INTERIM REPORT ON
CHEMICAL AND THERMAL ANALYSIS

Prepared for: CAPP International Consortium

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SYNOPSIS

Work during the past six months has included significant research in several areas aimed at further clarification of the aging and chemical failure mechanism of thermoplastics (PVDF or Tefzel) for pipes. Among the areas investigated were the crystallinity changes associated with both the Coflon and Tefzel after various simulated environmental exposures using X-Ray diffraction analysis. We have found that significant changes in polymer crystallinity levels occur as a function of the exposures. These crystallinity changes may have important consequences on the fracture, fatigue, tensile, and chemical resistance of the materials. We have also noted changes in the molecular weight distribution of the Coflon material using a dual detector Gel Permeation Analysis. Again these changes may result in variation in the mechanical and chemical properties in the material. We conducted numerous analytical studies with methods including X-Ray Diffraction, Gel Permeation Chromatography, Fourier Transform Infrared Spectroscopy, Thermogravimetric Analysis, and Differential Scanning Calorimetry. We investigated a number of aged samples of both Tefzel and Coflon that were forwarded from MERL. Pressurized tests were performed in a modified Fluid G, which we will call G2. In this case the ethylene diamine concentration was increased to 3 percent in methanol. Coflon pipe sections and powdered Coflon were exposed in pressure cells at 1700 psi at three separate test temperatures, 70 °C, 110 °C, and 130 °C. The primary purpose of the pressure tests in Fluid G2 was to further elucidate the aging mechanism of PVDF degradation.
1.0 High Pressure Aging of Coflon

1.1 Test Apparatus

Tests were performed in a high pressure Atlas cells which was graciously loaned to TRI for the CAPP project by CONOCO. The configuration for chemical exposure cell is indicated in Figure 1. The Atlas cell symmetry axis is horizontal, and the end plates are operated on edge, sealed against the body of the cell with O-rings. For these tests each cell was half filled with the EDA/MeOH liquid and pressurized to 1700 psi with CO₂ gas. Provisions for heating and monitoring the temperature of the liquid are incorporated into the cell. The cell is also plumbed to allow the gas pressure to be monitored and adjusted as necessary. Pressurization of the test chambers was accomplished using a double-piston high pressure intensifier.

![Figure 1 - Atlas Pressure Test Cell](image)

1.2 Sample Preparations

Two types of samples were prepared for exposure in the Atlas pressure cell. Coflon pipe sections, and powdered Coflon. The following sections describe how the two types were prepared.

1.2.1 Pipe Sections

Pipe sections were cut in approximately 2 mm lengths. These thin sections were chosen so that maximum deplasticization and diffusion of the ethylenediamine/methanol mixture could occur during the seven day test. Two pipe section specimens each were included in the 110 °C and 130 °C experiments. One of the two ring specimens was cut perpendicular to the pipe axis in order to allow free contraction. The other specimen was left intact to observe constrained contraction after exposure. Figure 1.2 shows both the cut and un-cut versions before exposure.
1.2.2 Powdered Materials

Powdered Coflon material was also used in order to facilitate deplasticization and diffusion of fluids into the polymer during the pressurized tests. The pipe section rings, described in Section 1.2.1, were cut into smaller sections and placed into a liquid nitrogen freezer mill for ten minutes. This process was repeated until enough powder was collected to perform the aging experiments (~20 g).

1.3 Test Conditions

Three temperatures were used in the high pressure Atlas cell tests, 70°C, 110°C, and 130°C. The Atlas cells were partially assembled and the powdered Coflon and Coflon pipe sections were placed inside. The cell was then completely bolted together. The cell was then filled half-way with a mixture of 3% EDA/97% MeOH. After reaching the appropriate temperature, 70°C, 110°C, or 130°C, CO₂ was introduced into the cell and the pressure raised to 1700 psi (117 bar). The cell was insulated with fiberglass and left for seven days. At the end of the seven day exposure the cell was drained and opened. Fluid samples were retained for analyses. The powdered Coflon was rinsed with methanol and dried in an oven for one hour at 70°C. Figure 1-3 shows the Atlas test cell after 70°C test with coflon powder.
2.0 Chemical Analysis Assessment of Aging Effects

We have conducted a variety of chemical analysis to detect changes in Coflon and Tefzel when aged. We have detected significant changes in molecular weight and percentage crystallinity for the materials. Using infrared spectroscopy we have noted decreases in plasticizer and formation of double bond character in Coflon. We have derived an activation energy in association with the crystallinity change of Coflon.

2.1 Gel Permeation Chromatography of Aged Coflon

The samples were run on a Model 100 Viscometer and Viscotek LR40 Refractometer using one 15 µm and one 10 µm mixed bed gel American Polymer Standards GPC column. The solvent was NMP (No Lithium Bromide was Required) and the flow rate was 0.6 mL/minute. The injection volume was 100 µL. The Detector Temperature was set at 35 °C and the columns were run at 80°C (in order to reduce solvent viscosity and maintain good resolution). Three hours dissolution time was initially allowed with all of the samples.

A universal calibration detector system was used for the analysis. This consists of a refractometer, and viscometer combination. Concentration distribution was calculated through the refractive index signal, and the viscosity was calculated directly through the Differential Wheatstone Bridge Viscometer. The molecular weight is calculated via the Universal Calibration technique. This allows us to measure absolute molecular weight and molecular size independent of the polymer calibration type.

The universal calibration curve is given with the viscometer chromatogram overlaid in Figure 2-1. The maximum error based upon the standards is 8.0% and the average error is 4%
Signal to noise was excellent on the Viscometer. There was some linear drift present on the Refractometer. Despite this drift, the signal to noise was applicable for good Universal Calibration reproducibility and accuracy.

The moderate intrinsic viscosity values coupled with linear Mark-Houwink plots (Figure 2-2). (over most of the molecular weight range) present in these samples indicate that the samples have a general linear structure. (There is no evidence of any aggregated or crosslinked material in these samples that seemed to be present in the first set of samples that was run. However, samples T73, T7D70, and T7D110 had different Mark-Houwink plot than that of samples T68, T70, and T72. This indicates that there is either the presence of a backbone modification or short chain branching difference between these samples, or that there is the presence of a contaminant in one of the two sample lots as shown by the Mark-Houwink Plot

![Mark-Houwink Plot](image)

Figure 2-2. Mark-Houwink Plot.

Samples run in duplicate gave similar findings. As in the case of the previous samples, dissolution percentage varied between sample lots with the worst recovery (estimated between 40-50% from samples T68, T73, T7D70, and T7D110.

Overlaying these samples (Figure 2-3) with the past samples that were run, confirm no aggregation compared to but continuity of structure with samples with T73, T7D70, and T7D110. T68, T70, and T72 seem to be different in structure somehow (i.e.) less compact or lighter on the backbone.
2.2 X-Ray Diffraction - Crystallinity Changes

X-Ray Diffraction was conducted on both samples from the exposed Tefzel and Coflon samples. The exposure fluids for the environmentally aged samples are detailed below.

(1) Fluid A- 100% Methanol
(2) Fluid B- 97/3 CH4/CO2 with saturated water vapor
(3) Fluid C- 97/3 CH4/CO2
(4) Fluid D- 94/5/1 CH4/CO2/ H2S
(5) Fluid E- 94/5/1 CH4/CO2/H2S with saturated water vapor
(6) Fluid F- As fluid E plus 1% ethylene diamine
(7) Fluid G- As fluid A plus 1% ethylene diamine
(8) Fluid H- As Fluid B plus 1% ethylene diamine
Percent crystallinity determinations on 16 polymer samples of PVDF were conducted. The percent crystallinity is calculated as the integrated area of the crystalline portion of the X-Ray diffraction pattern, as determined after background removal, divided by the total integrated area of the patterns. Correction factors for air scattering are also applied.

Results:

The percent crystallinities are very close between many of these samples. The X-Ray diffraction patterns show that samples T68, T70 and T77 have almost identical X-Ray diffraction patterns, and resulting percent crystallinity calculations. Samples T74 and T75 have only slightly lower percent crystallinity. Data plots are included in Appendix one.

Table 2-2. percent crystallinity for Coflon and Tefzel samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>% Crystallinity</th>
<th>Exposure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control  Tefzel</td>
<td>26.0</td>
<td>Laboratory Ambient</td>
</tr>
<tr>
<td>Control Coflon</td>
<td>41.5</td>
<td>Laboratory Ambient</td>
</tr>
<tr>
<td>T-66 Coflon</td>
<td>25.5</td>
<td>Fluid A 120 °C 22 days</td>
</tr>
<tr>
<td>T-68 Coflon</td>
<td>29.1</td>
<td>Fluid A 120 °C 126 days</td>
</tr>
<tr>
<td>T-70 Coflon</td>
<td>28.8</td>
<td>Fluid A 100 °C 126 days</td>
</tr>
<tr>
<td>T-73 Coflon</td>
<td>43.0</td>
<td>Fluid F 100 °C 3 months</td>
</tr>
<tr>
<td>T-74 Coflon</td>
<td>25.0</td>
<td>Fluid F 120 °C 2 weeks</td>
</tr>
<tr>
<td>T-75 Coflon</td>
<td>25.8</td>
<td>Fluid F 140 °C 3 days</td>
</tr>
<tr>
<td>T-76 Coflon</td>
<td>27.2</td>
<td>Fluid F 140 °C 5 days</td>
</tr>
<tr>
<td>T-77 Coflon</td>
<td>29.3</td>
<td>Fluid F 140 °C 8 days</td>
</tr>
<tr>
<td>T-79 Tefzel</td>
<td>18.0</td>
<td>Fluid F 100 °C 1 month</td>
</tr>
<tr>
<td>T-84 Tefzel</td>
<td>20.2</td>
<td>Fluid F 140 °C 8 days</td>
</tr>
<tr>
<td>T-88 Coflon</td>
<td>42.0</td>
<td>Fluid F 120 °C 1 week</td>
</tr>
<tr>
<td>T-89 Coflon</td>
<td>41.0</td>
<td>Fluid F 120 °C 3 week</td>
</tr>
<tr>
<td>T-90 Coflon</td>
<td>39.0</td>
<td>Fluid F 140 °C 2 week</td>
</tr>
<tr>
<td>T-92 Coflon</td>
<td>32.0</td>
<td>Fluid G 120 °C 2 week</td>
</tr>
</tbody>
</table>

Also included are the percent (%) Crystallinity on three powdered Coflon samples. percent crystallinity (microdiffraction) on cross section of Coflon tube. Three samples are Coflon Baseline Powder (CBP) (unaged), CP70 (aged at 70°C) and CP110 (aged at 110°C), and CP130 (aged at 130°C).

Coflon Powder Samples

The results on four powder samples, three aged in the Atlas cell, are shown below in Table 2-3
Table 2-3. percent crystallinity results for Coflon powder samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>% Crystallinity</th>
<th>Exposure</th>
</tr>
</thead>
<tbody>
<tr>
<td>CBP</td>
<td>41%</td>
<td>Laboratory Ambient</td>
</tr>
<tr>
<td>CP70</td>
<td>37%</td>
<td>Fluid G2 70 °C 1 week</td>
</tr>
<tr>
<td>CPI10</td>
<td>32%</td>
<td>Fluid G2 110 °C 1 week</td>
</tr>
<tr>
<td>CPI30</td>
<td>18%</td>
<td>Fluid G2 130 °C 1 week</td>
</tr>
</tbody>
</table>

The results show a decrease in crystallinity after aging (See Appendix One (Exhibit 1 and 6)).

The changes in crystallinity were used to construct an Arrhenius relationship as displayed in Figure 2-4. An activation energy of 3 kcal/mole was obtained for the process. This is a rather low activation energy indicating that the energy needed to initiate the degradation reaction is relatively small. Likewise, within the range tested, the effect of temperature on acceleration of the process is also relatively small.

We attempted to model the data considering the decrease in crystallinity to be a first order reaction. The reaction rate was taken to have an Arrhenius temperature dependence. With the data available this led to three equations and three parameters, two of which were known previously. The three equations were solved for the third parameter leading to three fairly different values for the reference reaction rate. We concluded that the model considered did not constitute an adequate representation of the data. In order to further define the mechanism additional testing at other temperatures will be needed.

![Figure 2-4. Plot of Arrhenius relationship of aged Coflon powder.](attachment:image.png)
The most interesting result of this analysis was the fact that, in addition to crystallinity changes upon heating, a structural change is also observed upon heating. The raw data composite plot containing the baseline, 70 °C, and 110 °C Coflon powder in Appendix One, shows the peak shift occurring at the higher 2θ range (lower d-spacing). In Table 2-4 we see the d-spacings in this region increasing upon heating.

Table 2-4. Shifted Peak D-Spacing for Coflon powder samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>D-Spacing for Shifted Peak</th>
<th>Δd</th>
</tr>
</thead>
<tbody>
<tr>
<td>CBP</td>
<td>2.19A</td>
<td></td>
</tr>
<tr>
<td>CP70</td>
<td>2.20A</td>
<td>+0.01 A</td>
</tr>
<tr>
<td>CP110</td>
<td>2.25A</td>
<td>+0.05A</td>
</tr>
<tr>
<td>CP130</td>
<td>Peak Not Significant</td>
<td>+0.05A</td>
</tr>
</tbody>
</table>

In the 2θ range between 15° and 35°, the peak positions remain stationary (see exhibit 1). The 2θ range between 35° and 60°, however, shows significant peak shifts to lower d-spacings upon heating. The 2θ range below 5° shows a large peak developing upon heating to 110°C.

It should be noted that although the bulk of CP130 sample showed less crystallinity and a greater amorphous area, there were tiny individual crystallites identified in this sample (but not in the other samples). See data image in Appendix One for 130 °C aged Coflon powder.

The peak shifts indicate structural changes occurring upon heating. One theory for this is that the peaks between 15° and 35° (stationary peaks) represent regular inter-chain distances which do not change upon heating. In contrast, the region between 35° and 60° represents a secondary structure based upon the dipole moment (and arrangement) of the fluorine atoms. These bonds have a typical disassociation energy of between 1.5 to 3 kcal/mole. The shift to higher d-spacing appears to be related to an increase in chain length, (or increase in distance between chain folds in the structure of the polymer) caused from the relaxing of the interchain or intrachain structure, in a way similar to a compressed spring relaxing. Notice that the intensity of the peaks in this region also decrease, which also indicates a decrease in crystalline structure associated with these d-spacings. From the X-Ray diffraction pattern in the region between 35° and 65° 2θ, we see both a chain length increase from the peak position change and a decrease in crystalline structure from the decrease in peak intensity.

**Tube Cross Section**

X-Ray diffraction patterns were taken by stepping from the outer to the inner wall of the tube cross section. The tube cross section wall thickness is 8 mm. The percent crystallinity results are as follows.
The crystallinity is highest at the extreme outer wall. It falls off quickly and then increases slowly to the inner wall.

Again, we are seeing structural changes as well as crystallinity changes over this cross section (see Appendix One). The pattern taken at the extreme outer wall of the tube shows a completely different and more ordered structure, as seen by the crystalline peaks emerging between $35^\circ$ and $60^\circ 2\theta$. This increased order could be due to differences in the fluorine dipole moments and atomic arrangement as discussed in the section above. The amorphous scattering is also increasing as shown by the increasing background intensity.

This experiment represents a classic skin-core X-Ray diffraction result. It does suggest that the structural change is due to a difference in the cooling rate between the two walls.

### 2.3 Elemental Analysis

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%F</th>
<th>Exposure</th>
</tr>
</thead>
<tbody>
<tr>
<td>CBP</td>
<td>40.77</td>
<td>3.91</td>
<td>&lt;0.02</td>
<td>54.64</td>
<td>Ambient Baseline Powder</td>
</tr>
<tr>
<td>T-66</td>
<td>37.91</td>
<td>3.13</td>
<td>&lt;0.02</td>
<td>59.05</td>
<td>Fluid A 120 °C 22 days</td>
</tr>
<tr>
<td>T-68</td>
<td>38.01</td>
<td>3.09</td>
<td>0.08</td>
<td>56.88</td>
<td>Fluid A 120 °C 126 days</td>
</tr>
<tr>
<td>T-72</td>
<td>39.96</td>
<td>3.48</td>
<td>0.09</td>
<td>56.26</td>
<td>Fluid F 100 °C 1 month</td>
</tr>
<tr>
<td>T-76</td>
<td>40.06</td>
<td>3.65</td>
<td>0.19</td>
<td>54.20</td>
<td>Fluid F 140 °C 5 days</td>
</tr>
<tr>
<td>T-79</td>
<td>36.30</td>
<td>2.75</td>
<td>0.03</td>
<td>60.29</td>
<td>Fluid F 100 °C 1 month</td>
</tr>
<tr>
<td>T-84</td>
<td>36.52</td>
<td>2.80</td>
<td>&lt;0.02</td>
<td>60.55</td>
<td>Fluid F 140 °C 8 days</td>
</tr>
<tr>
<td>T-85</td>
<td>40.63</td>
<td>3.82</td>
<td>0.11</td>
<td>53.79</td>
<td>Unaged</td>
</tr>
<tr>
<td>T-86</td>
<td>40.49</td>
<td>3.75</td>
<td>0.22</td>
<td>55.45</td>
<td>Unaged extruded bar</td>
</tr>
<tr>
<td>T-91</td>
<td>38.04</td>
<td>3.24</td>
<td>0.15</td>
<td>58.62</td>
<td>Fluid F 140 °C 5ksi 4 weeks</td>
</tr>
<tr>
<td>T-92</td>
<td>38.31</td>
<td>3.35</td>
<td>0.32</td>
<td>58.12</td>
<td>Fluid G 120 °C 5ksi 2 weeks</td>
</tr>
<tr>
<td>CP70</td>
<td>37.23</td>
<td>3.10</td>
<td>0.08</td>
<td>58.62</td>
<td>Fluid G2 70 °C 1 week</td>
</tr>
<tr>
<td>CP110</td>
<td>37.72</td>
<td>3.04</td>
<td>0.07</td>
<td>59.61</td>
<td>Fluid G2 110 °C 1 week</td>
</tr>
<tr>
<td>CP130</td>
<td>49.72</td>
<td>4.30</td>
<td>7.29</td>
<td>36.98</td>
<td>Fluid G2 130 °C 1 week</td>
</tr>
</tbody>
</table>

Two samples, T79 and T92 showed (initial results) total percentages (CHNF) of <95%. Carbon results were confirmed in reanalysis. Repeating fluorine using a smaller sample size resulted in a correspondingly greater value such that total percentages showed >99%. Fluorine recovery tended to be more difficult in the analysis. There are several samples (T68, T76, T85) whose total percentage values are <99%. The fluorine value is slightly low due to some
incomplete recovery of fluorine. Combustion difficulties also can result in slightly excessive nitrogen. Values greater than 0.1 are suspect. Reanalysis of samples whose initial nitrogen is >0.2 usually resulted in a lower value. We have not reanalyzed T76 and T86. The nitrogen value for T92 was the only one which consistently showed >0.3%.

2.4 FTIR and Gas Chromatography/Mass Spectroscopy

We investigated accelerated high temperature aging of the Coflon material and analyzed the liquid exudate using gas chromatography coupled with mass spectrometry. The gas chromatography/mass spectrum of the liquid is provided in Appendix 5. The database search in many instances provided the best match with a dibutyl ester of decanedioic acid. The search routine can only match those compounds catalogued in the library that is approximately seven hundred thousand compounds. The tests were conducted on liquids (methanol) after Atlas cell aging.

We have conducted FTIR analyses of Coflon and Tefzel test pieces after aging for selected times, temperature, and exposure conditions. We are interested in the chemical functionalities or changes the samples exhibited upon aging. We were also interested whether the recrystallized PVDF deposits exhibited a change from the α or the type 2 form to the β or type 1 crystalline form when the PVDF dissolved under strain. The FTIR results are contained in Appendix 5. One factor that was evident was a band that appeared at about 1650 cm⁻¹ and can be indicative of double bond formation. We have enclosed FTIR result on samples that were not included in the last interim report. Dr. Baye and Dr. Casisdy also conducted infrared testing included in this report.

3.0 Thermal Stability and Compositional Analysis

We have conducted thermal studies to examine the migration of residual volatile material from aged Coflon test pieces. This includes aged Coflon pipe sections, power, and specimens aged at MERL. After aging we would expect some if not all the plasticizer to be removed or displaced by other components in the case of liquid exposure and to simply diffuse out of the polymer in gaseous environments. The following information describes how some of these processes occur in plasticized PVDF.

3.1 Analyses of Aged Coflon Powder.

Several aspects of the aged powdered Coflon material were analyzed. First, the residual volatile content, including plasticizer, is shown in Table 3.1. The volatile content decreased upon aging at 70°C and 110°C. However, at 130°C the residual content increases significantly. This may be due to the presence of low molecular weigh products of the degradation reaction. In addition, we see from the DSC results, also in Table 3-1, that the number of components appearing after aging is increasing with exposure temperature.
Table 3-1. Aged Powdered Coflon Pipe Results

<table>
<thead>
<tr>
<th>Sample</th>
<th>Exposure</th>
<th>Residual Volatiles Including Plasticizer (%)</th>
<th>Weight Retention at 500 °C (%)</th>
<th>Weight Retention at 600 °C (%)</th>
<th>Melt Onset °C</th>
<th>Melt Peak °C</th>
<th>Heat of Fusion (J/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Baseline Coflon Powder</td>
<td>None</td>
<td>10.97</td>
<td>41</td>
<td>30</td>
<td>144</td>
<td>168</td>
<td>51</td>
</tr>
<tr>
<td>CP70</td>
<td>EDA/Methanol 7d at 70°C</td>
<td>3.52</td>
<td>52</td>
<td>34</td>
<td>162</td>
<td>172</td>
<td>77</td>
</tr>
<tr>
<td>CP110</td>
<td>EDA/Methanol 7d at 110°C</td>
<td>1.86</td>
<td>44</td>
<td>33</td>
<td>159</td>
<td>171</td>
<td>56</td>
</tr>
<tr>
<td>CP130</td>
<td>EDA/Methanol 7d at 130°C</td>
<td>9.13</td>
<td>55</td>
<td>50</td>
<td>51</td>
<td>86</td>
<td>224</td>
</tr>
</tbody>
</table>

Another outstanding feature is the weight retention at 500°C which remains fairly constant at around 33% except for the 130°C exposure where an increase to 50% was observed. This appears to be a verification that some degree of crosslinking is occurring resulting in a more stable char.

3.2 Analyses of Aged Coflon Pipe Sections

Again with the 130 °C aged Coflon pipe sections the weight retention at 600°C is greater than observed at the lower exposure temperatures. As shown in Table 3-2 we also detected, as with the powder, an increase in the number of endothermic events in the DSC data indicating conversion to a multi-component material.

Table 3-2 - Aged Coflon Pipe Section Results

<table>
<thead>
<tr>
<th>Sample</th>
<th>Exposure</th>
<th>Residual Volatiles Including Plasticizer (%)</th>
<th>Weight Retention at 500 °C (%)</th>
<th>Weight Retention at 600 °C (%)</th>
<th>Melt Onset °C</th>
<th>Melt Peak °C</th>
<th>Heat of Fusion (J/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Baseline</td>
<td>None</td>
<td>11</td>
<td>41</td>
<td>33</td>
<td>159</td>
<td>170</td>
<td>49 - 67</td>
</tr>
<tr>
<td>PS110</td>
<td>7d 110°C 3%</td>
<td>3</td>
<td>53</td>
<td>35</td>
<td>161</td>
<td>166</td>
<td>100</td>
</tr>
<tr>
<td></td>
<td>EDA/Methanol</td>
<td>169</td>
<td>172</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PS130</td>
<td>7d 130°C 3%</td>
<td>7</td>
<td>48</td>
<td>44</td>
<td>88</td>
<td>134</td>
<td>318</td>
</tr>
<tr>
<td></td>
<td>EDA/Methanol</td>
<td>192</td>
<td>195</td>
<td>2.5</td>
<td></td>
<td></td>
<td>2.7</td>
</tr>
</tbody>
</table>

3.3 Compositional Profile of Aged Coflon Permeation Specimens

In order to determine how high pressure permeation affects the composition of Coflon pipe, thermogravimetric analysis (TGA) was performed. This analysis provides information regarding how plasticizer and other volatile materials migrate through the pipe wall. High
pressure permeation testing was performed at MERL using 8 mm thick sections cut from Coflon pipe. After permeation testing the specimens were sent to TRI for analysis. An example of this behavior is shown in Table 3-3. Shown here are the results obtained from the high and low pressure side of a test specimen that was exposed to Fluid F for 4 weeks at 120 °C and 5 ksi. Note that the high pressure side contains less residual volatile material than the low pressure side.

Table 3-3. TGA test result for aged Coflon samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Exposure</th>
<th>Residual Volatiles Including Plasticizer (%)</th>
<th>Weight Retention at 500 °C (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T-51</td>
<td>Fluid F 120°C 5 ksi 4 weeks</td>
<td>8</td>
<td>37</td>
</tr>
<tr>
<td>T-51</td>
<td>Fluid F 120°C 5 ksi 4 weeks</td>
<td>12</td>
<td>N/A</td>
</tr>
<tr>
<td>T-74</td>
<td>Fluid F 120°C 5 ksi 2 weeks</td>
<td>7</td>
<td>35</td>
</tr>
<tr>
<td>T-75</td>
<td>Fluid F 140°C 5 ksi 3 days</td>
<td>8</td>
<td>36</td>
</tr>
<tr>
<td>T-76</td>
<td>Fluid F 140°C 5 ksi 5 days</td>
<td>9</td>
<td>36</td>
</tr>
<tr>
<td>T-77</td>
<td>Fluid F 140°C 5 ksi 8 days</td>
<td>8</td>
<td>37</td>
</tr>
</tbody>
</table>

3.3.1 Long-Term Coflon Methane Permeation Test Specimen

Thermogravimetric analysis was performed on a Coflon specimen, T98 (MERL C22), after long term methane permeation testing at 140°C and 5000 psi. This specimen was not exposed to any fluid so that all residual volatile material within the sample should be plasticizer. Sections one mm in thickness were cut from the surface of the high and low pressure sides of the specimen. These were then placed in the TGA sample compartment where a nitrogen atmosphere was maintained while ramping the temperature 10°C per minute.

The results of the TGA indicated that the high pressure side had a residual plasticizer content of 1.9 percent. In comparison the low pressure side had a plasticizer content of 0.95 percent. This may be due to some molecular "packing" phenomenon occurring on the high pressure side.

3.3.2 Long Term Coflon Methane/Methanol Permeation Test Specimen

An additional thermogravimetric analysis was performed on a Coflon specimen which was exposed to methane permeation testing at 140°C and 5000 psi. In this case the high pressure side was covered with methanol to investigate any synergy between methane and methanol. Figure 3-1 shows the cross sectional profile of the specimen. Eight 1 mm thick sections were cut from the specimen. These sections were cut from the surface of the high and low pressure sides of the specimen. These were then placed in the TGA sample compartment where a nitrogen atmosphere was maintained while ramping the temperature 10°C per minute. This allows the determination of a residual plasticizer profile through the wall thickness of the Coflon pipe permeation specimen. The onset temperature observed indicates that the residue coming off is primarily plasticizer and not methanol.
As shown in Figure 3-1, the highest residual plasticizer content was again found on the high pressure side. A transition point appears to occur midway through the specimen. For comparison a similar crossectioning for determination of plasticizer concentration profile was performed on unaged Coflon pipe. The results of this test indicated that the plasticizer concentration is very consistent through the wall thickness of the pipe. Differential Scanning Calorimetry (DSC) tests were also performed at the same wall depth locations. The DSC and TGA test results are summarized in Table 3-4.
Table 3-4. DSC and TGA test results from unaged Coflon Pipe.

<table>
<thead>
<tr>
<th></th>
<th>Melt Onset (°C)</th>
<th>Melt Peak (°C)</th>
<th>Heat of Fusion (J/g)</th>
<th>Residual Plasticizer (%)</th>
<th>Crystallinity (%)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 mm From I.D.</td>
<td>161</td>
<td>170</td>
<td>49.8</td>
<td>11.98</td>
<td>18</td>
</tr>
<tr>
<td>Interior of Pipe Wall</td>
<td>163</td>
<td>170</td>
<td>55.2</td>
<td>10.98</td>
<td>11</td>
</tr>
<tr>
<td>1 mm From OD</td>
<td>159</td>
<td>168</td>
<td>67.1</td>
<td>10.36</td>
<td>37</td>
</tr>
<tr>
<td>Average</td>
<td>161</td>
<td>169</td>
<td>57</td>
<td>11</td>
<td>22</td>
</tr>
</tbody>
</table>

* performed using X-Ray crystallography

As would be expected the heat of fusion and crystallinity appear to correlate. The highest crystallinity and heat of fusion were found on the O.D. of the pipe. The plasticizer concentration is relatively constant through the thickness of the unaged pipe but there does seem to be a trend toward higher concentrations near the I.D.

3.4 Residual Strain Before and After Aging Coflon Pipe Sections

Additional pipe sections were cut and placed into the high pressure test chambers along with the powdered Coflon for the 110°C and 130°C tests. Three sections were placed in the pressure cell during each experiment. Additional specimens were exposed to 110°C at atmospheric pressure with no fluid for comparison. After the one week aging cycles the sections were removed from the test cells and measured to determine shrinkage effects caused by exposure to the high temperature and pressure in the EDA/methanol mixture. Dimensional changes were primarily obtained for the 110°C test. Sufficient pipe section material was not available for the 70°C test. Pipe sections were placed in the 130°C test but were deformed and degraded so severely that only a few measurements were possible.

3.4.1 Residual Strain in Pre-Cut Pipe Sections

Two of the three specimens that were placed in the cells were cut perpendicular to the axis of the pipe prior to the test to allow for free contraction of the specimen. Immediately after cutting, the specimens contracted circumferentially approximately three percent corresponding to a residual hoop stress of 4500 psi. This residual stress is primarily due to thermal drawing history of the pipe during the extrusion process. Knowing the residual hoop stress we can calculate the internal pipe pressure needed to compensate for the residual stress by:

\[ q = \frac{qt}{r} \]

where:

- \( r \) = radius
- \( t \) = wall thickness
q = internal pressure

After exposure, the additional contraction of the cut sections was measured and found to have a total circumferential shrinkage of 17 percent for the specimens exposed to the EDA/methanol fluid at 110°C. Similarly an identical pre-cut specimen that was simply deplasticized for one week at 110°C had a circumferential shrinkage of 15 percent. It was also interesting to note that this specimen shrank 9.2% during the first ten minutes of the oven exposure indicating that most of the dimensional change is a result of thermal processing history. One dimension was measured for the specimen that was exposed to the EDA/methanol fluid at 130°C. The wall thickness was found to shrink by 7.2% after the seven day exposure. This was significantly higher than any of the other specimens tested. Table 3-5 summarizes the dimensional changes occurring in the various pipe sections tested.

<table>
<thead>
<tr>
<th>Wall Shrinkage (%)</th>
<th>Length Shrinkage (%)</th>
<th>Initial Circumferential Shrinkage Prior To Exposure (%)</th>
<th>Circumferential Shrinkage After Aging (%)</th>
<th>Weight Loss (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aged 7d 110°C</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3% EDA/Methanol; Pre-Cut</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Deplasticized 7d 110°C Pre-Cut</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Aged 7d 130°C</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3% EDA/Methanol</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Wall Shrinkage (%)</td>
<td>Length Shrinkage (%)</td>
<td>Circumferential Shrinkage After Aging (%)</td>
<td>Weight Loss (%)</td>
<td></td>
</tr>
<tr>
<td>Aged 7d 110°C</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3% EDA/Methanol; Cut After Exposure</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Deplasticized 7d 110°C Cut After Exposure</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

3.4.2 Residual Strains in Pipe Sections Cut After Exposures

The Coflon pipe specimens that were not cut perpendicular to the axis of the pipe were exposed to either an elevated temperature fluid or air atmosphere as described in Table 3-5. These specimens were intact and not as free to contract compared to the pre-cut specimens. Because of this constraint, we observed a significant decrease in the amount of circumferential shrinkage after aging (~1-3%). After making the dimensional measurements subsequent to aging, the specimens were then cut and measured. In this case the fluid exposed specimen had a
much smaller residual strain compared to the specimens conditioned in air at 110°C, (2.6% vs. 10.7% respectively). Figures 3-3(a-c) show the severe cracking observed in the 130 °C aged Coflon pipe sections. Figure 3-3 (c) is a comparison of unaged and aged Coflon.

Figure 3-3 (a-c). Cross section of 130 °C aged Coflon pipe section.

4.0 Degradation Kinetics and Mechanisms

Both old and new PVDF pipe were dissolved in NMP and films were cast on glass plates. After drying, small (1"x1") film sections were used for aging studies. In a typical experiment, a 100 mL, three neck, round-bottom flask was fitted with reflux condenser and argon inlet. The flask was charged with methanol (50 mL) and ethylenediamine (0.5 mL), and the film was added
much smaller residual strain compared to the specimens conditioned in air at 110°C, (2.6% vs. 10.7% respectively). Figures 3-3(a-c) show the severe cracking observed in the 130 °C aged Coflon pipe sections. Figure 3-3(c) is a comparison of unaged and aged Coflon.

Figure 3-3(a-c). Cross section of 130 °C aged Coflon pipe section.

4.0 Degradation Kinetics and Mechanisms

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to this mixture and heated to reflux. After a specified time interval the film was removed, washed with methanol, dried and analyzed by infrared spectroscopy.

PVDF films were aged under several different conditions and the results are summarized in Table 4-1. All of the films were discolored by the aging process, and films subjected to prolonged aging acquired a dark color and became brittle.

The infrared spectra of old PVDF films showed that peaks at 3370, 2149 and 1642 cm⁻¹ appeared after aging. A comparison of the infrared spectra of new PVDF film before and after aging showed that the absorption at 1642 cm⁻¹ moved to 1599 cm⁻¹. This may be due to reaction of the plasticizer.

Chemical analysis of aged old PVDF films indicated that composition is dependent on aging conditions (Table 4-1). In run 1 (10% ED, reflux, 24h), the film suffered a drastic reduction in percent fluorine (from 59.38% F in PVDF to 16.19%) accompanied by an increase in C, H, and N percentage. They also became extremely dark (black). Run 3 (1% ED, reflux, 16h) also resulted in some decomposition (lowered %F and raised values for % C, H and N). However, runs 2 and 4 (much milder conditions) left the PVDF analyses unchanged.

**Future Plans:**

1. The PVDF powder will be extracted with methanol, ethyl acetate, THF or 10% NaHCO₃ and aging studies will be repeated.
2. These films will be analyzed by infrared and NMR.

### Table 4-1. Chemical analysis of aged PVDF films.

<table>
<thead>
<tr>
<th>Run No</th>
<th>Solvent</th>
<th>Temp. (°C)</th>
<th>Time (hr.)</th>
<th>IR (cm⁻¹)</th>
<th>%C</th>
<th>%H</th>
<th>%N</th>
<th>%F</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>MeOH + 10% EDA</td>
<td>Reflex</td>
<td>24.0</td>
<td>3372(b), 2945(s), 2149(w), 1642(b)</td>
<td>56.34</td>
<td>4.27</td>
<td>7.16</td>
<td>16.19</td>
</tr>
<tr>
<td>2</td>
<td>MeOH + 1% EDA</td>
<td>Reflex</td>
<td>1.5</td>
<td>3378(b), 3021(s), 2979(s), 1642(w), 1407(s), 1208(w), 1027(b), 882(s)</td>
<td>37.79</td>
<td>2.90</td>
<td>0.04</td>
<td>59.58</td>
</tr>
<tr>
<td>3</td>
<td>MeOH + 1% EDA</td>
<td>Reflex</td>
<td>16.0</td>
<td>3384(b), 3021(s), 2979(s), 1601(b), 1453(s), 1410(b), 1264(b), 878(s), 837(s)</td>
<td>42.81</td>
<td>3.17</td>
<td>1.03</td>
<td>51.34</td>
</tr>
<tr>
<td>4</td>
<td>MeOH + 1% EDA</td>
<td>Room</td>
<td>16.0</td>
<td>3380(b), 3021(s), 2979(s), 1452(s), 1388(b), 1214(b), 1027(b), 886(s), 837(s)</td>
<td>37.86</td>
<td>2.87</td>
<td>0.13</td>
<td>59.60</td>
</tr>
</tbody>
</table>

### Table 4-1 (alternative format).

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Temp. (°C)</th>
<th>Time (hr.)</th>
<th>IR (cm⁻¹)</th>
<th>Elemental Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>MeOH + 1% EDA</td>
<td>Reflex</td>
<td>22.0</td>
<td>2945(b), 1599(s), 1400(s), 1182(b), 879(s), 838(s)</td>
<td>NA</td>
</tr>
<tr>
<td>MeOH + 1% EDA</td>
<td>Reflex</td>
<td>15.0</td>
<td>Almost Same as Above</td>
<td>NA</td>
</tr>
<tr>
<td>MeOH + 1% EDA</td>
<td>Room</td>
<td>22.0</td>
<td>Almost Same as Above</td>
<td>NA</td>
</tr>
</tbody>
</table>

19
5.0 Literature Search

A literature search was conducted using NERAC on flexible PVDF pipes. Below are the results of this search.

1. FLEXIBLE METAL PIPE LINED WITH >POLYVINYLIDENE< >FLUORIDE< AND HAVING HIGH BENDABILITY
   CAS SECTION- 156 CAS SUBSECTION- BBE - CAS 121-11121-261450 CA 121:261450
   NDN- 010-0572-4587-3
   NO-AUTHOR
   JPN. KOKAI TOKKYO KOHO, AUG 02 1994, 4 PP. PATENT NUMBER- 94213377
   PATENT APPLICATION NUMBER- 93-249954 GATE FILED- SEP 10 1993 DOCUMENT
   TYPE- PATENT CDDEN- JKXXAF LOCATION OF WORK- FR. PATENT CLASS-
   F16LB111400BA; B32B0150800BB; F16L0111000B, B29D0231800- PATENT ASSIGNEE(S)
   - ELF ATOCHEM S. A. SUBFILE CODE- APP PATENT APPLICATION PRIORITY-
   920910FR92-10010 ORIGINAL PATENT APPLICATION COUNTRY- JP LANGUAGE-
   JAPANESE NO-ABSTRACT

   Chemical Engineering Abstracts (CEA)

2. Surface treatment of chemical equipment with fluorinated plastics. - CEA 87-ORB
   CEB1802006024 NDN- 141-0009-3451-3
   Mieschke, W.

   ABBREVIATED JOURNAL TITLE- Chemie-Technik VOL. 15 NO. 11 1986 PP. 33-
   34,36,39 DOCUMENT TYPE- Journal ISSN- 0340-9961 CODEN- CMTKAT
   LANGUAGE OF ABSTRACT- English ORIGINAL LANGUAGE- German

   The >lining< of steel >pipes< and reactors with fluorinated plastics considerably reduces
   corrosion problems in chemical environments., The range of fluorinated plastics now available
   enables selection of material to suit particular process conditions., The most commonly
   encountered fluoropolymer is PTFE, which has exceptional chemical resistance., Other
   compositions now available include >PVDF<, FEP, and TFA.

3. Choosing fluoropolymers for >lining< >pipes< - CEA 85-00 CEB1612015743 NDN-
   141-0006-6025-5

   anonym
Fuer die Auskleidung von Stahlrohren stehen eine Reihe von Fluorpolymeren (PTFE, FEP, PFA, ETFE, ECTFE, >PVDF<) zur Verfügung. Dem Artikel ist zu entnehmen, welcher Kunststoff unter den Aspekten Chemikalienbeständigkeit, Temperaturbeständigkeit, Verarbeitbarkeit und mechanische Eigenschaften zu bevorzugen ist. (Materne)

Engineering Index

4. Thermal insulation for >flexible< >pipe< development, and testing - EIX
94-27 EIX94271289170 NDN- 017-0204-6436-1

Kalman, M.D.; Alexander, H L.; Belcher, J.R.; Loper, C.W.


DOCUMENT TYPE- CA, Conference Artie MONOGRAPH TITLE- American Society of Mechanical Engineers, Petroleum Division (Publication) PD ISBN- 0-7318-0946-3 CODEN-ASMPEX AUTHOR AFFILIATION- Wellstream Corporation, Panama City, FL, USA SPONSDR- ASME

CONFERENCE DATE- 19930131-19930204 CONFERENCE TITLE- Proceedings of the 16th Annual Energy - Sources Technology Conference and Exhibition CONFERENCE LOCATION- Houston, TX, USA CONFERENCE CODE NO. - 19938 LANGUAGE- English

This paper presents the method of calculating non-bonded >flexible< >pipe< thermal resistance, the results of efforts to develop suitable insulation materials to increase the thermal resistances of non-bonded >flexible< >pipe< and the results of tests to verify the thermal resistance of an insulated >flexible< >pipe< section. The thermal resistance of a pipe (expressed in units of m degree C/M) is a function of the >flexible< >pipe< layer materials thermal conductivities and thicknesses. The effects on thermal resistance of flooding the >flexible< >pipe< annuls with seawater resulting from outer sheath damage and burial of the pipe in soil are also considered.

Materials that were investigated include the thermoplastics normally used for >Flexible< >pipe< (HDPE), Nylon, >PVDF<), other solid thermoplastics (PVC, PP), and syntactic foams. The purpose of the investigations was to verify the thermal resistance of the conventional >flexible< >pipe< thermoplastics and develop a suitable material for providing additional thermal insulation that would be economical, manufacturable, chemically and thermally stable, and withstand the long term effects of hydrostatic pressure and moisture absorption at design water depth without significantly affecting the thermal performance of the material. The results of tests conducted to verify the thermal conductivity of the conventional >flexible< >pipe< materials and the materials being considered For thermal insulation are presented. Tests were conducted to verify the thermal resistance of a >flexible< >pipe< section by maintaining a constant temperature differential between the inside and outside of the >flexible< >pipe< and measuring the heat input
The increasing development of marginal offshore hydrocarbon deposits has resulted in production of increasingly corrosive fluids. This has increased the need for pipelines capable of operating at elevated temperatures in the presence of high concentrations of H\textsubscript{2}S and COS\textsubscript{2} gases. Conventional pipelines requires the use of stainless steels or corrosion resistant alloys which drives up the cost of materials and fabrication/installation of the system. The use of flexible pipe systems is assuming an important role in these applications where stainless steel and CRA carcass materials may be economically utilized. This paper discusses testing of flexible pipe carcass materials and a new PVOF homopolymer fluid barrier material.

For estimating the service performance of polyethylene (PE), polypropylene (PP), polyvinylchloride (PVC) and polyvinylidenfluoride (PVDF) as lining materials for pipes and tanks for corrosive chemicals, the test specimens cited in Table 1 have been stored in contact with different oxidizing acids. The period of storage was up to 24 months, the temperature of the chemicals has been adjusted to 23 or 40 degree C. 30% chromic acid, 80% sulphuric acid and 65% nitric acid have been chosen as oxidizing acids. All chemicals were of analytical grade for analysis. During the period of storage, the test specimens did not come into contact with metals. At the end of storage in chemicals, the molecular weight distributions have been determined by
gel permeation chromatography. In this connection, the test conditions indicated in Table 2 have been maintained. (Author abstract) 3 Refs. In German, English.

7. QUALITY IMPROVEMENTS, INNOVATIONS, AND SUCCESSFUL APPLICATIONS OF >PVDF< (PVFSUB2) LINED SYSTEMS. - EIX 85-04 EIX85040050387 NDN- 017-0044-1560-0

Werthmueller, Ernst

Publ by NACE, Houston, TX, USA p 89-97 1983 DOCUMENT TYPE- CA, Conference Paper AUTHOR AFFILIATION- Symalit Co, Lenzburg, Switz SPONSOR- AICHe, New York, NY, USA; NACE, Houston, TX, USA; Soc of Plastics Engineers, Brookfield, CT, USA, SPI, New York, NY, USA; SPI of Canada, Don Mills, Ont, Can; TAPPI, Atlanta, GA, USA CONFERENCE TITLE- Managing Corrosion With Plastics, Volume V. CONFERENCE CODE NO. - 04932 LANGUAGE- English

>Polyvinylidene< >fluoride< (>PVDF<) is a material distinguished by the tested, excellent corrosion and temperature resistance of a fluorine resin, without losing the technological advantages of a thermoplastic. The thermoplastic properties as well as the outstanding chemical resistance of >PVDF< led to this material being used more and more in lining metal containers, as well as in connection with glass Fiber reinforced plastics (FRP) as dual laminates. 4 refs.

Rubber K Plastics Research Abstracts

8. ASTM F 491-. SPECIFICATION FOR POLY (VINYLIDENE FLUORIDE) (>PVOF<) PLASTIC-LINED FERROUS METAL PIPE & FITTINGS RPA 95-05 538774 NDN-131-0107-5151-8

NO-AUTHOR


Details are given of >PVDF< plastic lined metal pipe and fittings, for use with corrosive gases and liquids. Photocopies and loans of this document are not available from Rapra. It may be purchased from BSI. Please contact Rapra for further details. NON POLYMER- METAL

9. GET THE MOST OUT OF PLASTIC LINEO PIPE - RPA 94-23 528177 NDN- 131-0106-5173-1

Button, L. W.; Henthorn, G. V.

The advantages of Fluoropolymer linings in metallic pipes are explained. Common fluoropolymers used in lined-pipe installations are listed. The use of fluoropolymer linings is discussed in detail, including standards, permeation mechanics, physical properties of the liner, laboratory programme, laboratory results, field test results, and design considerations. 10 refs.

Non polymer: Benzene; Chloroform; Chlorine; Hydrochloric Acid; Metal; Methyl ethyl ketone; Methylene chloride; Nitric Acid; Stainless steel; Sulphuric Acid; Water

10. MECHANICAL BEHAVIOUR OF COMPOSITE TUBES AFTER EXPOSURE TO CORROSIVE ENVIRONMENTS - RPA 94-07 505159. NDN- 131-0104-2191-9

Dudley, N.; Hoa, S. V.


Several hundred filament wound tubes made of fibreglass/vinyl ester with veil and PVC liners were exposed to hydrochloric acid, sulphuric acid, and water at room temperature and at 145F and 180F for up to 3 years. At different intervals of time, the tubes were emptied of their contents and observed visually for any degradation. Also, the tubes were subjected to axial tests, internal pressure to create hoop stress and internal pressure tests. High temperature (180F) had detrimental effect on the long-term performance of fibre reinforced plastic materials. Thermoplastic lined (PVC, > PVDF<) tubes had better environmental resistance than veil-lined tubes. Photographs show modes of failure. 1 ref.

11. DEMYSTIFY VACUUM CAPABILITIES OF PLASTIC-LINED PIPING - RPA 92-19 454292 NDN- 131-0084-7610-0

Jeglic, M. F.; Lindley, N. L.


A description is given of various operating conditions that can create vacuum within process piping, the theoretical and empirical capabilities of plastic-lined piping in vacuum service are presented, and guidelines are suggested which the chemical engineer should consider when
designing and specifying plastic-lined piping systems capable of withstanding possible vacuum conditions. 5 refl.

12. **ECONOMICAL, SAFE PLASTIC >LINING< FOR STEEL >PIPES< - RPA 92-11**
445175 NDN- 131-0083-9510-0

NO-AUTHOR

**DOCUMENT TYPE-** Journal  
**NAMED COMPANY-** CHEMPLAST INC.  
**SUBFILE CODE-** BR  
**TRADE NAME(S) -** ECONOSAFE; TEFOLON  
**ISSUE OF ORIGINATION-** 9207  
**LANGUAGE-** English (DEF)

Che mplast Mare Etters new Econosafe range of plastics materials for the >lining< of steel >pipes< is claimed to offer significant savings over pipe systems of exotic materials and rubber linings. Products are available in a wide range of plastic liner materials, all extruded to international standards, which are less costly than coated and stainless steel pipes. Brief details are given. NON POLYMER- STAINLESS STEEL; STEEL

13. **REDUCE LEAKS AND CORROSION WITH PLASTIC-LINED PIPING - RPA 91-15**
424035 NDN- 131-0081-8381-8

Lednicky, R.; Lindley, N. L.

**DOCUMENT TYPE-** Journal  
**CORPORATE AUTHOR-** DOW CHEMICAL CO.  
**SUBFILE CODE-** RR; MM  
**ISSUE OF ORIGINATION-** 9109  
**LANGUAGE-** English (DEF)

The capabilities and limitations of plastic-lined metallic piping are described and guidelines for its consideration, including liner selection, are presented. Techniques for improving sealability, such as system design, installation and maintenance practices, are discussed. Lining materials considered are PP, >PVDF< and PTFE. NON POLYMER- METAL

14. **LINING UP TO RESIST CORROSION - RPA 91-03**
411282 NDN- 131-0080-5632-8

Jones, M. E.

**DOCUMENT TYPE-** Journal  
**CORPORATE AUTHOR-** PERFORMANCE PLASTICS INC.  
**SUBFILE CODE-** RR, MM  
**ISSUE OF ORIGINATION-** 9103  
**LANGUAGE-** English (DEF)

Plastic linings For piping and equipment are reviewed. Assistance is given n choosing the type of lining for a particular application. NON POLYMER- STAINLESS STEEL; STEEL
A series of tables are presented to illustrate the chemical resistance of Solef PVDF virgin grades 1008, 1010, 1012 and 5008. The tables are divided into two parts. The left hand side gives the chemical resistance of solid Solef as it is used for tubes, fittings, linings, pumps etc., and the right hand side refers solely to powder coatings applied by electrostatic spraying or by fluidised bed. Methods of testing are described and symbols and evaluation criteria are explained.

Details are given of the specifications, quality and performance of the Fluoroline range of plastic lined ferrous pipework as supplied by BTR Silvertown Ltd. The plastic linings include PTFE, PFA, FEP, >PVDF< and PP. Design details are given of the pipes and fittings, and properties of the linings.

Details are given of the manufacture and properties of Fluoroline lined pipework. They consist of a seamless liner formed into Schedule 40 steel pipe by an exclusive process developed by Resistoflex Corp., which compensates for thermal expansion and contraction between the liner
and the steel. Four types of liner are available, i.e. Fluoroline T (PTFE/PFA), Fluoroline-F (FEP), Fluoroline-K (>PVDF<) and Fluoroline-P (PP). NON POLYMER- STEEL

18. GAFLON PTFE. LINED CHEMICAL EQUIPMENT - RPA 89-26 386720 NDN-131-0078-1071-4

NO-AUTHOR


Gafton PTFE pipes and Fittings are described. The products can be lined with a selection of corrosion resistant materials including PTFE, PFA, FEP, >PVDF< and PP. Products include lined flexible hoses, lined dip pipe, lined sight glasses, expansion bellows and PTFE sheet, rod and tube. Specifications For pipes and fittings are given with details of linings and steel components NON POLYMER- STEEL

19. INVESTIGATION OF DAMAGE TO ORGANIC MATERIALS USED IN CHEMICAL PLANT - RPA 85-28 301182 NDN- 131-0069-8893-3

Schindler, H.; Graefen, H.


By use of thirteen case histories, the authors discuss types of damage which can occur in polymeric components such as >linings<, >pipes<, pumps, fittings and the like used in chemical plant. Polymers covered are PTFE, >PVDF<, HDPE, PP, PVC, rubber and GRP. Reasons For damage are analysed, causes considered include incorrect construction, installation, unsuitability or overloading of materials

20. FLUOROPOLYMER LININGS. SELECTION FOR PERFORMANCE AND RELIABILITY - RPA 84-21 256500 NDN- 131-0067-2933-2

Jones, A. P.


Details are given of the use of Fluoropolymers for >pipe< >linings<. Materials generally available are identified and the design factors which influence their use, performance and reliability over the range of pipeline requirements are discussed. Information is given about
the various fluoropolymers including PTFE, perfluoro alkoxy, FEP, ETFE, ECTFE and >PVDF<. Fluorocarbon Ltd., is discussed with reference to its activities in >lining< of >pipes< and vessels.


NO-AUTHOR

1976 Burton-on-Trent, 1976-, looseleaf. 15/12/83. 42C38-6H-935 DOCUMENT TYPE- Library Edition CORPORATE AUTHOR- BTR SILVERTOWN LTD. TRADE NAME(S) - FLUOROLINE ISSUE OF ORIGINATION- 8408 LANGUAGE- English (DEF)

Presented in this folder is information on fluoridine products, which include corrosion-proof >pipes<, fittings and vessels, vessel >linings<, PTFE hose and end fittings and pumps. These products are fluoroplastic lined and are designed to withstand the corrosive influences of virtually all corrosive Fluids. Tabulated data, specifications, and diagrams are given.

22. >PVDF< LININGS OUTLAST EXOTIC METALS - RPA 83-22 238210 NDN- 131-0065-4673-0

NO-AUTHOR


A solution to the problem of pumping hot hydrochloric acid through pipework is briefly discussed. British Steel are using Fluoroline pipes lined with >PVDF< by BTR Silvertown NON POLYMER- HYDROCHLORIC ACID

23. ANTICORROSION: PIPING LINED WITH >PVDF< OR TEFLOM FEP - RPA 83-16 233486 NDN- 131-BBB4-9955-7

NO-AUTHOR


An account is given of the use of >PVQF< or Teflon FEP >linings< for >pipes< for corrosion resistant piping, particular reference being made to the products of Symalit SA. Topics discussed include the use of Armalon composite Film for >lining< >pipes<, Maxar composite film, glass
fibre reinforced \texttt{PVDF}\texttt{\textgreater} pipes and tubes, films of \texttt{PVDF\textless} with films of an elastomer. In particular piping for chemical plants and similar applications is considered.

24. \textbf{MILD STEEL PIPING WITH LOOSE THERMOPLASTIC LINERS} - RPA 77-02
101164 NDN- 131-0052-9325-0

SMITH, F.

1976 PRI, NORTH EASTERN SECTION; IME. PLASTICS LININGS & COATINGS IN PREVENTION OF CORROSION. NEWCASTLE UPON TYNE, MAR. 1976, PAPER 16, PP. 7. PREPRINT. 935 DOCUMENT TYPE - Conference NAMED COMPANY - ICI ISSUE OF ORIGINATION - 8201 LANGURGE - English (DEF)

(SYMPHOSIM) PROBLEMS ASSOCIATED WITH \texttt{PIPE\textgreater} LININGS\texttt{\textless} OF \texttt{PVDF}\texttt{\textgreater}, PTFE AND FEP ARE DISCUSSED. NDN POLYMER - STEEL; METAL

NDN- 131-0001-7369-1

NO-AUTHOR

PP. 9. 12INS. 28/5/74. 42C38(10)-BA312 DOCUMENT TYPE - Library Edition CORPORATE AUTHOR - BTR SILVERTOWN LTD., CHEMICAL PLANT DIV. NAMED COMPANY - BTR SILVERTOWN LTD., CHEMICAL PLANT DIV. TRADE NAME(S) - FLUOROLINE ISSUE OF ORIGINATION - 8201 LANGUAGE - English (DEF)

THE RANGE OF HOSE, \texttt{PIPE\textless} \texttt{PIPE\textgreater} FITTINGS AND \texttt{PIPE\textless} \texttt{LININGS\textless} AVAILABLE IN PTFE, FEP, POLYPROPYLENE AND \texttt{POLYVINYLIDENE\textless} FLUORIDE\texttt{\textless} ARE OUTLINED.

26. \textbf{FLUOROPLASTIC LININGS FOR CORROSIVE SERVICE} - RPA 73-02 26090
NDN- 131-0000-8425-6

ATKINSON, H. E.


CHARACTERISTICS OF THE MAJOR VARIETIES OF CORROSION RESISTANT FLUOROPLASTICS (I.E. PTFE, FEP, \texttt{POLYVINYLIDENE\textless} FLUORIDE\texttt{\textless}, PCTFE AND (PERFLUOROALKOXY) FLUOROCARBON RESIN ARE GIVEN. METHODS OF \texttt{LINING\textless} PIPE\texttt{\textless}, PIPE FITTINGS, VALVES, PUMPS,) AND TANKS AND COLUMNS, ARE DESCRIBED. TYPICAL CASE HISTORIES FOR \texttt{LININGS\textless} PIPE\texttt{\textless} ARE LISTED.
FACTORS WHICH DETERMINE THE TYPE OF MATERIAL TO BE USED ARE CONSIDERED. THEY INCLUDE CHEMICAL ENVIRONMENT, TEMP., PRESSURE, COMPONENT DESIGN, MECHANICAL ENVIRONMENT, FABRICATION TECHNIQUE AND QUALITY CONTROL INSTALLATION PRECAUTIONS, COMMERCIALLY AVAILABLE PRODUCTS AND INSTALLATION COST ARE ALSO DISCUSSED.

World Surface Coating Abstracts

27. >Polyvinylidene< >fluoride<: polymer suitable for fluid handling under extremely corrosive conditions. - WSC 78-00 61860 78/01860 NDN- 138-0001-8300-7

DILLEY, E. R.

ABBREVIATED JOURNAL TITLE- BHRA 2nd Internat. Conf. on Internal & External Protection of Pipes, Canterbury 1977, Paper H2, 10 pp. DOCUMENT TYPE- Journal; etc. MAIN HEADING- PIPELINE COATINGS; PIPES; POLYVINYLIDENE FLUORIDE; POWDER COATINGS ISSUE OF ORIGINATION- 7803 LANGUAGE- English (DEF)

>Polyvinylidene< >fluoride< may be used for lining steel pipe, as solid pipe and as a powder coating for pipe. It is highly corrosion-resistant and suitable for chemical plant.

Second Search

1. DEGRADATION OF >POLYVINYLIDENE< >FLUORIDE< CAPACITORS DURING >ACCELERATED< >TESTS< CAS SECTION- 076 CAS SUBSECTION- 003 - CAS 090-08 090-131368 CA 090:131368 NDN- 154-0469-7845-5

BUROUGH, S. W.; BRAMMER, H. G.; BURNHAM, JOHN

ANNU. PROC, RELIAB. PHYS. (SYMP.), 1978, VOL.16, PP.219-23 DOCUMENT TYPE- JOURNAL CODEN- ARLPBI LOCATION OF WORK- HUGHES AIRCRAFT CO., TECH. SERV. OIV., CULVER CITY, CALIF. SUBFILE CODE- PHY LANGUAGE- ENGLISH

NO-ABSTRACT

DESCRIPTOR(S) - >POLYVINYLIDENE< >FLUORIDE< CAPACITOR DEGRDN; >LIFE< TEST >POLYVINYLIDENE< >FLUORIDE< DEGRDN; ALUMINUM FLUORIDE FORMATION CAPACITOR.

2. >POLYVINYLIDENE< >FLUORIDE< FOR PIPING OPERATING UNDER HIGHLY CORROSIVE CONDITIONS. - EII 80-82 07350 NDN- 115-0079-7275-8

E. R. Dilley

30
The high crystal energy of polyvinylidene fluoride (PVDF) with the strength of the C-F bond make it probably the strongest of the fluoropolymers. Chemical resistance is outstanding, as one would expect of a fluoropolymer, so the material has a useful combination of properties to offer in the field of piping. A brief summary of PVDF's properties relevant to piping is given. It is concluded that PVDF is clearly a polymer with high potential for the protection or manufacture of piping systems where resistance to aggressive environment and/or where long >life< is required.

DESCRIPTOR(S) - APPLICATIONS; CONDITIONS; CORROSION; FLUOROPOLYMERS; LI; LINING, PIPE, PLASTIC; PIPE, STEEL; PIPING SYSTEMS; PLASTICS APPLICATIONS, PLASTICS, REINFORCED; POLYVINYLIDENE FLUORIDE SECTIONAL CLASSIFICATION CODE- CAL539, CAL545; CAL619, CAL817.

Engineered Materials Abstracts (EMA)

3. Cracking of >Poly(Vinylidene< >Fluoride<) Due to Chemical Attack. - EMA 93-12 199312-CI-P-2648  NDN- 104-0018-222S-9

Stirling, C. D., Van Tilburg, V. S. M., Miller, N. A.

JOURNAL- NAME- Polymers and Polymer Composites 1, (3) 1993 PP. 167-174 reference (s) FIGURES- Photomicrographs. DOCUMENT TYPE- Journal Article  ISSN- 0967-3911 AUTHOR AFFILIATION- Industrial Research (New Zealand), (All Authors) PUBLICATION COUNTRY- UK LANGUAGE- English

The effects of exposure of poly(vinylidene fluoride) (PVDF) to various chemical solutions have been investigated. Scanning electron microscopy (SEM) was used to examine the exposed samples for signs of chemical attack and cracking, while tensile and flexural bend tests were used to determine the effects of exposure on mechanical properties. Samples exposed to 58% sodium hydroxide exhibited both chemical attack and cracking together with a significant loss in ductility. Exposure to 16% sodium hypochlorite and concentrated sulphuric acid did not give either chemical attack or cracking but exposure to sulphuric acid did cause significant loss of mechanical properties. From the results obtained it was concluded that the cause of cracking in 58% sodium hydroxide was chemical embrittlement, whereby a brittle layer was formed on the surface of the material. This brittle layer subsequently cracked under applied tensile stress. The actual mechanism by which the brittle layer was formed was not established. The cracking was not considered to be due to environmental stress cracking as this requires the simultaneous action of tensile stress and cracking medium.
Two types of pipe were extruded from one special kind of granulate. Long-term tests with internal pressure were performed with water, p-xylene and acetic acid, in one case with pressure maintained at a constant level in the other case with constantly increasing pressure rates. In addition the uni-axial creep behaviour of injection-moulded and pressed material was examined. According to the degree of crystallinity of the semi-Fabrics and the kind of configuration within the different crystalline types it was found that creep compliances and failure times vary. For polymers like PVDF which show very flat time curves until rupture vs. applied stress, long-term tests with slowly increasing stresses and strains prove advantageous. Graphs, Photomicrographs.

A comparison of the mechanical, thermal and chemical properties of polyvinylidene fluoride (PVDF) with those of other thermoplastics shows that PVDF is often the best material for high-temperature applications (100 deg C to 140 deg C) in contact with corrosive fluids, e.g. mineral acids, saline solutions or oxidising agents. The reinforcement of PVDF tube by encasing it in thermosetting resin based GRP increases resistance to internal pressure. However, the difference in coefficient of linear expansion between the reinforcing material and the PVDF causes very high internal stresses to be set up as the temperature rises. Analysis of failures which have occurred during the service life of pipework shows that these were due to a combination of two factors: creep under compression of the PVDF at high temperature and tensile stress set up...
when returning to ambient temperature. The analytical basis of the problem is presented and the mechanical and thermal properties of each material are related to the overall behaviour of the composite material. 4 ref.--AA

DESCRIPTOR(S) - Corrosion resistance; Creep (materials); Failure; Pipe --Materials substitution; Polyvinylidene fluorides --Materials substitution; Thermal expansion; Thermal stresses

SECTION HEADING CODE- FI

SECTION HEADING- Engineering Components and Structures.

6. Development of Kynar >Polyvinylidene< >Fluoride<. - EMA 88-00 8803G1-P-0049 NDN- 104-0002-9543-4

Dohany, J. E


LANGUAGE- English

Kynar polyvinylidene fluoride was introduced in 1960. First commercial production began in 1965. For the first five years of its >life<, Kynar resin was used almost exclusively in the electrical and electronic market. In 1965, the architectural finish, Kynar 500, was introduced followed by first plan expansion in 1969. In 1975, Kynar 460 was introduced for high speed wire extrusion. In 1981, the Underwriter laboratories listed Kynar resins for use in plenum cables that can be used without conduit. In 1983, a new homopolymer series was introduced known as the series 700 and was Followed by Kynar Flex 2800 in 1984. To meet the demands of the growing markets, a new plant for Kynar resins was built and placed in operation in 1985. Also that year, new coating resins, Kynar SL and Kynar ADS, were introduced. Kynar polyvinylidene fluoride is a crystalline polymer that is produced by the emulsion polymerization technique. Typical properties are discussed. 3 ref.--AA

DESCRIPTOR(S) - End uses; Historical; Polyvinylidene fluorides --Development; Properties (attributes)

SECTION HEADING CODE- GI

SECTION HEADING- General and Nonclassified.

Rubber K Plastics Research Abstracts

7. DEVELOPMENTS IN FLUOROPOLYMER COATINGS - RPA 95-10 546171 NDN-131-0108-2543-5

Scheirs, J.; Burks, S.; Locaspi, A.
A review is presented of developments in Fluoropolymer coatings as the demand increases for outdoor durability. The new systems are based on copolymers such as polyfluoroethylene-co-vinyl ether and perfluoropolyethers, which can be used to coat temperature-sensitive substrates. This new generation of Fluorocarbon-based coatings are soluble in common organic solvents, curable at ambient temperatures, and have increased compatibility with hardeners. Automotive topcoat applications are described which upgrade the service >life< of engineering plastics, speciality paints for graffiti and corrosion resistance, and water-based dispersions for environmental compliance are all discussed, as well as advanced techniques for monitoring the weathering performance of these coatings. 53 refs.

DESCRIPTOR(s) - ACCELERATED; TEST; AUTOMOTIVE APPLICATION; BLEND; BUILDING APPLICATION; COATING; COIL COATING; COLOUR RETENTION; COMPANY; COMPATIBILITY; COPOLYMER; CORROSION RESISTANCE; DATA; DEGREE OF POLYMERISATION; DURABILITY; ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS; ENVIRONMENTAL LEGISLATION; ESCA, ESR; ESR SPECTROSCOPY; FLUOROETHYLENE COPOLYMER; FLUOROPOLYMER; FOURIER TRANSFORM INFRARED SPECTROSCOPY, FTIR; FUNCTIONALISATION, GLOSS; GRAPH; HARDENER; MECHANICAL PROPERTIES; MOLECULAR STRUCTURE; MOLECULAR WEIGHT; PAINT; PERFLUORINATION; PERFLUOROPOLYETHER; PLASTIC; PMMA; POLYETHYLENE; POLYMETHYL METHACRYLATE; POLYVINYLIDENE; FLUORIDE; PROPERTIES; PVDF; REVIEW, ROOM TEMPERATURE CURING; RTV; SOLUBILITY; SOLVENTLESS; TABLES; TECHNICAL; TENSILE STRENGTH; TEST METHOD; TESTING; TETRAFLUOROETHYLENE COPOLYMER; THEORY, THERMOPLASTIC; THERMOSET; WEATHER RESISTANCE; WEATHERING RESISTANCE IDENTIFIER(S) - COATINGS --fluoropolymers, FLUOROPOLYMERS --coatings GEOGRAPHIC DESCRIPTOR(S) - AUSTRALIA; EUROPEAN COMMUNITY; EUROPEAN UNION; ITALY; USA; WESTERN EUROPE SECTIONAL CLASSIFICATION CODE-42C38; 6A311 SECTION HEADING CODE- QB: KM.

8. GET THE MOST OUT OF PLASTIC LINED PIPE - RPA 94-23 528177 NDN- 131-0106-5173-1

Buxton, L. W., Henthorn, G. V

The advantages of fluoropolymer linings in metallic pipes are explained. Common fluoropolymers used in lined-pipe installations are listed. The use of fluoropolymer linings is then discussed in detail, including standards, permeation mechanics, physical properties of the liner, laboratory programme, laboratory results, field test results, and design considerations. 18 refs. NON POLYMER- BENZENE; CHLORIDE; CHLORINE; HYDROCHLORIC ACID; METAL; METHYL ETHYL KETONE; METHYLENE CHLORIDE; NITRIC ACID; STAINLESS STEEL; SULPHURIC ACID; WATER

Descriptor(s)- CHEMICAL; COMPANY, CORROSION; CORROSION RESISTANCE, COST; CRYSTALLINITY; DATA; DESIGN; ETHYLENE-CHLOROTRIFLUOROETHYLENE COPOLYMER; ETHYLENE-TETRAFLUOROETHYLENE COPOLYMER; FAILURE; FLUOROPOLYMER; GAS; GRAPH; HAZARDOUS MATERIAL; MECHANICAL PROPERTIES; PERFLUOROALKOXYALKANE COPOLYMER, PERFLUOROETHYLENE COPOLYMER, PERMEATION; PHYSICAL PROPERTIES; PIPE; PIPE LINING; PLASTIC; POLYPROPYLENE; POLYTETRAFLUOROETHYLENE; POLYVINYLIDENE CHLORIDE; >POLYVINYLIDENE< >FLUORIDE<; PP, PROPERTIES; PROPYLENE COPOLYMER; PTFE; PVDC; >PVDF<; SERVICE >LIFE<; STANDARD; STRESS CRACKING; TABLES; TEMPERATURE; TEST; THERMOPLASTIC; THICKNESS IDENTIFIER(S)- FLUOROPOLYMERS --pipe linings, LININGS --fluoropolymer linings; LININGS --pipes; PIPES --fluoropolymer linings; GEOGRAPHIC DESCRIPTOR(S) - USA SECTIONAL CLASSIFICATION CODE- 42C38; 6L12; 6H21 SECTION HEADING CODE- KM; QL; QI.

9. SOLEF >PVOF< ENGINEERING POLYMER STANDS THE TEST OF TIME...APPLICATIONS IN THE CHEMICAL AND ASSOCIATEO INDUSTRIES - RPA 92-23 458024 NDN- 131-0085-1342-9

NO-AUTHOR


Solef PVOF is a Fluorinated semi-crystalline thermoplastic material which offers chemical resistance to a wide spectrum of chemical products. It guarantees high reliability during a long service >life< and requires no special maintenance. It is lighter than steel and can reduce the weight and cost of a plants superstructure if used for solid structures or in conjunction with reinforced polyester. Examples are described and illustrated of the use of Solef PVDF in a variety of corrosive situations. NON POLYMER- BROMINE

Descriptor(s) - ACID RESISTANCE; ACID RESISTANT; APPLICATION; CASE HISTORY; CHEMICAL INDUSTRY; CHEMICAL RESISTANCE; COMPANIES;
Munekata, S.

The weather resistance of PVF and PVDF as coatings and the characteristics of such coatings are discussed and compared with those of recently developed fluoroethylene/vinyl ether copolymers (FEVE)s, which are soluble in organic solvents and curable over a wide range of temps. Data are provided on the properties of PVF, including Tedlar, PVDF, including Kynar-1120 and 1140, compatibility of FEVE with curing agents and pigments, adhesiveness of FEVE finish to various substrates, such as metals and polymers, and the mechanical properties and gas permeability of FEVE exposed in a Sunshine Weather-O-Meter. 14 refs. NON POLYMER- METAL

DESCRIPTOR(S) - >ACCELERATED< >TEST<; ADHESION; BONDING; COATING, COMPANIES, COMPANY; COMPATIBILITY; CURE, CURING, CURING AGENT; DATA; FLUOROETHYLENE COPOLYMER; FLUOROPOLYMER, GAS PERMEABILITY; MECHANICAL PROPERTIES; PIGMENT; PROPERTIES, >PVDF<; PVF; SOLUBILITY; SOLVENT; SUBSTRATE; TECHNICAL; TEST; TESTING; THERMOPLASTIC; ULTRAVIOLET IRRADIATION; UV LIGHT; VINYL ETHER COPOLYMER; VINYL FLUORIDE POLYMER; VINYLIDENE FLUORIDE POLYMER; WEATHERING IDENTIFIER(S)- FLUOROPOLYMERS, --properties; COATINGS --fluoropolymers; COATINGS, --properties; FLUOROPOLYMERS --coatings GEOGRAPHIC DESCRIPTOR(S) - JAPAN SECTIONAL CLASSIFICATION CODE- 42C38-6A3-9T SECTION HEADING- QB; KM; UA.

11. UNIQUE COMPLEX - RPA 89-01 366393 NDN- 131-0076-0758-1

NO-AUTHOR
Details are given of the activities of Elkaplast of France in the coextrusion of ABS and PVDF. Working under licence from Atochem, the company is the world's only manufacturer of such coextrusions. The laminates obtained are trade named Elkalite, and consist of 1 to 9 mm thick ABS sheet covered with a 50 to 300 microns layer of Foraflon PVDF. Applications include vehicle components and equipment housings. Some company information is presented on Elkaplast.

DESCRIPTOR(S) - ABS; >ACCELERATED<; TEST; AGEING; APPLICATION; CASING; CHEMICAL RESISTANCE IDENTIFIER(S) - COEXTRUSION - ABS, COEXTRUSION, --PVDF; COMPANY INFORMATION --Elkaplast GEOGRAPHIC DESCRIPTRQR(S) - FRANCE SECTIONAL CLASSIFICATION CODE - 42C21C391D11-623, 42C386-623; 06 SECTION HEAOING-OI; SC, KF, KN, KO, KM; CS.

12. FORAFLON >POLYVINYLIDENE< - A MATERIAL FOR CHEMICAL PLANT CONSTRUCTION - RPA 86-21 317104 NDN- 131-0071-4755-7

NO-AUTHOR

ABBREVIATED JOURNAL TITLE- Plastverarbeiter 1986 37, No.2, Feb. 1986, p.3-8-2 DOCUMENT TYPE- Journal TRADE NAME(S) - FQRAFLON ISSUE OF ORIGINATION-8611 LANGUAGE- German

Foraflon is a semi-crystalline homopolymer containing 59% fluorine and has a melting point of 170°C. Properties of this PVDF are discussed, mention being made of thermoplasticity, heat stability, chemical resistance, wear, service >life<, mechanical behaviour, electrical characteristics and solubility. Information on processing characteristics is given and application areas are outlined. Mention is made of its use in pipes, packaging, cables and wires and chemical plant.

DESCRIPTOR(S) - ABRASION RESISTANCE; CABLE, CHEMICAL PLANT; CHEMICAL RESISTANCE; CHEMICALLY RESISTANT APPLICATION, ELECTRICAL PROPERTIES; HEAT STABILITY; MECHANICAL PROPERTIES: MELTING POINT; PACKAGING; PIPE; PLI-“ISTICITY; PROCESSING; >PVDF<; SEMI-CRYSTALLINE; SERVICE >LIFE<; SOLUBILITY; THERMAL STABILITY; VINYLIDENE FLUORIDE POLYMER; WEAR; WIRE IDENTIFIER(S) - CHEMICAL RESISTANCE --PVDF; VENYLIDENE FLUORIDE POLYMERS GEOGRAPHIC DESCRIPTOR(S) - FRANCE SECTIONAL CLASSIFICATION CODE - 42C386; 42C386-935 SECTION HEADING- KM; UE.

13. PROGRAMME ON POLYMER AGEING EFFECTS - RPA 86-06 30603 NDN-131-0070-3720-0
A major research programme on the ageing of polymer blends has begun at the Polytechnic Institute of New York. Very brief details are provided.

A coating system having a claimed service life of 28 years and comprising undercoat/intermediate coat/topcoat containing polyvinylidene fluoride, has an intermediate coat composition consisting of a mixture of polyvinylidene fluoride with an acrylic polymer of MW 50,000 -200,000.
METHACRYLATE; OCTYL METHACRYLATE; FLUOROPOLYMER; ETHYL METHACRYLATE; HEXYL METHACRYLATE
SECTIONAL CLASSIFICATION CODE- 64  SECTION HEADING- Paints, etc. for other Specific Uses.

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Appendix One

X-Ray Diffraction Results
Baseline, 70 °C, and 110 °C powder. Scans decrease with increasing temperature.
Data from images indicating tiny crystallites in the 130 °C aged Coflin powder.
Baseline, 70 °C, and 110 °C powder. Scans decrease with increasing temperature.
Comparisons of Coflon pipe throughout the thickness.

Crystalline fractions similar

Additional peaks at outer edge
Cross section through coflon pipe, 1mm scan 25.4mm from outer edge.
Cross Section Throuth Coflon Pipe, 8 mm Thickness
Appendix Two FTIR Results
SAMPLE T-40 TEFZEL
SAMPLE T-54 COFLOM
Appendix Three  Gel Permeation Chromatography Results
Mark-Houwink Plot

Log(Molecular Weight)

Log(Imtrinsic Viscosity)

T68  T70  T72  T73  T7D  T7D110  T31-FA  T56-FA  T60-FH
Mark-Houwink Plot

Log(Molecular Weight)

Log(Initial Viscosity)
TRI Sample Analysis

GPC Characterization

Run Conditions
System: 100 Viscometer + RI
Solvent: NMP
Columns: 2 Amgel Mixed Bed
Concentration: 1.920 mg/mL
Flowrate: 0.592 mL/min
In. Vol.: 107.3 uL

Detector Temp.: 30.0 C
Column Temp.: 80.0 C

Universal Calibration Report
Mn: 115,400
Mw: 379,600
Mz: 814,500
Pd: 3.29

IVn: 1.011
IVw: 1.641
IVz: 2.215
Pd: 1.62

Rgn: 17.99
Rgw: 25.73
Rgz: 33.55
Pd: 1.43

MH Coefficients
0.4% Below 10,000 a: 0.622
7.0% Above 1,000,000 LogK: -3.192

Dual Detector Chromatogram

Mark-Houwink Plot

Molecular Weight Distribution
TRI Sample Analysis

GPC Characterization

Run Conditions
System: 100 Viscometer + RI
Solvent: NMP
Columns: 2 Amgel Mixed Bed
Concentration: 2.020 mg/mL
Flowrate: 0.593 mL/min
In. Vol.: 107.3 uL
Detector Temp.: 30.0 C
Column Temp.: 80.0 C

Universal Calibration Report
Mn: 101,900
Mw: 350,300
Mz: 762,800
Pd: 3.44

Mw: 294,700
1.2% Below 10,000
4.7% Above 1,000,000
LogK: -4.058

Mark-Houwink Plot

Molecular Weight Distribution
TRI Sample Analysis
GPC Characterization

Run Conditions
System: 100 Viscometer + RI
Solvent: NMP
Columns: 2 Amgel Mixed Bed
Concentration: 1.880 mg/mL
Flowrate: 0.594 mL/min
In. Vol.: 107.3 uL

Detector Temp.: 30.0 C
Column Temp.: 80.0 C

Universal Calibration Report
Mn: 85,600
Mw: 332,800
Mz: 784,400
Pd: 3.89

IVn: 0.648
IVw: 1.608
IVz: 2.423
Pd: 2.48

Rgn: 14.55
Rgw: 24.06
Rgz: 33.43
Pd: 1.65

Dual Detector Chromatogram

Mark-Houwink Plot

Molecular Weight Distribution
TRI Sample Analysis
name: T73-1

GPC Characterization

Run Conditions
System: 100 Viscometer + RI
Solvent: NMP
Columns: 2 Amgel Mixed Bed
Concentration: 1.930 mg/mL
Flowrate: 0.596 mL/min
In. Vol.: 107.3 uL
Detector Temp.: 30.0 C
Column Temp.: 80.0 C

Universal Calibration Report

Mn: 105,300  Mp: 268,300  MH Coefficients
Mw: 281,800  0.4% Below 10,000  a: 0.613
Mz: 503,100  2.1% Above 1,000,000  LogK: -2.913
Pd: 2.68

IVn: 1.678  IVw: 2.479
IVz: 3.398  Pd: 1.48

Rgn: 20.29  Rgw: 27.18
Rgz: 34.08  Pd: 1.34

Mark-Houwink Plot

Molecular Weight Distribution
TRI Sample Analysis

Run Conditions
System: 100 Viscometer + RI
Solvent: NMP
Columns: 2 Amgel Mixed Bed
Concentration: 2.030 mg/mL
Flowrate: 0.594 mL/min
In. Vol.: 107.3 uL

Detector Temp.: 30.0 C
Column Temp.: 80.0 C

Universal Calibration Report
Mn: 115,800
Mw: 310,500
Mz: 606,900
Pd: 2.68
M: 271,400
0.2% Below 10,000
3.4% Above 1,000,000
MH Coefficients
a: 0.568
LogK: -2.739

Mark-Houwink Plot

Molecular Weight Distribution

Dual Detector Chromatogram
TRI Sample Analysis
GPC Characterization

Run Conditions
System: 100 Viscometer + RI
Solvent: NMP
Columns: 2 Amgel Mixed Bed
Concentration: 2.040 mg/mL
Flowrate: 0.593 mL/min
In. Vol.: 107.3 uL

Detector Temp.: 30.0 C
Column Temp.: 80.0 C

Universal Calibration Report
Mn: 116,000
Mw: 285,400
Mz: 497,800
Pd: 2.46

Mw: 259,900
MH Coefficients
0.3% Below 10,000
a: 0.599
1.9% Above 1,000,000
LogK: -2.864

IVn: 1.662
IVw: 2.341
IVz: 3.053
Pd: 1.41

Rgn: 20.73
Rgw: 26.92
Rgz: 33.08
Pd: 1.30

Mark-Houwink Plot

Molecular Weight Distribution
Appendix Four  
Gas Chromatography / Mass Spectroscopy Results
### Sample CP-7D70

#### Library Search Results

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Quality: 50
ID: 1-Tetradecanamine, N,N-dimethyl-

Abundance Average of 20.545 to 20.553 min.: ROCK1B.D (+,-,*)

#25798: 1-Tetradecanamine, N,N-dimethyl- (*)
### Integration Params: current RTEINT parameters

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Operator : Ken McFerren
Acquired : 2 Jun 96 2:41 am using AcqMethod MEOHSCAN.M
Sample Name: EDA METHANOL 130C 1:20
Misc Info : w/ 125 ng/mL VOA Int Stnds
Vial Number: 18

Search Libraries:  C:\HPCHEM\DATABASE\NBS49K.L
                  C:\HPCHEM\DATABASE\WILEY.L
Minimum Quality: 50
Minimum Quality: 0

Unknown Spectrum: Apex minus start of peak
Integration Params: current RTEINT parameters

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Operator: Ken McFerren
Acquired: 2 Jun 96 1:38 am using AcqMethod MEOHSCAN.M
Sample Name: EDA METHANOL 110C 1:100
Misc Info: w/ 125 ng/mL VOA Int Stnds
Vial Number: 17

Search Libraries: C:\HPCHEM\DATABASE\NBS49K.L
C:\HPCHEM\DATABASE\WILEY.L

Unknown Spectrum: Apex minus start of peak
Integration Params: current RTEINT parameters

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Operator: Ken McFerren
Acquired: 2 Jun 96 3:45 am using AcqMethod MEOHSCAN.M
Sample Name: EDA METHANOL 110C 1:20
Misc Info: w/ 125 ng/mL VOA Int Stnds
Vial Number: 19

Search Libraries: C:\\HPCHEM\DATABASE\NBS49K.L
C:\\HPCHEM\DATABASE\WILEY.L
Minimum Quality: 50
Minimum Quality: 0

Unknown Spectrum: Apex minus start of peak
Integration Params: current RTEINT parameters

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Sun Jun 02 11:48:03 1996 Page 2
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Sample: T99 COFLON HP SIDE CH4/MEOH PERM
Size: 26.7540 mg
Method: 10R600
Comment: CH4 HTRU MEOH 5000 PSI 140C

File: C: CAPP93.01B
Operator: RAR
Run Date: 15-May-96 12:12

![TGA graph showing weight loss at 500°C with 9.023% loss (2.414 mg)]
Sample: T99 (MEAL C28) CENTER SECT.
Size: 27.5280 mg
Method: 10R600
Comment: THROUGH MEOH 5000 PSI GAS 140ºC

TGA

File: C:CAP93.08
Operator: RAR
Run Date: 28-May-96 11:57

Graph:
- 400.00ºC
  - 92.55%
- 500.00ºC
  - 42.17%
- 7.934 %
  - (2.184 mg)
Sample: T99 (MERL C28) LP SIDE SECT. 4
Size: 17.0160 mg
Method: 10R600
Comment: THROUGH MEOH 5000 PSI GAS 140C

File: C; CAPP93.06
Operator: RAR
Run Date: 28-May-96 06:07

Temperature (°C)

Weight [%]

400.00°C
94.80%

500.00°C
42.03%

5.541 %
(0.9429 mg)
Sample: T99 (HEAL C28) LP SIDE SECT. 3
Size: 24.7370 mg
Method: 10R600
Comment: THROUGH MEOH 5000 PSI GAS 140°C

File: C:CAPP93.05
Operator: RAR
Run Date: 25-May-96 12:33

Temperature (°C):

- 400.00°C, 95.21%
- 500.00°C, 42.62%

Weight (%):

- 100%
- 5.105 % (1.263 mg)
Sample: T99 (MERL C28) LP SIDE SECT. 2
Size: 21.3660 mg
Method: 10R600
Comment: THROUGH MEOH 5000 PSI GAS 140°C

File: C:\CAPP93.04
Operator: RAR
Run Date: 24-May-96 06:32

Weight [%]

Temperature (°C)

TGA V5.1A DuPont 210°C
Sample: COFLON PWDR BASELINE
Size: 21.6140 mg
Method: 10R600

File: C:CAPP97.06
Operator: RAR
Run Date: 31-May-96 15:22

Graph showing weight loss as a function of temperature. The graph indicates a weight loss of 10.97% (2.372 mg) at a certain temperature range.
Sample: COFLON PWDR AGED
Size: 24.2660 mg
Method: 10R600
Comment: AGED IN 3% EDA/97% MEOH 110°C 1700 PSI 7D

File: C:CAPP97.04
Operator: RAR
Run Date: 31-May-95 08:01

Temperature (°C)

Weight [%]

400.00°C
98.61%

500.00°C
44.17%

1.862 %
(0.4517 mg)
Sample: COFLON POWDER AGED
Size: 5.3000 mg
Method: 10R300
Comment: AGED 7D 70C 3% EDA / 97% ME0H 1700 PSI

File: CAPP99.04
Operator: RR
Run Date: 1-Jun-96 07:32

162.33°C
76.85J/g

172.54°C
Sample: COFLON POWDER AGED
Size: 7.7000 mg
Method: 10R300
Comment: AGED 7D 110C 3% EDA / 97% ME0H 1700 PSI

File: CAPP99.03
Operator: RR
Run Date: 1-Jun-96 05:54

Temperature (°C) vs. Heat Flow (W/g)

159.53°C
56.50J/g

181.16°C

170.95°C
Sample: COFLOX POWDER AGED
Size: 5.7000 mg
Method: 10R300
Comment: AGED 7D 130C 3%EDA/ 97% MEOH 1700 PSI

DSC File: CAPP99.01
Operator: RR
Run Date: 31-May-96 14:07

Temperature (°C)

Heat Flow (W/g)

164.89°C
7.265 J/g
170.92°C
Sample: CENTER 1 mm UNAGED COFLON PIPE
Size: 18.5560 mg
Method: 10R600
Comment: COFLON PIPE SECTION

File: C: CAPP100.02
Operator: RAR
Run Date: 2-Jun-96 06:18

Diagram:
- Weight [%] vs Temperature [°C]
- Drop at 10.98% (2.038 mg)
Sample: OUTER 1 MM UNAGED COFLON PIPE
Size: 23.8340 mg
Method: 10R600
Comment: COFLON PIPE SECTION

File: C:CAPP100.01
Operator: RAR
Run Date: 2-Jun-96 04:53

TGA V5.1A DuPont 210C

Weight [%]

Temperature (°C)

10.36 %
(2.469 mg)
Sample: INNER 1 MM UNAGED COFLON PIPE
Size: 4.5000 mg
Method: 10R300

DSC
File: CAPP101.03
Operator: RR
Run Date: 2-Jun-96 07:36

Heat Flow (W/g)

161.91°C
49.82 J/g

170.56°C

Temperature (°C)

0 50 100 150 200 250 300

General V4.1C DuPont 210
Sample: CENTER 1 MM UNAGED COFLON PIPE
Size: 5.0000 mg
Method: 10R300

File: CAPP101.02
Operator: RR
Run Date: 2-Jun-96 06:22

DSC

Heat Flow (W/g)

Temperature (°C)

162.76°C
55.20 J/g
170.76°C

General V4.1C DuPont 210C
Sample: OUTER 1 MM UNAGED COFLON PIPE
Size: 4.8000 mg
Method: 10R300

File: CAPP101.01
Operator: RR
Run Date: 2-Jun-96 04:59

Temperature (°C)

Heat Flow (kW/g)

158.88°C
67.12 J/g

168.07°C
Sample: AGED COFLOM PIPE SECTION
Size: 9.7000 mg
Method: 10R300
Comment: AGED 7D 110C 1700 PSI EDA/MEOH 3/97

File: CAPP101.05
Operator: RR
Run Date: 4-Jun-96 14:10

DSC

Heat Flow (W/g)

Temperature (°C)

160.69°C
99.95 J/g

171.92°C

166.23°C
Sample: COFON PIPE SECT. AGED 10R600 25:5910 mg
Size: AGED IN 3% EDA/97% MEDEH 110C 1700 PSI 70 CUT SPEC.
Method: 10R600 Run Date: 29-May-96 09:01
Comment: AGED IN 3% EDA/97% MEDEH 110C 1700 PSI 70 CUT SPEC.

TGA

Temperature (°C)

100
200
300
400
500
600
700
800

Weight (%) 0
20
40
60
80
100

3.371 % 0.8627 mg
Sample: COFLON PIPE SECTION
Size: 20.0180 mg
Method: 10R600
Comment: AGED 7D AT 130°C 1700 PSI 3% EDA / 97% MECH

TGA

File: C:CAPP97.07
Operator: RAR
Run Date: 1-Jun-96 07:29

Temperature (°C)

Weight [%]

6.743 %
(1.350 mg)

35.58 %
(7.121 mg)

43.74%
Sample: T98 COFLON (MERL C22)
Size: 39.8410 mg
Method: 10R600
Comment: LONG TERM CH4 PERMEATION TEST AT 5000 PSI 140°C LP SIDE

File: C: CAPP92.0408
Operator: RAR
Run Date: 15-May-96 10:03

TGA

Weight (%) vs. Temperature (°C)

0 100 200 300 400 500 600 700

TGA V5.1A DuPont 2100

0.9590 %

(0.3821 mg)
Sample: T98 COFLON (MERL C22)
Size: 28.2410 mg
Method: 10R500
Comment: LONG TERM CH4 PERMEATION TEST AT 5000 PSI 140°C HP SIDE

File: C: CAPP92.03B
Operator: RAR
Run Date: 15-May-96 07:34

Temperature (°C)

Weight [%]

100
90
80
70
60
50
40
30
20
10
0

1.963 %
(0.5545 mg)

TGA V5.1A DuPont 210K
Sample: T86 (M3179) UNAGED COFLON
Size: 42.1250 mg
Method: 10R600
Comment: NEW BATCH 6 MM EXTRUDED BAR N2 PURGE

File: C:CAPP92.018
Operator: RAR
Run Date: 14-May-96 13:33

Graph showing weight loss as a function of temperature. The graph indicates a significant weight loss at around 400°C, with a weight loss of 11.07% (4.562 mg).
Sample: T87 COFLON "NEW BATCH" 6 MM BAR
Size: 29.9400 mg
Method: 10R600
Comment: AFTER AGING 4 WEEKS 120-130°C (7% WT LOSS)

File: C: CAPP92.028
Operator: RAR
Run Date: 15-May-96 05:22

Graph showing weight loss as a function of temperature. At 273.0%, the weight loss is 0.8175 mg.
Sample: COFLON "OLD" BATCH PIPE
Size: 34.7750 mg
Method: 10A600
Comment: N2 PURGE

File: C: CAPP97.01
Operator: RAR
Run Date: 28-May-96 14:10

[Diagram of TGA analysis showing weight loss of 11.29% (3.925 mg) at 400°C]