CAPP
International Research Project on the Effects of Chemical Ageing of Polymers on Performance Properties

Interim Report on Chemical and Thermal Analysis
Interim Report On
Chemical And Thermal Analysis

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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) 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 small changes in the molecular weight distribution. Again these changes may result in variations 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, Ultra-Violet Scanning Analysis, GC/Mass Spectrometry, Differential Scanning Calorimetry and Thermomechanical Analysis. In the ultra-violet analysis we noted the presence of an absorption band indicative of triene formation. We investigated a number of aged samples of both Tefzel and Coflon that were forwarded from MERL. We also cast films at SWT and subjected these films to a refluxing methanol 1% ethylene diamine solution. An updated literature search was conducted using Dialog and DROLLS to identify any new papers that may have been published in the open literature since the start of this project. The updated literature search and abstracts are contained in the Appendix section of this report.

1.0 HIGH TEMPERATURE AGING OF COFLON FOR MECHANISTIC ANALYSIS

1.1 Test Apparatus

The apparatus for the experimentation involved a round bottom flask fitted with a reflux condenser for aging of the Coflon film. Into the reflux condenser the diamine containing methanol solution was charged, and the Coflon film was added.

1.2 Sample Preparations

The virgin Coflon was dissolved in hot DMAC and cast into thin films. The solvent was allowed to evaporate prior to the beginning of the aging experiments.
1.3 **Test Conditions**

The tests were conducted by immersing the Coflon films in the refluxing methanol ethylene diamine solution. The test films were removed periodically for later chemical analysis. Photographs that exhibit the visual changes of the Coflon upon aging follow on the next page.

2.0 CHEMICAL ANALYSIS ASSESSMENT OF AGING EFFECTS

2.1 **Gel Permeation Chromatography of Aged Coflon**

Gel Permeation Chromatography was conducted on a number of environmentally aged Coflon test pieces. The solvent was NMP and the flow rate was 2mL/minute. The injection volume was 100 uL the detector temperature was set at 30°C and the columns were run at 30°C.

<table>
<thead>
<tr>
<th>Sample Identification</th>
<th>Visual Appearance</th>
<th>Molecular Weights Mn,Mw, Mz</th>
<th>Exposure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control Coflon</td>
<td>White, Translucent</td>
<td>81,700-538,900, 3,217,000</td>
<td>Laboratory Exposure and Conditions</td>
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<tr>
<td>T-75 Coflon</td>
<td>White</td>
<td>177,460-536,509, 1,062,095</td>
<td>Fluid F, 140°C 3 day</td>
</tr>
<tr>
<td>T-77 Coflon</td>
<td>Brown</td>
<td>170,504-532,717, 1,052,564</td>
<td>Fluid F, 140°C 8 days</td>
</tr>
<tr>
<td>T-74 Coflon</td>
<td>White</td>
<td>152,905-534,361, 1,080,943</td>
<td>Fluid F, 120°C 2 weeks</td>
</tr>
<tr>
<td>T-87 Coflon</td>
<td>White</td>
<td>171,265-538,897, 1,067,315</td>
<td>27 days 120-135°C air oven</td>
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<tr>
<td>T-88 Coflon</td>
<td>White</td>
<td>177,880-549,827, 1,084,282</td>
<td>Fluid F, 120°C 5 ksi 1 weeks</td>
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<tr>
<td>T-89 Coflon</td>
<td>White</td>
<td>173,803-539,103, 1,073,977</td>
<td>Fluid F, 120°C 5 ksi 3 weeks</td>
</tr>
<tr>
<td>T-76 Coflon</td>
<td>White</td>
<td>173,329-534,908, 1,059,213</td>
<td>Fluid F, 140°C 5 day</td>
</tr>
<tr>
<td>T-53 Coflon</td>
<td>Brown</td>
<td>158,990-519,823-990,690</td>
<td>Fluid F, 120°C 5 ksi 4 weeks gas phase</td>
</tr>
</tbody>
</table>
After Aging in Methanol + 1% Ethylene Diamine at Reflux

PVDF Solution (NMP) Dried on Hot Plate for 3 hours (Temp. 150-160 °C)

New PVDF Films

Before Aging

For 17 Hours
For 4 Hours
For 3 Hours
For 2 Hours
For 1 Hour
2.2 X-Ray Diffraction - Crystallinity Changes

X-ray Diffraction was conducted on both samples from the exposed Tefzel and Coflon thermoplastics. 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
(9) Fluid I- MERL Formulated aromatic and aliphatic hydrocarbon solution

The percentage crystallinity was calculated on the samples. Twelve polymer samples were analyzed, ten PVDF (Coflon) samples and two Tefzel samples. The degree of crystallinity ranges from as low as 9% to as high as 48%. The Coflon samples were more crystalline. Below are the results of the analysis including the fluids the samples were exposed to prior to testing. Color changes were slight with both the Tefzel and Coflon materials in this batch of samples.

<table>
<thead>
<tr>
<th>Sample Identification</th>
<th>Visual Appearance</th>
<th>Percentage Crystallinity</th>
<th>Exposure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control Tefzel</td>
<td>White</td>
<td>26.0%</td>
<td>Laboratory Temperature and Conditions</td>
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<tr>
<td>T-105 Tefzel</td>
<td>White</td>
<td>9%</td>
<td>Fluid A, 6 days 140°C vapor pressure</td>
</tr>
<tr>
<td>Control Coflon</td>
<td>Translucent</td>
<td>41.5%</td>
<td>Laboratory Exposure and Conditions</td>
</tr>
<tr>
<td>T-73 Coflon</td>
<td>Beige</td>
<td>39%</td>
<td>Fluid F, 3 months 100°C</td>
</tr>
<tr>
<td>T-74 Coflon</td>
<td>Beige</td>
<td>35</td>
<td>Fluid F, 120°C, 2 weeks</td>
</tr>
<tr>
<td>T-75 Coflon</td>
<td>Beige</td>
<td>31% prev. 26%</td>
<td>Fluid F, 140°C 3 days</td>
</tr>
<tr>
<td>T-77 Coflon</td>
<td>Beige</td>
<td>34% prev. 29%</td>
<td>Fluid E, 140°C 8 days</td>
</tr>
<tr>
<td>T-87 Coflon</td>
<td>White</td>
<td>40%</td>
<td>After 27 days at 120-130°C in air oven</td>
</tr>
<tr>
<td>T-90 Coflon</td>
<td>Beige</td>
<td>29% prev. 39%</td>
<td>Fluid F, 140°C 5 ksi 2 weeks</td>
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<tr>
<td>T-91 Coflon</td>
<td>Beige</td>
<td>37%</td>
<td>Fluid F, 140°C 5 ksi 4 weeks</td>
</tr>
<tr>
<td>T-100 Coflon</td>
<td>Brown</td>
<td>39%</td>
<td>Fluid G, 65°C, reflux 2 weeks</td>
</tr>
<tr>
<td>T-102 Coflon</td>
<td>Beige</td>
<td>48%</td>
<td>Fluid I, 140°C, 5 ksi 10 weeks</td>
</tr>
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</table>
We can see clearly that all of the exposures for both materials had an effect on the percentage crystallinity. The greatest decrease in crystallinity for both polymers was observed with the Tefzel Fluid G exposure in methanol with ethylene diamine. We also note that the percentage of crystallinity for Coflon increases or decreases depending on the exposure. At the end of this report in Appendix we have attached a number of plots pertaining to the X-ray diffraction analysis of the Coflon and Tefzel test pieces.

2.3 FTIR and Ultraviolet Analysis

The Coflon films described in Section 1.0 were analyzed using Fourier Transform Infrared Spectroscopy (FTIR) and Ultraviolet (UV) spectroscopy. Changes in the molecular structure of PVDF were therefore determined as a function of exposure time. The aging media for the films was methanol with 1% ethylene diamine (EDA). Exposure times were 0, 1, 2, 3, 4 and 17 hours.

Film thicknesses were 0.01 mm for all tests. FTIR analyses were performed on a Nicolet 20 SX spectrophotometer in the transmission mode. A Varian DMS 200 UV/Vis spectrophotometer was used for the ultraviolet measurements.

FTIR Results

Appendix 4.1 displays the complete FTIR spectra for each Coflon film tested. Figure 2-1 compares an enlarged section of the baseline and 17 hour aged specimens. A clear indication of C=C formation is observed at 1700 cm\(^{-1}\). The decrease in the intensity of the peak at 1400 cm\(^{-1}\) may be due to loss of plasticizer (carbonyl) or carbon-fluorine.

Ultraviolet Results

The UV absorption at 290 nm of the PVDF films aged in methanol/EDA were recorded at room temperature. Absorption at this frequency is known to be associated with C=C double bonds, particularly conjugated trienes. The figure displayed in Appendix 4.2 shows the results for the five aged specimens and the baseline sample. An increase in the absorbance was found after aging of the PVDF films. This growth in absorbance translates into a decrease in percent transmittance in the samples. The absorbance change was found to increase with time of exposure to the methanol/amine mixture.

2.4 Gas Chromatography couples with Mass Spectrometry

We conducted GC/Mass Spec on Sample T-89 extracted with methanol. The extract showed primarily the presence of the dibutyl ester of decanedioic acid. Secondarily evident was the free acid decanedioic acid. The decanedioic acid is a degradation product of the plasticizer. The chromatograms are included in Appendix 4.4.
3.0 THERMAL STABILITY AND COMPOSITIONAL ANALYSIS

3.1 Creep Analysis using TMA

The Coflon films described in Section 1.0 were subjected to creep testing in TRI's 943 Thermomechanical Analyzer (TMA) after aging in methanol/EDA. The films were cut into 0.15 inch (3.8 mm) widths. The specimens were mounted in a tensile fixture. A dead load was applied sufficient to impose a stress of 500 psi. The TMA furnace was then placed around the sample compartment. The sample temperature was ramped up 10°C per minute to 150°C. In this manner the creep behavior was observed while the specimen was undergoing an increase in temperature.

Appendix Figure 4.1 displays the results for the unaged Coflon and three of the aged specimens. The specimen which was aged for 17 hours was too brittle to
fixture and is not indicated in Appendix Figure 4.1 for this reason. A significant increase in the resistance to creep with temperature was noted in the aged specimens. This behavior may be a result of dehydrofluorination and possible subsequent crosslinking within the polymer.

3.2 DSC of Aged Materials

A separate set of PVDF films was prepared for the DSC tests. The films were prepared as described in Section 1.0 with the exception of having a slightly thicker crosssection. These specimens were again exposed to methanol/EDA for 0.25, 1.0, and 15 hours respectively. After exposure, sections of the films were subjected to Differential Scanning Calorimetry (DSC) testing. Sections were cut from the dry aged films and placed into tared DSC sample pans. The sample temperature was raised at a constant rate of 10°C per minute to a maximum of 200°C. Melt onset onset, peak temperature, and heat of fusion were thus determined.

DSC Results

DSC plots are displayed in Appendix 4.2. Tabular data describing the melt aspects and heats of fusion is shown in Table 3.1.

<table>
<thead>
<tr>
<th>Coflon Film Sample</th>
<th>Melt Onset (°C)</th>
<th>Melt Peak (°C)</th>
<th>Heat of Fusion (J/g)</th>
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<tr>
<td>Baseline</td>
<td>161.45</td>
<td>173.74</td>
<td>58.49</td>
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<tr>
<td>Aged 0.25 hrs</td>
<td>162.11</td>
<td>177.12</td>
<td>56.89</td>
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<tr>
<td>Aged 1.0 hrs</td>
<td>160.76</td>
<td>174.55</td>
<td>68.21</td>
</tr>
<tr>
<td>Aged 15 hrs</td>
<td>153.87</td>
<td>169.39</td>
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</table>

A slight increase in melt temperatures was noted with the 0.25 hour aged specimen. At the 1 hour and 15 hour exposure level the temperatures appear to be decreasing. Most notable are the values obtained from the specimen which was aged 15 hours. Here the melt onset and peak temperatures dropped significantly as did the heat of fusion. This results lends credibility to the hypothesis that initial aging of PVDF in this fluid causes some crosslinking and at longer exposure times lower molecular weight species begin to appear which may lower the melt points and heats of fusion.
4.0 Appendix

4.1 TMA
Creep Behavior of Coflon Films
Aged in Methanol + 1% EDA

Percent Strain

Temperature (C)
4.2 FTIR and Ultraviolet Absorption Plots
FTIR Spectra
Coflon Film Unaged Baseline

Absorbance

Wavenumber (cm⁻¹)
FTIR Spectra
Coflon Film Aged 1 hr MeOH + 1% EDA Reflux
ITIR Spectra
Coflon Film Aged 2 hrs MeOH + 1% EDA Reflux

Wavenumber (cm⁻¹)

Absorbance

3900 3400 2900 2400 1900 1400 900 400
FTIR Spectra
Coflon Film Aged 3 hrs MeOH + 1% EDA Reflux
FTIR Spectra
Coflon Film Aged 4 hrs MeOH + 1% EDA Reflux
FTIR Spectra
Coflon Film Aged 17 hrs MeOH + 1% EDA Reflux

Absorbance

Wavenumber (cm\(^{-1}\))

3900 3400 2900 2400 1900 1400 900 400
Aged Coflon Films
Changes in UV Absorption at 290 nm
After Exposure to Methanol + 1% EDA

[Graph showing the percentage transmission at 290 nm over exposure time (hrs). The graph indicates a decrease in transmission from 38% to 35% over a 20-hour exposure period.]
4.2 DSC Plots
4.3 X-Ray Diffraction Plots
This plot shows differences between earlier & current T90
4.4  GC/Mass Spectrometry Chromatograms T-89
Operator: Don Clay  
Acquired: 4 Dec 96 5:09 pm using AcqMethod MEOHSCAN.M  
Sample Name: MeOH extract of PVDF 12/04/96  

### 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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Abundance

#35446: Decanedioic acid, dibutyl ester (*)

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tefzel
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| -- 1+2+3 LEVEL FINDS*------------------- | 0 | O7HLR-- | 0 |
| -- 1+2+3+4 LEVEL FINDS*----------------- | 0 | | |
CHEMICAL REACTIONS, AUSTRALIA.

CHEMICAL REACTANTS, FLUOROSILICONE SEALANT, FLUOROPOLYMERS, POLYMERS, DEGRADATION.

-- 24 - DESCRIPTOR CLASSIFICATION: UNCLASSIFIED
-- 25 - IDENTIFIERS: *WING FUEL TANKS, Q4-2817 FLUOROSILICONE SEALANT, RAAF F-111 AIRCRAFT, F-111 AIRCRAFT.
-- 26 - IDENTIFIER CLASSIFICATION: UNCLASSIFIED
-- 29 - INITIAL INVENTORY: 2
-- 32 - REREGRADE CATEGORY: F
-- 33 - LIMITATION CODES: 14
-- 35 - SOURCE CODE: 409014
-- 36 - ITEM LOCATION: DITIC
-- 40 - GEOGRAPHICAL CODE: AU
-- 41 - TYPE CODE: 8
-- 43 - IAC DOCUMENT TYPE:

******

-- 2 - OF 5
-- 1 - AD NUMBER: B209155
-- 2 - FIELDS AND GROUPS: 11/9, 11/10, 18/6
-- 3 - ENTRY CLASSIFICATION: UNCLASSIFIED
-- 5 - CORPORATE AUTHOR: BATTELLE MEMORIAL INST COLUMBUS OH RADIATION EFFECTS INFORMATION CENTER
-- 6 - UNCLASSIFIED TITLE: THE EFFECT OF NUCLEAR RADIATION ON FLUOROPOLYMERS.
-- 8 - ILL C CLASSIFICATION: UNCLASSIFIED

-- 9 - DESCRIPTIVE NOTE: MEMORANDUM KLP1.
-- 10 - PERSONAL AUTHORS: BROADWAY, N. J.; PALINCHAK, S.
-- 11 - REPORT DATE: JUN 30, 1989
-- 12 - PUBLICATION: 21 P MEDIA COST: $ 6.00 PRICE CODE: AA
-- 14 - REPORT NUMBER: NREL-TR-90-6
-- 16 - CONTRACT NUMBER: AF 33(616)-6564
-- 18 - MONITOR ACRONYM: XC
-- 19 - MONITOR SKILLS: USAF
-- 20 - REPORT CLASSIFICATION: UNCLASSIFIED
BECAUSE OF THEIR CHEMICAL INERTNESS, HIGH-TEMPERATURE RESISTANCE, AND GOOD ELECTRICAL PROPERTIES, SINCE THEY ARE RESISTANT TO FUMING NITRIC ACID AND OTHER OXIDIZING AGENTS, SUCH MATERIALS AS TEFLON AND KEL-F ARE ESSENTIAL MATERIALS FOR VALVES, HOSES, AND ELECTRICAL INSULATION. THEIR HEAT RESISTANCE HAS MADE THEM INVALUABLE FOR HIGH-TEMPERATURE APPLICATIONS. THIS RESISTANCE TO HEAT AND CHEMICALS IS DUE TO THE CARBON-FLUORINE BOND WHICH IS CHEMICALLY ONE OF THE MOST STABLE OF ORGANIC LINKAGES. BECAUSE OF THEIR IMPORTANT APPLICATIONS, THESE MATERIALS WERE INVESTIGATED FOR USE IN A RADIATION ENVIRONMENT. IT WAS FOUND THAT THE RADIATION RESISTANCE OF FLUOROPOLYMERS IS NOT AS GOOD AS THEIR CHEMICAL AND HEAT RESISTANCE. TEFLON HAS VERY POOR RADIATION RESISTANCE, WHILE THE OTHER FLUOROPOLYMERS ARE ONLY SLIGHTLY BETTER. THIS MEMORANDUM SUMMARIZES THE AVAILABLE DATA ON THE RADIATION STABILITY OF TEFLON AND KEL-F PLASTICS, AND KEL-F, POLYFLUOROBUTYL ACrylate (POLYFBA) HEXAFLUOROPENTAMETHYLENE ADIPATE (A POLYESTER), AND SILASTIC LS-53 (A FLUORINATED SILICONE) ELASTOMERS.

ABSTRACT: FLUOROPOLYMERS ARE USEFUL IN MISSILE CONSTRUCTION.
--16 - PROJECT NUMBER: 3005
--17 - TASK NUMBER: 50
--18 - MONITOR ACRONYM: WRDC

--19 - MONITOR SERIES: IR-69-4042
--20 - REPORT CLASSIFICATION: UNCLASSIFIED
--21 - SUPPLEMENTARY NOTE: ORIGINAL CONTAINS COLOR PLATES: ALL DTIC
-- REPRODUCTIONS WILL BE IN BLACK AND WHITE.
--22 - LIMITATIONS (ALPHA): DISTRIBUTION AUTHORIZED TO U.S. GOV'T.
-- AGENCIES ONLY: SPECIFIC AUTHORITY: PROPRIETARY INFO.: FEB 89. OTHER
-- REQUES BY SHALL BE REFERRED TO WRDC/MLDC, WRIGHT-PATTERSON AFB, OH
-- 45433-6533. THIS DOCUMENT CONTAINS EXPORT-CONTROLLED TECHNICAL DATA.
--23 - DESCRIPTORS: *BIODEGRADATION, *RUBBER COATINGS, *ELASTOMERS,
-- *MICROORGANISMS, *PAINTS, *SEALING COMPOUNDS, AGAR, AIR FORCE,
-- AIRCRAFT, BIOLOGY, CHEMICALS, COMPOSITE MAILKIALS, CORROSION
-- INHIBITION, DAMAGE, DEGRADATION, DETERIORATION, EFFICIENCY, ENZYMES,
-- FLUORINE COMPOUNDS, FUNGICIDES, GERMICIDES, HAZARDS, MAINTENANCE,
-- METABOLISM, NUTRIENTS, POLYSULFIDES, REMOVAL, REPAIR, SILICONES,
-- SOUP, SURFACES, TELFON, FLUOROPOLYMERES.
--24 - DESCRIPTOR CLASSIFICATION: UNCLASSIFIED
--25 - IDENTIFIERS: PL65502F, WWWD3055500, EXPORT CONTROL
-- FLUOROSILICONE POLYSULFIDE, POLYSULFIDE ELASTOMERS.
--26 - IDENTIFIER CLASSIFICATION: UNCLASSIFIED
--29 - INITIAL INVENTORY: 1
--33 - LIMITATION COUS: 3 57
--34 - SOURCE SERIES: F
--35 - SOURCE CODE: 397355

--36 - ITEM LOCATION: DTIC
--40 - GEOPOlITICAL CODE: 4902
--41 - TYPE CODE: 4
--42 - IAC ACCESSION NUMBER: PL-053019
--43 - IAC DOCUMENT TYPE:
-- PLASTEC-MICROFICHE
--44 - IAC SUBJECT TERMS: --CORROSION RESISTANCE, MICROBIOLOGICAL
-- DETERIORATION, BIODEGRADATION, POLYSULFIDES, FLUOROSILICONES,
-- SEALANTS, FLUOROCARBONS, MONITORING, DEGRADATION, FOURIER TRANSFORM
-- IR, SPECTROSCOPY, PAINTS, ENZYMES DEGRADATION, TELFON, ELASTOMERS,
-- PIPE, COATINGS, ZZ CONTROLLED USGO.;

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-- 4 OF 5
-- 1 - AD NUMBER: A294734
-- 2 - FIELDS AND GROUPS: 7/3, 7/4, 7/6, 8/8
-- 24/4
-- 3 - ENTRY CLASSIFICATION: UNCLASSIFIED
-- 5 - CORPORATE AUTHOR: COLD REGIONS RESEARCH AND ENGINEERING LAB
-- HANOVER NH
-- 6 - UNCLASSIFIED TITLE: SUSCEPTIBILITY OF ABS, FEP, TFE, FRP, PTFE,
-- AND PVC WELL CASINGS TO DEGRADATION BY CHEMICALS.
-- 8 - TITLE CLASSIFICATION: UNCLASSIFIED
-- 9 - DESCRIPTIVE NOTE: SPECIAL REP'T.

--10 - PERSONAL AUTHORS: KANNELY, THOMAS A.; PARKER, LOUISE V.
--11 - REPORT DATE: JAN, 1995
--12 - PAGINATION: 24 P. MLUVA COPY: 1 6.00 PRICE CODE: AA
--14 - REPORT NUMBER: CRREL-SR-95-1
--18 - MONITOR ACRONYM: 3FIM-ALC-LT, XA
LESS COMMONLY USED MATERIALS FOR CASING GROUNDWATER MONITORING WELLS: ACRYLONITRILE BUTADIENE STYRENE (ABS), FLUORINATED ETHYLENE PROPYLENE (FEP), FIBERGLASS-REINFORCED EPOXY (FRE), AND FIBERGLASS-REINFORCED PLASTIC (FRP), WITH TWO MORE COMMONLY USED CASING MATERIALS: POLYVINYL CHLORIDE (PVC) AND POLYTETRAFLUROETHYLENE (PTFE). THE SIX MATERIALS WERE EXPOSED TO 28 NEAR ORGANIC COMPOUNDS (INCLUDING ONE ACID) AND 10 EXTREMELY ACIDIC AND ALKALINE CONDITIONS FOR UP TO 112 DAYS. THIS WAS DONE TO SIMULATE SOME OF THE MOST AGGRESSIVE ENVIRONMENTS THAT MONITORING WELL CASINGS MAY BE EXPOSED TO. THE CASINGS WERE OBSERVED FOR CHANGES IN WEIGHT AND SIGNS OF PHYSICAL DEGRADATION (SWELLING, SOFTENING, DECREASE IN STRENGTH, DETERIORATION, OR DISSOLUTION). AS EXPECTED, THE TWO FLUORINATED POLYMERS (FEP AND PTFE) WERE THE MOST INERT MATERIALS TESTED. THEY WERE NOT DEGRADED BY ANY OF THE TEST CHEMICALS, ALTHOUGH SAMPLES EXPOSED TO A FEW ORGANIC CHEMICALS DID SHOW A SLIGHT WEIGHT GAIN (APPROX. 1%). AMONG THE NONFLUORINATED PRODUCTS TESTED, FRE WAS THE MOST INERT. THREE ORGANIC CHEMICALS CAUSED PARTICLES TO FLAKE FROM THE FRE SURFACE, FOLLOWED BY SEPARATION OF THE GLASS FIBERS, AND TWO ORGANIC CHEMICALS CAUSED WEIGHT GAINS EXCEEDING 10%. ALSO, HIGHLY ACIDIC CONDITIONS (pH<1) DEGRADED THIS MATERIAL, AND THIS MAY LIMIT THE USE OF THIS MATERIAL IN ACIDIC ENVIRONMENTS. JG

ABSTRACT: THIS STUDY COMPARES THE CHEMICAL RESISTANCE OF FOUR...
EPOXY RESINS: NAVAL EXPERIMENTAL RESIN C8/ISA AS A STRUCTURAL
MATERIAL AND FOR USE IN BLENDS AND COMPOSITES.

- TITLE CLASSIFICATION: UNCLASSIFIED
- DESCRIPTIVE NOTE: TECHNICAL REPORT.

- PERSONAL AUTHORS: TWARDOWSKI, T. E.; GEIL, P. H.

- REPORT DATE: JUN 01, 1990

- MEDIA COST: $ 6.00

- REPORT NUMBER: UIUC-NCAR-89-0019

- REPORT ACKNOWLEDGMENT: XN

- REPORT CLASSIFICATION: UNCLASSIFIED

- DESCRIPTORS: ADHESION, COMPOSITE MATERIALS, CONSTRUCTION
  MATERIALS, DEGRADATION, ENVIRONMENTS, EPOXY COMPOUNDS, EPOXY RESINS,
  FILLERS, FLAMES, FLUOROPOLYMERS, LIGHTWEIGHT, LOW ANGLES, MATERIALS,
  MEASUREMENT, MODIFICATION, POLYMERS, PROCESSING, RESISTANCE,
  SIZES (DIMENSIONS), STABILITY, THERMAL STABILITY, THERMOPLASTIC
  RESINS, WATER.

- DESCRIPTOR CLASSIFICATION: UNCLASSIFIED

- IDENTIFIERS: EPOXY RESINS.

- IDENTIFIER CLASSIFICATION: UNCLASSIFIED

- ABSTRACT: EPOXY THERMOSETS ARE CURRENTLY THE MATERIAL OF CHOICE
  FOR HIGH PERFORMANCE COMPOSITE MATERIALS. THEY HAVE HIGH MODULUS,
  LOW WEIGHT, EXCELLENT ADHESION TO THE FILLER COMPONENT, AND HIGH
  DIMENSIONAL AND THERMAL STABILITY AS WELL AS PROCESSING
  CHARACTERISTICS MUCH MORE TRACTABLE THAN THERMOPLASTICS.
  UNFORTUNATELY, EPOXIES ARE OFTEN ATTACKED BY ENVIRONMENTAL ELEMENTS,
  ESPECIALLY WATER, RESULTING IN A DEGRADATION OF PROPERTIES. AS SUCH,
  MODIFICATION OF EPOXIES TO IMPROVE THEIR ENVIRONMENTAL RESISTANCE
  IS VALUABLE CONSIDERATION. EPOXY RESINS INCORPORATING LARGE
  FLUORINE CONTENTS HAVE MEET THIS CHALLENGE, SHOWING LOW CONTACT
  ANGLE, LOW MEASURE UPTAKE AND IMPROVED FLAME RESISTANCE. WHAT

REMAINS IS THE NEED TO INVESTIGATE THE SUITABILITY OF SUCH RESINS
FOR USE IN STRUCTURAL MATERIALS.

- ABSTRACT CLASSIFICATION: UNCLASSIFIED

- INITIAL INVENTORY: 2

- LIMITATION CODES: 1

- SOURCE CODE: 422821

- ITEM LOCATION: UIUC/N11S

- GEOGRAPHICAL CODE: 1719

- TYPX CODE: 0

- IAC ACCESSION NUMBER: PL-053137

- IAC DOCUMENT TYPE:
  PLASTEC-MICROFICHE

- IAC SUBJECT TERMS: (U) MODIFIER INTERFACIAL STRENGTH,
  INTERFACE STRENGTH, CHARACTERIZATION, FLUORINATED EPOXY,
  GLASS TRANSITION, DSC,
  MOISTURE ABSORPTION, BLENDS, COMPOSITES, EPOXY 828, SWELLING, EPOXY
  828, AGING, STRESS STRAIN, ADHESION, THERMAL STABILITY, PROPERTY
  DEGRADATION, WATER IMMERSION, TENSILE PROPERTIES, ZZ UNLIMITED.

<<ENTER NEXT COMMAND>>
### Status: Path 1 of [Dialog]

### Status: Initializing Port COM2 using (Baud 19200 Handshake XON/XOFF DataBits 7 Parity Even StopBits 1)

### Status: Initializing modem ...
ATEI0V1X4&C1&D2
OK
### Status: Dialing primary number (448-4611)...
ATDT448-4611
PROTOCOL: LAP-M

COMPRESSION: NONE

CONNECT 19200
### Status: Connection established at 19200 baud
/ARQ

please type your terminal identifier
-3523:01-005-
please log in: DIALOG

DIALOG: call connected
### Status: Connected

DIALOG INFORMATION SERVICES
PLEASE LOGON:
******** HHHHHHHH SSSSSSSS?
### Status: Signing onto Dialog
********
ENTER PASSWORD:
******** HHHHHHHH SSSSSSSS? ********
Welcome to DIALOG
### Status: Connected

Dialog level 42.10.06D

Last logoff: 22nov96 09:36:40
Logon file405 22nov96 10:54:50
Banner display set OFF.
HILIGHT set on as '*'
KWIC is set to 5.
BLIP set on
NOTICE set ON to $25.00
You will be prompted to confirm each TYPE or PRINT request where
format charges exceeds $25.00
COST = ONESEARCH.
Please enter SUBACCOUNT name/number:
?bulluck/n7402
Is BULLUCK/N7402 the SUBACCOUNT you want to use? (Y/N)
?y
Subaccount is set to BULLUCK/N7402
SYSTEM: HOME
Menu System II: D2 version 1.7.8 term=ASCII
Terminal set to DLINK

*** DIALOG HOMEBASE(SM) Main Menu ***

Information:
1. Announcements (new files, free connect time, price changes, etc.)
2. Database, Rates, & Command Descriptions
3. Help in Choosing Databases for Your Topic
4. Customer Services (telephone assistance, training, seminars, etc.)
5. Product Descriptions
Connections:
6. DIALOG Menus(SM)
7. DIALOG Business Connection(R) and DIALOG Headlines(SM)
8. KR SourceOne(SM) Document Delivery
9. Data Star(R)
10. Other Online Menu Services & Files (MoneyCenter(R), OAG, TNT, etc.)

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/H = Help      /L = Logoff      /NOMENU = Command Mode

Enter an option number to view information or to connect to an online service. Enter a BEGIN command plus a file number to search a database (e.g., B1 for ERIC).

File 411:

DIALINDEX(R)

(c) 1996 Knight-Ridder Info

*** DIALINDEX search results display in an abbreviated ***
*** format unless you enter the SET DETAIL ON command. ***

?schemlit, chemeng, plastics, material

You have 43 files in your file list.
(To see banners, use SHOW FILES command)

?s ((pvdf or coflon or polyvinylidene()fluoride or tefzel) and (flexible()pip? or degradation))/ti,de,id

Your SELECT statement is:

s ((pvdf or coflon or polyvinylidene()fluoride or tefzel) and (flexible()pip? or degradation))/ti,de,id

<table>
<thead>
<tr>
<th>Items</th>
<th>File</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>INSPEC_1969-1996/Nov W3</td>
</tr>
<tr>
<td>10</td>
<td>EI Compendex(R)_1970-1996/Dec W4</td>
</tr>
<tr>
<td>31</td>
<td>World Surface Coatings Abs_1976-1996/Oct</td>
</tr>
</tbody>
</table>

>>>Term "ID" is not defined in file 144 and is ignored

14 144: Pascal_1973-1996/Oct

>>>Term "ID" is not defined in file 322 and is ignored

1 322: Polymer Online

>>>Term "DE" is not defined in file 340 and is ignored


>>>Term "ID" is not defined in file 340 and is ignored

2 340: CLAIMS(R)/US PATENTS ABS_1950-1996/SEP

>>>File 399 processing for PIP? stopped at

PIPERAZINYLALKANOYLIDHYDROBENZOFURANS

84 399: CA SEARCH(R)_1967-1996/UD=12522


6 6: NTIS 64-1996/Dec W5

2 293: Eng Materials Abs(R)_1986-1996/Dec

>>>Term "ID" is not defined in file 108 and is ignored

1 108: Aerospace Database_1962-1996/Nov

13 files have one or more items; file list includes 43 files. One or more terms were invalid in 26 files.
22nov96 10:57:23 User036172 Session D803.2
Sub account: BULLUCK/N7402
$1.50 Estimated cost File411
$0.60 TYMNET
$2.10 Estimated cost this search
$2.11 Estimated total session cost 0.051 Hrs.

SYSTEM:OS - DIALOG OneSearch
*File 399: Use is subject to the terms of your user/customer agreement. For format prices, including formats 6 & 8, see HELP RATES 399.
You have 13 files in your file list.
(To see file names, coverage dates, and copyright notices, enter SHOW FILES.)

Set Items Description
------- -------

?exs td228
Hilight option is not available in file(s) 6, 293, 399
HILIGHT set on as '**'
KWIC is set to 5.
KWIC option is not available in file(s) 6, 293, 399
>>>Term "ID" is not defined in one or more files
>>>Term "DE" is not defined in one or more files
>>>File 399 processing for PIP? stopped at PIPERAZINYLALKANOYLDIHYDROBENZO
FURANS
Processed 10 of 13 files ...
Completed processing all files
4969 PVDF/TI, DE, ID
 3 COFLON/TI, DE, ID
12502 POLYVINYLIDENE/TI, DE, ID
194611 FLUORIDE/TI, DE, ID
 9055 POLYVINYLIDENE/TI, DE, ID(W) FLUORIDE/TI, DE, ID
 141 TEFZEL/TI, DE, ID
121920 FLEXIBLE/TI, DE, ID
365537 PIP?/TI, DE, ID
 900 FLEXIBLE/TI, DE, ID(W) PIP?/TI, DE, ID
268842 DEGRADATION/TI, DE, ID
 272 ((PVDF OR COFLON OR POLYVINYLIDENE) FLUORIDE OR TEFZEL) AND (FLEXIBLE() PIP? OR DEGRADATION))/TI, DE, ID

?rd s1
>>>Duplicate detection is not supported for File 94.
>>>Duplicate detection is not supported for File 322.
>>>Duplicate detection is not supported for File 340.

>>>Records from unsupported files will be retained in the RD set.
...examined 50 records (50)
...examined 50 records (100)
...examined 50 records (150)
...examined 50 records (200)
...examined 50 records (250)
...completed examining records
S2 253 RD S1 (unique items)
?s s2/eng
>>>Term "ENG" is not defined in one or more files
S3 183 S2/ENG
?s s3 and py=1992:8888
>>>One or more prefixes are unsupported
>>> or undefined in one or more files.
Processed 10 of 13 files ...
Completed processing all files
 183 S3
13684327 PY=1992 : PY=8888
4/6/1  (Item 1 from file: 8)
04187181
Title: Piezo- and pyroelectric properties of dehydrofluorinated *PVDF* films
Conference Title: Proceedings of the 8th International Symposium on Electrets (ISE 8)

4/6/2  (Item 2 from file: 8)
03681363
Title: Advanced *flexible* *pipe* materials for aggressive hydrocarbon service

4/6/3  (Item 3 from file: 8)
03641106
Title: Application of TGA/FTIR to the thermal *degradation* mechanism of tetrafluoroethylene-propylene copolymers

4/6/4  (Item 4 from file: 8)
03623563
Title: XPS studies of radiation-induced structural changes in *polyvinylidene* *fluoride*

4/6/5  (Item 5 from file: 8)
03423047
Title: X-ray induced *degradation* of poly(vinylidene fluoride) films.

4/6/6  (Item 1 from file: 94)
03333595  JICST ACCESSION NUMBER: 96A0668980  FILE SEGMENT: JICST-E
Systematic Peptide Fragmentation of Polyvinylidene Difluoride(*PVDF* )-Immobilizes Proteins Prior to Microsequencing.

4/6/7  (Item 1 from file: 144)
12383888  PASCAL No.: 96-0030620
Thermal decomposition kinetics of a commercial fluropolymer

4/6/8  (Item 2 from file: 144)
12251841  PASCAL No.: 95-0476866
KeV ion beam irradiation of *polyvinylidene* *fluoride* (*PVDF*)

4/6/9  (Item 3 from file: 144)
12251412  PASCAL No.: 95-0476413
Temperature influence on the gas desorption from *polyvinylidene* *fluoride* (*PVDF*) irradiated with helium beams

4/6/10  (Item 4 from file: 144)
11583591  PASCAL No.: 94-0469531
A FTIR study of *PVDF* irradiated by means of swift heavy ions

4/6/11  (Item 5 from file: 144)
11504125  PASCAL No.: 94-0344727
Utilization of *PVDF* sensors to determine impact damage in graphite/epoxy plates by acousto-ultrasonic technique
Monosaccharide and oligosaccharide analysis of glycoproteins electrotransferred onto PVDF membranes

Retention of beer spoilage microorganisms by polyvinylidene fluoride microporous membranes with various retention ratings

TITLE: FAMILY FORTUNES

TITLE: RADIATION EFFECTS ON FLUOROPOLYMERS: RADIATION-INDUCED STRUCTURAL AND CRYSTALLINITY CHANGES OF TEFZEL

TITLE: COMPARISON OF HIGH PERFORMANCE, CLEAR FILMS USED FOR LONG TERM PRODUCTION

TITLE: POLYMER COMPOSITIONS INTENDED FOR THE MANUFACTURE OF CABLES AND FLEXIBLE PIPES AND ARTICLES BASED ON THESE COMPOSITIONS

TITLE: ACID-BASE INTERACTIONS AT POLYMER INTERFACES

TITLE: CRYSTALLISATION BEHAVIOUR AND PHASE DIAGRAM OF EXTENDED-CHAIN CRYSTALS OF POLY(VINYLIDENE FLUORIDE) UNDER HIGH PRESSURE

TITLE: RADIATION-INDUCED ENHANCEMENT OF CRYSTALLINITY IN POLYMERS

TITLE: ELECTROCHEMISTRY AS THE WAY TO TRANSFORM POLYMERS

TITLE: ELECTROCHEMICALLY INDUCED FUNCTIONALISATION OF FLUOROCONTAINING POLYOLEFINs

TITLE: BLENDS OF GLYCIDYL METHACRYLATE(GMA)/METHYL METHACRYLATE(MMA)
COPOLYMERS WITH *POLYVINYLIDENE* *FLUORIDE*

4/6/24 (Item 11 from file: 323)
00554472
TITLE: POLYMER SUPPORTS IN SYNTHESIS

4/6/25 (Item 12 from file: 323)
00549288
TITLE: YIELD BEHAVIOUR OF *PVDF* AND THE DEFORMATION PROCESS AT HIGH TEMPERATURE

4/6/26 (Item 13 from file: 323)
00509576
TITLE: STUDY OF THE THERMAL *DEGRADATION* OF POLYCHLOROTRIFLUOROETHYLENE, *POLYVINYLIDENE* *FLUORIDE* AND COPOLYMERS OF CHLOROTRIFLUOROETHYLENE AND VINYLIDENE FLUORIDE

4/6/27 (Item 14 from file: 323)
00496424
TITLE: RADIATION EFFECTS ON *PVDF*

4/6/28 (Item 15 from file: 323)
00492182
TITLE: GRAFTING OF SILICON PHTHALOCYANINE DICHLORIDE ONTO *PVDF* FILM SURFACES BY ULTRASOUND

4/6/29 (Item 16 from file: 323)
00474223
TITLE: PLASTICS HEAT EXCHANGERS

4/6/30 (Item 17 from file: 323)
00472912
TITLE: SEEKING THE PERFECT BLEND

4/6/31 (Item 18 from file: 323)
00470711
TITLE: X-RAY PHOTOELECTRON SPECTROSCOPY (XPS) STUDIES OF RADIATION-INDUCED STRUCTURAL CHANGES IN *POLYVINYLIDENE* *FLUORIDE*

4/6/32 (Item 19 from file: 323)
00448524
TITLE: REACTIONS OF ATOMIC OXYGEN WITH POLYMER FILMS

4/6/33 (Item 1 from file: 340)
2620850 9516422
C/POLYMER COMPOSITIONS INTENDED FOR THE MANUFACTURE OF CABLES AND *FLEXIBLE* *PIPES* AND ARTICLES BASED ON THESE COMPOSITIONS; *POLYVINYLIDENE* *FLUORIDE* BLEND

4/6/34 (Item 2 from file: 340)
2439164 9402057
C/METHOD FOR EXTRUDING POLYOLEFINS CONTAINING VINYLIDENE CHLORIDE POLYMERS; COATING EQUIPMENT WITH *POLYVINYLIDENE* *FLUORIDE* TO PREVENT *DEGRADATION* OF THE CHLOROPOLYMER
KeV-MeV ion irradiation of polyvinylidene fluoride (PVDF) films

Photodegradation of fluorocarbon resin films. A new evaluation method using an ESR spectrometer

Testing the utility of EIS measurements to predict and monitor the behavior of organically coated aluminum during atmospheric exposure

Swift heavy ion modification of polymers

Radical in heavy ion-irradiated polyvinylidene fluoride

Photoacoustic detection of the decomposition kinetics of polymers: interpretation of acoustic signals

Crystallite damage studies on irradiated poly(vinylidene fluoride)

Derivatization of polyvinylidene difluoride membranes for the solid-phase sequence analysis of a phosphorylated sea urchin embryo histone H1 peptide

Surface analysis of poly(vinylidene difluoride) membranes

Low dose γ-irradiation of some fluoropolymers: effect of polymer chemical structure
### Status: Path 1 of [Dialog]

### Status: Initializing Port COM2 using (Baud 19200 Handshake XON/XOFF DataBits 7 Parity Even StopBits 1)

### Status: Initializing modem ...
ATEIQOV1X4&Cl&02
OK
### Status: Dialing primary number (448-4611)...
ATDT448-4611
PROTOCOL: LAP-M

COMPRESS: NONE

CONNECT 19200
### Status: Connection established at 19200 baud

/ARQ

please type your terminal identifier
-3523:01-004-
please log in: DIALOG

DIALOG: call connected

### Status: Connected

DIALOG INFORMATION SERVICES
PLEASE LOGON:
******** HHHHHHHH SSSSSSSS?
### Status: Signing onto Dialog
********
ENTER PASSWORD:
******** HHHHHHHH SSSSSSSS? ********
Welcome to DIALOG

### Status: Connected

Dialog level 42.10.06D

Last logoff: 22nov96 11:01:38
Logon file405 22nov96 15:00:32
Banner display set OFF.
HILIGHT set on as '*'
KWIC is set to 5.
BLIP set on
NOTICE set ON to $25.00
You will be prompted to confirm each TYPE or PRINT request where format charges exceeds $25.00
COST = ONESEARCH.
Please enter SUBACCOUNT name/number:
?bulluck/n7402
Is BULLUCK/N7402 the SUBACCOUNT you want to use? (Y/N)
?y
Subaccount is set to BULLUCK/N7402
SYSTEM: HOME

Menu System II: D2 version 1.7.8 term=ASCII
Terminal set to DLINK

*** DIALOG HOMEBASE(SM) Main Menu ***

Information:
1. Announcements (new files, free connect time, price changes, etc.)
2. Database, Rates, & Command Descriptions
3. Help in Choosing Databases for Your Topic
4. Customer Services (telephone assistance, training, seminars, etc.)
5. Product Descriptions
Connections:
6. DIALOG Menus(SM)
7. DIALOG Business Connection(R) and DIALOG Headlines(SM)
8. KR SourceOne(SM) Document Delivery
9. Data Star(R)
10. Other Online Menu Services & Files (MoneyCenter(R), OAG, TNT, etc.)

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Enter an option number to view information or to connect to an online service. Enter a BEGIN command plus a file number to search a database (e.g., B1 for ERIC).

File 8:Ei Compendex(R) 1970-1996/Dec W4
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Set Items Description
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?k 03681363;k 03641106
S0 1 03681363
S0 2 03641106
?t 0/5/all

0/5/1
DIALOG(R) File 8:Ei Compendex(R)
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03681363 E.I. No: EIP93081046180
Title: Advanced flexible pipe materials for aggressive hydrocarbon service
Author: Hill, R.T.; Measamer, J.C.
Corporate Source: Wellstream Corp, Panama City, FL, USA
Conference Location: Singapore, Singapore Conference Date: 19930606-19930611
E.I. Conference No.: 18719
Publication Year: 1993
Language: English
Document Type: CA; (Conference Article) Treatment: X; (Experimental)
Journal Announcement: 9309W4
Abstract: 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 \( \text{H}_2\text{S} \) and \( \text{CO}_2 \) gases. Conventional pipelines require 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 as 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 PVDF homopolymer fluid barrier material. (Author abstract) 7 Refs.

Descriptors: *Offshore pipelines; Submarine pipelines; Corrosion resistance; Seawater effects; Plastic pipe
Identifiers: Flexible pipes; Plasticized homopolymer; Corrosive fluids; PVDF homopolymer, Carcass layer

Classification Codes:
619.1.2 (Pipe Materials)
619.1 (Pipe, Piping & Pipelines); 539.2 (Corrosion Protection); 511.2 (Oil Field Equipment)
619 (Pipes, Tanks & Accessories); 539 (Metals Corrosion & Protection);
511 (Oil Field Equipment & Production Operations)
61 (PLANT & POWER ENGINEERING); 53 (METALLURGICAL ENGINEERING); 51 (PETROLEUM ENGINEERING)

0/5/2
DIALOG(R) File 8: Ei Compendex(R)
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03641106 E.I. No: EIP93050995198
Title: Application of TGA/FTIR to the thermal degradation mechanism of tetrafluoroethylene-propylene copolymers
Author: Schild, H.G.
Publication Year: 1993
CODEN: JPACF CT ISSN: 0887-624X
Language: English
Document Type: JA; (Journal Article) Treatment: A; (Applications); G;
(General Review); X; (Experimental)
Journal Announcement: 9307W4
Abstract: Aflas, produced by Asahi Glass Co. Ltd., Tokyo, Japan and marketed in the United States by 3M, is a high-performance elastomeric tetrafluoroethylene (TFE)/propylene copolymer with superior high temperature stability, electrical, and chemical resistance properties. (Edited author abstract) 14 Refs.
Descriptors: *Copolymers; Polypropylenes; Polytetrafluoroethylenes; Pyrolysis; Thermoanalysis; Infrared spectroscopy
Identifiers: Aflas; Tefzel; Thermogravimetric analysis; Fourier transform infrared spectroscopy; Tetrafluoroethylene; Reaction mechanism
Classification Codes:
815.1.1 (Organic Polymers)
815.1 (Polymeric Materials); 802.2 (Chemical Reactions); 801.1 (Chemistry, General)
815 (Plastics & Polymeric Materials); 802 (Chemical Apparatus & Plants);
801 (Chemical Analysis & Physical Chemistry)
81 (CHEMICAL PROCESS INDUSTRIES); 80 (CHEMICAL ENGINEERING)

File 144: Pascal 1973-1996/Oct
(c) 1996 INIST/CNRS
Set Items Description
??? ----- ------------
?k 12383888
SO 1 12383888
Thermal decomposition kinetics of a commercial fluoropolymer (Tefzel) in the temperature range 359-550 Degree C by thermogravimetry (TG) and 235-270 Degree C by differential scanning calorimetry (DSC) was investigated under nitrogen flux. The temperature range studied (225-550 Degree C) includes the range of recommended continuous use and processing temperature of these commercial resins. The activation energy (DELTA E) for decomposition was 53.33 k.cal mole SUP -1 in TG at 10 Degree C/min rate, whereas it was 9.49 k.cal mole SUP -1 in DSC. The effect of heating rate on TG and DSC thermograms was studied.

English Descriptors: Fluoroelastomer; Ethylene copolymer; Tetrafluoroethylene copolymer; Commercial form; Thermal degradation; Kinetics; Rate constant; Activation energy; Heating rate; Experimental study

French Descriptors: Caoutchouc fluor; Ethylene copolymere; Ethylene(tetrafluoro) copolymere; Forme commerciale; Degradation thermique; Cinetique; Constante vitesse; Energie activation; Vitesse chauffage; Etude experimentale; Tefzel

Classification Codes: 001D09D03D
ABSTRACT: Tensile yield stress measurements on PVDF were carried out, covering as many as seven decades of strain rates at temps. from -50 to 150°C. The data were analysed on the basis of four Ree-Eyring processes acting in parallel, three of which were identified. At high temps. and low strain rates, a threshold yield stress was observed. A model was proposed, permitting description of the yield behaviour both at the threshold and in its adjacent strain rate dependent range. It consisted of a modification of the Ree-Eyring theory using an asymmetrical free energy barrier for the rate process. The data were consistent with the above model, which implied that yielding occurred only when the stress was higher than a threshold. In this case, a rejuvenation (or de-ageing) effect should be assumed in the deformation process which could consist of a melting of a chain segment located between two folds. 21 refs.

SUBJECT HEADING (RAPRA): TENSILE PROPERTIES, PVDF; VINYLIDENE FLUORIDE POLYMERS, tensile properties

GEOGRAPHIC LOCATION: BELGIUM; EUROPEAN COMMUNITY; EUROPEAN UNION; WESTERN EUROPE

DESCRIPTORS: DATA; DEFORMATION; FLUOROPOLYMER; FREE ENERGY; GRAPH; HIGH TEMPERATURE; LOW TEMPERATURE; MECHANICAL PROPERTIES; MELTING; MODEL; PLASTIC; POLYVINYLIDENE FLUORIDE; PROPERTIES; PVDF; STRAIN RATE; TABLES; TECHNICAL; TEMPERATURE; TENSILE PROPERTIES; THEORY; THERMAL DEGRADATION; THERMOPLASTIC; YIELD; YIELD STRESS

RAPRA CLASSIFICATION CODE: 42C386; 95111

CATEGORY CODES: UG; KM

0/5/2
DIALOG(R) File 323:RAPRA Rubber & Plastics
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00509576
TITLE: STUDY OF THE THERMAL DEGRADATION OF POLYCHLOROTRIFLUOROETHYLENE, POLYVINYLIDENE FLUORIDE AND COPOLYMERS OF CHLOROTRIFLUOROETHYLENE AND VINYLIDENE FLUORIDE

AUTHOR(S): Zulfiqar S; Zulfiqar M; Rizvi M; Munir A; McNeill I C

CORPORATE SOURCE: Quaid-i-Azam, University; Glasgow, University


CODEN: PDSTDW JOURNAL ANNOUNCEMENT: 9406 RAPRA UPDATE: 9410

DOCUMENT TYPE: Journal

LANGUAGE: English

SUBFILE: (R) RAPRA

ABSTRACT: A systematic study of the thermal degradation of a series of homopolymers and copolymers of chlorotrifluoroethylene and vinylidene fluoride was carried out using TGA and thermal volatilisation analysis(TVA). Volatile products were separated by sub-ambient TVA and characterised by means of IR spectroscopy and mass spectrometry. On degradation, polychlorotrifluoroethylene gave the monomer as the major product and carbon dioxide, C2F2Cl2, C3F5Cl and C2F3Cl3 in traces. Polyvinylidene fluoride formed hydrogen fluoride in appreciable amount along with the monomer and C4H3F3. The copolymers showed a similar type of degradation pattern. The structural changes which took place during degradation were also studied and mechanisms of formation of the various products are discussed. 12 refs.

SUBJECT HEADING (RAPRA): DEGRADATION, thermal, PCTFE, PVDF; CHLOROTRIFLUOROETHYLENE POLYMERS, thermal degradation; VINYLIDENE FLUORIDE POLYMERS, thermal degradation

IDENTIFIERS (Non-Polymer Terms): CARBON DIOXIDE; CHLORINE COMPOUND; CHLOROPENTAFLUOROBUTANONE; DICHLOORODIFLUOROPROPANE; FLUORINE COMPOUND; HYDROFLUORIC ACID; HYDROGEN FLUORIDE; TRICHLOROTRIFLUOROPROPANE; TRIFLUOROBUTADIENE
Polymer compositions based on fluoropolymers intended especially for the manufacture of electrical cables and of flexible pipes, comprising, by weight, (A) from 25 to 75% of PVDF homopolymer; (B) from 25 to 75% of a thermoplastic copolymer of VF2 and of at least one other fluoromonomer, exhibiting a content of 5 to 25% of this other monomer. This other fluorocomonomer may be in particular CTFE (chlorotrifluoroethylene), HFP (hexafluoropropylene) or TrFE (trifluoroethylene). Articles produced from these compositions have good mechanical properties at low temperature.

Exemplary Claim:
1. A polymer composition based on polyvinylidene fluoride (PVDF) homopolymer and at least one fluorocopolymer, comprising: a mixture of fluoropolymers comprised of, by weight: (A) from about 25 to about 75% of at least one PVDF homopolymer; and (B) from about 25 to about 75% of at least one thermoplastic copolymer comprising vinylidene fluoride (VF2) and from about 5 to about 25% by weight of at least one other fluoromonomer selected from the group consisting of hexafluoropropylene and chlorotrifluoroethylene.
Photoacoustic detection of the decomposition kinetics of polymers: interpretation of acoustic signals

AUTHOR(S): Kukreja, L. M.; Hess, P.
LOCATION: Institute of Physical Chemistry, University of Heidelberg, Im Neuenheimer Feld 253, Heidelberg, Germany, 69120
PAGES: 399-402 CODEN: ASUSEE ISSN: 0169-4332 LANGUAGE: English
SECTION:
CA237004 Plastics Manufacture and Processing
CA273XXX Optical, Electron, and Mass Spectroscopy, and Other Related Properties

IDENTIFIERS: polyimide degrdn kinetics photoacoustic detection, PVDF piezoelec transducer polyimide ablation

DESCRIPTORS:
Photoacoustic effect...
in laser-induced ablation of polyimides
Polyethers, polyimide-, properties... Polyimides,polyether-, properties...
laser-induced degrdn. of, PVDF for photoacoustic detection of
Ablation, laser-induced... Polymer degradation, laser-induced...
of polyimides, photoacoustic detection of
Kinetics of polymer degradation, photochem....
with laser source, of polyimides, photoacoustic detection of

CAS REGISTRY NUMBERS:
25036-53-7 25038-81-7 laser-induced degrdn. of, PVDF for photoacoustic detection of
24937-79-9 piezoelec. foil transducer, for photoacoustic detection of polyimide degrdn.

File 399:CA SEARCH(R) 1967-1996/UD=12522
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*File 399: Use is subject to the terms of your user/customer agreement.
For format prices, including formats 6 & 8, see HELP RATES 399.

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Set Items Description
s (photoacoustic and decomposition and kinetics and polymers)/ti
s 2924 PHOTOACOUSTIC/TI
s 43550 DECOMPOSITION/TI (SEE ?IGNORE)
110434 KINETICS/TI
125120 POLYMERS/TI (SEE ?CLASS)
1 (PHOTOACOUSTIC AND DECOMPOSITION AND KINETICS AND POLYMERS)/TI

?t 1/9
Crystallite damage studies on irradiated poly(vinylidene fluoride)

AUTHOR(S): Zhao Zhudi; Chu Jin; Chen Xinfang
SECTION: CA 36005 Physical Properties of Synthetic High Polymers
IDENTIFIERS: PVDF crystallite radiation damage, polyvinylidene fluoride, crystallite irradiation
DESCRIPTORS: Crystallites... in poly(vinylidene fluoride), damage of, by irradiation. Polymer degradation, radiochem. of poly(vinylidene fluoride), crystallite damage in Crystallinity... Heat of fusion and Heat of freezing... Polymer morphology, cryst., spherulitic... of poly(vinylidene fluoride), radiation effect on
CAS REGISTRY NUMBERS: 24937-79-9 crystallite damage of irradiated surfaces and analysis and membranes
?s (surface and analysis and membranes)/ti
195990 SURFACE/TI (SEE ?IGNOTE)
306491 ANALYSIS/TI (SEE ?IGNOTE)
53770 MEMBRANES/TI
S4 20 (SURFACE AND ANALYSIS AND MEMBRANES)/TI
?s s4 and poly
20 S4
909548 POLY
S5 6 S4 AND POLY
?s S5 and vinylidene
6 S5
13851 VINYLIDENE
S6 1 S5 AND VINYLIDENE
?t 6/9

Surface analysis of poly(vinylidene difluoride) membranes

SECTION: CA 238003 Plastics Fabrication and Uses
IDENTIFIERS: PVDF membrane surface analysis spectroscopy
DESCRIPTORS:
Membranes, microporous...
  hydrophilic and hydrophobic PVDF, surface anal. of, by x-ray, IR and mass spectroscopy
Fluoropolymers...
  membranes, hydrophilic and hydrophobic, surface anal. of, by x-ray, IR and mass spectroscopy
Surface analysis...
  of hydrophilic and hydrophobic PVDF membranes
Polymer degradation, radiochem....
  of hydrophilic and hydrophobic PVDF membranes, by x-ray
Surface structure...
  of hydrophilic and hydrophobic PVDF membranes, surface anal. of, by x-ray, IR and mass spectroscopy
Mass spectra, secondary-ion...
  of hydrophilic and hydrophobic PVDF membranes, surface structure in relation to
CAS REGISTRY NUMBERS:
24937-79-9 membranes, hydrophilic and hydrophobic, surface anal. of, by x-ray and IR and mass spectroscopy

### Status: Signing Off...

logoff
22nov96 15:09:01 User036172 Session D804.6
Sub account: BULLUCK/N7402
  $12.00 0.100 Hrs File399
  $5.85 3 Types
  $17.85 Estimated cost File399
  $1.20 TYMNET
  $19.05 Estimated cost this search
  $34.15 Estimated total session cost 0.168 Hrs.

### Status: Signed Off. (9 minutes)