Fluoropolymers for the Chemical Processing Industry Applications

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Abstract

Fluoropolymers are very effective in corrosion protection applications due to their exceptional chemical resistance, low permeability to liquid and gases, and excellent strength and toughness at elevated temperatures. Fluoropolymer coatings and liners have been used successfully in a variety of industries for several years. This presentation reviews the performance of fluoropolymers in several chemical environments and describes the industrial case histories. The main focus of this work is the ECTFE resin; the liners manufactured from this resin performed very well for more than 20 years in several industrial applications.

Keywords: liners, plastic, protection, fluoropolymers, ECTFE**.**

Introduction

The U.S. Federal Highway Administration study (1), initiated by NACE International and released in 2002, shows that the direct cost associated with metallic corrosion in the U.S. is $276 billion, approximately 3.1% of the nation's Gross Domestic Product. This study also shows that utilities, transportation, and production & manufacturing account for most of the annual industrial corrosion cost.
The use of proper materials can significantly lower the expenses caused by corrosion. Fluoropolymers are suitable for corrosion protection applications due to their excellent chemical resistance to a variety of chemicals over a wide range of concentrations and temperatures, as well as their low permeability, high temperature thermal stability, surface smoothness, high purity, and non-burning characteristics. They can successfully replace metals and in many cases, they are the only viable protection against highly corrosive and aggressive chemicals.
Fluoropolymers can be applied on metals, concrete, or FRP if high mechanical strength is required.

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Description of Fluoropolymers

The first fluoropolymer, polytetrafluoroethylene (PTFE) was discovered by Dr. Roy Plunkett from DuPont in 1938. This discovery was followed by the development of other fluoropolymers in late 40's. Initially, PTFE was used in military applications, but other industrial applications were developed in the late 40's. Most of the thermoplastic fluoropolymers were synthesized in the 50's and 60's, which triggered the development of the corrosion protection applications of these resins.
The fluoropolymers used for corrosion protection can be divided into two groups: fully fluorinated and partially fluorinated polymers. The first group of polymers, also called perfluoropolymers, consists of PTFE, FEP, and PFA. The partially fluorinated polymers, commonly used in the Chemical Processing Industry (CPI) applications, are PVDF, ECTFE, and ETFE. When compared to the partially fluorinated resins, the fully fluorinated polymers have higher upper service temperatures and better chemical resistance at higher temperatures, but lower mechanical properties. The structure of fluoropolymers is as follows:

Polymer	Monomer(s)
Fully Fluorinated Polymers	
PTFE	$CF_2=CF_2$
PFA	$CF_2=CF_2$ + $CF_2=CF\text{-}O\text{-}(CF2)_n\text{-} F$ *n = 1, 2,3*
FEP	$CF_2=CF_2 + CF_2=CF\text{-}CF_3$
Partially Fluorinated Polymers	
PVDF	$CF_2=CH_2$
ETFE	$CH_2=CH_2 + CF_2=CF_2$
ECTFE	$CH_2=CH_2$ + CF2=CFCl

Table 1 - Chemical composition of fluoropolymers

Properties of Fluoropolymers

The C-F bond is the strongest in organic compounds, stronger than C-H or C-C bonds. Since more energy is required to break the C-F bond, fluoropolymers have a very good thermal stability and excellent chemical resistance.
The first fluoropolymer, PTFE, has an extremely high molecular weight and its' melt viscosity is about a million times higher when compared to the other polymers. Because of this extremely high melt viscosity and chain stiffness, the PTFE resin can not be processed using common melt-processable techniques.

The substitution of the fluorine atoms or the addition of side groups, allowed the synthesis of a variety of melt-processable fluoropolymers. These modifications improved the mechanical properties of fluoropolymers, but their chemical resistance and thermal stability were lowered.
There is no single material which will provide sufficient and cost effective corrosion protection. Fluoropolymers, due to their good chemical resistance and thermal stability, are one of the best choices in applications where good corrosion protection is required. In many cases, they are the only viable solution. The advantages of fluoropolymers as corrosion protection liners and coatings can be summarized as follows:

- good chemical resistance & low permeation
- low surface energy
- surface smoothness
  - low friction, excellent scaling and bio-film formation resistance
- high purity
- water repellent, anti-staining
- high temperature thermal stability
- excellent weathering resistance
- exceptional electrical properties
- non-burning & low smoke generation
- excellent UV and visible light resistance
- good light transparency

To choose the right fluoropolymer for a certain application, several performance criteria have to be considered. Table 2 lists the maximum service temperatures of fluoropolymers. This table shows that perfluoropolymers have a much higher temperature limit when compared to the partially fluorinated resins. However, the better mechanical properties of partially fluorinated resins (see table 3) give them a substantial advantage in free-standing structures, including piping systems.

Fluoropolymer	Max Temperature Rating [°C]
PTFE	260
PFA	260
MFA	235
FEP	210
ETFE	175
ECTFE	175
PVDF	150

Table 2 - Maximum temperature ratings of fluoropolymers.

Fluoropolymer	Tm (°C)	Elastic Modulus (MPa)
PTFE	> 320	500
Hyflon® PFA	305	600-500
Hyflon MFA	265 - 275	500-400
Halar® ECTFE	240	2000-1500
Solef®/Hylar® PVDF	170	2200-1700

Table 3 - Mechanical properties of Solvay Solexis fluoropolymers (2).

It is important to stress that there are substantial differences even between polymers which belong to the same group of resins. There are several grades of PVDF, ECTFE, and perfluoroalkoxy resins. Different grades of the same group of polymers were synthesized in order to get better performance for specific applications. In most cases, the modified grades were obtained by utilizing different co-monomers or by changing polymerization conditions. One of the examples showing the difference between polymers of the same fluoropolymer group is the surface smoothness of perfluoroalkoxy resins. Table 4 shows that the MFA resin has a much smoother surface when compared to the other PFA polymers. This makes this grade a good choice in applications where the elimination of scaling or biofouling is very important.

Material	Rms (Å)
Standard PFA	755
High-Purity PFA	290
MFA	161

Table 4 - Surface smoothness of perfluoroalkoxy resins (3).

Chemical resistance & permeability

It is not a main goal of this paper to compare all performance data of fluoropolymers. However, since most of the CPI applications require using materials with very good chemical resistance and low permeation rate, a brief summary of the compatibility of fluoropolymers in different chemicals is included in this work. Table 5 summarizes the performance of fluoropolymers in acids, bases, and oxidizers. With few exceptions, mainly PVDF in strong acids and bases, fluoropolymers perform very well when exposed to these chemicals. Table 6 contains the chemical resistance of the ECTFE and MFA resins to several very aggressive chemical compounds at elevated temperatures. Both resins performed very well in these tests: their weight gain was negligible and the resins retained most of their mechanical properties. Please note that the color change of the Halar surface exposed to concentrated sulfuric and nitric acids did not affect the properties of the ECTFE resin.

	Temp. (°C)	*MFA PFA PTFE*	ECTFE	ETFE	PVDF
ACIDS					
98% Sulfuric	104	+	+	+	S
37% Hydrochloric	104	+	+	+	+
BASES					
50% Sodium Hydroxide	104	+	+	+	A
50% Sodium Hydrosulfide	104	+	+	S	A
OXIDIZERS					
100% Chlorine	93	+	S	S	S
60% Sodium Chlorate	93	+	+	S	S
65% Nitric Acid	93	+	S	A	A
OTHER FLUIDS					
Crude Oil	140	+	+	+	+
Ethanol	100	+	+	+	+

+: Unaffected, **S**: Slightly Affected, **A**: Attacked

Table 5 - The effect of chemical exposure; 264-hour immersion test.

Chemical Name	ECTFE	MFA	Color After Exposure
37% HCl @100°C	0.2	0.0	ECTFE - light brown
65% HNO_3 @ 65°C	0.2	0.0	ECTFE - yellow
65% HNO_3 @ 100°C	0.8	0.0	No discoloration
98% H_2SO_4 @ 140°C	0.2	0.0	ECTFE - black
Oleum - 20% SO3 @ 40°C	0.2	0.0	ECTFE - black
NaClO (5% chlorine)@ 100°C	0.0	0.0	No discoloration
NaClO (10-15%) @ 100°C	0.1	0.1	No discoloration
CH_3OH @ 65°C	0.6	0.1	No discoloration
CH_3OH @ 120°C	0.5	0.0	No discoloration

** The change in tensile modulus, tensile strength at break, yield stress, and elongation was insignificant in all cases*

Table 6 – 30-day chemical immersion test - Weight Gain [%] (4).

In most of the chemical resistance testing, polymers are exposed to pure reagents or to concentrated compounds. Based on this data, some resins might be excluded from the applications where they are exposed to dilute solutions of the same chemicals. Halar ECTFE performs poorly when exposed to methylene chloride at 40°C and it can not be recommended for corrosion protection in these conditions. However, the ECTFE resin can be successfully used when in contact with 2% CH_2Cl_2 at much higher, 100°C temperature (5).

Good chemical resistance does not always guarantee that parts made from the resin will perform well in specific applications. The other factor which has to be considered is the permeation resistance of the polymer. A high permeation rate through the coating or the liner can cause the chemical attack of the substrate and consequently the failure of the part.
Figure 1 compares the permeation resistance of ECTFE, ETFE, FEP, and MFA fluoropolymers in chlorine (6). This graph clearly shows that the ECTFE resin has the lowest permeation rate of all tested polymers.
The effect of permeation rate on the performance of dual laminate pipes was studied by the Swedish Corrosion Institute (7). The dual laminate pipes, having 1.5 mm thick ECTFE and FEP liners, were exposed to wet chlorine gas at (80-85)°C for 2.5 years. These pipes were evaluated after the exposure and the amount of chlorine permeated through the liner was calculated. The SCI testing, summarized in Table 7, determined that that the top 0.5 mm of the ECTFE liner was chemically attacked and the FEP resin was not chemically affected at all.
However, it was calculated that the amount of chlorine permeated through FEP was about 30 times higher when compared to the ECTFE resin. As a consequence of its poor permeability, the FEP liner developed blisters and delaminated from FRP. At the same time, the bond strength between the ECTFE liner and FRP was not affected.

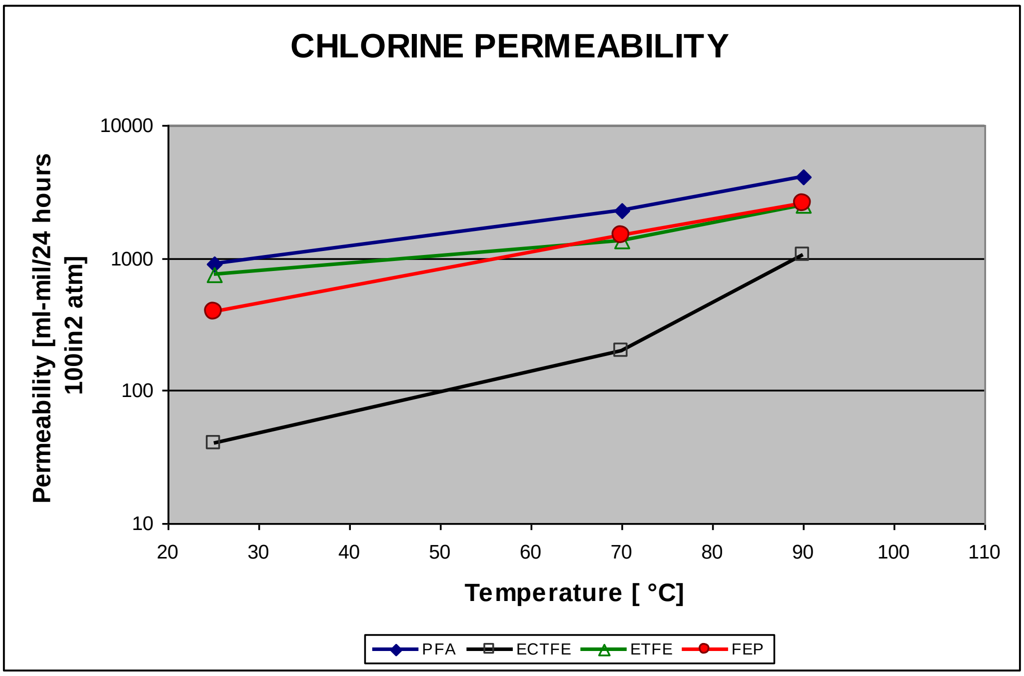

Figure 1 - **Fluoropolymer films - chlorine permeability.**

	FEP/FRP	ECTFE/FRP
Melting Point (0-0.5 mm layer)	260°C	229°C
Melting Point (0.5-1 mm layer)	261°C	240°C
Visual Evaluation	Blisters up to 5 mm in diameter, (0.5 - 0.8) mm of the liner depth	Whitening up to 0.5 mm of the liner depth
Total permeated chlorine through the liner	310 g/m^2	10 g/m^2
Bond strength between the liner and FRP after 2.5 years of exposure	Loss of adhesion	Not affected

Conditions: Wet chlorine gas; T=80-85°C, tested for 2.5 years; 1.5 mm thick liners of FEP & ECTFE

Table 7 - The Swedish Corrosion Institute testing - evaluation of dual laminate pipes after 2.5 years of exposure to wet chlorine

The permeation resistance depends on the temperature to which the polymer is exposed. For example, PVDF has excellent permeation resistance to small molecules like nitrogen, oxygen, or hydrogen. However, the permeation rate of chemicals through PVDF increases significantly at elevated temperatures (8).

Fluoropolymers applications

There are several application methods and fabrication techniques of fluoropolymers that can be used in corrosion protection applications. The choice of the fabrication technique depends on the particular application and sometimes on the preference of the final user. These techniques include:

- Dispersion coating & solution casting
- Powder coating & rotolining
- Thick sheet structures
- Lined vessels, rail cars, and iso-containers
  - Sheet lining
  - Dual laminates
  - Loose lining
- Piping
  - Dual laminate piping
  - Metal lined piping
  - Thick wall, unsupported piping
  - Dual containment piping
- Tower packing
- Heat exchangers
  - Pumps (diaphragm & ball valve), valves, and fittings
  - Solid parts – injection molded or machined from thick stock shapes
  - Lined parts – transfer molding & fabric backed
- Internals for reactors & scrubbers

The fluoropolymer applications include the semiconductor & electronic, oil & gas, chemical processing, automotive, wire & cable, architecture & building, aerospace, and pharmaceutical industries. This paper focuses on the typical CPI applications where fluoropolymers are used in the production processes or in the storage and transport equipment. These applications include:

- General Corrosion Protection Equipment
- Corrosion Protection Equipment in Specific Industries:
    - Chlor-Alkali
    - Pulp & Paper
    - Sodium Chlorate
    - Mining
    - Sulfuric Acid Manufacturing
    - Power Industry
    - Bio-Fuel
    - UV and Photovoltaic
    - Geothermal Energy
    - Water Treatment
    - Mining

Case Histories

The performance data helps when selecting the right material for the particular application. However, performance in real life applications is always the best test. This paper focuses on the ECTFE resin, the fluoropolymer which has been successfully used in industrial applications for more than 20 years. The initial characterization of the ECTFE polymer performed in the 70's, confirmed by the testing performed later on, showed that this resin has excellent chemical and permeation resistance.
When combined with good mechanical properties, low brittleness temperature, exceptional surface smoothness, low notch sensitivity, excellent impact strength and abrasion resistance, this resin was quickly identified as the material of choice for the chemical processing industry. Several large industrial applications of ECTFE resin date back to the early 80's.
The industrial applications include reactors, scrubbers, electrolytic cell liners, transport and storage tanks, tower packing, pipe, and fittings, all exposed to very aggressive chemicals.
During the past few years, we have been working with end users and fabricators in order to identify the case histories where the ECTFE resin was used in industrial applications. We identified more than 40 such applications, not including powder coating. This presentation includes a summary of the most interesting applications.

A. Summary of the ECTFE case histories

The Halar ECTFE case histories identified by us and described in this work should help to confirm what type of chemistries ECTFE can be successfully used in. Examples of successful ECTFE applications include:

1. Transport and storage of aggressive chemicals such as HF, H_2SO_4, HCl, sodium hypochlorite, chlorine, and herbicides & pesticides. The sulfuric acid applications include:
   - 11 sheet-lined vessels exposed to different concentrations of acid at temperatures as high as 135°C. Several of these tanks and trailers, installed from 1989-1992, are still in use.
   - More than 100 ECTFE/FRP dual laminate plating tanks were installed since 1993. The liners are exposed to 20% sulfuric acid @ 105°C and all tanks are still in good condition.
   - 98% H_2SO_4, semiconductor grade sheet-lined tank container; installed in 1990, still in satisfactory condition.

2. Chlorine Scrubbers:
   - 3 ECTFE/FRP scrubbers were installed in the UK in 1985 and 1988. They performed very well after 10-15 years, when they were inspected. However, we were not able to obtain any information about the current status of these applications.
   - The scrubber installed in the USA in 1984 is still in use, but we did not get permission to publish this case history.
   - Vessels installed in Belgium and the Netherlands will be further described in this paper. Both applications were already published in the March and October 2009 issues of the MP magazine.

3. Chlorine Dioxide Bleaching Towers – the first Halar lined structure was installed in 1998. It performed very well for 7.5 years until the plant was shut down. Four other towers are still in service; they were installed from 5 to 9 years ago and did not require any maintenance.

4. Piping, Electrolytic Cell Covers, and Reactors in the sodium chlorate and chlor-alkali manufacturing processes.

5. Dual Laminate Chimney Stack in Denmark. This application was published in the December 2009 issue of POWER Magazine; it is also summarized in this paper.

B. Chlorine Scrubber at Monsanto, Belgium

Summary of the application:

- ECTFE/FRP vessel was installed in Belgium in 1987
- Conditions: 22% NaOH to scrub chlorinated solvents, HCl, and molecular chlorine; gas stream velocity = 9000 m^3/hour; pH equal to or higher than 12, operating temperature = 40°C
- PVDF could not be used because of alkaline environment
- FRP structures were tried before installing ECTFE vessel, but they failed after a few years of service
- the ECTFE liner was in excellent condition after 14 years of continuous service when the plant was shut down

One of the reasons why the ECTFE resin was selected for this application was its excellent permeation resistance to chlorine and hydrochloric acid, the best of all fluoropolymers, at temperatures as high as 100°C. The two graphs below clearly show that the ECTFE resin has the lowest permeation rate at 45°C, slightly above the operating temperature in the Monsanto plant (6, 8).

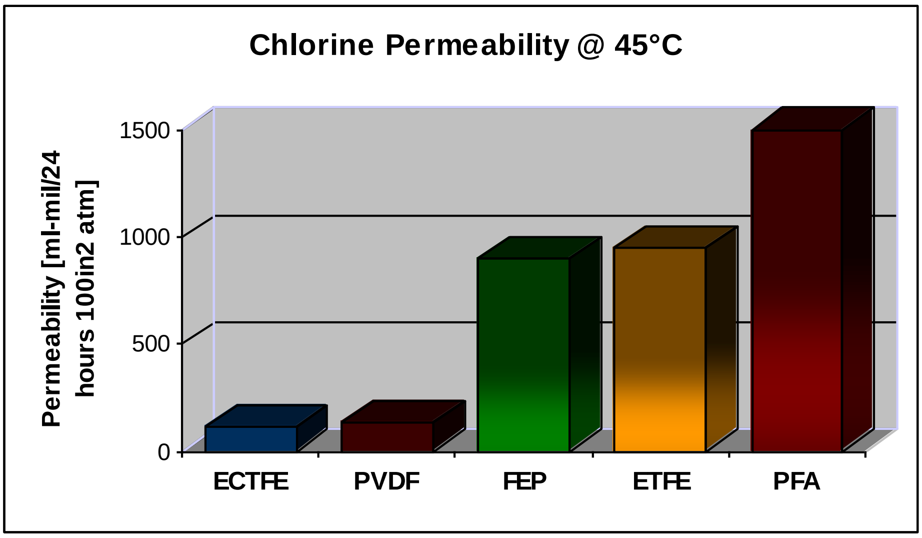

Figure 2 - Chlorine gas permeability of 10 mil thick films

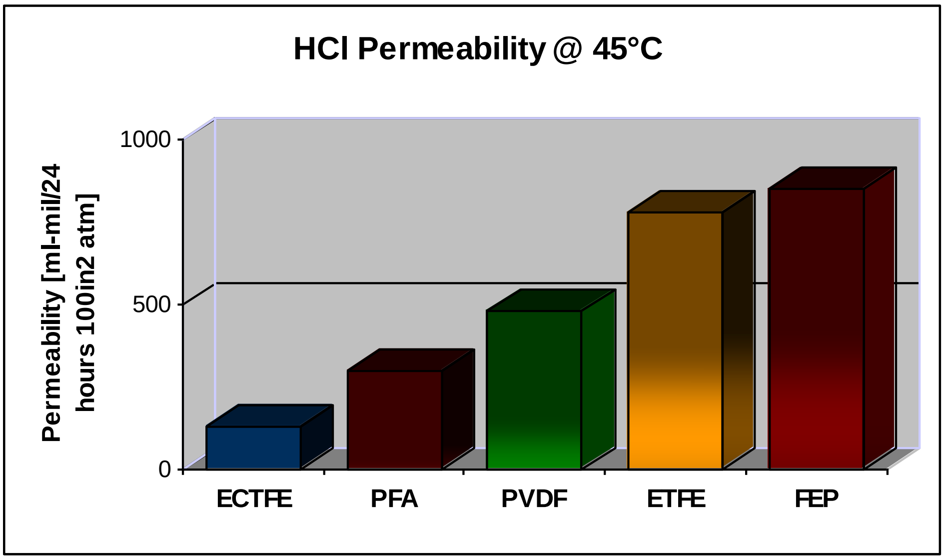

Figure 3 - HCl gas permeability of 10 mil thick films

Before installing the liner, Monsanto exposed individual ECTFE coupons and ECTFE blind flanges for 6 to 12 months in-situ. When no color change or any noticeable degradation was observed after this test period, Monsanto decided to go ahead with an ECTFE lined FRP scrubber. More details of this case history were published in the March 2009 issue of the MP magazine, the NACE publication (9).

C. ECTFE/FRP Chimney Stack in Denmark

The power station in Nordjutlandwerk was designed to burn crude refinery residue with high sulfur content. In 1991 it was outfitted with a 112 m high concrete chimney stack, internally protected with acid resistant brick. However, shortly after going on stream, it appeared that the condensate of sulfuric acid quickly attacked the inner brick lining. During 1995, the decision was made to replace the failing brick lining with the ECTFE/FRP structure. The 2.3 mm thick liner was used to build a 4.3 m diameter, 92 m high chimney stack, prefabricated in 7 modular sections (10). The fabrication was performed by Tunetanken (Denmark) with help from PRP, Finland.

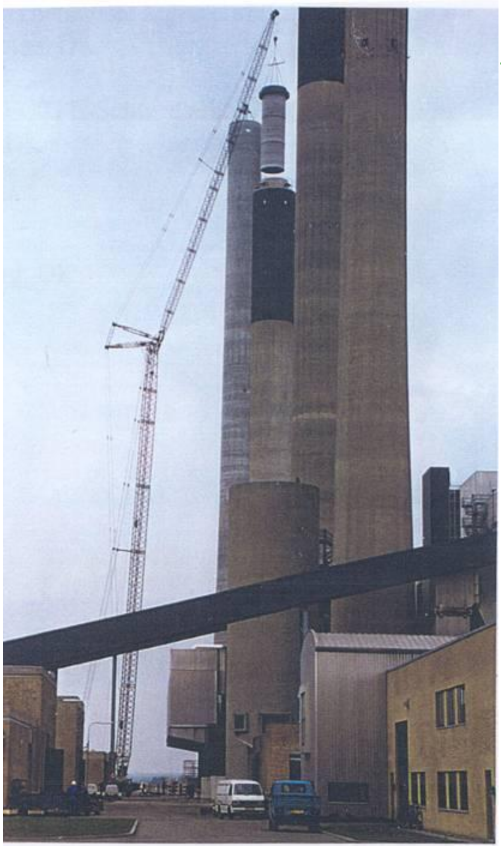

Figure 4 - Installation of the chimney in 1995

		Start-up	Max. Service
O_2	%	2 – 20	2 – 6
H_2O	%	0 – 3	6 – 9
SO_2	ppm	0 – 200	50 - 200
SO_3	ppm	0 – 40	5 – 15
No_x	ppm	0 – 100	20 - 50
HCl	ppm		max. 200
HF	ppm		max. 25
Temperature	°C	10 – 110	100 - 110
Temperature rise		0.7 °C/min. max.	
Temperature drop		0.7 °C/min. max.	
Fluegas flow	$Nm^3/min.$	$1 - 9.10^5$	$9 - 10.10^5$
Fluegas speed	m/s		28 max.
under pressure	Pa		175
over pressure	Pa		375

Table 9: Maximum Operating Conditions

The inspection performed by Tunetanken in September 2006 revealed that the ECTFE liner was in excellent condition and looked practically new.

Figure 5 – ECTFE liner after 10 years of service

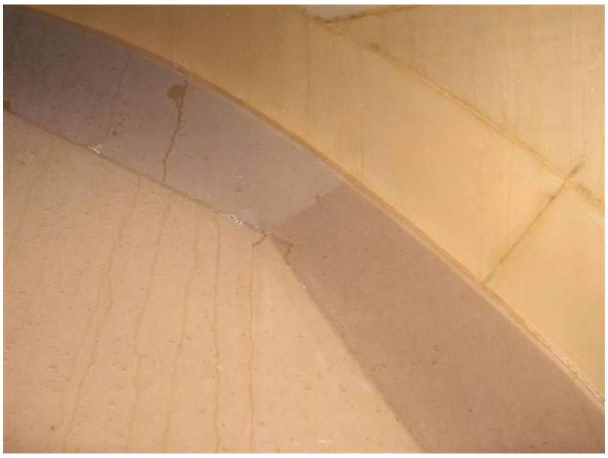

Figure 6 – ECTFE liner after 10 years of service

The liner was also evaluated by the Danish Technological Institute in 2008. The results of the DTI evaluation showed that the liner was in excellent condition after approximately 13 years of service. The conclusions of the Danish Technological Institute were summarized in the May 2009 letter from Mr. Torben Martens Knudsen, the Project Manager of DTI's Plastics Technology:

> "Samples taken from the ECTFE lining of the stack at Nordjyllandsvaerket Power Plant have been tested last year. Two drilled out samples were taken at level 24 and analyzed using IR-spectroscopy at test samples taken from inside surface and from the middle part of the lining. There were no sign of degradation of the polymer. Polished cross-sections of the samples were examined in microscope; a darker color was observed in a depth of 0.1 mm from inside surface. The joint between lining and glass fiber-polyester layer was fully intact. It was concluded that the ECTFE-lining was not influenced by the service conditions after approx. 13 years of service."

More details of this case history can be found in the article published in the December 2009 issue of POWER magazine (11).

D. Chlorine Dioxide Bleaching Towers

The first ECTFE/FRP upflow tube was installed in the Muskegon mill in May 1998. It replaced the FRP structure which failed after less than 2 years of service. The total installation cost including equipment, labor, and material was US$ 210,579,00. It was calculated that the return of investment was less than 2 months.

Figure 7 – Outside view of the Upflow Tube

The Muskegon mill was shut down in 2005, which allows us to take samples for analysis. We also took pictures of the liner after more than 7 years of service. The photographs showed that the majority of the liner was in excellent condition.

The tensile properties measurement and DSC analysis of the liner showed that the properties of the ECTFE resin were not affected after 7.5 years of chlorine dioxide service. It was also determined that the total permeation of the chemicals through the portion of the liner, taken from the bottom of the column (the most affected part due to the highest concentration of chlorine dioxide), was about 50% of the liner thickness. This suggests the ECTFE liner would perform very well for at least 7 more years.

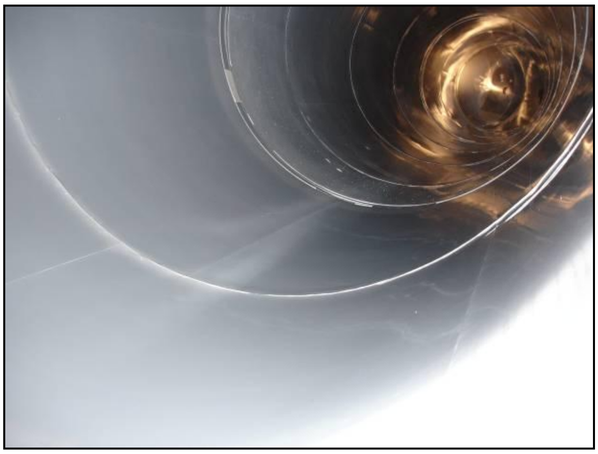

Figure 8- Inside view of the ECTFE liner

Figure 9 - Inside view of the ECTFE liner after 7.5 years of service (bottom)

The second Halar/FRP bleaching tower was installed in the International Paper mill in France in 2000. It was inspected in 2009 and based on the statement of the plant engineer, no damage, no wear, and no discoloration of the liner was observed.

There are three other ECTFE/FRP bleaching towers installed in the USA. All of them are in service for more than 5 years; they perform very well and did not require any repair or maintenance.

E. ECTFE LINED TANK IN CAUSTIC SERVICE IN NETHERLANDS

This ECTFE lined vessel was a large holding tank which received a continuous flow of alkaline based fluids in addition to active chlorine. The liner was also exposed to sodium hypochlorite, the product of the reaction between chlorine and caustic soda. The design temperature of the vessel was 70°C with operating temperature between 40°C and 50°C.

The 2 m diameter and 3.5 m long steel vessel was lined with the ECTFE by Mainchem, UK and it was put in service in the vinyl chloride monomer plant in Botlek, Netherlands in June 1983. This vessel was in continuous service until February 2007 when it was replaced by the FRP/ECTFE dual laminate tank. It was discovered during the inspection in 2005 that, probably due to mechanical abuse, the liner was damaged around the flange and partially disbonded from the metal. Back in 1983, the VCM plant was owned by AKZO Zoutechemie, but since 2000 it has been a part of Shin-Etsu Chemicals Co. Ltd.

We received samples of the ECTFE after the vessel was replaced by the new FRP/ECTFE tank. Several tests were performed on the liner to determine the extent of chemical attack. This data showed that the properties of the ECTFE resin were only slightly affected or not affected at all after 23 years of service. The resin retained most of its tensile properties, which is illustrated on the graph presented below.

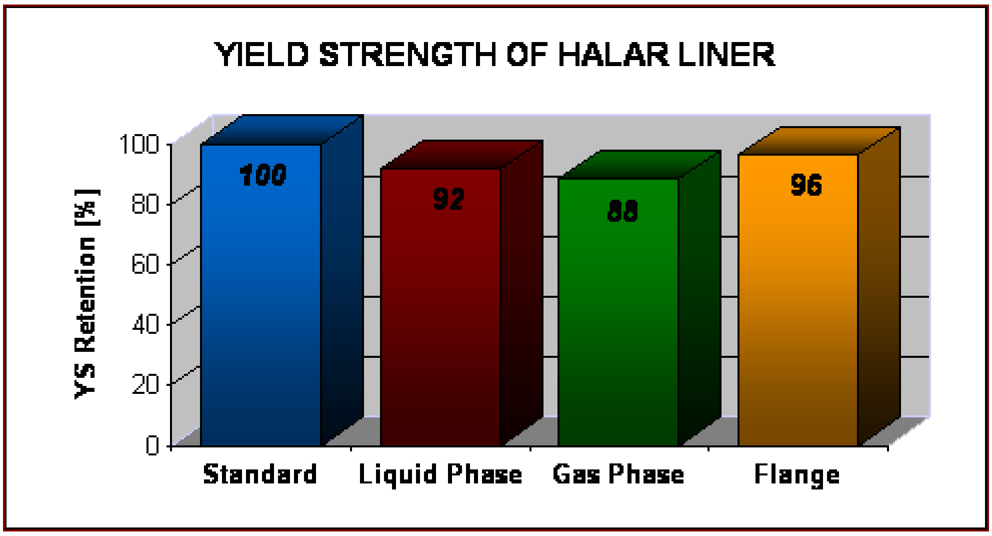

Figure 10 - Yield Strength of the liner after 23 years of service. The samples were taken from four different parts of the vessel

The cross section of the liner, as illustrated on the microscopic picture below, shows no signs of chemical attack, with the permeation front reaching a maximum of 10% of the liner thickness.

Figure 11 - The cross section of the liner after 23 years of service

This case history and more detailed results of the testing performed on the liner after 23 years of service was published in the October 2009 issue of the MP magazine (12).

F. OCEAN GOING TRANSPORT SHIPS

Steel and its alloys, based on their mechanical properties, are ideal materials of construction for large transport vessels. However, they have one major drawback which is well known - their limited chemical resistance. As a result, chemical shippers revert to suitable polymeric linings for transporting specialty chemicals. Seatrans, the Bergen, Norway based company, chose the ECTFE polymer to line large tanks to be installed on ocean-going chemical transport ships. As a result of the close collaboration between Seatrans and Ausimont, 636 chemicals, both organic and inorganic, were approved to be transported in the ECTFE lined vessels. These chemicals included: H_2SO_4 (up to 98%), HCl, NaOH, $AlCl_3$, and $FeCl_3$.

The first ship was equipped with two, 250 m^3 ECTFE lined tanks in 1991. Since then, Seatrans added three other ships, containing more than ten ECTFE lined tanks. These tanks have a total capacity of more than 6,000 m^3. The sheet lining of the tanks was done by PRP in Finland. All ships are sailing 7 days a week and the owner of the fleet is satisfied with the performance of the ECTFE resin. Inspection of the tanks on the ship built in 1993 was performed in 2005 and no change of the ECTFE liner was observed.

Figure 12 - Sheet lining of the tank, to be installed on the ship

Figure 13 - One of the ships containing the ECTFE lined tanks

Conclusions

Fluoropolymer has an excellent chemical resistance, very low permeability to liquid and gases, as well as very good strength and toughness at elevated temperatures. They have been widely used in chemical production, transport, and storage systems. Several case histories of the ECTFE polymer were identified during the last few years. These case histories, summarized above, show that the ECTFE pipes, liners and vessels performed very well in many industrial applications. It provided excellent corrosion protection from very aggressive chemicals for several years, and in many cases, for more than 20 years.

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