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**ENVIRONMENTAL
RESTORATION
PROGRAM**

**Data Analysis of a High-Density
Polyethylene Geomembrane Located
in Waste Area Grouping 6
at Oak Ridge National Laboratory**

M. Rochelle Martino

MANAGED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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Energy Systems Environmental Restoration Program
ORNL Environmental Restoration Program

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M. Rochelle Martino

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EXECUTIVE SUMMARY

The Environmental Restoration Program at Oak Ridge National Laboratory is monitoring the material properties of the High Density Polyethylene (HDPE) cap at Waste Area Grouping 6 for degradation due to exposure to the environment. This report compares data from the unexposed geomembrane from the manufacturer's Geomembrane Certificate of Analysis to data accumulated between March 1990 and March 1994 on selected mechanical, physical, and thermal properties. Additional data from the evaluation of the geomembrane's stress cracking resistance are used to make recommendations on further testing and monitoring of the HDPE material.

The material properties of tear resistance, uniaxial yield and break stress, density and carbon black content were within an acceptable range of the minimum average values recommended by National Sanitation Foundation International Standard 54-1993 and two manufacturers of HDPE: Gundle Lining Systems Inc., and National Seal Co. The latter three properties did not deteriorate over time when compared to the manufacturer's certificate of analysis. The results of the bent strip test showed failure of all specimens within 170 hours, compared to the recommended time of 1500 hours without brittle failure recommended by NSF. The Notched Constant Tensile Load test reported transition times of 7 and 8 hours for a passive and active sample of the exposed HDPE liner. The percent crystallinity was found to be within the typical range of 40-60% for 80-mil HDPE; however, samples with high crystallinity, greater than 50%, tend to have poor stress crack performance. This was reinforced by the geomembrane's poor performance in the stress crack resistance tests.

As a result of these data, the continued monitoring of the HDPE cap is recommended in order to determine the durability and longevity of the geomembrane material. Quality control measures must be enacted immediately if a definitive, scientific study is to be conducted that will be beneficial to manufacturers, installers, and users. The development of a reliable, reproducible data base will assist in predicting the performance of HDPE in other applications in the future.

1. INTRODUCTION

The Environmental Restoration Program at Oak Ridge National Laboratory is monitoring the High Density Polyethylene (HDPE) cap at Waste Area Grouping 6 for degradation because of exposure to the environment. These semiannual inspections, which began in 1990, are used to locate cracks, tears, seam failures, and punctures in the cap material. Supplementing the visual inspections is analysis of "sacrificial" liner samples for such physical properties as thickness, density, tear resistance, carbon black content, yield and break stress, yield and break elongation, shear, and peel. The bent strip test and the Notched Constant Tensile Load (NCTL) test, which were initiated in November 1993 by the Geosynthetic Research Institute (GRI) at Drexel University, are used to evaluate the stress crack resistance.

This report compares data from the unexposed geomembrane to data accumulated from March 1990 through March 1994 on selected mechanical, physical, and thermal properties of the HDPE cap material to determine if degradation occurred during the five years of exposure to the environment. Recommendations on further testing and monitoring of the HDPE material are made based on the results.

2. SITE DESCRIPTION

Waste Area Grouping 6, a waste disposal site at Oak Ridge National Laboratory, contains Solid Waste Storage Area (SWSA) 6, the Explosive Detonation Trench, and the Emergency Waste Basin (see Fig.1). SWSA 6 opened in 1969 and was used until 1986 as a shallow land burial site for solid wastes, including hazardous and radioactive materials disposed of in unlined trenches and auger holes. In May of 1986, SWSA 6 was temporarily closed because of the Resource Conservation and Recovery Act (RCRA) regulated wastes (lead, xylene, and toluene) disposed of at the site. It was reopened a few months later, but the presence of the RCRA-regulated materials necessitated closure under RCRA.

In 1989 an interim closure of SWSA 6 under RCRA was accomplished by placement of an 80-mil HDPE cover over 8 acres containing the RCRA waste. The life expectancy of the exposed cap was five years, so a postclosure surveillance and maintenance plan was developed for the monitoring of the cap's durability for that period.

3. METHODOLOGY

Between March 1990 and March 1994, semiannual samples from the exposed HDPE cap material have been analyzed by three different laboratories. The samples exhumed between March 1990 and September 1992 were analyzed by the same laboratory herein referred to as Lab 1. Samples from August 1993 and March 1994 were analyzed by a second laboratory, which will be designated as Lab 2. The samples from November 1993 were analyzed by GRI which is identified as Lab 3. All samples were exhumed from both the actual cap material—termed "active samples"—and the unconfined cap material—termed "passive samples". Whether all the testing laboratories received a passive and an active sample cannot be determined, except in the case of GRI, where one of each was definitely received. Samples from the geomembrane were analyzed by the manufacturer before installation and were documented on the manufacturer's Geomembrane Certificate of Analysis.

The samples were analyzed for mechanical, physical, and thermal properties. Mechanical properties include uniaxial yield stress and elongation, uniaxial break stress and elongation, tear resistance, and stress-crack resistance. Because of several instances of stress-cracking of the geomembrane, GRI initiated the evaluation of stress-crack resistance in November 1993 which involved two test methods: the bent strip test and the Notched Constant Tensile Load (NCTL) test. The physical properties analyzed include thickness and density, while the thermal properties include percent carbon black and percent crystallinity, the latter of which was also initiated by GRI in November 1993.

3.1 MECHANICAL TESTS

The mechanical testing covered in this report includes (1) uniaxial tensile tests (ASTM D 638 Type IV) and tear resistance tests (ASTM D 1004), which are basic index tests, and (2) stress-crack resistance using two test methods: the bent strip test (ASTM D 1693) and NCTL test (ASTM D 5397).

3.1.1 Uniaxial Tensile Test—ASTM D 638-91

This test method covers the determination of the tensile properties of unreinforced and reinforced plastics in the form of standard dumbbell-shaped test specimens when tested under defined conditions of pretreatment, temperature, humidity, and testing machine speed.

Ten dumbbell-shaped specimens are die cut using an ASTM D 638 type IV die. Five of the samples are cut with the longitudinal axis parallel to the machine direction, MD, and five samples are cut with the longitudinal axis perpendicular to the machine direction, XD. The test specimens must conform to the dimensions shown in Fig. 2. The test specimens are conditioned at $73.4 \pm 3.6^\circ\text{F}$ and $50 \pm 5\%$ relative humidity for 40 h before the test. Tests are conducted in the standard laboratory atmosphere of $73.4 \pm 3.6^\circ\text{F}$. After the specimen is placed in the grips of the testing machine, a jaw separation of 2 in./min. shall be used, thereby subjecting the specimen to increased stress. The jaws are slowly separated until the specimen breaks. The tensile strength is calculated by dividing the maximum load in lbf by the original minimum cross-sectional area of the specimen in in^2 . The result is expressed in lbf/in^2 (psi).

When a nominal yield or break load less than the maximum is present and applicable, it may be desirable also to similarly calculate the corresponding yield stress or break stress and report it to three significant figures. The elongation percent is calculated using the increase in length of the specimen expressed as a percentage of the original gage length. When a yield or breaking load less than the maximum is present and of interest, it is desirable to calculate and report both percent yield elongation and percent break elongation.

ORNL-DWG 94M-18041

Specimen Dimensions for Thickness, T, in. (mm) Type IV		
Symbols	Dimensions	Tolerances
W-Width of narrow section	0.25 (6)	± 0.02 (± 0.5)
L-Length of narrow section	1.30 (33)	± 0.02 (± 0.5)
WO-Width over-all, min.	0.75 (19)	± 0.25 (± 6.4)
LO-Length over-all, min	4.5 (115)	no max.
G-Gage length	1.00 (25)	± 0.005 (± 0.13)
D-Distance between grips	2.5 (64)	± 0.2 (± 5)
R-Radius of fillet	0.56 (14)	± 0.04 (± 1)
RO-Outer radius	1.00 (25)	± 0.04 (± 1)

Fig. 2. Tension test specimens dimensions.

3.1.2 Tear Resistance —ASTM D 1004-90

This test method covers the determination of the tear resistance of flexible plastic film and sheeting at very low rates of loading (51 mm (2 in.)/min.) The test is designed to measure the force to initiate tearing. The specimen geometry of this test method produces a stress concentration in a small area of the specimen. The maximum stress, usually found near the outset of tearing, is recorded as the tear resistance in newtons (or pounds-force).

Ten specimens, five in the machine direction and five in the cross machine direction, are cut with a die having the dimensions shown in Fig. 3. The test specimens are conditioned at $73.4 \pm 3.6^\circ\text{F}$ and $50 \pm 5\%$ relative humidity for 40 h prior to the test. Tests are conducted in the standard laboratory atmosphere of $73.4 \pm 3.6^\circ\text{F}$ and $50 \pm 5\%$ relative humidity. A jaw separation of 1 in. shall be used, with the rate of travel of the power-activated grip to be a constant speed of 2 in./min. After complete rupture of the specimen, the maximum tearing load in pounds shall be noted from the dial scale or recorder chart and recorded.

Fig. 3. Die for tear test specimen.

3.1.3 Bent Strip Test—ASTM 1693-70

This test method covers the determination of the susceptibility of ethylene plastics to environmental stress-cracking when subjected to conditions of stress and in the presence of environments such as soaps, wetting agents, oils, or detergents. Under these conditions, ethylene plastics may exhibit mechanical failure by cracking.

The condition desired is selected from the standard test conditions shown in Table 1. Specimens are cut to the dimensions given in Fig. 4A, and each conditioned specimen is given a controlled imperfection (notch) on one surface as shown in Fig. 4A. Ten such prepared specimens, with the controlled imperfection up, are placed in the slots provided in the bending clamp shown in Fig. 5. Not more than ten minutes after the specimens have been bent into the holder, the holder is inserted into the test tube as shown in Fig. 4C. Then the tube is filled immediately to approximately 13 mm (0.5 in.) above the top specimen with fresh reagent at a temperature of $73.4 \pm 1.8^\circ\text{F}$. The tube is closed with a foil-wrapped cork and immediately placed in the constant temperature bath at the preselected test temperature. The controlled imperfections should not touch the test tube during the test. The test specimens are inspected at the end of the intervals specified for the material under test (48 h, if not otherwise stated) and the total number of failures is recorded. The pass criteria is no failures within the stipulated time of 1500 h (NSF 54, 1993).

Table 1. Standard test conditions

Condition	Specimen thickness		Notch depth		Bath Temp ($^\circ\text{C}$)
	mm ^a	in.	mm ^a	in.	
A	min	3.00	0.120	0.50	50
	max	3.30	0.130	0.65	
B	min	1.75	0.070	0.30	50
	max	2.00	0.080	0.40	
C ^b	min	1.75	0.070	0.30	100 ^b
	max	2.00	0.080	0.40	

^aDimensional values are not exactly equivalent. However, for referee purposes the metric units shall apply.

^bAt a temperature of 100°C , a full-strength reagent, rather than an aqueous solution of a reagent, is generally used because solutions tend to change their compositions by water evaporation losses during the period of test.

Dimensions		
	mm	in.
A	38 ± 2.5	1.5 ± 0.1
B	13 ± 0.8	0.5 ± 0.03
C	See Table 1	
D	See Table 1	
E	16.5	$6\frac{1}{2}$
F		
(outside)	16	
(inside)	11.75 ± 0.05	0.463 ± 0.002
G	10	
H	15	$37/64$
I	2	0.081 (12 B&S)
J	Ten 5-mm holes	Ten $3/16$ " holes,
	15-mm centers	$19/32$ " centers

Fig. 4. Test equipment.

Fig. 5. Bending clamp assembly.

3.1.4 Notched Constant Tensile Load Test—ASTM D 5397 - 93

This test method is used to develop test data from which the susceptibility of polyolefin geomembrane sheet material to stress cracking under a constant tensile load condition and an accelerated environmental condition can be evaluated. This test measures the failure time associated with a given test specimen at a specified tensile load level. The method is intended as an index test and may be used for grading polyolefin geomembrane sheets in regard to their stress cracking sensitivity.

This test method subjects a dumbbell-shaped, notched test specimen from a polyolefin sheet to a constant tensile load in the presence of a surface-active agent and at an elevated temperature. The time to failure of the test specimen is recorded. The results of a series of such tests conducted at different stress levels are presented by plotting stress level against failure time for each stress level on a log-log axis.

3.2 PHYSICAL TESTS

The physical tests covered in this report include density (ASTM D 1505-85) and thickness (ASTM D 751).

3.2.1 Density—ASTM D 1505-85

This test method is based on observation of the level to which a test specimen sinks in a liquid column that exhibits a density gradient in comparison with standards of known density. A constant temperature density column is prepared by filling a graduated cylinder with fluids of continuously varying density so that a density gradient is achieved throughout the height of the cylinder. Four samples approximately 0.2 in. in width are cut in any shape. Each sample is carefully lowered into the density column along with glass beads of known density. The locations of the beads and the samples are noted using the graduations etched in the cylinder, and the density is calculated.

3.2.2 Thickness—ASTM D 751-89

The gage used for the measurement of thickness shall be of the deadweight type equipped with a dial graduated to read directly to 0.025 mm (0.001 in.). The presser foot shall be circular, with a diameter of 9.52 ± 0.03 mm (0.375 ± 0.001 in.). The presser foot and moving parts connected therewith shall be weighed so as to apply a total force of 1.7 ± 0.1 N (6 ± 0.1 oz.), equivalent to a pressure of 23.5 ± 0.5 kPa (3.4 psi) to the specimen. The presser foot and anvil surfaces shall be plane to within 0.0025 mm (0.0001 in.) and parallel to each other to within 0.0001 in. The gage shall be calibrated for the actual load exerted by the presser foot by means of any device so arranged as to measure the total vertical force exerted by the presser foot at the several gage readings or presser foot levels selected for calibration. The presser foot shall be brought to each calibration level from a higher one.

The coated fabric is placed upon the anvil of the gage so that it is smooth, but without tension. The presser foot is lowered upon the material gradually (without impact), allowed to rest upon it for 10 s, and the reading is observed on the dial. Similar measurements are made at not less than five different places uniformly distributed over the surface of the coated fabric, exclusive of the area adjacent to either selvage and within one tenth the width of the fabric or within 2.5 m (100 in.) of either end of a roll or piece. The average of five or more measurements is reported as the average thickness.

3.3 THERMAL TESTS

The thermal tests covered in this report include percent crystallinity using a differential scanning calorimeter, and percent carbon black using two test methods: thermogravimetry analysis and ASTM D 1603.

3.3.1 Percent Crystallinity using a Differential Scanning Calorimeter

The percent crystallinity in the geomembrane was measured by GRI using a differential scanning calorimeter (DSC). In this test method, a small solid specimen, approximately 3.0 mg, is heated from room temperature to 200°C at a rate of 20°C/min. under a nitrogen atmosphere. A DSC thermal curve of temperature versus heat flow is generated. The area under the curve represents the energy necessary to melt the crystal phase and is directly proportional to the percent crystallinity.

3.3.2 Percent Carbon Black using Thermogravimetry Analysis

Thermogravimetry Analysis was used by GRI to determine the percent carbon black in the geomembrane. A weighed specimen (approximately 25 mg) is heated from room temperature to 650°C at a rate of 20°C/min. in a nitrogen atmosphere. As the sample is heated, the weight is monitored and a graph of temperature versus weight percent is generated. The weight percent is constant until it reaches the decomposition temperature of polyethylene resin. At this temperature, around 400°C, a single weight drop occurs until there is no more resin. The weight percent is again a constant with increasing temperature, and the point on the graph where this occurs indicates the percent carbon black.

3.3.3 Percent Carbon Black Content —ASTM D 1603-76

This test method covers the determination of the carbon black content in polyethylene, polypropylene, and polybutylene plastics. Determinations of carbon black are made gravimetrically after pyrolysis of the sample under nitrogen.

A known quantity of resin is placed in a flowing, oxygen-free, nitrogen atmosphere in a 600°C tube furnace. The sample remains in the oven for 15 min., allowing all the polyethylene to boil off, leaving only the carbon black. The carbon black is cooled for 15 min. and weighed, and the percent carbon black is calculated.

4. HDPE GEOMEMBRANE DATA EVALUATION

4.1 MECHANICAL TESTS

4.1.1 Uniaxial Tensile Test

Tables 2 and 3 list the data accumulated from March 1990 to March 1994 on the mechanical properties of average yield and break stress and average yield and break elongation in the machine and cross machine directions, respectively. The data in the first line of each table are taken from the Geomembrane Certificate of Analysis. The Geomembrane Certificate of Analysis is a document from the manufacturer certifying the initial properties of the unexposed HDPE material before it is shipped to the job site. In each table, there are two data points, designated as Sample 1 and Sample 3, for the testing in November 1993 by GRI. As stated in Sect. 3, the Geosynthetic Research Institute is known to have received an active and a passive sample. Table 4 lists selected specifications of 80-mil HDPE from two manufacturers, Gundle Lining Systems, Inc., and National Seal Co., and from the National Sanitation Foundation (NSF). The NSF standards provide a reliable and dependable means for industry to furnish materials of known and consistent quality. The values listed in the table are the minimum average values.

The graphs in Figs. 6 and 7 represent the average yield stress versus exposure time in the machine and cross machine directions, respectively. The variability of the data can be attributed to the manufacturing process itself, specimen preparation, environment of testing, laboratory error, and equipment error. In the manufacture of HDPE, a series of warm and cold spots are common, resulting in areas of high and low crystallinity. Thus a variability in the tensile properties of the material will occur depending upon the degree of crystallinity. As stated in Sect. 3, three different laboratories were involved in testing the mechanical, physical, and thermal properties of the geomembrane during the five years of exposure. When a comparison of data from several testing laboratories is attempted, care must be exercised to ensure that all samples are prepared in exactly the same way unless the test is to include the effects of sample preparation. Similarly, extreme care must be taken to secure the maximum degree of uniformity in details of treatment, laboratory environment, and testing.

The values of yield stress in Table 2 fall within an acceptable range of the minimum average values for yield strength listed in Table 4. With an average of 2778 psi for the last three data points in the machine direction (from Aug. and Nov. 1993 and March 1994), the percent difference between this average and the original value of 2487 psi dated March 1989 is calculated to be 11.7%. In the cross machine direction, the average of the last three data points is 2937 psi, with a calculated percent difference of 15.2% between this average and the original value. Thus the recent data from August and November of 1993 and March of 1994 do not differ significantly from the original data from the unexposed liner. Based on this data, there has been relatively little variation of the yield stress over the five year period of exposure. The similarity between the data from the active and passive samples analyzed by GRI in Nov. 1993 indicates that the imposed stress of the cap during the five years of exposure has had little affect on the yield stress of the HDPE material.

Figures 8 and 9 represent the graphs of average break stress versus exposure time in the machine and cross machine directions, respectively. Again, there is a variability in the data for the same reasons as stated above. The values of break stress in Table 2 fall within an acceptable range of the minimum average values for break strength listed in Table 4. A value of 3693 psi is calculated for the average of the last three data points in the machine direction. The percent difference between this average value and the original value of 4336 psi is 14.8%. In the cross machine direction, the average of the last three data points is 4177 psi with a percent difference of 7.1% between this average and the original value. The recent values do not differ significantly from the data obtained from the certificate of analysis, thus the tensile property of break stress has not changed significantly in the five years of exposure. The similarity between the data from the active and passive samples analyzed by GRI in November 1993 indicates that the imposed stress of the cap during the five years of exposure has had little affect on the break stress of the HDPE material.

A graphical representation of yield and break elongation versus exposure time was not prepared because of the unusually high values for break elongation reported in the manufacturer's certificate of analysis and by two of the three testing laboratories. The high values could be the result of an improper gage length used in the calculations. The gage length should be 1.0 in. if extensometers are used and 2.5 in. if extensometers are not used. The gage length used for the calculations cannot be determined because it is rarely reported on the data sheets by the laboratory. From Table 4, the minimum average values of break elongation are 700% reported by the two manufacturers and 560% reported by NSF. More than half of the break elongation data points in Table 3 are not within a reasonable range of these values. Because of the uncertainty of gage length and the unusually high data points, a comparison of the break elongation data to the original data was not made. Because gage length is also used to calculate yield elongation, this data was also excluded from any comparison to the original data. The two data points from GRI are very similar, indicating that the imposed stress of the cap has had little affect on the yield and break elongation over time.

Table 2. Average yield stress and break stress for HDPE cap samples

Lab No.	Date	Yield stress (psi)		Break stress (psi)	
		MD ^a	XD ^b	MD	XD
-	2/89 ^c	2487	2550	4336	4498
1	3/90	2609	2627	3996	4172
1	9/90	2586	2554	4093	3605
1	5/91	2400	N/A	3903	N/A
1	1/92	1915	1939	3280	3268
1	9/92	1439	1463	2902	N/A
2	8/93	2677	2812	3173	4148
3	11/93				
	Sample 1	2950	3230	4120	4530
	Sample 3	2980	3230	4340	4490
2	3/94	2706	2769	3787	3853

^amachine direction^bcross-machine direction^cunexposed cap; data from manufacturer's Geomembrane Certificate of Analysis**Table 3. Average yield elongation and break elongation for HDPE cap samples**

Lab No.	Date	Yield elongation (%)		Break elongation (%)	
		MD ^a	XD ^b	MD	XD
-	2/89 ^c	22.7	21.6	1299	1431
1	3/90	16.7	15.7	1259	1395
1	9/90	17.3	18.1	1376	1161
1	5/91	N/A	N/A	123	96
2	8/93	24.2	23.3	718	906
3	11/93				
	Sample 1	17.1	15.2	620	660
	Sample 3	16.9	15.0	670	660
2	3/94	22.3	22.8	626	658

^amachine direction^bcross-machine direction^cunexposed cap; data from manufacturer's Geomembrane Certificate of Analysis

Table 4. Minimum average values of selected specifications of 80-mil HDPE

	Density	Strength Yield/Break	Elongation Yield/Break	Tear Resistance	Carbon Black	Environ. Stress Crack Resistance
	ASTM 1505 (g/cm ³)	ASTM D638 (lb)	ASTM D638 (%)	ASTM D1004 (lb)	ASTM 1603 (%)	ASTM 5397 (hrs)
Gundline HD ^a	0.94	2375/3900	13/700	60	2 - 3	1500
HDPE 80 ^b	0.94	2200/3800	13/700	60	2.0	200
NSF ^c	0.94	2350/3800	12/560	52	2 - 3	1500

^aGundle Lining Systems, Inc.^bNational Seal Co.^cNational Sanitation Foundation International Standard 54-1993

Fig. 6. Average yield stress (MD) vs exposure time.

Fig. 7. Average yield stress (XD) vs exposure time.

ORNL-DWG 94M-10846

Fig. 8. Average break stress (MD) vs exposure time.

ORNL-DWG 94M-10845

Fig. 9. Average break stress (XD) vs exposure time.

4.1.2 Tear Resistance

Only recent data is available for the tear resistance of the HDPE geomembrane over its five-year exposure period; therefore, a representative graph of tear resistance versus exposure time is not included in this report. Table 5 lists the tear resistance evaluated in August and November 1993 and March 1994. The values are very similar and are all within a reasonable range of the minimum average values in Table 4. The passive and active samples tested by GRI are in close agreement, indicating that the imposed stress of the cap has had little affect on the tear resistance over time.

Table 5. Average tear resistance

Average tear resistance (lb)			
Lab No.	Date	MD ^a	XD ^b
2	8/93	65.8	65.0
3	11/93		
	Sample 1	72	69
	Sample 3	70	69
2	3/94	65.4	63.5

^amachine direction

^bcross-machine direction

4.1.3 Bent Strip Test

Environmental stress cracking is of major concern when polymeric materials are used and is most commonly associated with semicrystalline geomembranes. Among the semicrystalline polymers, HDPE has the greatest percent crystallinity. Table 6 is a summary by Koerner (1993) after Geoservices, Inc. (1989), of the occurrences of HDPE geomembrane stress cracking in the field. The table shows 17 sites with incidents of stress cracking involved exposed HDPE geomembranes.

Table 6. Summary of stress cracking survey, after GeoServices Inc. (1989)
 (Parentheses indicate number of sites with stress cracks)

Type of installation			Environment			Exposed liner	Covered liner	Years in service
Solid waste	Liquid waste	Total	Hot	Cold	Both			
2(0)	7(3)	9(3)	1(0)	3(3)	5(0)	9(3)	0(0)	7-10
17(2)	6(2)	23(4)	16(3)	4(1)	3(0)	19(4)	4(0)	2-6
4(0)	14(10)	18(10)	11(9)	0(0)	7(1)	18(10)	0(0)	1-8

In a 1994 report, Hsuan reported that ten specimens from an active sample and a passive sample of the exposed HDPE geomembrane were tested for either a pass or fail designation. The results of the test, listed in Table 7, show that all the specimens failed at 170 h. According to NSF (1993), the test should run for 1500 h. without brittle failure, so the extremely short time of 170 h indicates that the stress crack resistance is very low. There is no way to assess the degree of stress-crack resistance from the results of this test because it is a qualitative rather than a quantitative test. Because both the active and passive samples failed at 170 h, the results suggest that the imposed stress resulting from confinement during the five years of exposure has not affected the stress-crack behavior of the geomembrane.

Table 7. Bent strip test results, after Hsuan (1994)

Sample No.	No. failed / No. tests at length of test (hrs)	Pass or Fail
1	10/10 At 170 hrs	Fail
3	10/10 At 170 hrs	Fail

4.1.4 Notched Constant Tensile Load Test

The Geosynthetic Research Institute used the NCTL test to quantify the stress-crack resistance of Samples 1 and 3. The specimens were stressed to a prescribed percentage of their yield stress, and the failure times were recorded. Figs. 10 and 11 represent the plot of the percent yield stress versus average failure time on a log-log scale for Samples 1 and 3, respectively. The ductile region where the slope of the line is shallow and the brittle region where the slope of the line is steeper are very distinct areas of the graphs. The region between these trends is the transition region. According to Hsuan (1994), the most important information yielded by the NCTL test is the onset of the brittle failure region of the curve because it signifies the transition of the material into a brittle state in which stress cracking can occur. Two values from the curve, the transition time and the transition stress, are used to define the geomembrane's stress-crack resistance. Generally, the longer the transition time and the lower the transition stress, the better is the HDPE geomembrane's stress-crack resistance (Koerner et al., 1993).

Table 8 lists the transition values and the slopes from the graphs in Figs. 10 and 11. The data show that both samples have almost identical brittle slopes and very similar transition times. The similarity is further illustrated in the superimposed response curves of Fig. 12. This similarity reinforces the results from the bent strip test that the two samples are virtually indistinguishable from each other. Thus, the imposed stress because of confinement during the five years of exposure has not affected the stress-crack behavior of the geomembrane.

Table 8. Results of NCTL test, after Hsuan (1994)

Sample No.	Ductile slope	Brittle slope	Transition stress (%)	Transition time (h)
1	-0.052	-0.37	38	7
3	-0.046	-0.36	40	8

Using the NCTL test, Koerner et al.,(1993) analyzed eighteen commercially available sheet materials and 7 field-exhumed HDPE geomembranes for stress-crack resistance. The survey led to the recommendation that, for an acceptable stress-crack-resistant HDPE geomembrane, the test results should indicate a transition time of > 100 h. Based on this recommendation, the transition times of 7 and 8 h for the two samples indicate that both samples are very sensitive to stress cracking. In the future, because of this high sensitivity, the geomembrane cap will experience additional stress cracks whenever stress is imposed on it (Hsuan 1994). The exposed HDPE caps have experienced numerous incidences of stress cracking in the five years of exposure.

Fig. 10. Results of NCTL tests, ductile-to-brittle curve of Sample No. 1. (Hsuan 1994)

Fig. 11. Results of NCTL tests, ductile-to-brittle curve of Sample No. 3. (Hsuan 1994)

Fig. 12. Superimposed ductile-to-brittle curves of samples No. 1 and No. 3. (Hsuan 1994)

4.2 Other Selected Material Properties

A comparison of recent data and the data from the Certificate of Analysis for the material properties of thickness, density, and carbon black content are included in Table 9. The variability of the data values can be attributed to the same reasons cited in Sect. 4.1.1. The data shows no appreciable deterioration of the properties due to the five years of exposure. The data for density and percent carbon black are in an acceptable range of the minimum average values listed in Table 4. The percent crystallinity is within the typical range of 40–60% (Hsuan, 1994). Samples with high crystallinity (greater than 50%), however, tend to have poor stress crack performance. This was reinforced by the geomembranes poor performance in the stress crack resistance tests.

Table 9. Selected material properties of geomembrane cap material.

Property	Unit	C.O.A. ^a 2/89	Lab 2 8/93	Lab 3 11/93	Lab 2 3/94
thickness	mils	81.90	81.7	79.75	81.2
density	g/cm ³	0.94	0.948	0.949	0.948
carbon black	%				
TGA				2.7 ^b (2.6) ^c	
ASTM D1603		2.21	2.22		2.61
crystallinity	%			46 ^b (49) ^c	

^aManufacturer's Geomembrane Certificate of Analysis

^bSample 1

^cSample 3

5. CONCLUSION AND RECOMMENDATIONS

The mechanical, physical and thermal properties of an exposed 80-mil HDPE liner at Oak Ridge National Laboratory were monitored from March 1990 to March 1994 and compared to the manufacturer's original Geomembrane Certificate of Analysis dated February 1989. Sacrificial active and passive liner samples were taken from the exposed cap semiannually and analyzed according to applicable ASTM methods by three different testing facilities.

The properties of uniaxial yield and break stress over the five-year exposure period do not differ significantly from the original data in the manufacturer's certificate of analysis. The percent differences between the certificate of analysis and the average of the three most recent data values are 11.7% and 15.2% for the yield stress in the machine and cross machine directions, and 14.8% and 7.1% for the break stress in the machine and cross machine directions, respectively. The reported data values fall within an acceptable range of the minimum average values recommended by NSF International Standard 54-1993, Gundle Lining Systems, Inc., and National Seal Co.

An evaluation of the yield and break elongation over time was not possible because of uncertainty in the parameters used to calculate the data from the different laboratory testing facilities. More than half of the reported data for break elongation is not within an acceptable range of 700%, reported by the two manufacturers listed in Table 4, or 560% recommended by NSF. The results of GRI's tests on the active and passive samples were very similar, indicating that the imposed stress of the cap has had little affect on the yield and break elongation over time.

The recent available data for the tear resistance is in an acceptable range of the values reported in Table 4. The passive and active samples tested by GRI are in close agreement, indicating that the stress of the cap has not affected the tear resistance of the HDPE geomembrane over time.

Evaluation of the geomembrane's stress-crack resistance was initiated by GRI in November 1993 using the NCTL test and the bent strip test. The results of testing an active and passive sample from the exposed cap material indicate that the material is highly susceptible to stress cracking based on the NCTL test transition time of only 7 and 8 h, compared to a recommended time of at least 100 h. Because of this high sensitivity, the geomembrane cap will experience additional stress cracks whenever stress is imposed on it. Both samples also failed the bent strip test, failing at 170 h compared to the recommendation by NSF of no failures within the stipulated time of 1500 h. Based on the similar results of the two samples tested, there is no distinction between the confined and unconfined sample. Thus the imposed stress resulting from confinement during the five years of exposure has not affected the stress-crack behavior of the geomembrane.

A comparison of the recent data and the data from the Geomembrane Certificate of Analysis for the material properties of thickness, density, and carbon black content indicate no appreciable deterioration of these properties over time. The density and the percent carbon black are in an acceptable range of the minimum average values listed in Table 4. The two values of percent crystallinity (46% and 49% for samples No. 1 and No. 3, respectively) are within the typical range of 40-60% for 80-mil HDPE; however, samples with crystallinity

greater than 50%, tend to have poor stress-crack performance. This was confirmed by the geomembrane's poor performance in the stress crack resistance tests.

Because the performance of the cap material was shown to be highly sensitive to stress cracking, this is an area of major concern. The exposed HDPE caps have experienced numerous incidences of stress cracks in the five years of exposure. For stress cracking to occur in a semicrystalline geomembrane material like HDPE, the material must fulfill two conditions (Koerner et al., 1993). First, it must be under some form of stress, and, second, it must have a point at which a crack can initiate. The crack initiation point could be at an imperfection, scratch, carbon black agglomeration, or other anomaly but will most likely be at the location of a seam. Cracks are likely to develop at seams because of the natural discontinuity of the overlap configuration used to seam geomembranes and also possibly because of overheating of fusion seams or excessive grinding associated with extrusion flat or fillet seams. A recent EPA Technical Guidance Manual (U.S. EPA 1991) focuses on proper geomembrane seam fabrication and inspection techniques that should alleviate these types of field installation problems.

The continued monitoring of the HDPE cap is recommended. The benefit of this ongoing monitoring is that determining the durability and longevity of the HDPE geomembrane will allow the best decision to be made on its use in future remediation projects. A study such as this, which monitors as many properties as were covered in this report, is not available in the literature and would be very useful to manufacturers, installers, and users.

With regard to the future sampling, testing, and monitoring of the geomembrane, quality/control measures must be implemented immediately if a definitive, scientific study is to be conducted. When the semiannual sacrificial samples of the geomembrane are obtained, duplicate samples from the same general area (ensuring similar stress histories) should be taken and distributed to two independent laboratories that have a history of using the applicable ASTM test methods outlined in Sect. 3. A passive sample should also be included but not labeled as such. A specified individual should be appointed to accompany the personnel collecting the liner samples and to monitor the data as they become available. If the results from the two labs differ when the data are compared, this individual is responsible for contacting the facilities and alerting them to the suspect data. The facilities can then recheck the values and possibly rerun the test if sufficient material is still available for analysis. The development of a reliable, reproducible data base such as this will assist in predicting the performance of HDPE in other applications in the future.

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