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# A Guide to Pall Corporation's Fluoropolymer Filters:

Proper Installation, Start-up and Operation to Prevent Membrane Dewetting.

*Robert Gieger, Ph.D and Tom Gutowski, M.S.*

In today's semiconductor fabrication processes, filter cartridges constructed from all fluorocarbon components are employed to filter high purity chemicals. The strict requirements for clean materials, as well as compatibility issues, often necessitate the use of fluorocarbon materials. PTFE is an excellent material for filter membranes and is widely used for bulk, distribution, and point-of-use chemical filtration applications.

Many applications involve the filtration of aqueous chemicals, which include etching, cleaning, photoresist development, etc. Since the PTFE membranes are hydrophobic, prewetting with low surface tension fluids such as isopropanol (IPA) is required. Prewetting can be best accomplished by vacuum-drawing or pumping the low surface tension fluid through the filter. This is especially recommended for 0.05 µm filters, which are more difficult to wet due to the tighter pores. While the prewetting procedure is well established, environmental regulations limiting the amount of organic vapors in the workplace air are forcing users to reduce the utilization of IPA and other solvents for prewetting purposes.

Attempts at prewetting the hydrophobic filters by submerging the filters in water pressurized above the bubble point of the membrane has had limited success. Until now, packaging and fluid shipment and purity issues have hampered providing hydrophobic filters prewet so that they can be installed directly into tools and distribution systems.

## Prewet Filter Cartridges

In order to minimize installation difficulties and environmental exposure to organic prewet fluids, Pall Corporation has introduced Prewet Emflon®, Prewet UltiKleen™, and Prewet Fluoryte™ filters for use in ultra high purity (UHP) semiconductor chemical and water applications. The prewet manufacturing method yields filters which are packaged in sterile UHP DI water, without any additives, and can be utilized in chemicals without the need to prewet utilizing a low surface tension fluid.

Following filter manufacture, they are tested for integrity with an alcohol-based fluid, by measuring diffusional flow characteristics. The integrity test of every filter insures 100% integrity of filters from the manufacturing facility. The filters are then rinsed with filtered (0.04 micron) UHP DI water ( 18 megohm-cm), and placed in a fluorocarbon bag filled with 0.04 micron filtered UHP water, then delivered in a sealed, sterile, fluorocarbon package to the customer, in a prewet state. There are no materials in the package other than fluorocarbon (of the filter and bag) and UHP water.

## Sterility of Water in Packaged Filters

In order to assess shelf life, packaged prewet Emflon® and prewet UltiKleenTM filters were kept in storage for 3, 6, 9, 12, 18 and 24 months, and the water sampled for viable bacteria. The water samples were incubated in the growth media for 5 days at 25°C, and examined for growth of colonies.

**Table 1: Prewet Emflon®, Prewet UltiKleen™ Viable Organisms After 5 Day Incubation at 25°C**

| Shelf Life Period | Viable Organisms |
|---|---|
| 3 months | 0 |
| 6 months | 0 |
| 9 months | 0 |
| 12 months | 0 |
| 18 months | 0 |
| 24 months | 0 |

Sampling of the ultrapure water contained in the packaging at intervals of 3, 6, 9, 12, 18 and 24 months shelf life for microbial growth resulted in no viable organisms detected. Table 1 on the previous page summarizes the data.

In order to assess shelf life, packaged prewet FluoryteTM filters were kept in storage for 1, 2, 6, 12, and 24 months, and the water sampled for viable bacteria. The water samples were incubated in the growth media for up to 5 days at 30°C, and examined for growth of colonies.

Sampling of the ultrapure water contained in the packaging at intervals of 1, 2, 6, 12, and 24 months shelf life for microbial growth resulted in no viable organisms detected. Table 2 summarizes the data.

**Table 2: Prewet Fluoryte™**
**Viable Organisms After 5 Day Incubation at 25°C**

| Shelf Life Period | Viable Organisms |
|---|---|
| 1 month | 0 |
| 2 months | 0 |
| 6 months | 0 |
| 12 months | 0 |
| 24 months | 0 |

This confirms the guaranteed shelf life of 12 months with respect to sterility. The sterility of the samples during shelf life means that microbial contamination will not occur, eliminating the need for a preflush or sanitization of the filters prior to being used on line.

## Stability of Prewet Condition

To determine the effect of shelf life on prewet condition, the filters from the shelf life testing noted in the above paragraph were subjected to differential pressure measurement. If the filters were not completely prewet, high differential pressures would be observed. Differential pressure was measured by flowing the UHP water through the filters at varying flow rates, both ascending and descending, and measuring differential pressure using a pressure transducer. Flow was monitored with a rotameter.

By measuring the differential pressure in UHP water directly out of the package, stability of the prewet condition was confirmed through one-year shelf life. The differential pressure data also confirmed that the prewet process had no effect as compared to conventionally prewet cartridges. The differential pressure data are summarized in Table 3. The differential pressures measured indicate that dewetting of the hydrophobic membrane did not occur over the 24-month shelf life testing.

**Table 3: Differential Pressure (psid/gpm)**

| Shelf Life Period | 0.01 µm Prewet Emflon® filter | 0.05 µm Prewet UltiKleen™ filter |
|---|---|---|
| 3 months | 0.85 | 0.75 |
| 9 months | 0.85 | 0.75 |
| 12 months | 0.85 | 0.75 |
| 24 months | 0.85 | 0.75 |

## Installation and Operational Precautions

Fluoropolymer filters are sold as either dry filters or as prewet filters. If the filter is received in the dry form it must be prewet with a low surface tension fluid and flushed to completely remove that fluid so that it does not contaminate the fluid being filtered. This flushing needs to be extensive and if the end user does not have sufficient means of monitoring the filter effluent for purity, then there may be a risk of contaminating the final fluid that is being filtered and ultimately the product being processed.

There are some precautions that must be undertaken when fluoropolymer membrane filters are used. These precautions are critical when filtering fluids with a surface tension >28 dynes/cm. The water wet hydrophobic PTFE membrane will easily accept gas into the pores of the membrane causing dewetting. Dewetting is defined as the previously fluid wet PTFE membrane returning to a hydrophobic state coupled with the inability of the membrane to pass fluid. Once the membrane dewets, it is necessary to rewet with the low surface tension fluid and flush. In some instances the filter may have to be thoroughly dried at elevated temperature for 24 hours and then rewet; a process that costs time and money.

The source of this gas that would cause dewetting could be from the atmosphere, long air filled lines leading to the housing in which the filter is installed, or it could come from the degassing of water used to flush the cartridge or chemicals that give off gas, either from trapped microbubbles (sulfuric acid) or as a breakdown product of the chemical (hydrogen peroxide).

After the initial prewet and flush, or the removal of the filter from the prewet packaging, the filter must be immediately placed in the housing and the fluid started flowing through the filter. If the filter sits exposed to the atmosphere, the wetting fluid may gravity flow from the membrane and cause dewetting. This may only partially dewet the membrane but reducing the effective filtration area compromises the performance of the filter in the form of high differential pressure and low flow rates.

Precautions are necessary when introducing the fluid to the filter; this is critical when the filter is in a water-wet state, such as after the prewet step or removal from the prewet packaging. It takes very little pressure to force the water from the pores of PTFE membrane. Typically only 2-3 psig is sufficient to start the membrane dewetting. If a fluid is introduced rapidly into a housing, and venting is not sufficient to evacuate the gas from the housing at the same rate that it is being introduced, the resultant backpressure could easily force air through the membrane and cause the PTFE membrane to dewet.

In automatic chemical delivery systems with diaphragm pumps that are controlled by pneumatic solenoids and pneumatic flow valves, this build up of backpressure can be rapid and can be a major source of PTFE membrane dewetting in chemical delivery systems. The introduction of the chemical must be controlled so as to avoid any backpressure in the filter housing. Pumps should be started at low pump stroke cycles and if there is a valve on the outlet of the pump/inlet of the housing, it should be used in a manual mode and slowly opened to let chemical into the housing at a rate equal to the flow of air through the inlet vent. Outlet valves and vents should be open so that the fluid will easily flow through the filter. Once the filter and housing are completely filled with fluid the inlet valve can be completely opened, the vent valves closed and the pump brought up to normal operating conditions. In some chemical filtration application the inlet vent may need to have a constant bleed. If off gassing or release of bubbles from the chemical is suspected, then accumulated gas in the housing could blind off the filter or cause dewetting of the membrane.

Two other factors that can dewet the filter in chemical delivery systems and in recirculation baths is the changing of chemical drums or the chemical itself. In recirculating chemical baths chemical is often changed to maintain its strength and purity. During this change it is important to recognize that this is a potential time for the filter to dewet. Precautions need to taken to minimize this dewetting opportunity. If air is introduced into the line or the housing is drained, precautions need to be taken when reintroducing the chemical. When the system is restarted it is necessary to make sure that the system chemical is introduced slowly and entrained air is vented out thoroughly. Proper venting eliminating any gas backpressure on the upstream side of the filter and prevents dewetting of the filter.

Ultrapure water (UPW) and certain chemicals may outgas if the circumstances are right. If UPW systems that do not contain degasification units the UPW may become fully saturated with either air or nitrogen. When UPW is pressurized by a pump to be distributed in the piping system and recirculated through the supply and return loops, it can become supersaturated and when pressure is released or there is a drop in pressure, such as downstream of a filter or within the filter membrane, degassing or the formation of bubbles caused by cavitation will occur. This degassing can happen during the flushing of a filter if the pressure drop across the filter is high. This degassing will cause bubbles to form either within the membrane on the downstream side of the membrane. Bubbles on the downstream side of the membrane could potentially be adsorbed back into the PTFE membrane causing dewetting.

In the case of a chemical causing dewetting, the same phenomena as occurs with UPW can occur with chemicals that have high levels of dissolved gas or suspended microbubbles. Sulfuric acid can have entrained gas as microbubbles, not the same as dissolved gas, and when the acid is filtered these microbubbles may be retained by the PTFE membrane and cause dewetting. Another chemical that is common for wet cleans is hydrogen peroxide; both in SC1 (ammonium hydroxide and hydrogen peroxide) and in SC2 (hydrochloric acid and hydrogen peroxide). Hydrogen peroxide will decompose and release oxygen. It will also react with organics and particles trapped by the filter to decompose them and in this reaction release. Both of these gasses can be trapped in the matrix of the PTFE membrane resulting in dewetting.

## Conclusion

Although unlikely, filters containing hydrophobic PTFE membrane may dewet under certain circumstances. Precautions in the installation and operation of these filters need to be taken in order to prevent dewetting of the membrane in order to maintain optimal filter performance.

1. Water wet PTFE membrane filters should not be exposed to any potential drying.

2. When starting flow through a newly installed prewet filter in an empty housing it is necessary to adequately vent the housing to prevent any backpressure within the housing. This backpressure could dewet the filter.

3. Process fluid flow needs to be introduced slowly to the filter for the same reason as in #1.

4. Precautions need to be taken when changing chemicals both in chemical distribution systems and point of use applications. The introduction of air in lines can potentially dewet the PTFE membrane.

5. Filter systems and housings that contain chemicals that can potentially outgas should have proper venting so that accumulated gas can escape.

6. Sizing of filters for proper flow should be addressed so that excessive pressure drops across the filter membrane do not cause degassing and the formation of bubbles.

Pall has introduced a modified PTFE membrane that eliminates the possibility of in-service de-wetting. With the introduction of the PALL Excellar® filter, installation and operating precautions typically associated with standard hydrophobic PTFE membrane filters is no longer a concern. The PALL Excellar filter was developed to overcome the propensity of hydrophobic PTFE to dewet and cause filter performance problems.

### References:


- Filterite® Electronics Fluoropolymer Products Technical Performance Guide, pg. 15
- Pall Microelectronics Contamination Control for the Microelectronics Industry, pg. 30
- Pall STR-PUF-30, "Eliminating the need to prewet hydrophobic filters with low surface tension fluids", O'Sullivan, J; Gotlinsky, B., 1994


### Pall Product Data Sheets

E28A – Emflon® Filter

E75A – UltiKleen™-CDS

E84A - UltiKleen™-Excellar Filter and Kleen-Change® Assemblies

E87A – Fluoryte™ High Flow Filter

E97 – Prewet UltiKleen and Kleen-Change Filters

E98 - UltiKlee


Pall Corporation

2200 Northern Boulevard
East Hills, New York 11548-1289 USA

1.800.360.7255 toll free (Only in US)
1.516.484.5400 phone
1.516.625.3610 fax


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