INTERIM REPORT ON
CHEMICAL AND THERMAL ANALYSIS

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SYNOPSIS

During the past six months we have conducted significant research in several domains in order to clarify and understanding the aging and chemical failure mechanism of thermoplastics (PVDF or Tefzel) for pipes. We organized numerous analytical studies with methods including Fourier Transform Infrared Spectroscopy, Dynamic Mechanical Analysis, Differential Scanning Calorimetry, and Stress Relaxation experiments. In addition we have reanalyzed previous thermogravimetric data concerning the rate of deplastization of Coflon pipe. We investigated a number of aged samples of both Tefzel and Coflon that were forwarded from MERL. We conducted stress relaxation experiments of Coflon pipe at several temperatures and determined an activation energy. We also examined the dynamic mechanical response PVDF during deplasticization and during methanol plastization. We performed numerous DSC analyses to research the changing crystalline morphology. We have noted significant changes in crystallinity upon aging for both PVDF and Tefzel. Little variation in elemental composition was noted for many of the aged Coflon and Tefzel samples tested.
1.0 Stress Relaxation of Coflon Pipe

We determined the stress relaxation characteristics of Coflon pipe sections in a splitting tension test emulating 200 psi differential internal pressure. The testing was conducted at three temperatures in order to determine the activation energy for the process. An activation energy was determined for the process of 3 Kcal/mol. Any activation below 5 Kcal/mol indicates that no primary bond breaking is occurring. The plots for the stress relaxation experiments are included in Appendix 4.

1.1 Elemental Analysis of Aged Coflon and Tefzel

Elemental analysis was accomplished on some of the aged Coflon and Tefzel samples obtained from MERL at the last CAPP meeting. Color changes were apparent with both the Tefzel and Coflon materials however there was little effect on the elemental composition of the material. Previously we reported substantial elemental changes in Coflon after exposure to a combination of temperature, H2S and aliphatic amine. We postulated at the last CAPP meeting that this was an elimination dehydrohalogenation type mechanism. This reaction occurs either in the presence of bases such as amines or hydroxides. These types of reactions are commonly known to occur with compounds such as PVC. Very little in the literature describes these processes occurring with PVDF. Considering the initial results after only thermal exposures it appears that Coflon and Tefzel are both relatively stable and undergoing no substantial elemental changes or reactions. This indicates that either the aliphatic amine and or the H2S are essential in addition to thermal activation to produce substantial elemental changes in Coflon. Some of the elemental results from the most recent tests are included below.

<table>
<thead>
<tr>
<th>Sample Identification</th>
<th>Visual Appearance</th>
<th>%C, %H, %N, %F</th>
<th>Exposure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control Tefzel</td>
<td>White</td>
<td>36.33, 2.74,</td>
<td>Laboratory Temperature and Conditions</td>
</tr>
<tr>
<td></td>
<td>Translucent</td>
<td>0.00, 61.04</td>
<td>8 days methanol, 140°C, unstrained 5 ksi</td>
</tr>
<tr>
<td>T-9 Tefzel</td>
<td>White</td>
<td>36.19, 2.80,</td>
<td>14 days methane, 140°C, strained 7% 5 ksi</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.00, 61.07</td>
<td></td>
</tr>
<tr>
<td>T-19 Tefzel</td>
<td>White</td>
<td>36.54, 2.86,</td>
<td>Laboratory Exposure and Conditions</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.00, 61.04</td>
<td></td>
</tr>
<tr>
<td>Control Coflon</td>
<td>White</td>
<td>40.76, 4.11,</td>
<td>14 days Nitrogen, 140°C, 7.5% strain</td>
</tr>
<tr>
<td></td>
<td>Translucent</td>
<td>0.00, 55.29</td>
<td></td>
</tr>
<tr>
<td>T-7 Coflon</td>
<td>Very Light Brown</td>
<td>37.87, 3.33,</td>
<td>14 days methanol, 140°C, 7.5% strain, 5 ksi</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.01, 58.40</td>
<td></td>
</tr>
<tr>
<td>T-8 Coflon</td>
<td>Brown</td>
<td>37.54, 3.29,</td>
<td>8 days methanol, 140°C, unstrained reached</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.01, 58.82</td>
<td></td>
</tr>
<tr>
<td>T-12 Coflon</td>
<td>Brown</td>
<td>38.12, 3.28,</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.01, 58.95</td>
<td></td>
</tr>
</tbody>
</table>
We conducted elemental analysis of a second batch of samples of aged Coflon and Tefzel test articles. This group of materials were again aged in various chemical and physical environments. From the Table it is obvious we are not observing any tremendous decreases in F as was observed for the initial Exxon test piece. This may be due to the severity of the conditions involved with the Exxon test piece.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Visual Form</th>
<th>%C, %H, %N, %F</th>
<th>Exposure</th>
</tr>
</thead>
<tbody>
<tr>
<td>T-28 Coflon</td>
<td>Light Brown</td>
<td>37.74, 3.23</td>
<td>6 days fluid A, 140°C vapor pressure</td>
</tr>
<tr>
<td>T-29 Tefzel</td>
<td>White</td>
<td>37.18</td>
<td>6 days fluid A, 140°C vapor pressure</td>
</tr>
<tr>
<td>T-30 Coflon</td>
<td>White Porous</td>
<td>37.86, 3.18</td>
<td>13 days Fluid A, 140°C vapor pressure</td>
</tr>
<tr>
<td>T-31 Tefzel</td>
<td>White Porous Foam</td>
<td>38.02, 3.13</td>
<td>13 days Fluid A, 140°C vapor pressure</td>
</tr>
<tr>
<td>T-36 Coflon</td>
<td>Brown Wax Porous Foam</td>
<td>58.39, 3.18, 0.03, 56.36</td>
<td>13 days Fluid A, 140°C vapor pressure in glass ended cell, residue from evaporated fluid at end of test</td>
</tr>
<tr>
<td>T-41 Coflon</td>
<td>Dark Brown</td>
<td>41.80, 3.42</td>
<td>4 weeks Fluid F, 120oC, 5kpsi, tested in gas phase</td>
</tr>
<tr>
<td>T-42 Tefzel</td>
<td>White</td>
<td>36.28, 2.71, 0.04</td>
<td>4 weeks Fluid F, 120oC, 5kpsi, tested in gas phase</td>
</tr>
<tr>
<td>T-52 Tefzel</td>
<td>White</td>
<td>36.22, 2.81, 0.01, 60.82</td>
<td>4 weeks (gas phase) Fluid F, 120oC, 5kpsi, subsequent permeation in methane at 140oC 5ksi</td>
</tr>
<tr>
<td>T-53 Coflon</td>
<td>Dark</td>
<td>39.62, 3.69, 0.03</td>
<td>4 weeks (gas phase) Fluid F, 120oC, 5kpsi, subsequent permeation in methane at 140oC 5ksi</td>
</tr>
</tbody>
</table>

*Also found 1.29 % Sulfur
Very few changes either elementally or visually were observed for the Tefzel material. It is commonly believed that the darkening of the Coflon is associated with dehydrohalogenation. We observed test specimens black or very dark in color that exhibited only minor changes in elemental composition. Considering the results it is imperative to test further samples under increasingly aggressive conditions to determine when dehydrohalogenation becomes a major mode of degradation.

1.2 Dynamic Mechanical Analysis of Coflon Pipe and Computer Accelerated Life Development Software

Dynamic Mechanical Analysis (DMA) of the Coflon material was carried out and is contained in Appendix 3. The initial flexural modulus was in good agreement with previously published information. Upon heating the flexural modulus drops to a low of 268 MPa. After which it was noted that the flexural modulus increased to 312 MPa. We believe that the increase is due to the deplasticization. We also noted that the tan delta increases (0.105) until the sample reaches 100°C and then decreases to (0.100). After a long period we again see the tan delta rise during deplasticization to (0.102), as the sample becomes more lossy.

We also examined the viscoelastic response of Coflon using DMA while the sample was immersed in methanol at room temperature. During the first eighteen hours of immersion we notice a decrease in the flexural modulus indicating plasticization with methanol had occurred in the Coflon. The flexural modulus of the Coflon decreased over thirteen percent in the initial eighteen hours while immersed in methanol.

The sample was then removed from methanol and the dynamic mechanical analysis was continued in air for the next twenty two hours. We noticed an increase in the flexural modulus to a higher level than the initial value. The initial flexural modulus value had increased 1.3% at the end of twenty-two hours from the initial value. The increase in the flexural modulus at the end of the test could be due to the leaching of the plasticizer, an increase in crystallinity, or both of these processes.

A computer program has been written for construction of an accelerated test plan for thermoplastic pipe. The duty profile for the pipe is input and an activation energy is also included for the principle mode of degradation finally an acceleration factor is calculated for specific environmental exposures. We will present the program at the meeting and discuss what other exposures or information is needed from the clients to complete the computer program. The aspiration of the program is to assist the project clients in developing a comprehensive composite unit accelerated life prediction test plan based on the mission profile as well as user defined laboratory testing conditions. A complete mission profile for the thermoplastic pipe is necessary for the program. This software program upon completion will hopefully also allow the clients to forecast a commensurate age for thermoplastic pipes that have been in service and exposed to variant environmental conditions.

1.3 Differential Scanning Calorimetry of Aged Coflon and Tefzel
We conducted DSC on many of the forwarded samples of Tefzel and Coflon. The DSC results of the Coflon materials indicated considerable variability in crystallinity depending on aging conditions. We generally observed an increase in the heat of fusion as a function of heating time and temperature. Some of this effect may be attributable to the loss of plasticizer. The residue from the evaporated fluid after 14 days exposure to fluid A at vapor pressure indicates the presence of three compounds with vastly different melt temperatures from Coflon. The three compounds are possibly plasticizer, the solvent, and the decomposed polymer. Based on the DSC information we are not observing any change from the \( \alpha \) form to the \( \beta \) crystalline form. The \( \beta \) form has a melting point of 190°C. We are clearly observing some changes in the relative amount of crystallinity of the Coflon based upon exposure. The DSC results for the samples, the heat of fusion's, the exposure conditions, and percentage crystallinity are given below.

<table>
<thead>
<tr>
<th>Sample Identification #</th>
<th>Exposure Conditions</th>
<th>Heat of Fusion</th>
<th>Percentage Crystallinity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coflon Baseline</td>
<td>Not aged</td>
<td>67.59 J/g</td>
<td>64.6%</td>
</tr>
<tr>
<td>Tefzel Baseline</td>
<td>Not aged</td>
<td>39.00 J/g</td>
<td></td>
</tr>
<tr>
<td>T-26 Coflon</td>
<td>14 days Fluid B,</td>
<td>77.96 J/g</td>
<td>74.5%</td>
</tr>
<tr>
<td></td>
<td>140°C 5,000psi</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T-30 Coflon</td>
<td>13 days Fluid A,</td>
<td>48.30 J/g</td>
<td>46.2%</td>
</tr>
<tr>
<td></td>
<td>140°C vapor pressure</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T-31 Coflon</td>
<td>13 days Fluid A,</td>
<td>44.23 J/g</td>
<td>42.3%</td>
</tr>
<tr>
<td></td>
<td>140°C vapor pressure</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T-32 Coflon</td>
<td>13 days Fluid A,</td>
<td>67.16 J/g</td>
<td>64.2%</td>
</tr>
<tr>
<td></td>
<td>140°C vapor pressure</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T-36 Residue from</td>
<td>13 days Fluid A,</td>
<td>3 peaks none</td>
<td>0</td>
</tr>
<tr>
<td>evaporated fluid at end of test</td>
<td>140°C vapor pressure</td>
<td>near the 150-160°C melt onset of Coflon</td>
<td></td>
</tr>
<tr>
<td>T-29 Tefzel</td>
<td>6 days fluid A,</td>
<td>51.50 J/g</td>
<td>32.1% increase in crystallinity over unaged</td>
</tr>
<tr>
<td></td>
<td>140°C vapor pressure</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T-33 Coflon</td>
<td>13 days Fluid A,</td>
<td>30.54 J/g</td>
<td>29.2%</td>
</tr>
<tr>
<td></td>
<td>140°C vapor pressure</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Using the methods cited in the literature we dissolved PVDF in hot dimethylacetamide (DMAC) and cast a film drying the film at \( \approx 70°C \) for 16 hours. We next performed FTIR and DSC analysis of the resultant film. Films were prepared for virgin PVDF, and aged PVDF (T-28) and compared with a sample of PVDF not dissolved and cast from PVDF. Solution of the PVDF into boiling DMAC was effected within thirty minutes. The DSC analysis (Appendix 1) and the FTIR analysis (Appendix 2) indicate some differences in Tg and the integration areas of the two DSC peaks around 160°C.
This could indicate some conversion from the alpha or beta form to the gamma form with resultant permeation and physical property changes. We also performed a derivative analysis of the DSC data in order to clearly resolve the peaks in the analyses.

1.4 Fourier Transform Spectroscopy 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 mass spectrum of the liquid exudate is provided in Appendix 5. The database search base 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.

We have conducted FTIR analyses of Coflon and Tefzel test pieces after aging for selected times, temperature, and exposure conditions (Appendix 2). We are interested in the chemical functionality’s 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. Below is a table that summarizes the results of the FTIR analyses conducted to date.

<table>
<thead>
<tr>
<th>Sample Identification #</th>
<th>Exposure Conditions</th>
<th>Visual Appearance and Observations</th>
<th>Chemical Analysis and Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>T-32 Coflon</td>
<td>14 days at 140°C</td>
<td>Solid retrieved by evaporation from fluids after exposure</td>
<td>Broad hydroxy functionality, decreased carbonyl absorption</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Polyethylene or polypropylene type spectrum, hydrocarbon type</td>
</tr>
<tr>
<td>T-36 Coflon</td>
<td>Residue from evaporated fluid A 140°C vapor pressure exposure with Coflon</td>
<td>Mixture of solids in fluid and retrieved from fluids after exposure, brown waxlike substance</td>
<td>Spongy residue deposits from bottom of cell: white, porous, extremely physically degraded</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Splitting of 1200cm⁻¹ peak, decreased carbonyl absorption</td>
</tr>
<tr>
<td>T-31 Coflon</td>
<td>Collection of deposited Coflon 13 days Fluid A exposure 140°C vapor pressure</td>
<td>White, translucent, good integrity</td>
<td>Few minor changes</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Hydroxy functionality evident, decreased carbonyl</td>
</tr>
<tr>
<td>T-29 Tefzel</td>
<td>Tefzel, 6 days fluid A 140°C vapor pressure exposure</td>
<td>White, translucent, good integrity</td>
<td>Few minor changes</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Hydroxy functionality evident, decreased carbonyl</td>
</tr>
<tr>
<td>T-35 Coflon</td>
<td>Debris collected from pressure cell after 14 days fluid A 140°C vapor pressure test of Coflon</td>
<td>Film from cell walls, light brown like paper</td>
<td>Few minor changes</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Hydroxy functionality evident, decreased carbonyl</td>
</tr>
<tr>
<td>Sample Type</td>
<td>Description</td>
<td>Physical/Optical Properties</td>
<td>Chemical Analysis</td>
</tr>
<tr>
<td>-------------</td>
<td>-------------</td>
<td>----------------------------</td>
<td>-------------------</td>
</tr>
<tr>
<td>T-30 Coflon</td>
<td>Remains of Coflon 4 point bend</td>
<td>White, spongy porous shell of sample fractured during test</td>
<td>Decreased carbonyl absorption</td>
</tr>
<tr>
<td></td>
<td>Sample tested at 23°C, after 14 days in fluid B at 140°C 5000 psi</td>
<td>White, opaque, with good integrity, ductile</td>
<td>Decreased Carbonyl absorption, new peaks at lower wavenumbers indicating increased hydrocarbon presence</td>
</tr>
<tr>
<td>T-26 Coflon</td>
<td>Residue of filtered methanol from 13 days exposure with Coflon sample 140°C vapor pressure</td>
<td>Extremely porous and brittle light brown material</td>
<td>Broad hydroxy absorption, decreased carbonyl absorption</td>
</tr>
<tr>
<td>T-32 Coflon</td>
<td>Unstrained Coflon bar fluid A at 140°C vapor pressure for 14 days</td>
<td>Dark skin to material, good physical integrity</td>
<td>Decreased carbonyl absorption, broad peaks at high wavenumbers</td>
</tr>
</tbody>
</table>
1.5 Appendix One- DSC Results
Sample: CAPP T-36 COFLON
Size: 8.4000 mg
Method: 10R250
Comment: RESIDUE FROM EVAP FLUID AFT. 14D FL. A 140C VAP. PRES. EXP
Sample: T-33 COFLON
Size: 6.3000 mg
Method: 10R300
Comment: 4 PT BND 12% STRAIN FLUID A 140°C VAP. PRESS.14D

DSC
File: CAP-DSC79.08
Operator: RR
Run Date: 1-May-95 14:58

Heat Flow (W/g)

Temperature (°C)

-150 -50 50 150 250

0 0.15 0.25

0.05 0.15

-0.05 -0.15

Deriv. Heat Flow (W/g/°C)

62.42°C 32.35J/g
165.50°C 30.75J/g
121.95°C
174.81°C

General V2.2A DuPont 9900
Sample: T32
File: CAP-DSC79.04
Size: 2.5000 mg
Operator: RR
Method: 10R250
Run Date: 25-Apr-95 08:59
Comment: RESIDUE FILTERED FROM METHANOL. 13D EXP COFLON 140C VAP PRESSURE

Temperature (°C)

Heat Flow (W/g)

Deriv. Heat Flow (W/g/°C)

151.70°C
67.41 J/g

164.70°C
Sample: CAPP T-31 COFLON
Size: 4.2000 mg
Method: 10R250
Comment: DEPOSITED FROM 13 D FLUID A EXP. 140C VAPOR PRESSURE

DSC
File: CAP-DSC79.02
Operator: RR
Run Date: 7-Apr-95 13:01

Heat Flow (W/g)

1.5
1
0.5
0
-0.5
-1
-1.5
-2

Temperature (°C)

150 200 250

General V2.2A DuPont 9900

161.21°C
44.82 J/g
171.09°C
Sample: T-30
Size: 2.4000 mg
Method: 10R250
Comment: REMAINS OF COFLON 4 PT BEND AFTER 13D IN FLUID A AT 140C VAP PRS

Heat Flow [W/g]

-3 -2 -1 0 1 2

Temperature (°C)

-150 -100 -50 0 50 100 150 200 250

155.19°C
48.74 J/g

166.90°C

Deriv. Heat Flow [W/g/°C]

-0.15 -0.05 0 0.05 0.25

General V2.2A DuPont 9900

File: CAP-DSC79.03
Operator: RR
Run Date: 13-Apr-95 14:50
Sample: T-26 COFLON
Size: 4.3000 mg
Method: 10R300
Comment: COFLON TESTED AT 23C AFT. 140 IN FLUID B AT 140C 5000 PSI

Deriv. Heat Flow (W/°C)

159.39°C
76.78J/g

168.83°C

Temperature (°C)

Heat Flow (W)

1

-1

-2
Sample: T-26 COFLON
Size: 4.3000 mg
Method: 10R300
Comment: COFLON TESTED AT 23°C AFT. 14D IN FLUID B AT 140°C 5000 PSI
Sample: PVDF RECRYS. FROM DMAC (T-28)
Size: 1.3000 mg
Method: 10R250
Comment: N2 PURGE

File: CAPP-DSC87.02
Operator: RAR
Run Date: 31-May-95 10:46

162.06°C
40.96 J/g

170.17°C
Sample: PVDF RECRYS. FROM DMAC (VIRGIN)
Size: 1.0000 mg
Method: 10R250
Comment: N2 PURGE

File: CAPP-DSC87.01
Operator: RAR
Run Date: 31-May-95 10:46

157.21°C
68.10 J/g

166.91°C

Heat Flow (W/g)

Deriv. Heat Flow (W/g°C)

Temperature (°C)

General V2.2A DuPont 9900
Sample: COFLON BASELINE NOT AGED
Size: 3,3000 mg
Method: 10R, 300

DSC

162.40°C
65.84 J/g

167.01°C
1.6 Appendix Two- FTIR Results
T3b FLUID A 140C 14 DAYS

[Graph showing absorption spectrum with wavenumber axis from 400 to 4000 cm⁻¹, and absorbance values at various points.]
SAMPLE T-28 FILM DMAC
1.7 Appendix Three- DMA Results
Sample: COFLON IN METHANOL
Size: 13.30 x 5.57 x 3.46 mm
Method: 1 HZ AMBIENT
Comment: SUBMERGED IN METHANOL

Amplitude (p-p) = 0.50 mm

- 904.6 MPa
- 916.0 MPa
- 785.6 MPa

Time (min)

DMA v3.0L
Sample: COFLON
Size: 13.40 x 5.57 x 3.46 mm
Method: 1 Hz ISOTHERM ELEV TEMP
Comment:

File: E:CAP-DMA45.02
Operator: RR
Run Date: 25-Apr-95 09:34

Amplitude (p-p) = 0.50 mm

E' (MPa)

Temperature (°C)

Tan Delta

Log [Time (min)]

267.9 MPa

311.6 MPa
1.8 Appendix Four- Pipe Stress Relaxation Results
1.9 Appendix Five- Mass Spectra of Exudate From Coflon
Library Searched: C:\HPCHEM\DATABASE\NBS49K.L
Quality: 38
ID: Decanedioic acid, dibutyl ester

Scan 3178 (36.556 min): RRSAMPLE.D (*)

Abundance

Scan 3146: Decanedioic acid, dibutyl ester (*)

Abundance

m/z --> 0 40 60 80 100 120 140 160 180 200 220 240 260 280 300

m/z --> 0 40 60 80 100 120 140 160 180 200 220 240 260 280 300
2.0 Thermogravimetric Evaluation of Deplasticization of Coflon
2.0 Thermogravimetric Evaluation of Deplasticization of Coflon
Additional analysis of thermogravimetric data has been conducted. We are currently developing detailed examination of the deplasticization rates for Coflon. From this information we are calculating this diffusion coefficient of the plasticizer from the Coflon. These rates will be useful in future tests should preconditioning of specimens be required.

We reanalyzed the thermogravimetric information and recalculated the times associated with deplastization for the Coflon pipe materials at specific temperatures. Below is the information that relates deplasticization of the Coflon to time and temperature.

Deplasticization of Coflon

\[ E(D) = 11.4 \text{ kcal/mol} \]
Coflon Deplasticization at 104°C
Coflon Deplasticization at 130°C
Coflon Deplasticization
at 158C

Percent Weight Loss

Root Time (minutes)^1/2
Coflon Deplasticization at 215°C

Percent Weight Loss

Root Time (minutes)^{1/2}
Coflon Deplasticization at 264°C

Percent Weight Loss

Root Time (minutes)^1/2

0 20 40 60 80 100 120 140