

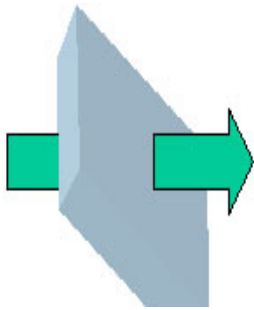


Teflon®

Nonstick & Industrial Coatings

Teflon® Finishes in the Chemical Processing Industry

Permeation—Its Effects on Teflon® Fluoropolymer Coatings



Permeation

- Penetration of a barrier (polymer layer) by chemical vapors or gases.
- Function of the particular permeant:
 - Solubility in the polymer
 - Diffusion through the polymer's intermolecular spaces

Introduction

Fluoropolymers are virtually inert substances that can withstand high temperatures. These two properties make them ideal for use as coatings to protect metals from corrosive attack. It is important to realize that fluoropolymer coatings are used where other polymers could not possibly survive, that is, under extremely hostile conditions. Under such conditions, permeation assumes a much more important role in the determination of performance.

This technical bulletin deals with the *effects* of permeation, not the theory itself (Fick's first law, Henry's Law, etc.). Further, it deals with the effects of permeation in the context of the harsh chemical environment to which coated parts are exposed (and not, for example, to the common thin plastic wraps used in the food packaging industry). The first portion of this bulletin discusses permeation through free-standing films, which relates to plastics and composites such as dual laminates. In the second portion, the effects of permeation are addressed as related to coatings bonded to metal substrates.

Permeation describes the transfer of gases and vapors in barrier materials such as polymeric plastics. As shown in **Figure 1**, the process involves: (1) dissolving the penetrant in the barrier material, (2) diffusion of dissolved penetrant through the material as a result of the concentration gradient, and (3) evaporation of the penetrant from the opposite side of the material.

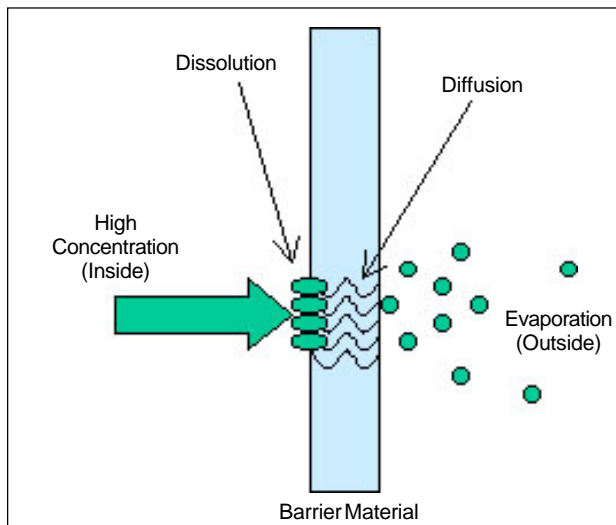
Permeation is generally regarded as an important consideration in determining the performance of plastics or composites, and for good reason. All polymers are permeable, and structures such as dual laminates or sheet linings are essentially free-standing polymeric materials.

Coated metal vessels, on the other hand, are a composite consisting of a relatively thin coating bonded to a metal shell. The major constituent of the coating is a fluoropolymer, which is permeable. The steel shell, however, is not permeable (at least not to the gases and vapors encountered in chemical processing).

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Figure 1. The Process of Permeation



Thus, whereas the concern for plastic and composite structures relates to the exterior emanation of chemical vapors that have permeated the plastic, the concern for coated metal vessels relates to the chemical attack on the steel from vapors permeating the fluoropolymer coating. This bulletin addresses these concerns and provides a discussion on coatings technology that helps explain how bonded linings made from fluoropolymer coatings are ultimately permeated, and how new advances in coatings technology allows us to manage these permeation effects.

Factors That Affect Permeation

Permeation involves a combination of physical and chemical factors¹ as shown in **Table 1**. Note that all the Factors in **Table 1** are represented as a positive (+) change, i.e., an increase. The effect of this change can be an increase (+) or decrease (-) in the rate of permeation, depending on the respective factor. For example, increasing the concentration, temperature, or pressure increases the rate of permeation. Increasing the polymer thickness decreases the rate of permeation.

Table 1
Permeation Variables

Factor	Change	Effect on Permeation
Permeant Concentration	+	+
Temperature	+	+
Pressure	+	+
Permeant/Polymer Chem. Similarity	+	+
Voids in Polymer	+	+
Permeant Size/Shape	+	-
Polymer Thickness	+	-
Polymer Crystallinity	+	-
Polymer Chain Stiffness	+	-
Polymer Interchain Forces	+	-

In general, *crystallinity* can range from the perfect order of carbon atoms in a diamond to completely noncrystalline, or amorphous, substances such as glass. Polyvinylchloride (PVC) polymers are considered noncrystalline. Fluoropolymers and polypropylene resins are semicrystalline, meaning they have domains (separate regions) of crystalline and amorphous character. The ratio of these domains is determined by the chemical makeup of the polymer as well as how it was processed (rapid cooling, for example, freezes the polymer chains in an amorphous state whereas slow cooling allows the molecules to arrange themselves in crystalline patterns). As the crystallinity increases, the related intermolecular volume decreases. This tighter packing of the molecules restricts the permeant from passing through.

Large size molecules, especially those with more complex, branched configurations are likely to be sterically hindered and thus diffuse slower than small molecules with simple configurations.

If the permeant is *chemically similar* to the barrier coating, i.e., has similar chemically functional groups and similar polarity, it will likely be more soluble in the polymer. This will cause the polymer to swell, resulting in an increase in the molecular space between the polymer chains and thus increase the rate of permeation. Note that the effect of chemical similarity counteracts that of crystallinity.

Increasing the *polymer chain stiffness* imparts resistance to bending. Increasing the *interchain forces* (Van der Waals, hydrogen bonding) increases the molecular attraction between adjacent polymer molecules. Both of these factors result in lower molecular mobility. The polymer molecules are less likely to separate to allow the permeant molecule to pass through, thus decreasing the rate of permeation.

Voids obviously relate to the quality of the polymer and the fabrication process. Voids are not considered in permeation theory, but they are a fact of commercial life.

In addition to these factors, consideration must also be given to environmental stress cracking. Stress cracks can occur when a polymer is under a small load condition for a prolonged time. Crystallinity, molecular weight, absorption of chemicals, mechanical or thermal stress, and processing conditions can all affect stress cracking². Stress cracks, like voids, allow the mass transport of chemicals through the coating.

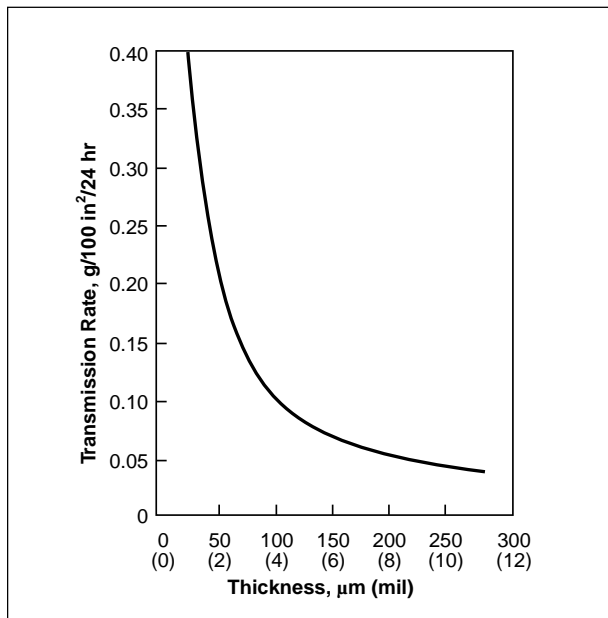
Permeation Data

Permeation data is often presented for water vapor (H_2O) as shown in the representative examples **Figures 2** and **3** below. The rate of diffusion is measured against the film thickness of the polymer films, at some defined temperature. Other kinds of graphs show how permeation varies with temperature, at a given film thickness. The atmospheric gases (O_2 , CO_2 , and N_2) are often presented this way. **Figure 4** represents an example of a more technical treatment of data³. The empirical data quantify and confirm, as was stated above, that permeation decreases with increasing film thickness and increases with increasing temperature.

It is important to exercise some care in the interpretation of these kinds of data. Casual observation may lead to the conclusion that Product A is more or less permeable than Product B, but upon further consideration it becomes clear that such a conclusion generalizes a variety of factors that the data may not necessarily support.

For example, the data are only valid for the substances measured, namely water vapor, MEK, or the atmospheric gases, and only under the conditions of the test design used for the measurements. If these were the only substances involved in the process, and if the process had some correlation to the test method, the data would be more meaningful. But most processes involve other substances, for which permeation test data are not available, and also mixtures of other substances for which permeation test data are even less likely to be available.

Figure 2. Water Vapor Transmission Rate of Teflon® FEP Film at 40°C (104°F) per ASTM E96 (Modified)

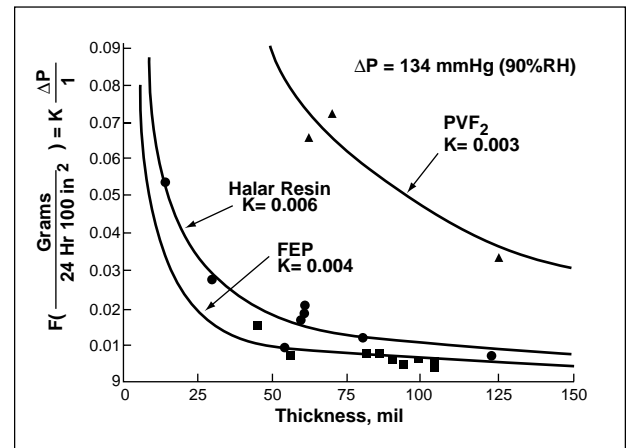


Source: DuPont

Because there are a variety of factors that can affect permeation, any generalizations based on simple water vapor, gas, or single chemical data, therefore, are likely to be misleading.

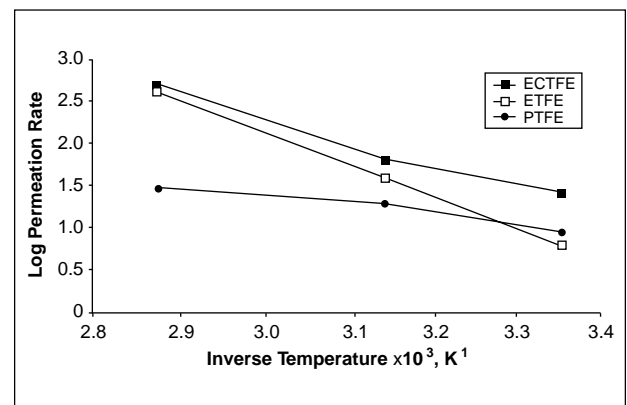
Permeation is a *rate* function. It is a measure of the quantity (grams) of material that traveled through a given area of film (cm^2) over some time interval (hours). The time interval actually used in the laboratory test is mathematically normalized to 24 hours to make it easier to compare materials, but keep in mind that the graphs illustrate *how fast* a substance diffuses through the film. Saying that Film A is more or less permeable than Film B is accurate, as long as it is understood that the *rate* of diffusion is being discussed. Most processes operate longer than 24 hours, so over extended time intervals any slight differences in the rates of permeation between two materials become less important. It's just a matter of time before the effects of permeation make their presence known in either case.

Figure 3. Moisture Vapor Permeability Rate vs. Thickness at 60°C (140°F)



Source: Ausimont Literature, 6/89 GB204

Figure 4. Methyl Ethyl Ketone Permeation, 250 μm (10 mil) thick



Source: DuPont

The graphs in **Figures 2** and **3** show a dramatic drop in permeability with increasing film thickness, the value of which depends on the temperature. Note, however, that the permeation rates at the higher thicknesses are not zero. In other words, the data does not say the fluoropolymer resins are impermeable. Thicker films retard the rate of permeation, but do not stop it. Permeation is unavoidable in free-standing plastic films, including fluoropolymer films as thick as 4,450 μm (180 mil). But it can be managed.

Permeation Data Concerns

- Little useful, hard data available
- Thin film (<250 μm) data is typically misleading
- Atmospheric gas data is typically misleading (N_2 , O_2 , CO_2 , H_2O)
- Mixtures can permeate differently and/or selectively vs. single chemicals
- Laboratory data recommended only for qualitative comparison
- Best data from test of actual component in exact process conditions

Permeation vs. Chemical Reactivity

A fundamental assumption in the formal treatment of permeability is the absence of chemical reactivity between the penetrant chemical and the polymer material through which it diffuses. As illustrated in **Figure 5**, chemical reactions with the polymer itself or with fillers or other additives can create voids, which then become pathways for the mass transport of the penetrants. This is the case with many organic polymers in chemical service, especially at elevated temperatures. What finally emerges from the other side can be an entirely different species.

If chemical reactivity is found to occur, permeability is no longer the sole issue. For example, **Figure 6** illustrates a failure of fiberglass reinforced plastic (FRP). FRP is constructed from fiberglass cloth impregnated with a nonfluoropolymer resin, usually a phenolic, modified phenolic, and sometimes a vinyl ester. The FRP duct as shown is used in the Semiconductor Industry to exhaust the chemical by-products from the etching processes for the manufacture of computer chips. Here, a white crystalline material is observed emerging. The composition of the white material is unknown, so it is not possible to say whether it is a permeant chemical from the process or some other substance that resulted from a chemical reaction within the FRP itself. In either case, however, the duct is leaking and may represent a potential hazard to personnel.

Other plastics such as polypropylene often fail by becoming brittle, and are then prone to cracking. This type of failure relates to the poorer chemical resistance of polypropylene polymer.

These kinds of observations characterize the complexity of the situation in the Chemical Processing Industry, where it is often difficult to distinguish the effects of permeation from those of chemical compatibility.

Permeation Through Free Films vs. Coatings

Unlike the common plastics just discussed, *Teflon*[®] fluoropolymers are exceptionally resistant to chemical attack, even at high temperatures. The likelihood of a chemical reaction between the permeant with the pure polymer itself, therefore, is very low. Thus, as illustrated on the left side of **Figure 7**, there is a high probability that the process of permeation will leave both the permeant and the film unchanged.

Figure 5. Chemical Reactions

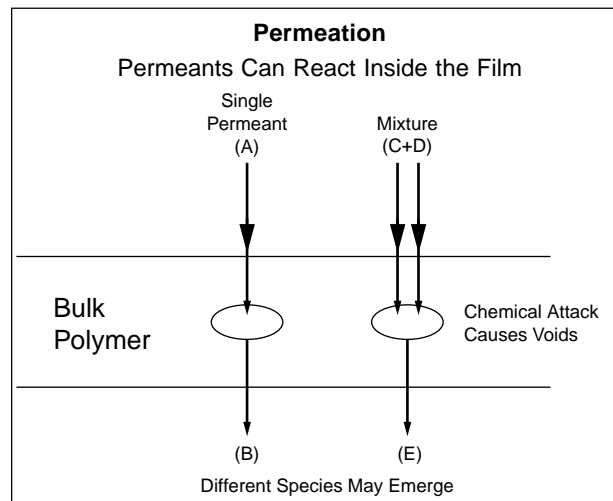


Figure 6. FRP Failure



Photo courtesy of Fab-Tech, Inc., Colchester, VT

But what happens when this irresistible force of permeation reaches the impenetrable wall of a steel vessel? It stops—at least in the sense that it does not emerge from the other side. At this point the permeant chemical has few options. Depending on its degree of chemical similarity, it will become absorbed to some extent within the polymer until a saturation point is reached. However, since the types of process chemicals and the conditions inside the vessel (temperature, pressure, and concentration) fluctuate over time, molecular diffusion continuously occurs in both directions.

Coatings vs. Linings

It is important to distinguish a coating from a lining. A “lining” refers to a protective layer of material, and usually a *thick* layer of material, *applied to the inside surface of a vessel*. It does not matter what kind of material is applied, or how it is applied. For example, the lining can be made from extruded sheets of fluoropolymer (or other plastic), thermoformed to the interior contours of the vessel, and glued in place (with welded seams). Or it can be a *Teflon*[®] coating applied and baked on to the interior surface. In both cases the vessel is said to be lined because the material is on the inside of the vessel. On the other hand, the agitator blade for that vessel is described as “coated” (or having a *Teflon*[®] coating)—where the film covers the *outside* surface.

The distinction between (interior) linings and coatings is important because the failure mechanisms are drastically different. The effect of permeation on linings is far more severe than on coatings.

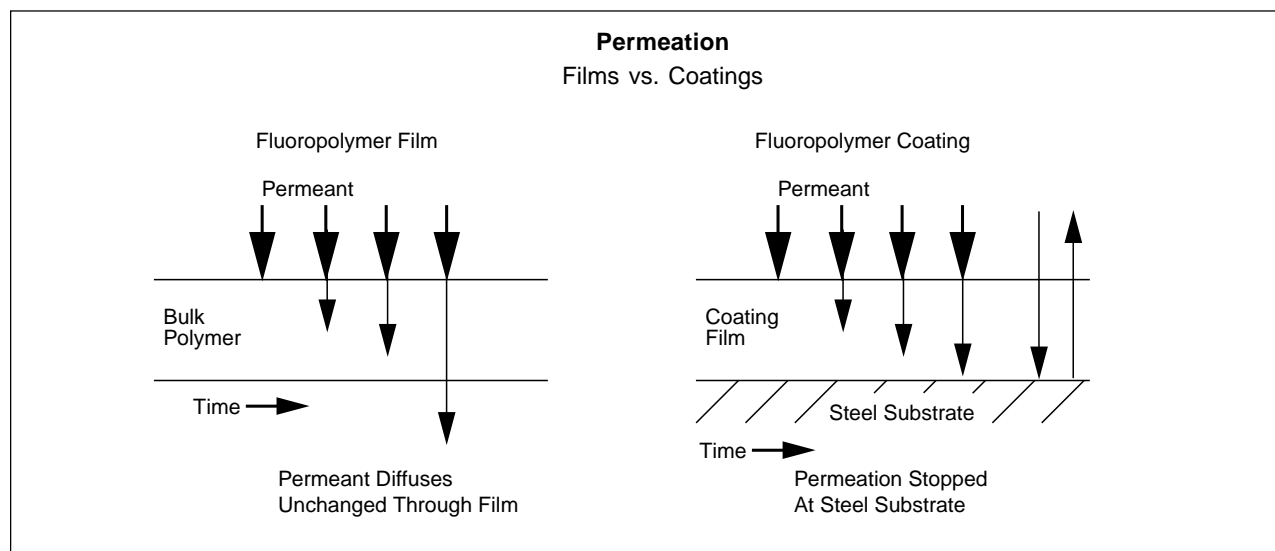
But to better understand this, it is first necessary to know something about the technology of fluoropolymer coatings.

Fluoropolymer Coatings/Linings

Fluoropolymers have a high degree of nonstick character, thus by themselves do not adhere well to metal (or other) substrates. A primer coat is necessary to achieve adhesion, and by default it cannot be a fluoropolymer. There are a variety of tough, temperature-resistant organic resins that have excellent adhesion to metals and that also have good chemical resistance. A primer, therefore, contains a blend of the fluoropolymer with another organic resin. When applied to the metal and fusion bonded (baked at high temperature), the organic resin bonds with the steel. The fluoropolymer component surrounds the organic resin molecules, providing a shield from chemical attack. It also fuses with the fluoropolymer topcoat subsequently applied to create a chemically resistant surface layer. This constitutes the coating system.

The development of the adhesive bonds with the metal substrate depends on how well the substrate was prepared prior to application of the coating. Contaminants such as dust, oils (even from fingerprints), or ionic residues from aqueous cleaning solutions that remain on the surface will prevent the primer from bonding at those points. Roughening the surface, preferably by grit blasting, increases the surface area and provides additional sites for adhesive bonding to occur. A clean, roughened surface, therefore, is essential for good adhesion.

Figure 7. Permeation in Fluoropolymer Films vs. Coatings



Another critical factor that affects adhesion is the baking process. Fluoropolymer resins are thermoplastic. Melt-processable varieties (including ETFE, PFA, and FEP) liquefy above their melting points, but the viscosity of the molten resin is very high. It takes time for this viscous fluid to flow together to form a properly fused, cohesive film. From an adhesion standpoint, enough time is also needed for the molten resin to flow into and wet the valleys of the rough surface profile and for the organic resin in the primer to form the adhesive bonds. An adequate dwell time at or above a specific temperature (depending on the particular resin) is essential, therefore, to develop the full integrity of the coating.

From a practical standpoint, metal parts come in different sizes and masses, and the temperatures in baking ovens are seldom constant everywhere within. These variations can significantly affect the quality of the bake, and therefore the performance of the final coating. To compensate for these effects, a robust coating system is needed, i.e., one having the thermal stability to withstand prolonged exposures at high bake temperatures without drips, sags, blisters, or polymer degradation.

No coating process is perfect (and this applies to all coatings, not just fluoropolymers). A single coat will almost always contain pinholes, and pinholes allow gross mass transport of chemicals through the film (which makes the discussion of *permeation* effects academic). Fortunately, multiple coats cover up the pinholes, and routine quality control spark-testing on the final system assures this is so. Micropores, however, are more insidious. Micropores can be any microscopic void in the coating. They arise from minute impurities inevitably encountered during normal production conditions and are more commonly found at the interface between the primer and the metal substrate due to contamination from processing and handling. Spark tests will not detect them. As will be discussed later, these micropores are believed to be the locus of severe failure of the lining in service.

Effects of Permeation on Fluoropolymer Coatings

Regardless of differences in the rates of permeation, penetrant chemicals will ultimately reach the metal interface where they can react with the molecular functionalities in the primer that form the adhesive bonds. If the adhesive bonds were destroyed the coating would obviously lift away from the metal. In practice, however, this is seldom the case because of the chemical shielding effect due to the presence of the fluoropolymer. What usually happens is that

the adhesive bonds become solvated (hydrated in aqueous systems)⁴. Although they remain mostly intact they may be considerably weakened. The effect of hydration is temperature dependent, almost insignificant at room temperature and increasing in severity as the boiling point of water is approached. At the boiling point, where water is now in equilibrium with its vapor, the rate of permeation increases drastically. Thus water alone is capable of significantly weakening the organic resin adhesive bonds. Peel strength values, for example, show a significant decrease after exposure to several hours of immersion in boiling water. However, a day or so after exposure the peel strength values show considerable recovery, although not to their original values.

The discussion above describes what happens when a coating (as on the agitator blade) is exposed to a permeation situation. The factors listed in **Table 1**, for a specific exposure situation, determine the overall performance of the coating. In practice, fluoropolymer coatings on parts that are immersed in some chemical mixture perform very well for a long time (unless the coating was poorly applied initially). Most failures involve other factors, such as abrasion resistance where the coating is mechanically removed slowly over time.

Effects of Permeation on Fluoropolymer Coatings Used as Linings

The situation is much different when the same coating system is used as an interior lining. In this case, after some period of exposure at elevated temperatures, the lining will start to blister. The number and size of these blisters can vary. Sometimes many small blisters form, as illustrated by a clear PFA lining in **Figure 8**. Over time, these small blisters will continue to grow and (as evident in **Figure 8**) begin to merge into larger ones. Sometimes only one or a few very large blisters form initially. In either case, given sufficient time of exposure, large sections of lining can completely delaminate from the metal surface. Note, however, that this phenomenon is only observed when the liquid inside the vessel is at a high temperature.

Obviously, this phenomenon cannot be explained by the mere hydrolytic weakening of primer bonds due to permeation. Coatings do not fall off agitator blades, nor then should they delaminate from the interior wall of a vessel. The fact that they do requires a more complex explanation, and the mechanism postulated for lining failure includes, in addition to permeation, a combination of osmosis and thermodynamics.

As permeant chemicals and moisture travel through the film they dissolve any ionic impurities that are present and, eventually, transport them to a micropore, as schematically illustrated in **Figure 9**. Micropores within the film itself are not critical because the plastic matrix is a nonreactive fluoropolymer. At the metal interface, however, they are much more critical because this is where the adhesive bonds are. The accumulation of ionic species inside the micropore at this point is driven by the temperature differential between the warm coating surface on the interior of the vessel (t_{Process}) and the steel with a cooler exterior temperature (t_{Ambient}). Fluoropolymers, being excellent thermal insulators, allow this differential to exist. The gaseous permeant, therefore, can condense to a liquid. The net result is the creation of a region of higher ionic concentration, which sets up an osmotic cell⁵. The osmotic pressure is strong enough to disbond the primer coat and thus lift the film from the metal.

Over time the area of disbonding continues to enlarge, eventually forming a visible blister. Remembering that permeation and the hydrolytic weakening of primer bonds increase with increasing temperature, the disbonding effect from moisture is accelerated as the temperature increases. Upon

Figure 8. Initial Blisters in Lining

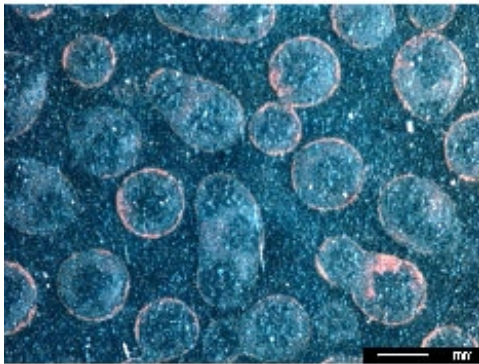
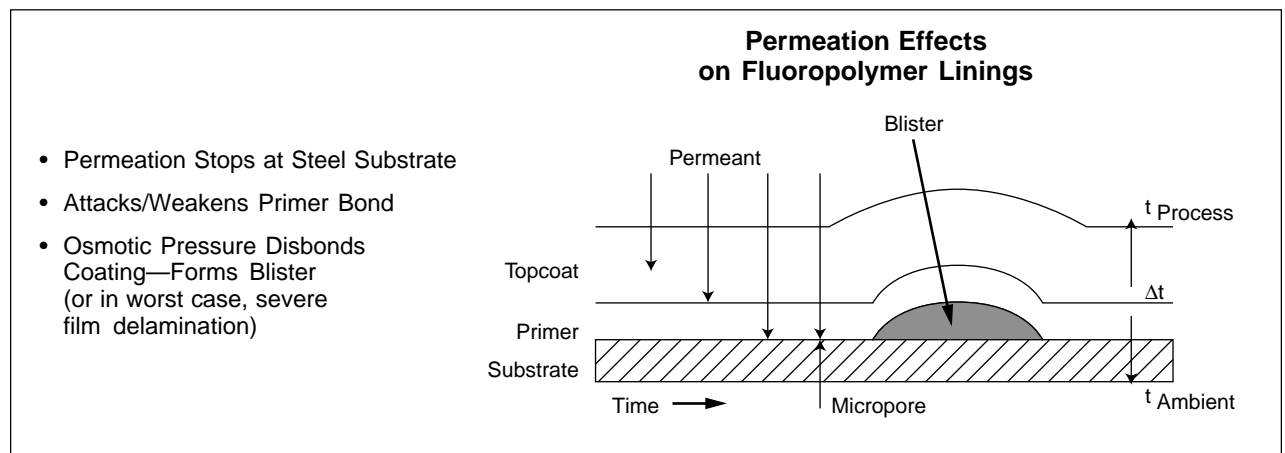


Figure 9. Mechanism of Lining Failure



reaching the boiling point of water, catastrophic failure (severe blistering or total delamination) can occur within a few days for a poorly designed or poorly applied coating system (at 1,000 μm [40 mil] thickness; thinner films fail faster). Thus, when fluoropolymer coatings fail as linings in a chemical exposure, they almost always do so by delamination from the metal substrate.

In the laboratory, the Atlas Cell Test (also called a Blind Flange Test), **Figure 10**, is probably the best prognosticator of the performance of a lining system. This device simulates the conditions of an actual lined vessel, especially the thermodynamics because the process temperature of the contents inside the vessel can be carefully controlled and measured against the ambient air temperature.

Coated (lined) panels are clamped to each open side of the glass vessel and sealed with a rubber gasket. Any liquid or mixture can be tested by adding a sufficient amount to cover the bottom two-thirds of the panels so that the effect on the coating in the liquid and vapor phases, as well as at their interface, can be observed. The apparatus additionally includes a heating element to heat the liquid, a thermometer to measure temperature, and a reflux condenser to return condensed vapors. The cell can be operated for months with little attention, although it is often more frequently dismantled to better observe progress.

Boiling water is always used as the first test medium because it is safe to handle and quite effective in defining performance. Typically, most fluoropolymer linings of 1,000 μm (40 mil) thickness start to blister after only 2–3 weeks (335–500 hours) when exposed to boiling water. The pictures in **Figures 8** and **11** were obtained from this test. If a coating candidate shows promise, additional acids and bases or other chemicals can then be tested.

Figure 10. Atlas Cell Test Apparatus

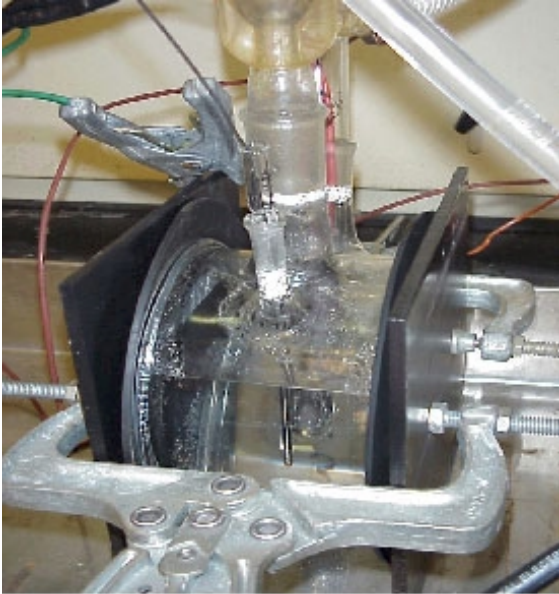
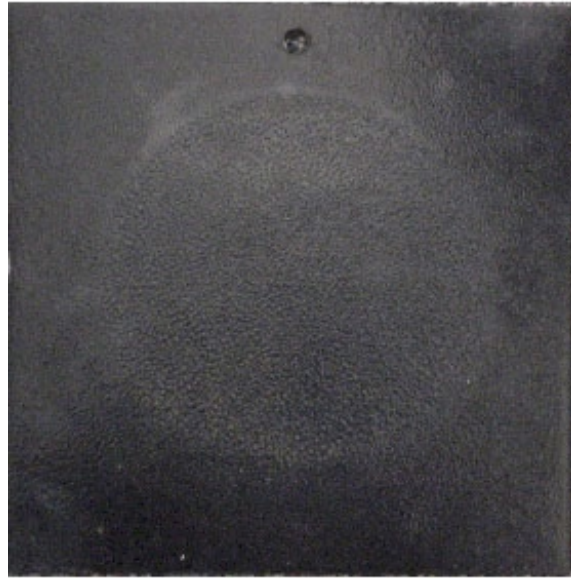


Figure 11. Atlas Cell Test Panel, 1,000 μm (40 mil) PFA, 2-Week Boiling Water Exposure



Although the Atlas Cell Test closely simulates actual field parameters and conditions, there is no consensus on its correlation to actual field performance. One possible reason is that most fluoropolymer linings evaluated to date only last a relatively short time in this test (2–3 weeks). It may be that this level of performance is simply inadequate to compensate for the actual variables such as the specific chemicals, their concentrations, and especially the temperatures and pressures of real processes. However, as will be discussed next, new technologies that are now lasting much longer in this test are currently under field evaluation and the expectation is that a better correlation will be found.

Managing Permeation: Teflon® Coating Technology

It is obvious by now that regardless of the slightly different rates of permeation through various materials, some species will eventually get through to the interface between the metal substrate and the coating or lining and cause problems. The problem is especially acute for linings because they are the most expensive to install, yet most likely to fail prematurely.

It does not matter which kind of fluoropolymer is used; they are all permeable. Thus the only recourse for the corrosion engineer who requires a fluoropolymer lining for chemical protection is to use a thicker one. Thick linings do slow the permeation process and extend the useful life of the lined vessel.

For example, many vessels are lined with fluoropolymer sheet linings that are 1.5 to 2.25 mm (60 to 90 mil) thick. However, sheet linings are very expensive and have their own limitations (namely seam cracking and disbonding of the neoprene or epoxy adhesive).

Fluoropolymer coatings used for linings are less expensive, but until recently there were no commercial products that performed reliably. This is because it is difficult to apply coatings on the interiors of vessels and the maximum obtainable film build is generally only around 1,000 μm (40 mil). Furthermore, the currently available coatings are limited to essentially pure fluoropolymers (although some applicators add small amounts of pigments for color). After final curing, these coatings have the same basic properties as extruded films—including the same degree of permeability. Thus, since even 2 mm thick sheet linings have limited service lifetimes, it would be unreasonable to expect a 1 mm thick coating to perform as well—it certainly would not be expected to perform any better.

The challenge, therefore, is to focus on the effects of permeability, and that inevitably leads to coatings technology. Coatings technology has the unique advantage of versatility. Unlike sheet linings made of pure bulk polymers, for example, where the only means of retarding permeation is to make them thicker, coatings can be modified with special additives to enhance a desired property without sacrificing the basic performance properties of the

fluoropolymer. In the case of primers, additional other nonfluoropolymer resins are added to provide excellent adhesion, as well as pigments and other fillers that act as barriers to permeation. The result is a strongly bonded primer coat that better resists the effects of hydrolytic weakening from permeation.

Special pigments are selected for the topcoats that significantly retard permeation by forcing the vapors to penetrate the film using a zigzag pathway, as illustrated in **Figure 12**. Adding such fillers has the same effect as increasing the thickness of the fluoropolymer, but it is more efficient and far more cost effective. Such additives, of course, must be chosen judiciously because the wrong choice can actually increase the rate of permeation by forming channels between the nonstick fluoropolymer resin and the surface of the additive. It is the *coating system*, therefore, that determines performance, not just the particular polymer upon which it is based.

DuPont has over 40 years of coating experience on a vast array of miscellaneous industrial parts used in a wide variety of service conditions, as well as on millions of frying pans coated with *Teflon*[®] and SilverStone[®] nonstick. This long-term success has relied on developing primer technologies that form exceptionally strong bonds with metal substrates and topcoats that are both durable and functional. It is this expertise that was incorporated into the design of the *Teflon*[®] coatings and linings dedicated to the Chemical Processing Industry, and is what brought new products such as the Aqueous and Powder “Ruby Red” coatings to this market. As the Atlas Cell Test performance shown in **Figures 13** and **14** attests, these new “Ruby Reds” are far superior in performance than any other fluoropolymer coating ever tested by DuPont.

Figure 12. Special Fillers Force Permeants to Take a Much Longer, Zigzag Path

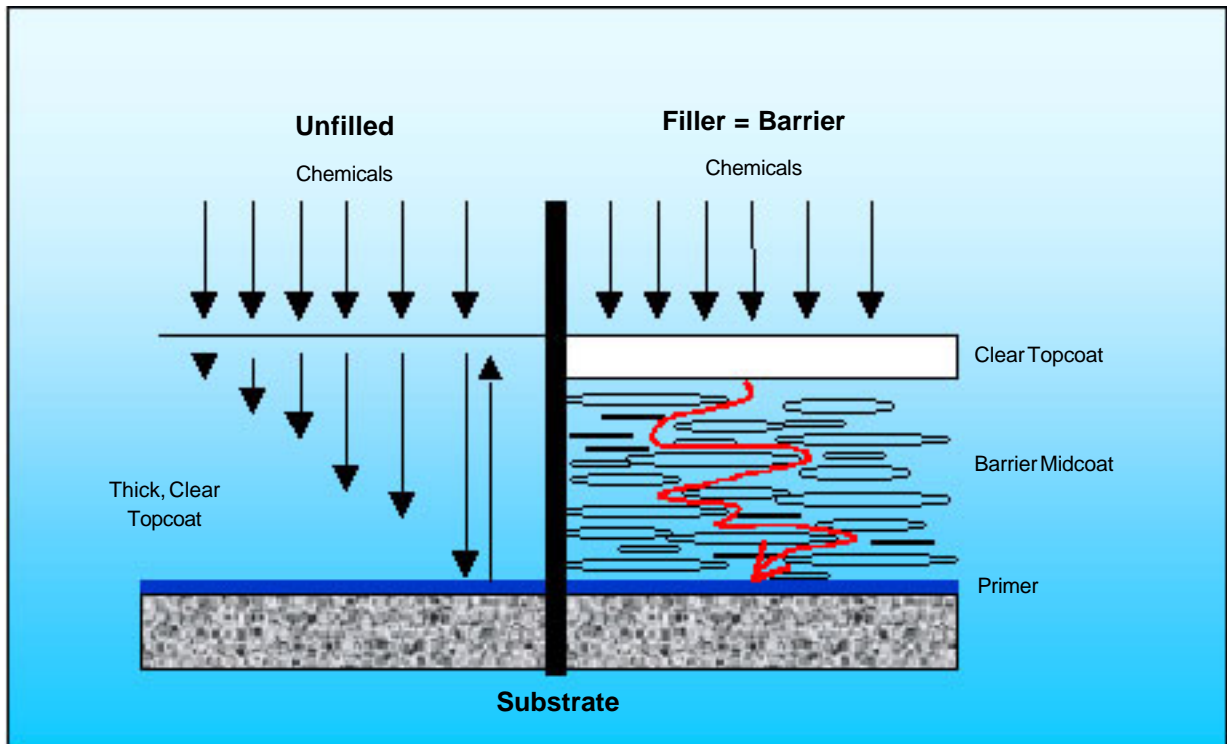


Figure 13. Atlas Cell Tests, Boiling Water, 1,000 μm PFA Linings

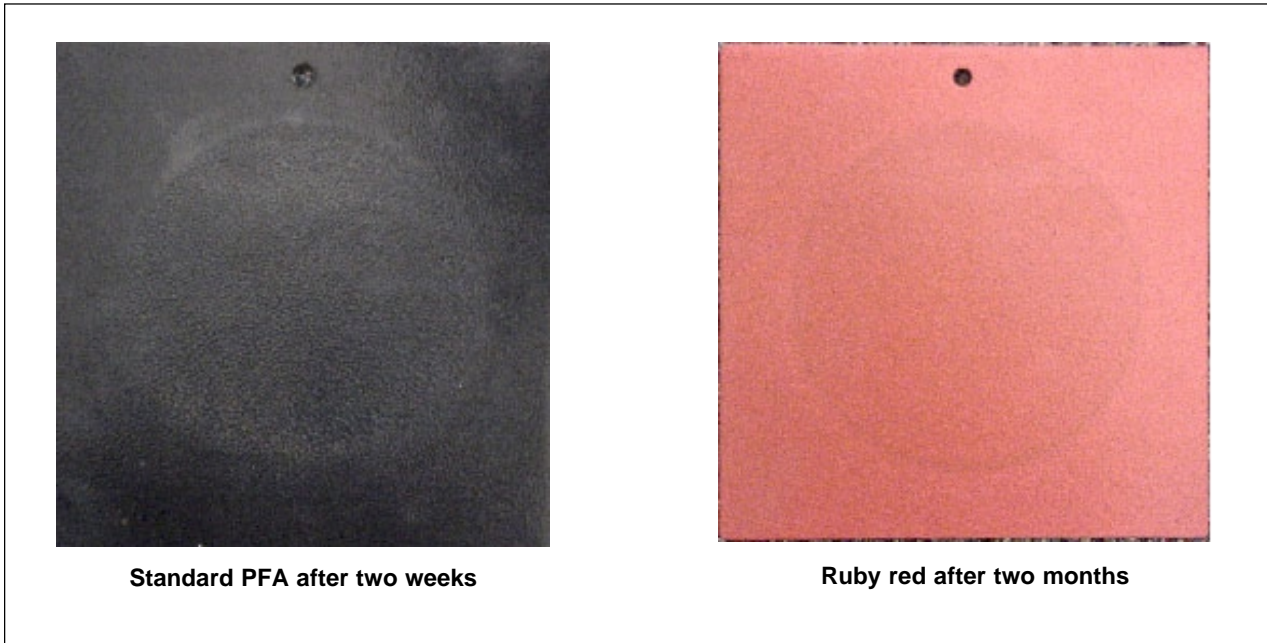
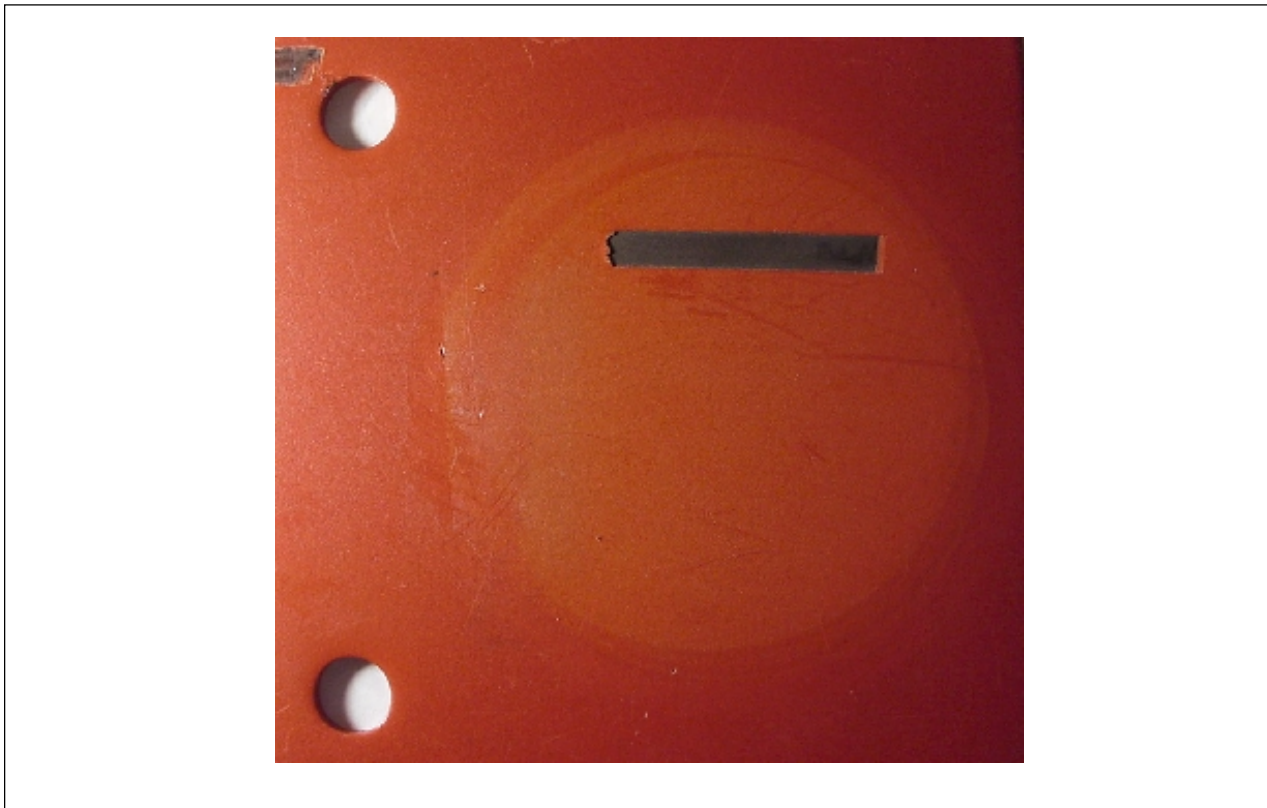


Figure 14. Ruby Red Prototype, 445 μm (17.5 mil) Thick, After 1-Year Atlas Cell Exposure in Boiling 0.05N Hydrochloric Acid



Summary

All polymers are permeable and, although many of the factors that affect permeability are known, there is often no test data available that are meaningful for the specific conditions of a specific process.

Using laboratory test data based on simple atmospheric gases or other individual chemicals to predict performance in harsh chemical environments encountered in the Chemical Processing Industry can be risky. Chemical compatibility of the plastics and any additives with the process chemicals must also be considered, because chemical attack can be far more detrimental to performance than mere permeation. Fluoropolymers have outstanding chemical resistance. When integrated into a well-designed and properly applied coating system, *Teflon*[®] coatings from DuPont provide a superior alternative to any of the competitive coatings based on the pure resins and approach the performance of sheet linings. These new coatings clearly demonstrate that the effects of permeation can be managed. And while they may not be the final solution to the permeation problem, the evidence so far strongly indicates they are a quantum leap better in performance than anything else on the market.

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