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Soil Burial Tests:

Effect of Soil Burial Exposure on the Properties of Structural Grade Reinforced Plastic Laminates

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This paper is a summary of the effects of long-term soil burial on the properties of reinforced plastic laminates. The interactions of reinforcement, resin, and fabrication technique, as they relate to the laminates' ability to resist the effects of soil burial, are discussed. The method of soil burial attack is explained. Recommendations for the best laminate construction and methods of protection from the soil environment are made. These include:

(i) Where there is a need for a laminate structure with the maximum initial mechanical properties and the largest percent retention of these values, a fully cured laminate with fiberglass cloth reinforcement and an epoxy resin matrix is recommended.

(ii) Laminates made with an epoxy resin matrix or a polyester resin matrix show the same rate of degradation due to soil burial.

(iii) The use of resin-rich face plies in a sandwich construction with fiberglass chopped strand mat (FCSM) as the core plies showed better resistance to the effects of soil burial than does an all-FCSM laminate.

I. INTRODUCTION

The effects of long-term soil burial on reinforced plastic laminates is of major significance because of their present and projected increased use in intimate soil contact. Reinforced plastic manholes and pipe structures are two examples of projected uses that depend upon the application of these soil burial data.

II. CHEMISTRY OF REINFORCED PLASTIC LAMINATES

Laminates discussed herein are made of two or more layers of reinforcement with a resin matrix. The physical, mechanical, chemical, and electrical properties of the resultant laminate are dependent upon the selection of the proper reinforcement, resin system, and fabrication method.

2.1 *Resins*

This paper concerns itself with laminates made of polyester and epoxy resin matrices.

2.1.1 *Polyester Resins*

Polyester resins are polycondensation products of a polycarboxylic acid and a polyhydroxy alcohol. These polyesters can be characterized as possessing a number of ester groups ($-\overset{\overset{\textstyle O}{||}}{C}-O-$) and ethylenic groups ($-\overset{|}{C}=\overset{|}{C}-$) in the polymer chain. The long linear chains are crosslinked by vinyl monomers. The polyester resins in the buried laminates almost exclusively contain styrene as the vinyl monomer.

The following is a brief description of the three polyester resins evaluated.

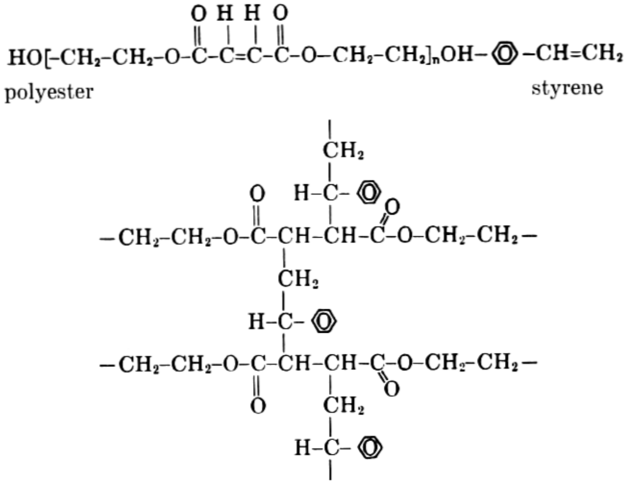
(*i*) A general purpose orthophthalate polyester resin that typically contains styrene monomer.

(*ii*) A fire-retardant polyester resin based upon chlorendic anhydride with styrene monomer added.

(*iii*) An acrylic-modified polyester resin and methyl methacrylate monomer, to which was added styrene monomer.

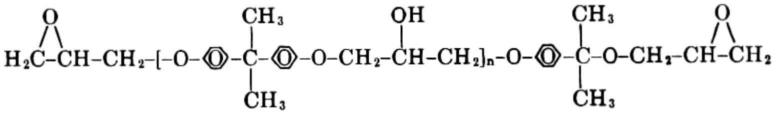
2.1.1.1 *Polyester Initiators.* Polyester addition polymerization is initiated by free radicals, which are obtained by the breakdown of peroxides. There are several different catalysts with a wide range of activation temperatures. The selection of the catalyst is dependent upon the desired curing conditions. Methyl ethyl ketone peroxide (DDM) and benzoyl peroxide (BPO) were the catalysts selected in this program.

2.1.1.2 *The Polyester-Styrene Curing Mechanism.*[1]

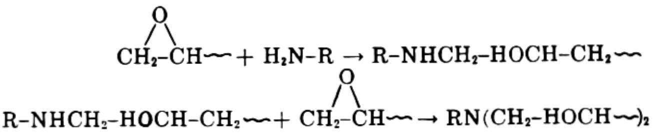


2.1.2 *Epoxy Resins*

Epoxy resins are available in a wide range of molecular weights. The epoxy resins referred to in this part of the soil burial program are the reaction products of epichlorohydrin and bisphenol A. A typical epoxy resin has the following structural formula.



These epoxy resins were cured by amine catalysts. The reaction is shown by the following schematic representation.



Two commercially available epoxy resins were evaluated: (*i*) a diglycidyl ether of bisphenol A, a liquid resin with a viscosity of 10,000 to 16,000 centipoises; (*ii*) the same base resin as above but diluted with 11 percent butyl glycidyl ether, a reactive diluent, to an average viscosity of 6000 centipoises.

2.1.2.1 *Epoxy Curing Agents.* Epon Curing Agent "Z", a commercial eutectic blend of methylene dianiline (MDA) and metaphenylene diamine (MPDA), and Epon Curing Agent "D", a commercial product of 2-ethyl hexoic acid salt of tris (dimethylaminomethyl) phenol,[2] and diethylene triamine (DTA) are the curing agents used in the curing of the epoxy resin laminates.

2.1.2.2 *Epoxy Additives.* The resin matrix in one of the laminates contained 50 parts of a saturated polyester resin added to 50 parts of the higher viscosity epoxy resin. Saturated polyesters impart flexibility and impact resistance to cured epoxy resins.

2.2 *Reinforcements*

Three classes of fibrous reinforcements were evaluated: glass fibers, a polyester fiber, and an acrylonitrile-vinyl chloride copolymer fiber. The glass fiber reinforcements were fabricated into three different forms: continuous filament woven cloth (FC), continuous filament rovings woven into a cloth (FWR), and glass rovings chopped into 2-inch strands and formed into a mat (FCSM). The fiberglass mat in some cases was the sole reinforcement in the laminate. It was also used as the core plies of reinforcement in laminates of sandwich type construction, Fig. 1. The fiberglass as woven roving is used only as the face plies of the sandwich construction.

The polyester fibers and the acrylonitrile-vinyl chloride copolymer fibers were used in a mat form as the face plies of the sandwich construction only.

III. TEST METHODS

3.1 *Laminate Fabrication*

The laminates were molded in a press under heat and pressure. The polyester resin laminates were cured at 245°F for 7 minutes. The

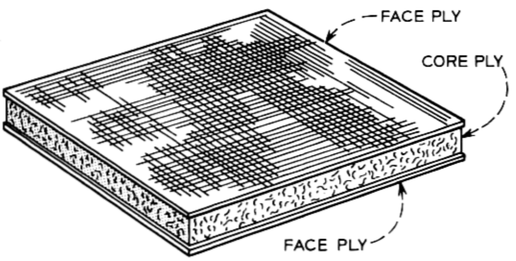


Fig. 1—Sandwich construction type laminate.

epoxy resin laminate molding conditions varied with the catalyst system. The individual laminate number and fabrication data are in Table I.

3.2 *Sample Preparation and Burial*[3]

The laminates were cut into specimens 1 inch by 4 inches and attached by means of polyethylene rivets to the polyethylene tubes. Thirty replicates of each laminate were exposed at each depth at each site.

3.3 *Test Procedure*

Following exposure, a 1/4 inch strip was cut from each side of one- and two-year soil burial specimens to reduce the "edge effect." This reduced the test specimens to 1/2 inch width. The sides of the four-, six-, and eight-year specimens were polished with an emery wheel to 1-inch width.

The flexural strength and the flexural modulus of elasticity tests were performed in accordance with ASTM D790.

A condensation and comparison of the values obtained are in Tables II, III, and IV.

IV. DISCUSSION OF DATA

4.1 *Test Selection*

In a study of this nature one is concerned with: (*i*) the effects of the soil environment on the reinforcement, (*ii*) the resin matrix, (*iii*) the interface between the resin matrix and the reinforcement, and (*iv*) the manner in which these forces come to bear on a structural laminate.

Flexural tests subject the specimen to tensile stresses in the outer extreme fiber and to compression in the inner extreme fiber. The tensile property is largely determined by the reinforcement whereas the compressive strength is dependent on the resin.

The measured resultant combination of these forces is an indication of the effects of soil burial conditioning on the fiber-reinforced plastic (FRP).

4.2 *Control—Shelf Aging at Murray Hill, N. J.*

Representative samples of the specimens buried at the two test sites were shelf aged in a Standard Laboratory Atmosphere per ASTM D618. The samples were tested as received, and after 2, 4, 6, and 8 years of shelf aging.

The greatest initial strength was exhibited by the fiberglass cloth laminates, followed by laminates of sandwich construction consisting of woven fiberglass rovings as the face plies and fiberglass chopped strand mat as the core plies. Laminates made entirely of FCSM and laminates made of FCSM as the core plies and polyester fiber mat or acrylonitrile-vinyl chloride copolymer fiber mat as face plies showed the lowest initial mechanical strengths. In this last group of laminates, the all-fiberglass chopped strand mat with epoxy resin matrix showed approximately 30 percent greater initial strength than the same construction using a polyester resin matrix.

The all-FCSM polyester resin matrix laminate and laminate with the polyester fiber mat or acrylonitrile-vinyl chloride copolymer fiber mat face plies all had initial flexural strengths in the range of 36,000 psi.

The four- and eight-year data showed a wide range of values that may be attributed to deviations in the samples rather than the effect of ambient conditions on the laminate. Samples may differ as to the degree of cure and location of the particular sample in the laminate.

4.3 *Soil Burial Effects*

4.3.1 *Visual Examination*

In practically all cases, gross examination of the specimens subjected to soil burial showed no severe attack upon the physical integrity of the laminate. A few laminates showed abnormalities traced to improper laminate preparation. For example, Laminate No. 157 showed a tendency to delaminate between the first and second plies of fiberglass cloth. The comparatively high initial flexural strength indicates that the laminate is adequately cured and that the delamination is probably due to insufficient resin or poor resin wet-out in the base laminate. Laminate No. 155 developed many hairline cracks due to shrinkage in resin-rich areas on the surface of the laminate. Laminate 169, the only laminate to show a significant staining, was made of an epoxy resin and a saturated polyester resin additive.

4.3.2 *Mechanical Properties*

The rate and percent degradation of the laminate mechanical properties due to soil burial is independent of soil depth and test location.

4.3.3 *Resin Systems Evaluation*

All laminates made using FCSM were compared in order to assess the effect of soil burial on the various resin systems. Data are tabulated in Table II. Although the two epoxy resin based laminates showed the highest initial flexural strength, these data show that the epoxy resin and the polyester resin laminates both exhibited the same degree of mechanical degradation. After eight years of soil burial these laminates maintained 65–70 percent of their initial flexural strength.

4.3.4 *Reinforcement Systems Evaluation*

Table III lists data obtained on laminates of the same polyester resin matrix and different reinforcements. This table shows the effect of soil burial as it relates to the reinforcement.

The evidence leaves no doubt that the laminates made of all fiberglass cloth reinforcement have superior resistance to the effect of long-term soil burial to any other reinforcement or combinations of reinforcements evaluated. The fiberglass cloth laminates show the highest initial flexural strength (54,000 psi) and a retention of 80 to 85 percent of their initial mechanical properties. The FCSM laminates maintained 55 to 60 percent of their original mechanical properties. The utilization of a fiberglass woven roving fabric as the two face plies and the fiberglass mat as the core plies enhanced the laminates' ability to resist degradation. This was particularly true with an epoxy resin as the matrix in which it retained at least 75 to 80 percent of its original properties. The data also indicate that the use of polyester fiber mat or acrylonitrile-vinyl chloride copolymer fiber mat face plies did show an improvement in soil burial resistance over laminates made of all-fiberglass chopped strand mat. These data demonstrate that the nature of the reinforcement has a greater influence on the laminates' ability to resist the effects of soil burial than does the resin.

4.3.5 *Cure*

Table IV demonstrates that the degree of cure is directly related to the laminates' ability to resist the effect of long-term soil burial.

V. CONCLUSIONS

A fully cured laminate with fiberglass cloth reinforcement and an epoxy resin matrix is recommended where there is need for a laminate structure with high initial mechanical strength and little change with time.

The fiberglass cloth is woven from long lengths of continuous filament yarn presenting relatively few filament ends to the hostile environment. Fiberglass chopped strand mat is made of many 2-inch lengths of fiberglass yarn. Each filament end presents loci for chemical attack by the environment, largely by capillary action, and thereby weakening of the bond between the glass fiber and the resin.[4] Laminates with an all fiberglass mat construction suffered the greatest damage due to the effect of soil burial regardless of the choice of resin.

Laminates with an epoxy resin matrix possess higher initial mechanical properties than do laminates made of a polyester resin matrix. However, both of these systems show the same rate of degradation due to soil burial. The study shows that to derive the best properties from the laminates the resin matrix must be fully cured.

The use of a polyester fiber mat, an acrylonitrile-vinyl chloride copolymer fiber mat, or fiberglass woven roving face plies in a sandwich construction with FCSM as the core plies showed better resistance to the effects of soil burial than does an all-FCSM laminate. The polyester fiber mat and the acrylonitrile-vinyl chloride copolymer fiber mat resin-rich surfaces act as a protective barrier for the ends of the glass fibers of the FCSM.

No precautions were taken to protect the exposed ends of fibers on the edges of the buried laminate. Although the exposed edges were machined from the soil burial samples to reduce the edge effect, the damaging effect of the soil burial had already been done. If only one surface of the laminate with protected edges were subjected to soil conditioning the effects on the laminate would be significantly less.

In considering the information in this evaluation program we must acknowledge the program's limitations. The laminates selected for the soil burial program were representative of the best glass, glass finish, resin, and fabrication technology available at the outset of the program in 1958. Since then there have been many new and significant developments in the above technologies. The following recent developments are cited to indicate the extent and areas that have shown advancement:

(*i*) "S" glass fibers—a new high-strength glass.

(*ii*) A thermochemical desizing agent that reduces or eliminates the need to use high-heat treating to remove the starch sizing from the glass fiber. Heat treatments of this nature degrade the mechanical properties of the glass fibers.

(*iii*) Sheet molding compounds (SMC) and parts made from SMC present a new technology in its entirety, along with its unique resin systems.

The soil burial program of these materials will be continued, reflecting the latest developments in the FRP field.

VI. ACKNOWLEDGMENTS

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4. Asbee, K. H. G., and Wyatt, R. C., "Water Damage in Glass Fibre/Resin Composites," Proc. Royal Soc., A312 (1969), pp. 553–564.

TABLE I—LAMINATE COMPOSITION AND FABRICATION DATA*

Laminate No.	151	152	153	154
Reinforcement				
Face ply	—	—	1 ply PM	1 ply AM
Core plies	4 plies FCSM	4 plies FCSM	4 plies FCSM	4 plies FCSM
Face ply	—	—	1 ply PM	1 ply AM
Resin Matrix				
Resin	Ortho P'ester	Ortho P'ester	Ortho P'ester	Ortho P'ester
Catalyst	0.05% BPO	1.0% DDM	1.0% DDM	1.0% DDM
Cure				
Time-Temp	7 min, 245°F	7 min, 245°F	7 min, 245°F	7 min, 245°F

Laminate No.	155	156	157	158
Reinforcement				
Face ply	1 ply FWR	—	—	—
Core plies	1 ply FCSM	11 plies 181FC	11 plies 181FC	11 plies 181FC
Face ply	1 ply FWR	—	—	—
Resin Matrix				
Resin	Ortho P'ester	Ortho P'ester	Ortho P'ester	Chlor P'ester
Catalyst	1.0% DDM	1.0% DDM	0.5% ATC	0.5% BPO, 0.5% DDM
Cure				
Time-Temp	7 min, 245°F	7 min, 245°F	7 min, 245°F	7 min, 245°F

Laminate No.	159	160	161	162
Reinforcement				
Face ply	—	—	—	1 ply PM
Core plies	4 plies FCSM	4 plies FCSM	4 plies FCSM	4 plies FCSM
Face ply	—	—	—	1 ply PM
Resin Matrix				
Resin	Chlor P'ester	Acryl P'ester	8 Epoxy	8 Epoxy
Catalyst	1.0% DDM	1.0% DDM	20 phr Z	20 phr Z
Cure				
Time-Temp	7 min, 245°F	7 min, 245°F	90 min, 180°F	90 min, 180°F
Post-cure				
Time-Temp	—	—	120 min, 300°F	120 min, 300°F

Laminate No.	163	164	165	166
Reinforcement				
Face ply	1 ply AM	1 ply FWR	—	—
Core plies	4 plies FCSM	1 ply FCSM	4 plies FCSM	4 plies FCSM
Face ply	1 ply AM	1 ply FWR	—	—
Resin Matrix				
Resin	8 Epoxy	8 Epoxy	5 Epoxy	8 Epoxy
Catalyst	20 phr Z	20 phr Z	20 phr Z	13 phr D
Cure				
Time-Temp	90 min, 180°F	90 min, 180°F	90 min, 180°F	30 min, 245°F
Post-Cure				
Time-Temp	120 min, 300°F	12 min, 300°F	120 min, 300°F	—

Laminate No.	167	168	169	170
Reinforcement				
Face ply	1 ply FWR	—	—	1 ply FWR
Core plies	1 ply FCSM	4 plies FCSM	4 plies FCSM	1 ply FCSM
Face ply	1 ply FWR	—	—	1 ply FWR
Resin Matrix				
Resin	Ortho P'ester	60 Part 5 Epoxy	50 Part 8 Epoxy	8 Epoxy
Catalyst	1.0% DDM	40 Part V125	15 Part Sat P'ester	20 phr Z
Cure				
Time-Temp	7 min, 245°F	30 min, 250°F	4 hrs, 158°F	90 min, 180°F
Post-Cure				
Time-Temp	—	—	—	120 min, 300°F

* See appended list of abbreviations.

TABLE II—COMPARISON OF RESIN MATRICES* (FCSM REINFORCEMENT)

Laminate No.	151	152	159	160	161	166	169	165	168
Resin	Ortho P'ester	Ortho P'ester	Chlor P'ester	Acryl P'ester	8 Epoxy	8 Epoxy	8 Epoxy Sat. P'ester	5 Epoxy	5 Epoxy
Catalyst or curing agent	BPO	DDM	DDM	DDM	Z	D	DTA	Z	V125
Initial flex str avg, psi	33,800	34,100	33,500	35,800	43,800	49,600	13,600	50,100	37,700
% Change:									
MH-shelf aging, 8 yrs	+7.1	+8.5	+1.5	−5.6	−11.6	−8.5	−38.8	−19.2	−17.5
Ga-topsoil, 8 yrs	−39.6	−42.2	−42.4	−40.3	−41.3	−33.9	+16.9	−33.5	−35.3
Ga-subsoil, 8 yrs	−41.7	−34.6	−42.7	−92.2	−36.5	−25.9	+14.0	−34.3	−35.0
NM-topsoil, 6 yrs	−34.3	−32.3	−31.3	−38.3	−32.9	−30.4	+13.2	−33.5	−29.4
NM-subsoil, 6 yrs	−31.4	−33.7	−31.9	−34.1	−30.6	−32.1	+15.4	−29.1	−30.0

* See appended list of abbreviations.

TABLE III—COMPARISON OF REINFORCEMENTS* (ORTHOPHTHALATE POLYESTER/1.0% DDM RESIN MATRIX, CURED AT 245°F FOR 7 MINUTES)

Laminate No.	152	153	154	155	156
Reinforcement					
Face plies	—	1 ply PM	1 ply AM	1 ply FWR	—
Core plies	4 plies FCSM	4 plies FCSM	4 plies FCSM	1 ply FCSM	11 plies 181FC
Face plies	—	1 ply PM	1 ply AM	1 ply FWR	—
Initial flex str avg, psi	34,100	33,800	33,200	46,100	57,300
% Change:					
MH-shelf aging, 8 yrs	+8.5	−8.0	+2.7	−14.5	−3.3
Ga-topsoil, 8 yrs	−42.2	−37.3	−29.5	−32.1	−4.4
Ga-subsoil, 8 yrs	−34.6	−38.2	−31.3	−25.4	−8.7
NM-topsoil, 6 yrs	−32.3	−31.4	−23.2	−34.1	−5.6
NM-subsoil, 6 yrs	−33.7	−25.4	−17.2	−29.1	−9.2

* See appended list of abbreviations.

TABLE IV—EFFECT OF DEGREE OF CURE OF EPOXY MATRIX*

Laminate No.	164	170
	Same Formulation (See Table I)	
Cure		
Time-Temp	90 min, 180°F	90 min, 180°F
Post-Cure		
Time-Temp	12 min, 300°F	120 min, 300°F
Initial flex str avg, psi	47,900	50,000
% Change:		
MH-shelf aging, 8 yrs	−27.8	+5.2
Ga-topsoil, 8 yrs	−29.6	−11.8
Ga-subsoil, 8 yrs	−25.1	+8.6
NM-topsoil, 6 yrs	−28.6	+5.8
NM-subsoil, 6 yrs	−24.6	+2.0

* See appended list of abbreviations.

ABBREVIATIONS

Reinforcements

AM—Acrylonitrile-vinyl chloride copolymer fiber mat
FC—Fiberglass cloth, continuous filament, style 181
FCSM—Fiberglass chopped strand mat, 2 oz per sq/ft
FWR—Fiberglass woven roving
PM—Polyester fiber mat

Resins

Acryl P'ester—Acrylic modified polyester resin
Chlor P'ester—Chlorendic anhydride based polyester resin
Ortho P'ester—Orthophthalate based polyester resin
Sat P'ester—Saturated polyester flexibilizer
5 Epoxy—Diglycidyl ether of bisphenol A, diluted to 6000 centipoises with butyl glycidyl ether
8 Epoxy—Diglycidyl ether of bisphenol A, liquid resin with a viscosity of approximately 13,000 centipoises

Catalysts and Hardeners

ATC—A paste of 50 parts benzoyl peroxide and 50 parts diallyl phthalate
BPO—Benzoyl peroxide
D—2-ethyl hexoic acid salt of tris (dimethylaminomethyl) phenol
DDM—60% solution of methyl ethyl ketone peroxide in dimethyl phthalate
DTA—Diethylene triamine
V125—Liquid polyamide hardener
Z—Blend of methylene dianiline (MDA) and methaphenylene diamine (MPDA)