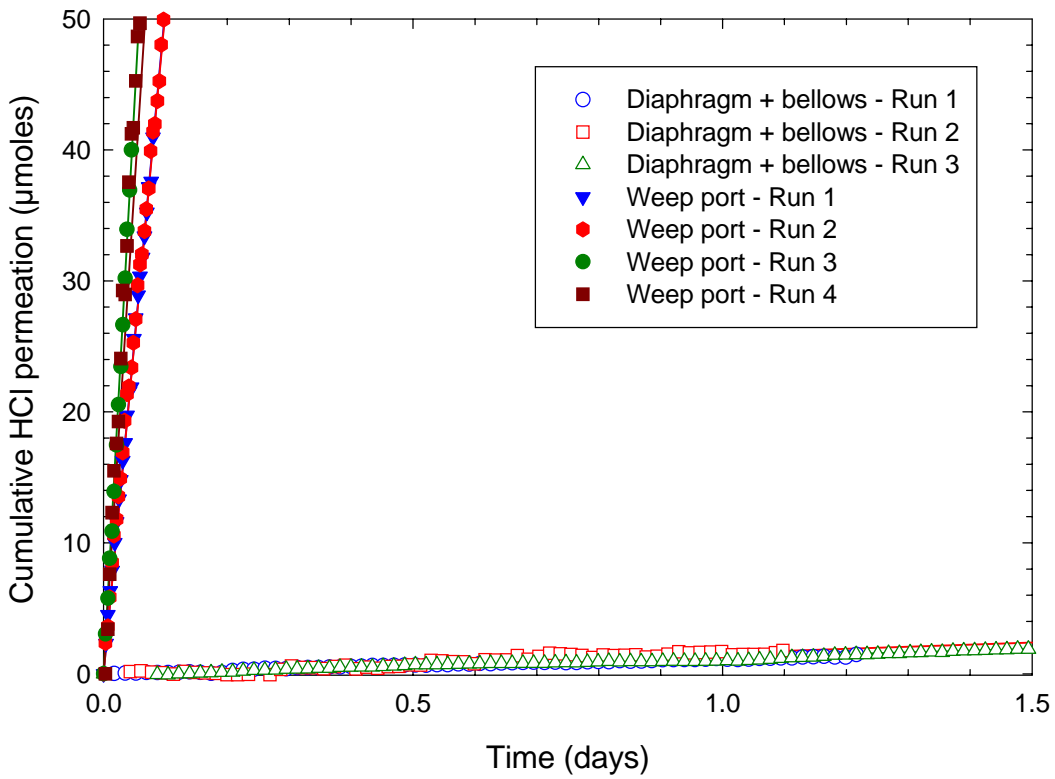
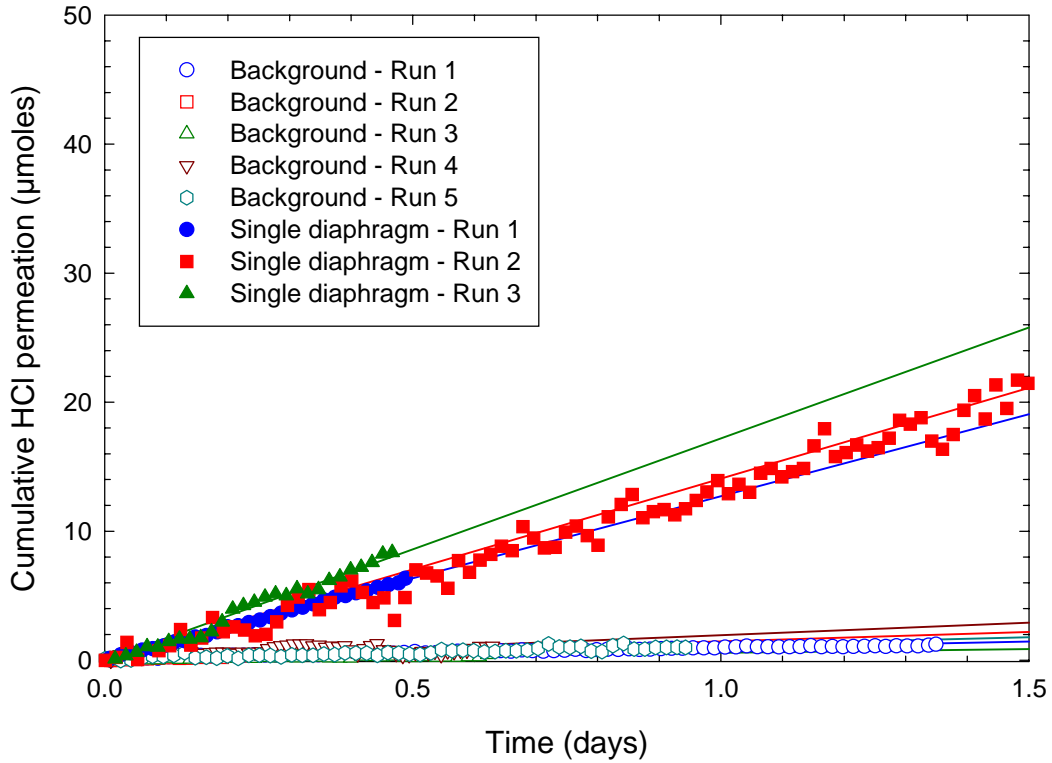


Table 1 summarizes the permeation rates calculated for the three configurations. The permeation rate through the weep port was about 40 times faster than through the single diaphragm. The diaphragm-bellows-weep port combination was most effective in reducing potential spring corrosion. The degree of improved corrosion resistance offered by this combination relative to a single diaphragm cannot be determined directly because the spring exposure for the diaphragm-bellows-weep port combination ( $1.3 \pm 0.2$   $\mu\text{moles/day}$ ) was indistinguishable from the system background ( $1.2 \pm 0.5$   $\mu\text{moles/day}$ ).

**Table 1: HCl permeation through different valve configurations**

Configuration	Potential spring exposure ( $\mu\text{moles/day}$ )	
	Average	Standard deviation
Single diaphragm	14.7	2.3
Diaphragm, bellows, weep port	1.3	0.2
Weep port	630	210
Background	1.2	0.5

**Figure 2: HCl permeation in different valve configurations**



It is possible to estimate the protection offered by the diaphragm-bellows-weep port combination by using the spring exposure rates measured for the single diaphragm and weep port configurations. In the diaphragm-bellows-weep port combination design, HCl that permeates through the valve diaphragm can either permeate through the bellows or exit through the weep hole. Hence, the valve can be modelled as a resistor (the valve diaphragm) followed by two resistors in parallel (the weep port and the bellows).

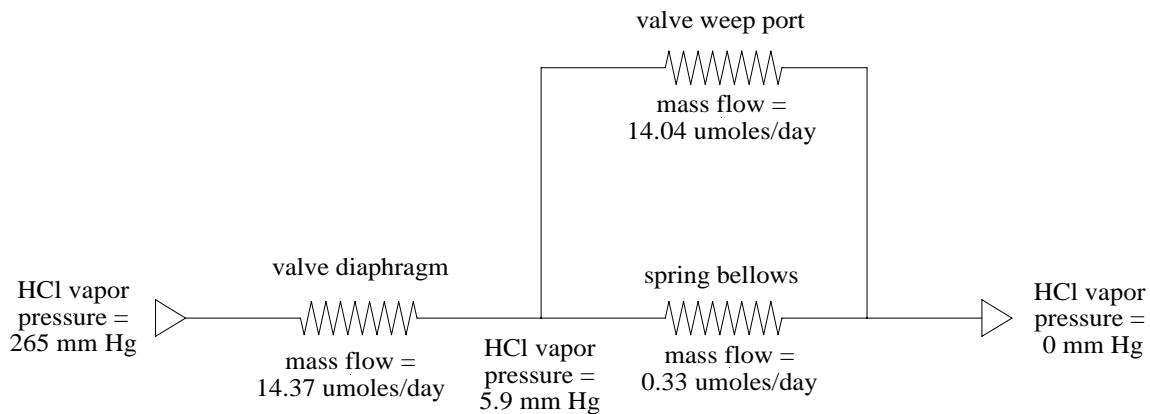
The weep port resistance can be determined as follows:

$$\begin{aligned} \text{Weep port resistance} &= \frac{\text{Driving force (HCl vapor pressure at } 30^\circ\text{C)}}{\text{Mass flow rate}} \\ &= \frac{265 \text{ mm Hg}}{630 \mu\text{moles/day}} \\ &= 0.42 \text{ mm Hg-days}/\mu\text{mole} \end{aligned}$$

The diaphragm and bellows are made of the same material and have essentially the same surface area. Hence, their resistances are essentially the same, 18.03 mm Hg-day/ $\mu$ mole, when calculated the same way. This resistance is 43 times that of the weep port.

These resistances can be used to predict the mass flow rates in the valve as shown in Figure 3. The majority of the HCl exits from the valve through the weep hole rather than permeating through the bellows. The predicted HCl flow through the bellows is 0.33  $\mu$ moles/day. This potential spring exposure is 1/44 of the exposure in the single diaphragm valve (14.7  $\mu$ moles/day). If spring life is inversely proportional to spring exposure to HCl vapors, springs in the diaphragm-bellows-weep port valve design should last 40 to 50 times longer than springs in the single diaphragm valve design.

**Figure 3: UPX valve permeation model and predictions**



## **Reduction of gas absorption in photoresists**

Photoresists used in semiconductor processing are often transported from a storage vessel to the substrate by pressurizing the storage vessel with the gas, typically nitrogen. Unfortunately, the photoresist solutions are often made using solvents that have high gas solubilities. When the solutions are exposed to pressurized gas during dispensing, they absorb some of the gas. If exposed for extended periods, they can become saturated with the gas at the delivery pressure. Because the pressure is reduced when the solution is dispensed onto the substrate, the fluid becomes supersaturated with gas and bubbles can form.

If a polymer film is placed between the liquid and the pressurizing gas, the gas must pass through the film before it contacts the liquid. Since polymers typically have lower gas permeabilities than liquids, these films can greatly reduce the rate at which gas is absorbed. Protective films of this sort are commonly used to prevent meat from spoiling by reducing the rate at which oxygen comes into contact with the meat. It should be noted, however, that films of this sort do not completely eliminate gas permeation.

This paper discusses the theory of gas transport and absorption into stagnant liquids with and without protective polymer films surrounding the liquid. The examples describe the transport of nitrogen into propylene glycol monomethylether acetate (PGMEA) with and without protective layers. The protective layers examined include those in two types of NOWPAK<sup>®</sup> containers manufactured by ATMI Packaging (Bloomington, MN).

## **Absorption of gas into a stagnant liquid**

Absorption of gas into a stagnant liquid involves two steps. First, the gas must dissolve into the surface of the liquid. The amount of gas that will dissolve in a liquid at equilibrium is proportional to the partial pressure of the gas in contact with the liquid, as described by Henry's Law [1]. As the partial pressure of a gas is increased, the amount of gas that can be dissolved increases.

Dissolution is followed by diffusion of the gas into the bulk of the liquid. Diffusion of the gas into the bulk of the liquid is a non-steady-state process. Assume that a liquid with a constant gas concentration throughout is suddenly exposed to an elevated gas pressure. The surface of the liquid solution absorbs gas very rapidly until the concentration equilibrates with the increased gas pressure. Because a very steep concentration gradient is formed near the liquid surface, the gas begins to diffuse further into the liquid. As the gas diffuses the concentration gradient becomes less steep. Since the rate of diffusion is proportional to the slope of the concentration gradient, the rate of diffusion slows.

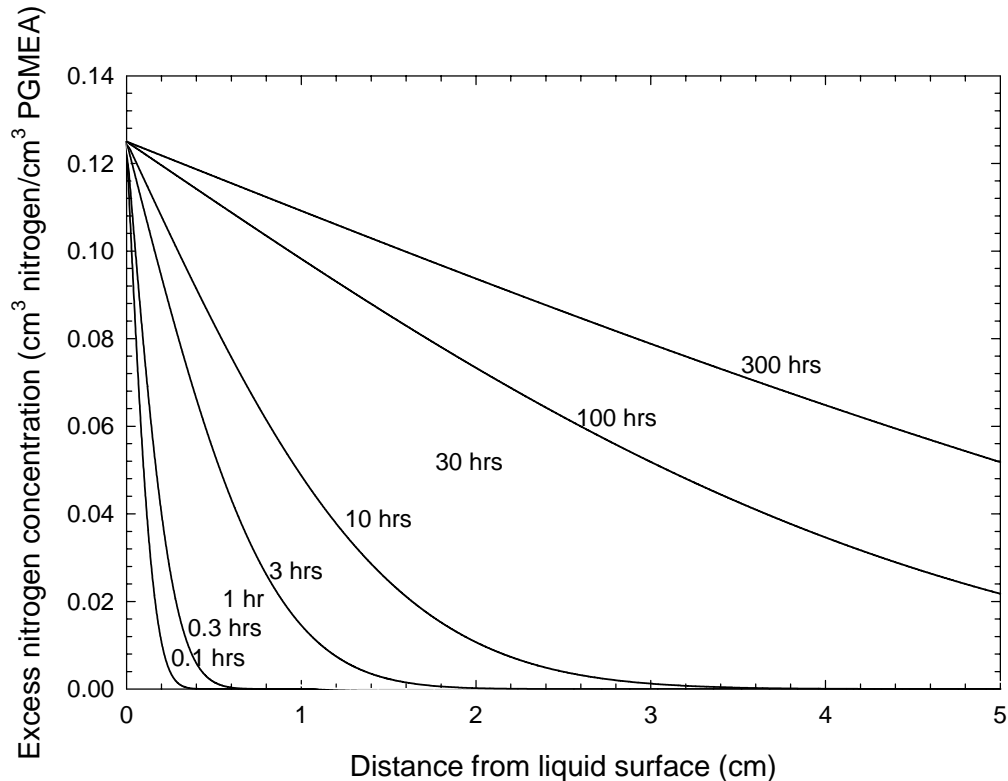
The gas concentrations in the liquid as a function of time and location can be calculated if it is assumed that the liquid is a semi-infinite medium (i.e. the concentration far into the liquid stays at the initial conditions) at rest (no convection) [2]. Consider, for example, the concentration of nitrogen gas in PGMEA saturated with 1 atmosphere absolute when the nitrogen pressure is raised suddenly to 1.5 atmospheres absolute. The effective driving force of the system is 0.5 atmospheres. Figure 4 shows the "excess" nitrogen concentration in the PGMEA, that is, the

concentration of nitrogen in excess of the concentration in equilibrium with 1.0 atmospheres absolute.

The Henry's constant used in this example was determined using the method of Osburn and Markovic. They showed that Henry's constants for inert gases in pure organic liquids at 20°C can be estimated if the surface tension of the liquid is known [3]. The surface tension of PGMEA measured at 20°C using a Du Nouy ring tensiometer is 28.5 dynes/cm [4]. The estimated Henry's constant for this surface tension is 880 atm/mole fraction. This constant yields a gas solubility of 0.125 cm<sup>3</sup> of gas (at STP) per cm<sup>3</sup> of liquid when the liquid is saturated with a nitrogen partial pressure of 0.5 atmospheres.

The diffusion coefficient used to generate the curves shown in Figure 4 was  $1.8 \times 10^{-5}$  cm<sup>2</sup>/sec. This estimated value was determined by multiplying the diffusion coefficient of nitrogen in water by the ratio of the viscosity of water to that of PGMEA.

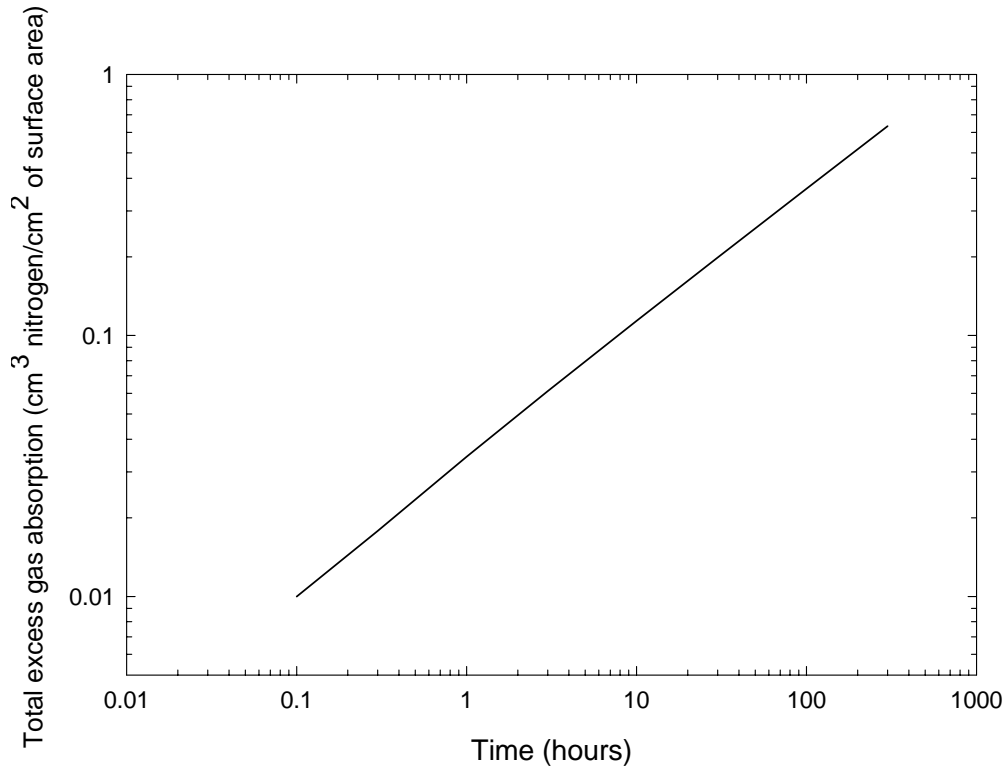
**Figure 4: Diffusion of nitrogen into stagnant PGMEA**



The eight curves in Figure 4 describe the concentration gradient in the fluid at different times. The curves indicate that the surface of the liquid is always saturated and there is initially a very steep concentration gradient. Initially, all the absorbed gas is near the surface. The steepness of the gradient decreases with time as the gas penetrates further into the liquid.

The total amount of gas absorbed into the solution at any time can be determined by numerically integrating the concentration profiles shown in Figure 4. The result of this integration is shown in Figure 5, which presents the total amount of gas absorbed per unit area of interface between the gas and the liquid. The total amount of absorbed gas increases with the square root of time.

**Figure 5: Total nitrogen absorption into PGMEA over time**

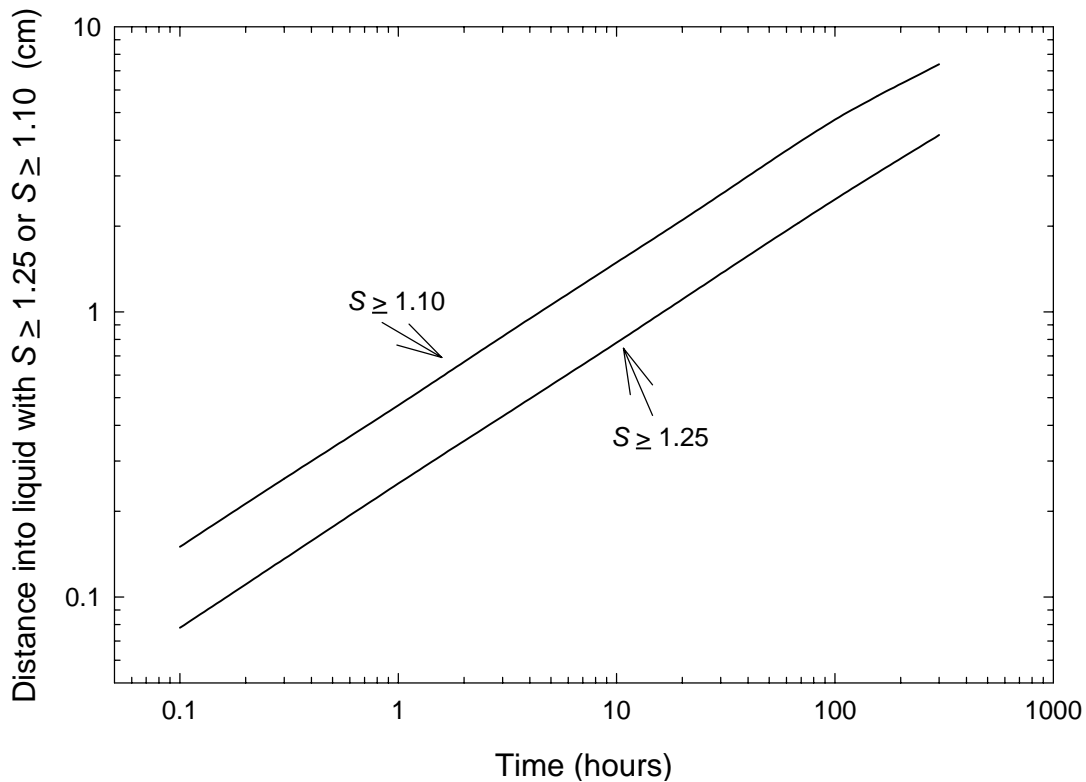


The saturation ratio ( $S$ ) is the ratio of the amount of gas dissolved in the liquid to the equilibrium amount of gas dissolved in the liquid at a standard state, a pressure of 1.0 atmosphere absolute in this example. It is an indicator of the likelihood that bubbles will form in the liquid when the pressure is reduced to 1.0 atmosphere. When  $S$  is greater than 1, the liquid is supersaturated and bubbles can form. The higher the saturation ratio, the more likely it is that bubbles will form.

The information in Figure 4 can also be used to determine how quickly the gas penetrates into the liquid and saturates it. When equilibrium is achieved at 1.5 atmospheres absolute, the saturation ratio is 1.5. Hence, a saturation ratio of 1.25 implies that the nitrogen concentration reached 50% of the fully saturated concentration at 1.5 atmospheres. Figure 6 presents the depth at which the saturation ratio has reached either 1.1 or 1.25. After 1 hour the saturation depths for  $S = 1.1$  and 1.25 are approximately 0.5 and 0.2 cm, respectively. After 100 hours the depths are approximately 4.7 and 2.5 cm.



**Figure 6: Rate at which nitrogen saturates PGMEA**



### **Transport of gas through a polymer film**

Before examining the case of gas transport through a polymer film into a stagnant liquid, the simpler case of gas transport through a thin polymer film into another gas will be examined. In this example, it will be assumed that the upstream and downstream pressures of the diffusing species are held at separate constant values.

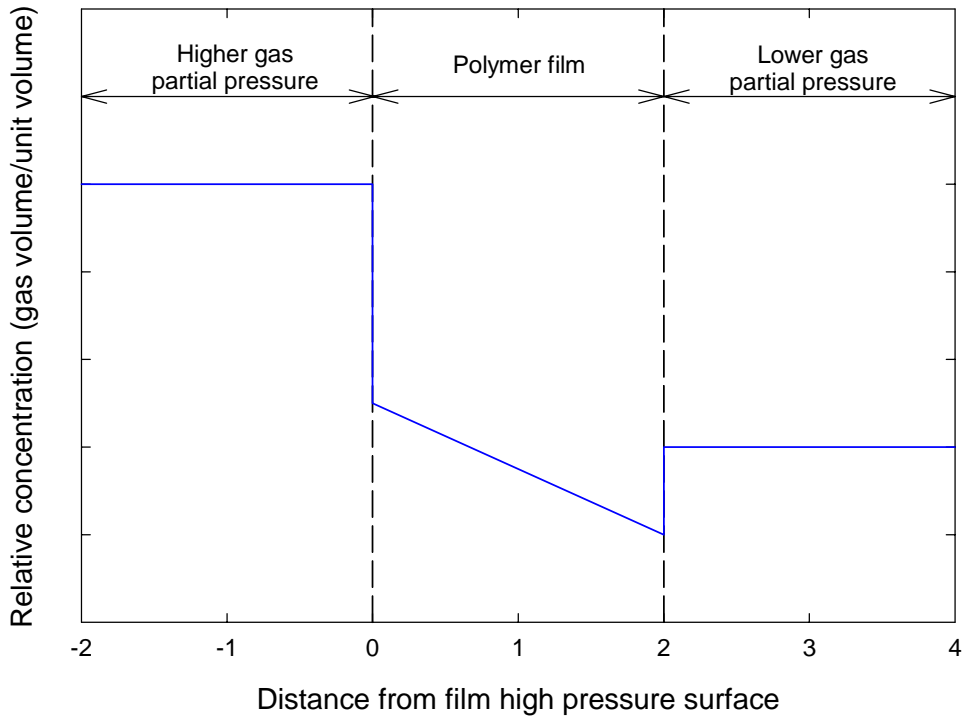
The process of gas transport through a polymer films consists of three steps:

- Adsorption or dissolution of the diffusing gas into the polymer film,
- Diffusion of the gas through the polymer film,
- Desorption of the gas from the polymer film.

The absorption/desorption equilibrium relationships are described by a process analogous to Henry's Law, described above, and result in discontinuities in concentration at both gas-solid interfaces.

Because in this example the partial pressure of the diffusing gas is held constant on each side of the film and because the film is thin, the concentrations throughout the film rapidly reach steady state. At this point the transport rate becomes invariant with time and concentration profiles like those shown in Figure 7 result.

**Figure 7: Steady-state diffusion of gas through a polymer film**



**Film permeabilities**

The permeability of gases through polymer films can be determined using ASTM method D1434-76 [6]. This method was used to measure the permeability of the polymer films used in NOWPAK<sup>®</sup> containers. The films have the permeabilities listed in Table 2 [7]:

**Table 2: Film permeabilities**

Film	Permeability	
	cm <sup>3</sup> /100 in <sup>2</sup> - day - atm	cm <sup>3</sup> /cm <sup>2</sup> - day - atm
Polytetrafluoroethylene (PTFE)	233	0.36
Polyethylene (PE)	195	0.30
Polyethylene/Nylon laminate (PE/Ny)	5.8	0.0090

Film permeability is a combination of both the solubility of the gas in the film and the gas diffusion coefficient through the film, as described above. These values were not known for the NOWPAK<sup>®</sup> films. However, the diffusion coefficient of gases in glassy polymers is typically between 10<sup>-6</sup> and 10<sup>-7</sup> cm<sup>2</sup>/sec [9]. In this study it was assumed that the nitrogen diffusion coefficient was 10<sup>-6</sup> cm<sup>2</sup>/sec in PTFE and PE films and 10<sup>-7</sup> cm<sup>2</sup>/sec in the PE/Ny film. The solubilities calculated using the measured permeability, film thickness and these diffusion coefficients are shown in Table 3.

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**Table 3: Film properties used in this study**

Film	Thickness (cm)	Diffusion coefficient (cm <sup>2</sup> /sec)	Gas solubility @ 1 atm (cm <sup>3</sup> gas/cm <sup>3</sup> polymer)
PTFE	0.0064	10 <sup>-6</sup>	0.027
PE	0.0076	10 <sup>-6</sup>	0.027
PE/Ny	0.0076	10 <sup>-7</sup>	0.0078

Note that the assumption of the diffusion coefficient has a major effect on the calculated gas concentrations within the film. It also affects the rate at which gas enters the liquid when the gradient in the film is non-linear. However, once the concentration gradient approaches linearity, the gas transport rate is determined solely by the permeability, which was experimentally measured. Therefore, errors due to inaccurate diffusion coefficients are small.

### **Effect of films on gas absorption into a stagnant liquid**

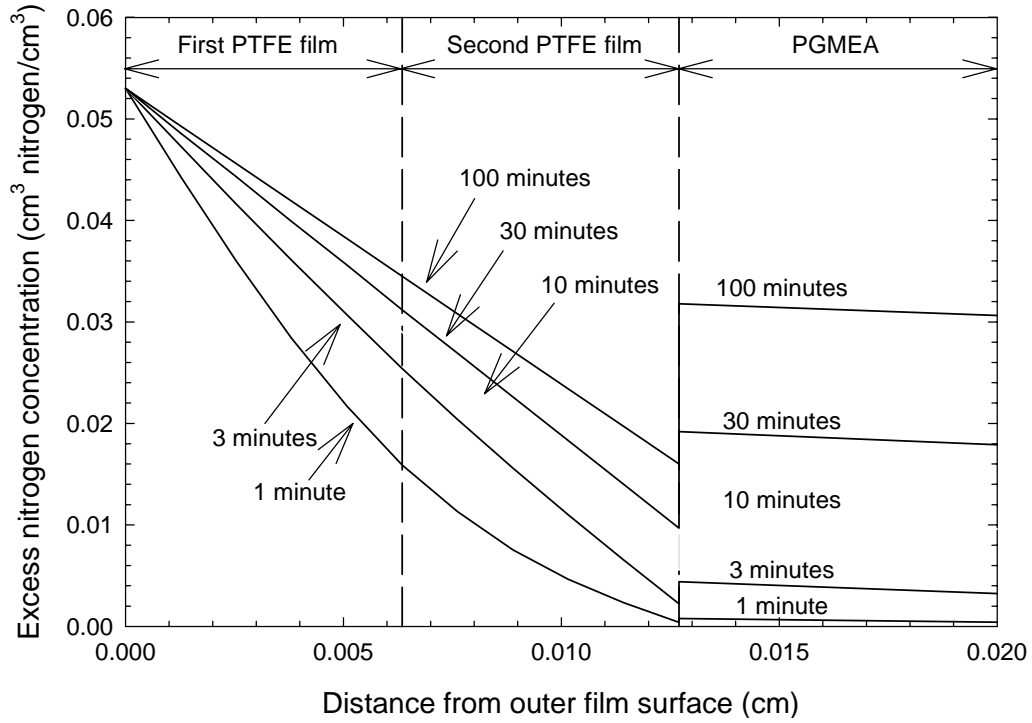
NOWPAK<sup>®</sup> containers incorporate two layers of film. In one product, two layers of PTFE are used. A second product incorporates one layer of PE and a layer of PE/Ny. When two layers of protective polymer film are placed between the pressurized gas and an organic liquid, the gas must go through a second adsorption/diffusion/desorption process before it diffuses into the liquid.

Absorption in this situation is not steady state and an analytical solution is not available. However, it can be determined numerically using a technique described by Geankoplis [8]. This technique allows calculation of the concentration gradient across the film and the liquid using the explicit numerical method.

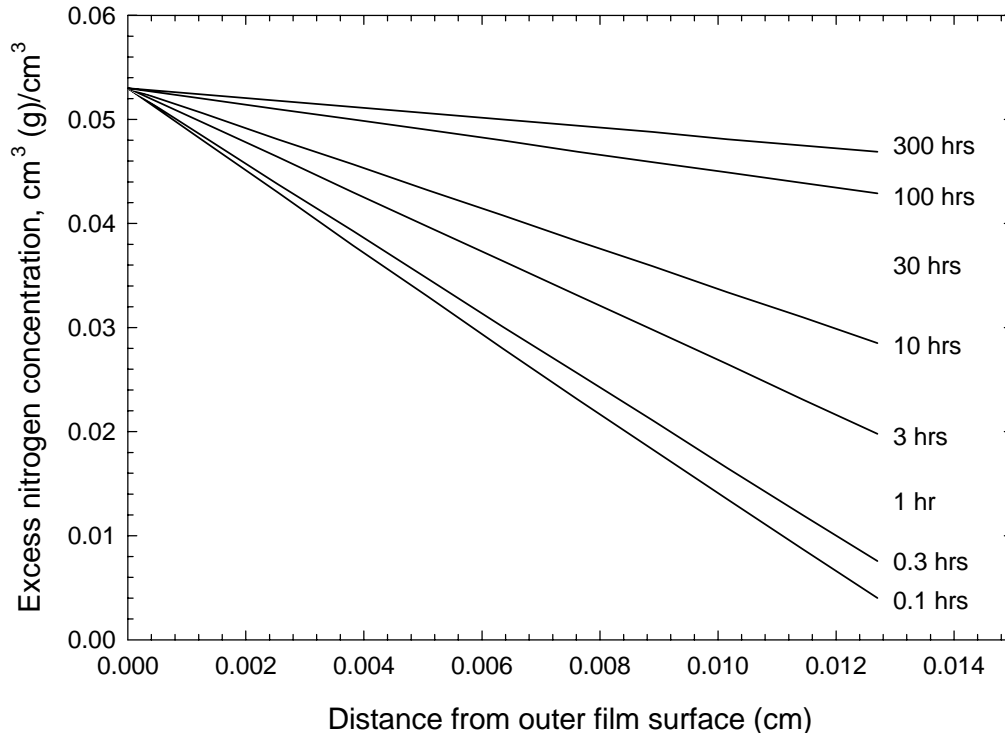
Nitrogen gradients in a system in which PGMEA is surrounded by the two layers of PTFE film used in NOWPAK containers is shown in Figure 8. The pressure on the outside of the film is 1.5 atmospheres absolute and the PGMEA is initially in equilibrium with nitrogen at 1.0 atmospheres absolute. The figure presents dissolved gas concentrations in the film and near the surface of the PGMEA at several times shortly after the pressure is increased. Note that essentially linear gradients are established in the film within the first few minutes. Also note that the concentration in PGMEA at the film surface is higher than in the film. This is because nitrogen is more soluble in PGMEA than in the PTFE film.

Concentration gradients within the film as a function of time for times up to 300 hours are shown in Figure 9. The gradients are essentially linear and decrease in steepness with time. At first thought, one would expect the gradients to be exponential like those shown in Figure 4 since the diffusion is not steady state. However, the gradients are essentially linear because the film is very thin relative to the liquid and transport in the liquid is much faster than in the film. Note that the concentration on the liquid side of the film increases with time. It is approximately 0.0040 cm<sup>3</sup> nitrogen/cm<sup>3</sup> PTFE after 0.1 hours of pressurization and 0.047 cm<sup>3</sup>/cm<sup>3</sup> PTFE after 300 hours of pressurization, a 12-fold increase.

**Figure 8: Diffusion of nitrogen into PGMEA through two layers of PTFE film during the first 100 minutes**

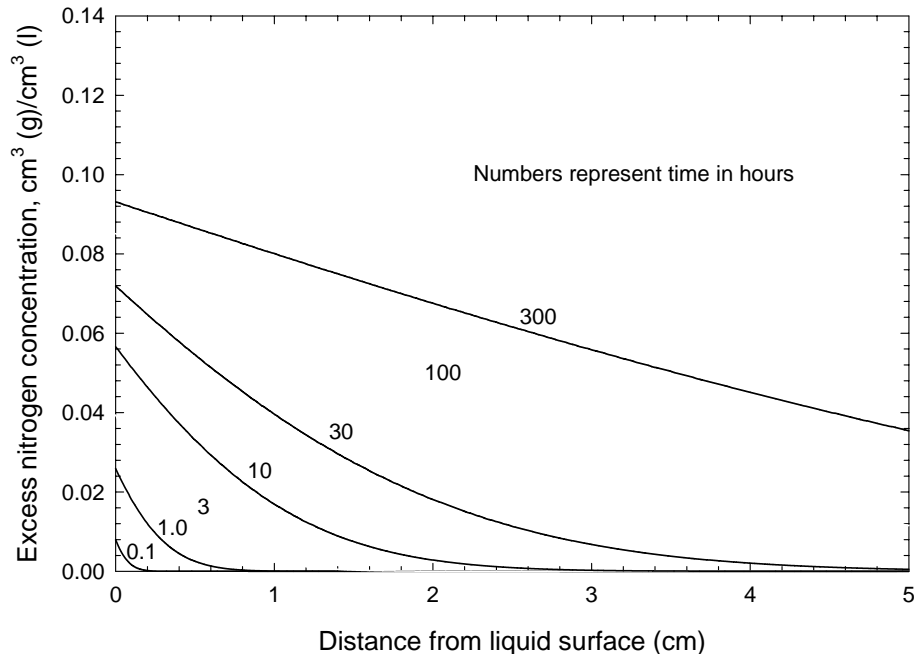


**Figure 9: Concentration gradients in PTFE film over time**



Concentration gradients in the liquid are shown in Figure 10. They show the expected exponential decay. The concentration at the surface of the liquid (which is in equilibrium with the gas concentration in the film at the surface) increases with time. It reaches a concentration of  $0.039 \text{ cm}^3 \text{ nitrogen/cm}^3 \text{ liquid}$  in 3 hours and a concentration of  $0.072 \text{ cm}^3 \text{ nitrogen/cm}^3 \text{ liquid}$  in 30 hours. Note that the concentration does not reach a saturation ratio of 1.25 ( $0.625 \text{ cm}^3 \text{ excess nitrogen/cm}^3 \text{ liquid}$ ) until the system has been pressurized for more than 10 hours.

**Figure 10: Concentration gradients over time in PGMEA protected by PTFE film**

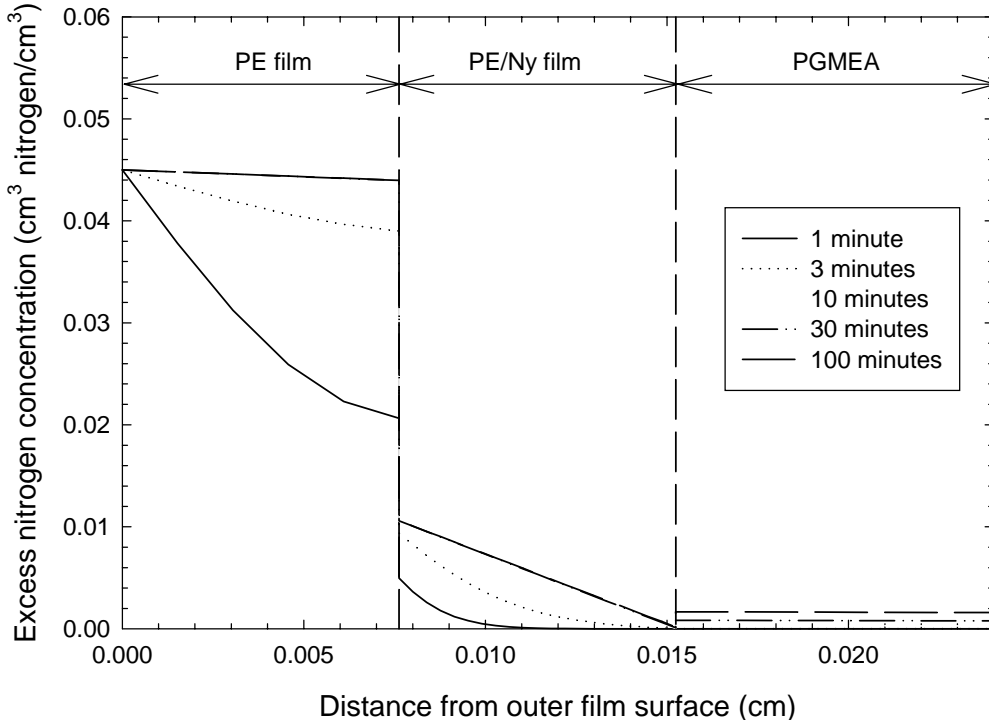


Concentration gradients in the films and PGMEA for the case of PGMEA protected by PE + PE/Ny films are shown in Figures 11 through 13. Figure 11 shows the concentration gradients in the films and near the PGMEA surface during the first 100 minutes following pressurization. The discontinuity in the concentrations at the interface between the PE and PE/Ny films is the result of different gas solubilities in the 2 films. Gradients in both films become essentially linear in just a few minutes. The approach to linearity is slower in the PE/Ny film than in the PE film and the concentration gradient is steeper because of the lower assumed diffusion coefficient in the PE/Ny film.

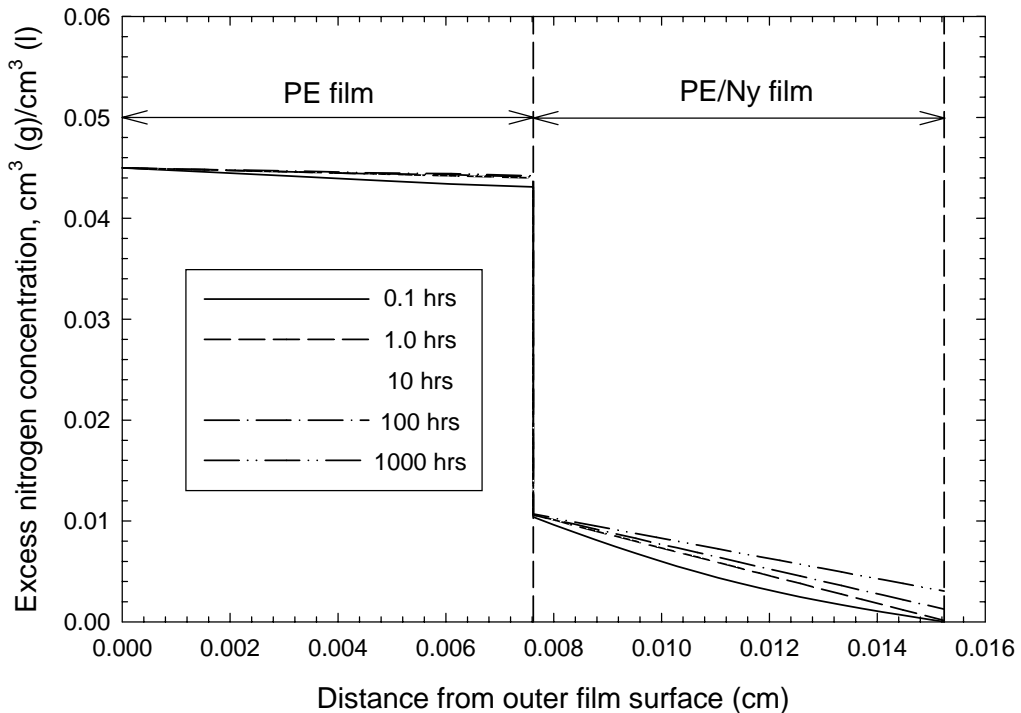
Figures 12 and 13 present the calculated concentration gradients in the films and PGMEA for times up to 1000 hours. Concentration gradients in the films are again essentially linear with time after the first few minutes. Gradients in the PGMEA are considerably more linear than in the cases of either unprotected liquid or liquid protected by PTFE films. This is because diffusion through PGMEA is much more rapid than diffusion through the films and diffusion through the PTFE films is more rapid than through the PE + PE/Ny film combination. Note that the concentration in the PGMEA at the liquid surface does not reach the concentration required to achieve a saturation ratio of 1.1 ( $0.025 \text{ cm}^3/\text{cm}^3$ ) until around 600 hours.

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**Figure 11: Diffusion of nitrogen into PGMEA through PE + PE/Ny films during the first 100 minutes**



**Figure 12: Concentration gradients in PE + PE/Ny films over time**



**Figure 13: Concentration gradients in liquid protected by PE + PE/Ny films over time**

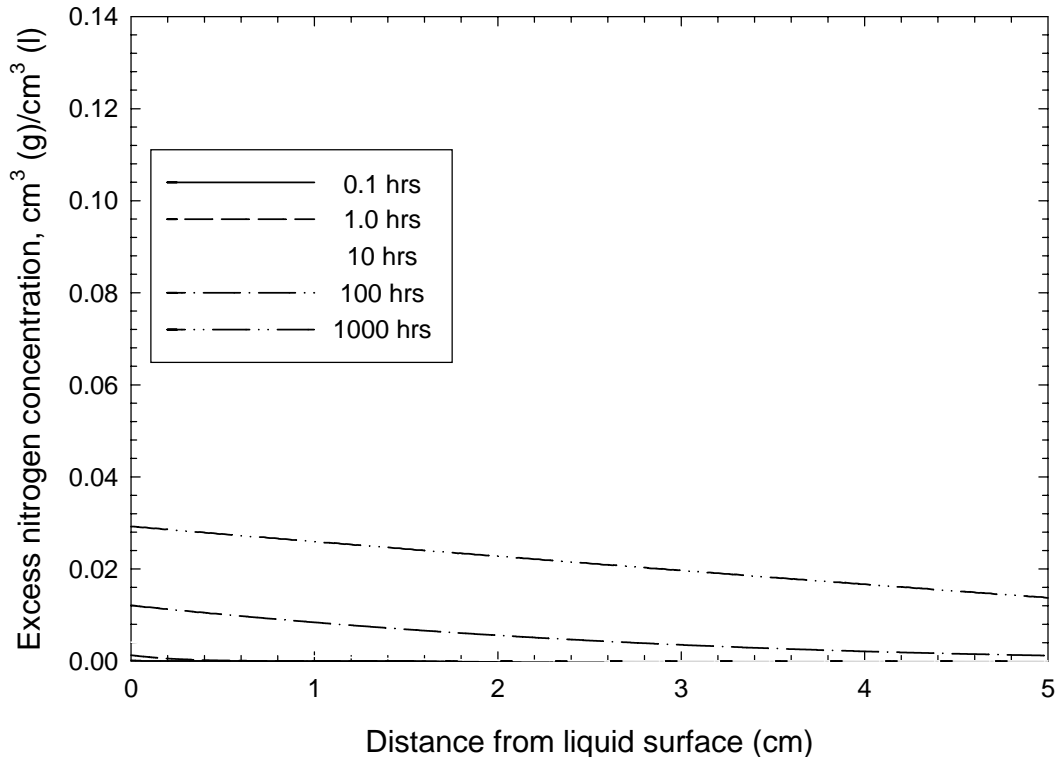
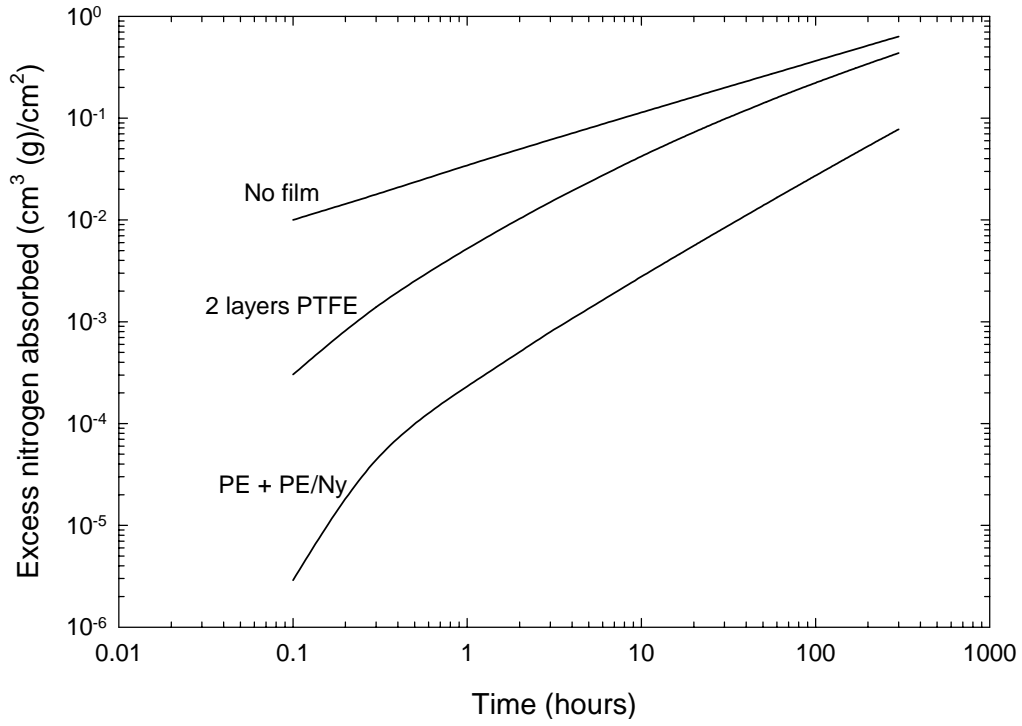


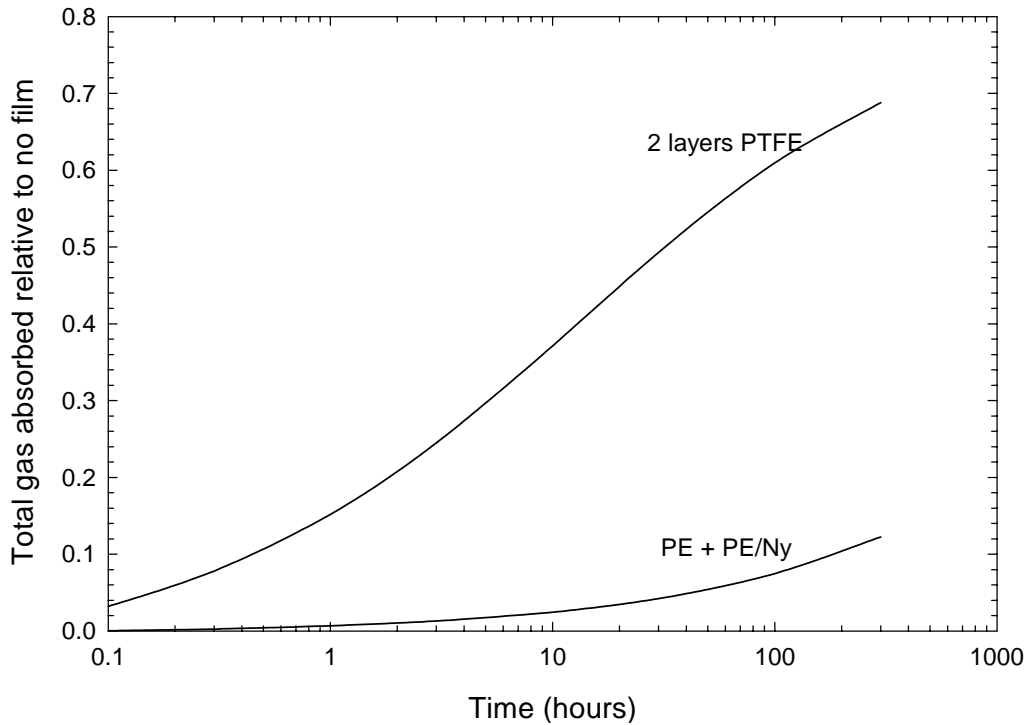
Figure 14 compares the amount of gas absorbed by PGMEA with and without protective films. Although both sets of films reduce gas absorption considerably, the PE+PE/Ny combination is more effective than the dual layer PTFE. The degree of protection offered is shown in Figure 15 that presents the ratio of the amount of gas absorbed into PGMEA with films present to the amount absorbed without films present. In the case of the PTFE films, the expected relative amounts of gas absorbed at 10 and 100 hours of pressurization are about 37% and 61% of that absorbed with no film present. In the case of the PE+PE/Ny film combination, the relative absorption is only 10% after 200 hours.

Figures 14 and 15 both indicate that the effectiveness of the films in reducing absorption decreases with time because the resistance to absorption in the liquid increases as the liquid absorbs nitrogen. The rate of absorption into PGMEA with no protective film (Figure 5) decreases with the square root of pressurization time. Since the resistance of the film remains constant and the resistance of the liquid increases over time, the relative benefit of the film decreases with time.

**Figure 14: Comparison of gas absorption into PGMEA with and without protective films**



**Figure 15: Reduction of gas absorption by polymer films**





The rates at which nitrogen penetrates into PGMEA with and without protective films present are compared in Figure 16. Graphs depicting the depths of penetration to saturation ratios  $\geq 1.25$  and  $\geq 1.10$  are shown. No data for the PE+PE/Ny film combination are shown in Figure 14A because the saturation ratio does not reach 1.25 at the PGMEA surface in 1000 hours. Saturation ratios in liquids protected by both film sets are considerably lower than in PGMEA with no film, especially when the PE+PE/Ny film combination is used. Hence, the films greatly reduce the probability of bubble formation in the liquid when the pressure is reduced.

## **The effect of convection**

It should be emphasized that the examples provided in this report are for absorption into a stagnant liquid. If the liquid is agitated, the rate of absorption will be faster both with and without films present. However, the increase in absorption rate will be greatest in the case of an unprotected liquid because the resistance of the liquid to absorption will be greatly reduced by convection while the resistance of the films will be unchanged. Hence, films will be even more effective at reducing absorption if convection is present in the liquid.

## **Summary**

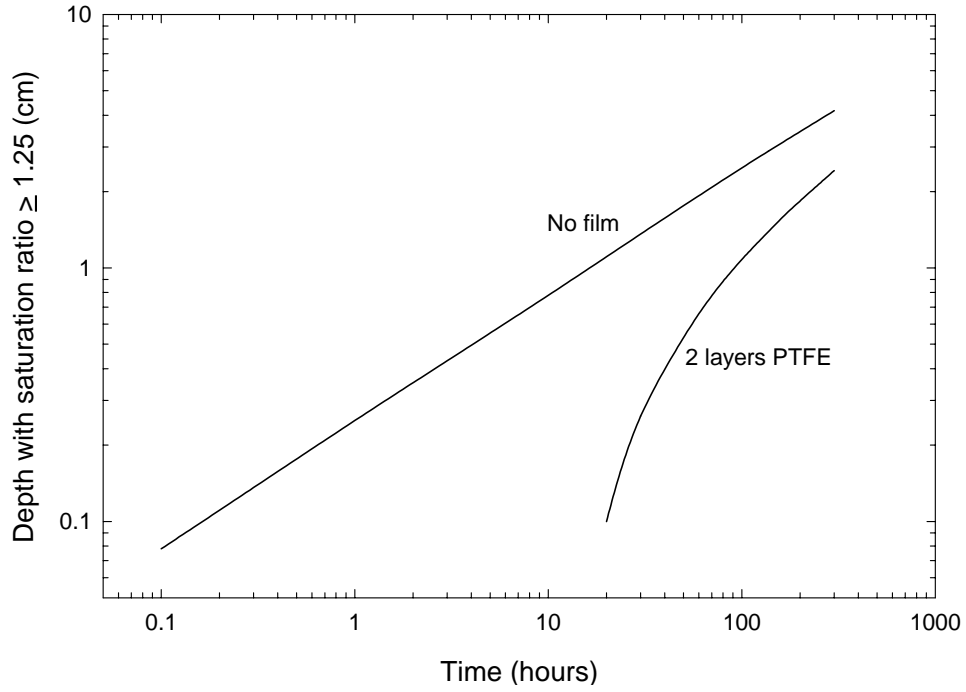
Unwanted permeation of gases can cause major problems in semiconductor processing. Examples of two problems and how they can be alleviated are discussed in this paper. In the first example, the likelihood of spring corrosion due to HCl permeation in two valve designs is compared. In one design, HCl that passes through the valve diaphragm can attack the spring. In the second design, the spring is surrounded by a bellows and the space between the diaphragm and the bellows is vented by a weep port. If spring life is proportional to HCl exposure, springs in valves with the diaphragm-bellows-weep port design should last 40 to 50 times as long as those in valves with the single diaphragm design.

In a second example, mass transport theory was used to calculate the rates at which PGMEA absorbs pressurized gases with and without protective polymer films between the fluid phases. The films examined were the dual-layer polytetrafluoroethylene and polyethylene + polyethylene/nylon combination used NOWPAK<sup>®</sup> containers. Calculations were performed using experimentally determined permeabilities of the films. Both polymer films significantly reduce the rate of nitrogen absorption and, hence, the probability of bubble formation when the pressure on the liquid is reduced. The PE + PE/Ny combination was shown to be the more effective film pair, reducing gas absorption in 7 days to  $< 10\%$  of that absorbed by unprotected liquid.

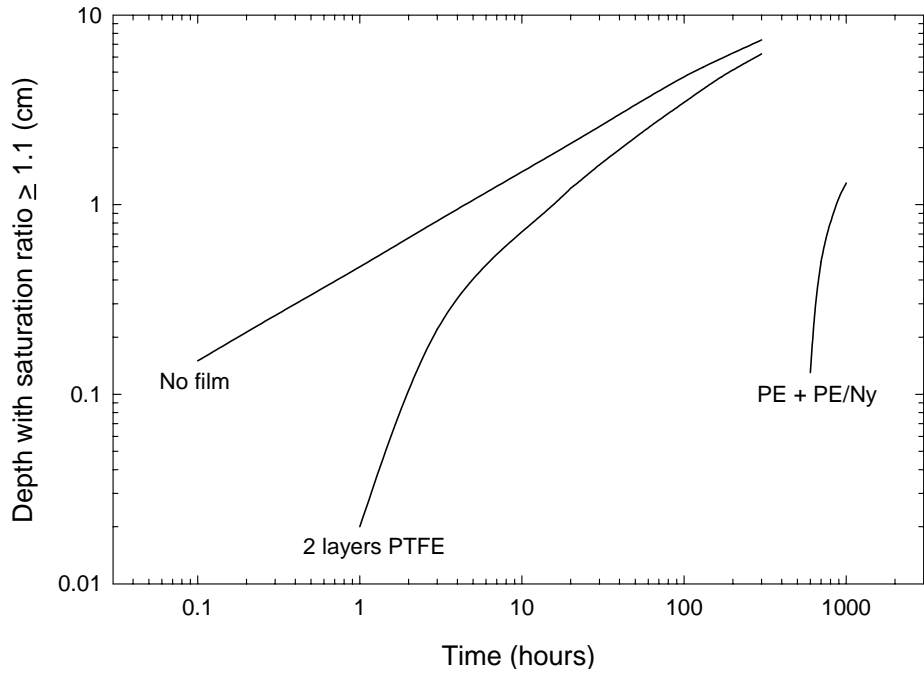
## **Acknowledgements**

The author would like to thank Saint Gobain Performance Plastics for sponsoring the study to evaluate valve designs for their resistance to HCl permeation and ATMI Packaging for sponsoring the study to assess the effectiveness of films in reducing gas absorption into solvents.

**Figure 16: Comparison of PGMEA saturation rates with and without protective films**  
A. Saturation ratio = 1.25



B. Saturation ratio = 1.10



## **Biography**

Don Grant is president of CT Associates, Inc., which performs contract research and development in contamination control, particle measurement and control, filtration and chemical engineering. He has more than 25 years of experience in analysis and purification of fluids and is the author or co-author of more than 100 technical papers and presentations. He has an M.S. in mechanical engineering from the Particle Technology Laboratory at the University of Minnesota and a B.S. in chemical engineering from Case Western Reserve University. He is a recipient of the Maurice Simpson Award from the Institute of Environmental Sciences.

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