Copyright © 1972 American Telephone and Telegraph Company
THE BELL SYSTEM TECHNICAL JOURNAL
Vol. 51, No. 1, January, 1972
*Printed in U.S.A.*

## Soil Burial Tests:

# Effect of Soil Burial Exposure on the Properties of Rubber, Crosslinked Polyethylene, and Vulcanized Wire Coatings

By G. H. BEBBINGTON

(Manuscript received August 24, 1971)

*Vulcanized rubber and polyethylene coated wires, molded specimens, and rubber-to-metal bonded test samples were buried in both alkaline and acid soils to determine the effects of these environments. Results show that:*

(*i*) *Most wire samples suffered a loss in insulation resistance, but retained their physical strength.*

(*ii*) *Insect and rodent chewing have caused varying test results in the dumbbell-shaped molded samples.*

(*iii*) *Rubber-to-metal bonds have failed due to corrosion in most samples using aluminum and cold-rolled steel, while stainless steel samples have retained strong bonds. The adhesion of insulations to aluminum conductors has also failed.*

(*iv*) *Black, chemically crosslinked polyethylene has been essentially unaffected by soil exposure.*

*Soil burial test exposures of improved and more recent materials are continuing.*

## I. INTRODUCTION

Rubber and other vulcanized polymers play an important role in the construction of a great deal of the hardware and equipment used in the Bell System. The uses range from wire and cable jackets and insulations to molded goods, such as the floor tiles in phone booths, splicing tape, underground conduit and couplings, waveguide supports, and manhole seals. Through the years more and more of the plant equipment has been and will be buried, necessitating the development of vulcanizate compounds that can withstand the rigors of different soil environments to provide twenty to thirty years service life.

O. DeVries reported the destruction of 22 percent of the hydrocarbons of "fresh raw rubber" by fungi in two years and 30 percent in five years.[1] But one must question the purity of any sample of natural rubber (NR) and assume that the diluents therein have promoted this degree of consumption, for it has been found that most pure synthetic polymers, by themselves, are generally resistant to fungi, i.e., polymers do not serve as a source of carbon for the growth of fungi.[2,3] However, they do not inhibit their growth. For this reason, the assorted ingredients that are added in making up a useable compound must be selected with care to be sure that they are not biologically susceptible. Although individual components may be resistant, in combination with others they may be susceptible to deterioration. Therefore, many combinations must be studied to find the most resistant vulcanizates.

Soil burial tests on various designs of tape, underground drop wire, and coaxial cable and on other experimental materials were started in 1929 and continued until late 1954. The results were evaluated and new soil burial tests were proposed to investigate additional materials and structures of possible interest for Outside Plant use. For this purpose, the Bell Laboratories Rubber Research Group supplied the below-listed items which were buried in 1958 in the moist, highly acid soil of the Bainbridge Environmental Test Plot in Georgia. Matched control specimens of all samples were put into storage for future comparative testing.

(*i*) *Wire Coatings*. Specimens consisted of 22 vulcanized wire samples (Table I) using both #22 AWG electrical-grade aluminum and 0.027-inch lead- and brass-coated copper-steel wire conductors. The coatings included 10 jacket compounds utilizing 7 different rubber polymers, and 6 insulation compounds made from 3 rubbers. Several fungicides were added to one of the insulations and one of the jacket formulations. A 48-volt dc potential was applied to half of the buried specimens.

(*ii*) *Molded Goods*. Dumbbell-shaped test specimens were cut from vulcanized sheets of 7 then-current rubber molding compounds (#260–266) utilizing 5 different polymers. Additional rubber and crosslinked polyethylene (XPE) molding compounds (#290–299) were added later for a total of 17 compounds in test. The formulations for the above are listed in Table II.

(*iii*) *Adhesion Studies*. Various vulcanized rubber-to-metal bonded specimens (#267–281) were made up using 3 different rubber compounds (Table III), 3 metals (aluminum, cold-rolled steel, and stainless steel sheet), and several proprietary bonding systems.

In 1959, crosslinked polyethylene insulated wires #485–490 (Table IV) were added to the test. Further additions of vulcanized test samples (#322–324), shown in Table V, were made when the new ethylene-propylene rubber polymers (EPM and EPDM) became available.

Duplicate specimens of most of the above materials were buried in 1960 in the dry, extremely alkaline soil of the Roswell Environmental Test Plot in New Mexico. Later additions were made in 1965.

## II. VULCANIZED INSULATED WIRES

### 2.1 *Rubber Covered Wires*

### 2.1.1 *Exposure Results*

While the specimens under potential usually have shown more degradation than those without potential, essentially no significant difference in capacitance or in insulation resistance was found between the samples aged with and without the applied 48-volt dc potential. However, because of the drastic reduction of insulation resistance that developed in the buried rubber covered wires during the first year, the potential had to be removed from most of these wires. The total current drain exceeded the fuse capacity of the test area. After a year and a half, only 6 of the original 22 types of rubber coverings (Table I) could be continued in the test with an applied potential. These were compounds #305, 311, 314, 315, 317, and 321. Interestingly, only #305 contained a fungicide, while both #317 and #321 were on aluminum conductors.

Because of the generally poor condition of the samples and the deterioration in electrical properties, only the 1-kHz measurements for capacitance, conductance, and insulation resistance were performed. The nonblack butyl (IIR) insulation #312, for example, was found to be discolored and cracked after only one year in the soil. After two years of exposure, the IIR jacket #313 could not even be measured on the test equipment. Conversely, the heat and water resistant chlorosulfonated polyethylene elastomeric (CSM) jacket #314 and #321 was the one exception since the insulation resistance increased somewhat on both aluminum and copper conductors.

In the current testing program one fungicide, copper-8-quinolinolate, was found to inhibit microbiological attack to a considerable degree

TABLE I—VULCANIZED WIRE JACKET AND INSULATION COMPOUNDS

| Compound Type | NR Ins | NR Jkt | SBR Ins | SBR Ins | SBR Ins | SBR Ins | SBR Jkt | CR Jkt | CR Jkt | CR Jkt | CR Jkt | NBR Jkt | IIR Ins | IIR Jkt | CSM Jkt | SI Jkt |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ingredients<br>Copper-Steel Cond (Aluminum Cond) | 300 | 301 (316) | 302 | 303 | 304 | 305 | 306 (317) | 307 (318) | 308 (319) | 309 | 310 | 311 | 312 | 313 (320) | 314 (321) | 315 |
| *Elastomers* | | | | | | | | | | | | | | | | |
| Reclaimed Rubber (NR) | | 29.0 | | | | | | | | | | | | | | |
| Natural Rubber—Smoked Sheet (NR) | 100.0 | 85.0 | | | | | | | | | | | | | | |
| Styrene-butadiene Rubber GR-S 1007 (SBR) | | | 100.0 | 100.0 | 100.0 | 100.0 | | | | | | | | | | |
| Styrene-butadiene Rubber GR-S 1712 (SBR) | | | | | | | 100.0 | | | | | | | | | |
| Polychloroprene W (CR) | | | | | | | | 100.0 | 100.0 | 100.0 | 100.0 | | | | | |
| Butadiene-acrylonitrile Copolymer 1001 (NBR) | | | | | | | | | | | | 100.0 | | | | |
| Isobutylene-isoprene Copolymer 035 (IIR) | | | | | | | | | | | | | 100.0 | 100.0 | | |
| Chlorosulfonated Polyethylene Elastomer 20 (CSM) | | | | | | | | | | | | | | | 100.0 | |
| Silicone Rubber 80 (SI) | | | | | | | | | | | | | | | | 100.0 |
| *Fillers* | | | | | | | | | | | | | | | | |
| Calcium Carbonate | | | 55.0 | 55.0 | 55.0 | 55.0 | | | | | | | 90.0 | | | |
| Zinc Oxide | 67.0 | 25.0 | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 | 3.0 | | | | 5.0 | 5.0 | 5.0 | | |
| Hard Clay | | | | | | | 30.0 | 40.0 | 40.0 | 40.0 | 40.0 | | | | 60.0 | |
| Ex. Light. Calcined Magnesium Oxide | | | | | | | | 3.0 | | | | | | | | |
| Easy-processing Channel (EPC) Black | | 27.0 | | | | | | | | | | | | | | |
| Fast-extruding Furnace (FEF) Black | | | | | | | 25.0 | | | | | 60.0 | | 50.0 | 10.0 | |
| Semi-reinforcing Furnace (SRF) Black | | | | | | | 25.0 | 52.0 | 52.0 | 52.0 | 52.0 | | | | | |
| *Reinforcers and Plasticizers* | | | | | | | | | | | | | | | | |
| Mineral Rubber | 47.0 | | 50.0 | 50.0 | 50.0 | 50.0 | | | | | | | | | | |
| High Styrene Resin | | | 27.0 | 27.0 | 27.0 | 27.0 | | | | | | | | | | |
| Hydrogenated Rosin | | | | | | | | | | | | | | | 2.5 | |
| Aromatic Hydrocarbon Resin | | | | | | | | | | | | | | | 10.0 | |
| Sulfonic Acid—Paraffin Oil | | 1.08 | | | | | | | | | | | | | | |
| White Petroleum Grease | | | | | | | | | | | | | | | 3.0 | |
| Dibutyl Phthalate | | | | | | | | | | | | 15.0 | | | | |
| Light Process Oil | | | | | | | | 17.86 | 17.86 | 17.86 | 17.86 | | | | 7.0 | |

## TABLE I—VULCANIZED WIRE JACKET AND INSULATION COMPOUNDS (Continued)

| Ingredients | Compound Type | NR Ins | NR Jkt | SBR Ins | SBR Ins | SBR Ins | SBR Ins | SBR Jkt | CR Jkt | CR Jkt | CR Jkt | CR Jkt | NBR Jkt | IIR Ins | IIR Jkt | CSM Jkt | SI Jkt |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Copper-Steel Cond (Aluminum Cond) | 300 | 301 (316) | 302 | 303 | 304 | 305 | 306 (317) | 307 (318) | 308 (319) | 309 | 310 | 311 | 312 | 313 (320) | 314 (321) | 315 |
| *Age Resistors* | | | | | | | | | | | | | | | | | |
| 65 N-phenyl-beta-naphthylamine/35 dppd | | 1.0 | | 0.5 | 0.5 | 0.5 | 0.5 | 3.0 | | | | | | | | | |
| Sym. dibetanaphthyl-ppd | | 1.0 | 0.90 | 0.5 | 0.5 | 0.5 | 0.5 | | | | | | | | | | |
| Trimethyl dihydroquinoline | | | | | | | | | | | | | 3.0 | | | 2.0 | |
| N-phenyl-alpha-napthylamine | | | 1.35 | | | | | | | | | | | | | | |
| N-phenyl-beta-napthylamine | | | | | | | | | 2.0 | 2.0 | 2.0 | 2.0 | | | | | |
| Nickel dibutyl dithio carbamate | | | | | | | | | | | | | | | | 1.0 | |
| NN'-bis (1-ethyl-e-methyl pentyl) ppd | | | | | | | | | | | | | | 1.2 | 1.2 | | |
| Paraffinic wax—Heliozone—E. I. duPont | | 1.0 | 4.5 | 5.0 | 5.0 | 5.0 | 5.0 | | | | | | 4.0 | | | 2.0 | |
| Paraffinic wax—Sunwax 4414–Sun Oil | | | | | | | | 3.0 | | | | | | | | | |
| Paraffinic wax—Herron wax #24—Herron Bros. | | | | | | | | | 4.0 | 4.0 | 4.0 | 4.0 | | | | | |
| Paraffinic wax—AA-1177 wax—Allied Asphalt | | | | | | | | | | | | | | 4.8 | 4.8 | | |
| Paraffin wax — — —Esso | | | | | | | | | 4.0 | 4.0 | 4.0 | 4.0 | | | | | |
| *Fungicides* | | | | | | | | | | | | | | | | | |
| Copper-8-quinolinolate | | | | | | | 1.0 | | | | | 2.0 | | | | | |
| N-trichloromethylthio-4-cyclohexane 1,2 dicarboximide | | | | | 2.0 | | | | | | | | | | | | |
| Zinc dimethyl-dithio carbamate + zinc-2-mercapto benzothiazole | | | | | | | | | | | 2.0 | | | | | | |
| Dihydroablethylammonium pentachlorophenoxide | | | | | | 2.0 | | | | | | | | | | | |
| *Curing Agents* | | | | | | | | | | | | | | | | | |
| 2-Mercaptobenzothiazole | | | | | | | | | | | | | | | 1.0 | | |
| pp'-Dibenzoylquinonedioxime | | | | | | | | | | | | | | 6.0 | | | |
| Di-ortho-tolylguanidine | | 0.22 | 0.18 | | | | | | 0.5 | 0.5 | 0.5 | 0.5 | | | | | |
| Lead monoxide | | | | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 | | | | | | | 40.0 | | |
| Benzothiazole disulfide | | | | | | | | | | | | | | | 0.5 | | |
| 2-Mercaptiomidazoline | | | | | | | | | 0.4 | 0.4 | 0.4 | 0.4 | | | | | |
| Poly-para-dinitrosobenzene | | | | | | | | | | | | | | 0.4 | | | |
| Red lead oxide | | | | | | | | | | 20.0 | 20.0 | 20.0 | | 9.0 | | | |
| 2-Napthalenethiol | | 0.45 | 0.75 | 0.8 | 0.8 | 0.8 | 0.8 | 1.5 | | | | | | | | | |
| Stearic Acid | | 1.0 | — | 1.0 | 1.0 | 1.0 | 1.0 | | | | | | 1.0 | 1.5 | 2.0 | | |
| Sulfur | | 4.0 | 5.1 | 4.0 | 4.0 | 4.0 | 4.0 | 2.0 | 1.0 | 1.0 | 1.0 | 1.0 | 2.0 | 1.5 | 2.0 | | |
| Dipentamethylene thiuram tetrasulfide | | | | | | | | | | | | | | | | 0.5 | |
| Tetra methyl thiuram monosulfide | | 0.33 | 0.43 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 | | | | |
| Tetra methyl thiuram disulfide | | | | | | | | | | | | | | | 1.0 | | |

### TABLE II—VULCANIZABLE MOLDING COMPOUNDS–FORMULATIONS

| Number | 260 | 261 | 262 | 263 | 264 | 265 | 266 | 290 | 291 | 292 | 293 | 294 | 295 | 296 | 297 | 298 | 299 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ingredients / Compound | M210C | M206 | M247 | M226 | M246 | M252 | M252A | | | | H-60-16 | M269 | H-61-8<br>H-61-9 | H-60-8 | H-61-9 | M278 | H-64-1 |
| *Polymer* | | | | | | | | | | | | | | | | | |
| Natural Rubber-Smoked Sheet (NR) | 100.0 | | | | | | | | | | | | | | | | |
| Styrene-butadiene Rubber-1004 (SBR) | | 100.0 | | | | | | | | 25.0 | | | | | | | |
| Polychloroprene Rubber-GN (CR) | | | 100.0 | | | | | | | | | | | | | | |
| Butadiene-acrylonitrile Rubber-1041 (NBR) | | | | 100.0 | | | | | | | | | | | | | |
| Butadiene-acrylonitrile Rubber-1042 (NBR) | | | | | 100.0 | | | | | | | | | | | | |
| Isobutylene-isoprene Copolymer-218 (IIR) | | | | | | 100.0 | 100.0 | | | | | | | | | | |
| Polyethylene-DYOB (PE) | | | | | | | | 100.0 | | | | | | | | | |
| Polyethylene-DYNH (PE) | | | | | | | | | 100.0 | 75.0 | | | | | | | |
| Ester Urethane Rubber-509 | | | | | | | | | | | 100.0 | | | | | | |
| Polychloroprene Rubber-WX (CR) | | | | | | | | | | | | 100.0 | | | | | |
| Ethylene-propylene Copolymer-60 (EPM) | | | | | | | | | | | | | 100.0 | | | | |
| Chlorosulfonated Polyethylene-40 (CSM) | | | | | | | | | | | | | | 100.0 | | | |
| Ethylene-propylene-diene Terpolymer-1070 (EPDM) | | | | | | | | | | | | | | | 100.0 | 100.0 | |
| Ether Urethane Rubber-C | | | | | | | | | | | | | | | | | 100.0 |
| *Fillers* | | | | | | | | | | | | | | | | | |
| Easy-processing Channel (EPC) Black | | | 20.0 | 50.0 | | | | | | | | 25.0 | | | | | |
| Fast-extruding Furnace (FEF) Black | | | | | 40.0 | | | | | | | | | | | 20.0 | |
| Medium-thermal (MT) Black | | | | | | | | 418.0 | 100.0 | 100.0 | 100.0 | 55.0 | 100.0 | 25.0 | | 20.0 | |
| Semi-reinforcing Furnace (SRF) Black | 47.0 | 45.0 | | | | 50.0 | 50.0 | | | | | | | | | | 40.0 |
| High-abrasion Furnace (HAF) Black | | | | | | | | | | | | | | | 50.0 | | 15.0 |
| Hard Clay | | | 80.0 | | | | | | | | | | | | | 125.0 | |
| *Plasticizers* | | | | | | | | | | | | | | | | | |
| Polyethylene-8406 | | | | | | | | | | | | | | | | | 3.0 |
| Pine Tar | 2.0 | | | | | | | | | | | | | | | | |
| Light Process Oil | | 15.0 | 12.0 | | | 5.0 | 5.0 | | | | | | | | 20.0 | | |
| Para Coumarone-indene Resin P-10 | | | | 20.0 | | | | | | | | | | | | | |
| Mineral Rubber | | | | | | | | | | | | 10.0 | | | | | |
| Polyether-TP-90B | | | | | | | | | | | | 10.0 | | | | | |
| Aromatic Type Oil-53 | | | | | | | | | | | | | | | | 30.0 | 10.0 |

### TABLE II—VULCANIZABLE MOLDING COMPOUNDS–FORMULATIONS (Continued)

| Number | 260 | 261 | 262 | 263 | 264 | 265 | 266 | 290 | 291 | 292 | 293 | 294 | 295 | 296 | 297 | 298 | 299 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ingredients / Compound | M210C | M206 | M247 | M226 | M246 | M252 | M252A | | | | H-60-16 | M269 | H-61-8<br>H-61-9 | H-60-8 | H-61-9 | M278 | H-64-1 |
| *Protectants* | | | | | | | | | | | | | | | | | |
| Phenyl-beta-napthylamine (PBNA) | 1.0 | | | | | | | | | | | | | | | | |
| 65%PBNA/35% Diphenyl-p-phenylenediamine | | | 3.0 | | | | | | | | | 3.0 | | | | | |
| Nickel Dibutyl Dithiocarbamate | | | | | | | | | | | | | | 3.0 | | | |
| Paraffinic Wax Blend #1* | | 3.0 | | | | | | | | | | | | | | | |
| Paraffinic Wax Blend #3† | | | 4.0 | | | | | | | | | 3.0 | | | | | |
| Paraffinic Wax Blend #6‡ | | | | | | 6.0 | 6.0 | | | | | | | | | | |
| *Vulcanizing Agents* | | | | | | | | | | | | | | | | | |
| Stearic Acid | 3.0 | | 0.5 | 1.5 | 1.0 | | | | | | | 0.5 | | | | 1.0 | |
| Tetramethyl Thiuram Disulfide | 0.1 | | | | 2.5 | 1.0 | | | | | | | | | | | |
| 2-Mercaptobenzothiazole | 1.0 | | | | | | | | | | | | | | 0.5 | 1.5 | 1.0 |
| Zinc Oxide | 5.0 | | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 | | | | | 5.0 | | | 5.0 | 5.0 | |
| Magnesium Oxide | | | 4.0 | | | | | | | | | | | | | | |
| Zinc Chloride 2 Benzothiazolyl Disulfide | | | | | | | | | | | | | | | | 0.35 | |
| Tetramethyl Thiuram Monosulfide | | 0.4 | | 0.5 | | | | | | | | | | | 1.5 | | |
| Benzothiazyl Disulfide | | | | | | | | | | | | | | 0.5 | | | 3.0 |
| Di-ortho-tolyguamidine Salt of Dicatechol Borate | | | 0.25 | | | | | | | | | | | | | | |
| Piperidinium Pentamethylene Dithiocarbamate | | | 0.3 | | | | | | | | | | | | | | |
| 4,4′-Dithiodimorpholine | | | | | 1.5 | | | | | | | | | | | | |
| Diabenzoyl-p-quinone Dioxime | | | | | | | 3.0 | | | | | | | | | | |
| Red Lead Oxide ($Pb_3O_4$) | | | | | | | 10.0 | | | | | | | | | | |
| Tetra Methyl Thiuram Disulfide | | | | | | | | | | | | 0.5 | | | | | |
| Sulfur | 3.0 | 2.0 | | 1.25 | | 1.5 | | | | | | | | | 1.5 | 2.5 | 1.5 |
| Dicumyl Peroxide-R (96-99%) | | | | | | | | 4.0 | 4.0 | 4.0 | 4.0 | | 4.0 | | | | |
| 2-Mercaptiomidazoline | | | | | | | | | | | | 1.0 | | | | | |
| Lead Monoxide-Litharge | | | | | | | | | | | | | | 20.0 | | | |
| Dipentamethylene Thiuram Tetrasulfide | | | | | | | | | | | | | | 0.75 | | | |
| Zinc Dimethyldithiocarbamate | | | | | | | | | | | | | | | | 1.5 | |

* Heliozone—E. I. duPont
† Herron Wax #24—Herron Bros.
‡ AA-1177-20 [Wax + 20% N,N′-bis (1-ethyl-e-methyl) p-phenylene diamine-Allied Asphalt]

TABLE III—RUBBER COMPOUNDS USED IN BONDING STUDY

| Ingredients | M-206 | M-210C | M-204 |
|---|---|---|---|
| Styrene butadiene copolymer—SBR 1004 | 100.0 | — | — |
| Natural rubber—smoked sheet | | 100.0 | — |
| Polychloroprene—GNA | | | 100.0 |
| Black—SRF | 45.0 | 47.0 | — |
| Black—HAF | | | 50.0 |
| Light process oil | 15.0 | — | 15.0 |
| Pine tar | | 2.0 | — |
| Paraffinic wax | 3.0 | — | — |
| Zinc oxide | 5.0 | 5.0 | 5.0 |
| Stearic acid | | 3.0 | 0.5 |
| Phenyl beta naphthylamine | | 1.0 | 1.5 |
| Sulfur | 2.0 | 3.0 | — |
| Tetramethylthiuram monosulfide | 0.4 | — | — |
| Benzo thiazyl disulfide | 0.4 | — | — |
| 2-Mercapto benzothiazole | | 1.0 | — |
| Tetramethylthiuram disulfide | | 0.1 | — |
| Magnesia—extra light calcined | | | 4.0 |
| Diorthotolyl guanidine salt of dipyrocatechol borate | | | 0.4 |

in the styrene-butadiene rubber (SBR) insulation #305. This same fungicide offered no protection, however, to the polychloroprene (CR) jacket #310, although it has been reported as giving CR "complete protection against fungus attack."[4]

In general, the mechanical properties of the rubber wire coatings that were intact and not damaged by insects or micro-organisms were not seriously affected. The natural rubber compounds #300, 301, and 316 showed some softening and the SBRs #302–306 and 317 changed little, while most of the polychloroprenes tended to harden. The CR (#310) containing copper-8-quinolinolate fungicide, however, showed some softening. The butadiene-acrylonitrile copolymer (NBR) compound #311 hardened more than the other compounds. Its compression resistance measurement (see Appendix A) doubled in only one year. This degree of hardening was not expected.

Although the adhesion* of the wire coating to the conductor varied considerably, in general it appeared that the burial did not reduce adhesion materially. The exceptions were those wires in which the conductor had corroded and those with the blistered jackets, which

---

* Adhesion test—measurement of the resistance in pounds required to pull a 1-3/8 inch length of conductor free from 3/8 of an inch of insulation.

### TABLE IV—CROSSLINKED POLYETHYLENE WIRE JACKETS

| Ingredients | #485 | #486 | #487 | #488 | #489 | #490 |
|---|---|---|---|---|---|---|
| | | | *Composition in Parts* | | | |
| Polyethylene DYNH | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| Medium Color Channel Black | 2.0 | 2.0 | 2.0 | — | 2.0 | 2.0 |
| Medium Thermal Black | — | 50.0 | 100.0 | — | — | 50.0 |
| Calcium Carbonate | — | — | — | 50.0 | — | — |
| Dicumyl Peroxide—T—(90–92%) | 2.5 | 2.5 | 2.5 | 2.5 | — | — |
| 2,5-Di-*t*-butylperoxy-2,5-Dimethyl Hexane Powder | — | — | — | — | 4.0 | 4.0 |
| | | | *Extrusion Conditions* | | | |
| Cylinder Temperature °F | 210 | 210 | 200 | 230 | 190 | 260 |
| Head Temperature °F | 270 | 275 | 280 | 270 | 270 | 275 |
| Worm Speed RPM | 15 | 15 | 12 | 17 | 17 | 15 |
| | | | *Compression (1-inch jaw-2000 lbs)* | | | |
| Original | 1110 | 940 | 610 | 690 | 1030 | 820 |
| Original + 2 days 212°F oven | 1360 | 970 | 590 | 640 | 1250 | 780 |
| 8 years—New Mexico | 1190 | 1020 | 619 | 62 | 1409 | 844 |
| 11+ years—shelf-aged control | — | — | — | 721 | — | — |
| | | | *Mooney Scorch (320°F-small rotor at 2 rpm)* | | | |
| Minimum (M) | 4.4 | | | | 4.2 | |
| Time to minimum (TM) | 1.0 | | | | 2.4 | |
| TM + 2 point rise | 3.9 | | | | 8.0 | |
| TM + 5 point rise | 5.2 | | | | 12.2 | |
| TM + 10 point rise | 6.3 | | | | 17.9 | |
| TM + 35 point rise | 11.5 | | | | 66.0 | |

TABLE V—RECENT ADDITIONAL WIRES IN TEST
(Ethylene-propylene Copolymer and Diene-modified Terpolymer)

| | Compound | #322 | #323 | #324 |
|---|---|---|---|---|
| Ingredients | Type | EPDM Jkt | EPM Jkt | EPM Ins |
| Ethylene-propylene-diene-terpolymer (EPDM) Rubber 1040 | | 100.0 | — | — |
| Ethylene-propylene-copolymer (EPM) Rubber 404 | | | 100.0 | 100.0 |
| Fast-extruding furnace (FEF) Black | | 100.0 | 25.0 | — |
| High abrasion-low structure-furnace (HAF-LS) Black | | | 50.0 | — |
| Semi-reinforcing furnace (SRF) Black | | | | 3.0 |
| Treated Aluminum Silicate Complex | | | | 110.0 |
| Trimethyl Dihydroquinoline | | | 1.0 | 1.0 |
| Zinc Oxide | | 5.0 | 5.0 | 5.0 |
| Naphthenic Petroleum Oil, ASTM Type 3 | | 50.0 | 25.0 | — |
| Paraffinic Petroleum Oil, ASTM Type 4 | | | | 35.0 |
| Stearic Acid | | 1.0 | — | — |
| Sulfur | | 2.0 | 0.3 | 0.3 |
| Dicumyl Peroxide T (90-93%) | | | 4.25 | 4.25 |
| Red Lead Oxide | | | | 3.75 |
| Dibasic Lead Stearate | | | | 0.5 |
| 2-Mercaptobenzothiazole | | 2.0 | — | — |
| 50% Zinc Dimethyldithiocarbamate ±50% Tetramethylthiuramdisulfide | | 4.0 | — | — |
| Tellurium Diethyldithiocarbamate | | 1.33 | — | — |

Compression Resistance (1-inch jaw, 2000 psi) after 4 years subsoil burial

| | | #322 | #323 | #324 |
|---|---|---|---|---|
| Roswell, N. M. | (alkali soil) | 645 | 779 | 677 |
| Bainbridge, Ga. | (acid soil) | 663 | 409 | 260 |

were all specimens with the applied potential. The test showed that aluminum conductors of this type should not be buried since they corroded rapidly during exposure. Some parts of the aluminum conductors under the polychloroprene jackets #318 and #319 were completely corroded in less than a year. The above test results are given in Table VI.

Figure 1a shows a reeled length of the blistered SBR insulated wire #302A-1 and Fig. 1b is an enlargement of one of these blisters. This type of blister was common also on the A-1 samples (under potential) #303, 304, 308, and 310.[5] The copper-steel conductors under the rubber insulation of some of these were found to contain pits due to corrosion in their copper coatings. But not all blistered specimens had corrosion pits, nor did all pitted specimens have blistered insulations. Pronounced

## TABLE VI—TEST RESULTS ON RUBBER COVERED WIRE AFTER ONE YEAR SOIL BURIAL AT BAINBRIDGE

| | Compression Resistance | | | | | Adhesion | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | After Exposure | | | | | After Exposure | | | |
| Sample | Original | With Potential | % Change | Without Potential | % Change | Original | With Potential | % Change | Without Potential | % Change |
| 300 NR I | 1700 | 1475 | −13.2 | 1385 | −18.5 | 8.0 | 6.9 | −13.8 | 2.7 | −66.3 |
| 301 NR J | 1785 | 1785 | 0 | 1850 | +3.6 | 9.1 | 3.9 | −57.1 | 4.1 | −55.0 |
| 302 SBR I | 1005 | 1010 | +1.0 | 970 | −3.5 | 8.0 | 5.0 | −37.5 | 6.5 | −18.8 |
| 303 SBR I* | 870 | 1230 | +41.4 | 995 | +14.4 | 7.6 | 6.5 | −14.5 | 8.9 | +17.1 |
| 304 SBR I† | 1040 | 910 | −12.5 | 995 | −4.3 | 4.4 | 3.2 | −27.3 | 3.8 | −13.6 |
| 305 SBR I§ | 1105 | 1205 | +9.1 | 1265 | +14.5 | 7.2 | 8.9 | +23.6 | 7.0 | −2.8 |
| 306 SBR J | 1415 | 1310 | −7.4 | 1335 | −5.7 | 4.5 | 3.9 | −13.3 | 3.7 | −17.8 |
| 307 CR J | 900 | 1370 | +52.2 | 1265 | +40.6 | 0.6 | 3.8 | +533.3 | 2.2 | +266.7 |
| 308 CR J | 1105 | 1595 | +41.3 | 1540 | +39.4 | 3.8 | 5.7 | +50.0 | 3.9 | +2.6 |
| 309 CR J‡ | 1395 | 2005 | +43.7 | 1775 | +27.2 | 9.7 | 9.8 | +1.0 | 7.2 | −25.8 |
| 310 CR J§ | 1425 | 1110 | −22.1 | 800 | −43.9 | 7.5 | 3.8 | −49.3 | 3.0 | −60.0 |
| 311 NBR J | 1360 | 2415 | +77.6 | 2780 | +104.4 | 1.5 | 9.0 | +500.0 | 7.4 | +393.3 |
| 312 IIR I | 920 | 1105 | +20.1 | 1115 | +21.2 | 10.1 | 8.1 | −2.0 | 7.4 | −36.6 |
| 313 IIR J | 1090 | 835 | −23.4 | 1170 | +7.3 | 6.3 | — | — | 12.4 | +96.8 |
| 314 CSM I | 980 | 1335 | +36.2 | 1330 | +35.7 | 10.2 | 8.0 | −21.6 | 9.4 | −7.8 |
| 315 SI — | 510 | 540 | +5.9 | 510 | 0 | 0 | 0.9 | | — | |
| 316 NR J | 1785 | 1280 | −28.3 | 1540 | −13.7 | | — | | — | |
| 317 SBR J | 1415 | 1055 | −25.4 | — | — | | — | | — | |
| 318 CR J | 900 | — | — | — | — | | — | | — | |
| 319 CR J | 1105 | — | — | — | — | | — | | — | |
| 320 IIR J | 1090 | 790 | −27.5 | 740 | −32.1 | | — | | — | |
| 321 CSM J | 980 | 800 | −18.4 | 800 | −18.4 | | — | | — | |

* N-trichoromethylthio-4-cyclohexane 1, 2-dicarboximide
† Dihydroabiethylammonium pentachlorophenoxide
‡ Zinc dimethyldithiocarbamate and Zinc 2-Mercaptobenzothiazole
§ Copper-8-quinolinolate
I—Insulation J—Jacket
Note: All conductors are copper except #316 through #321 which are aluminum.

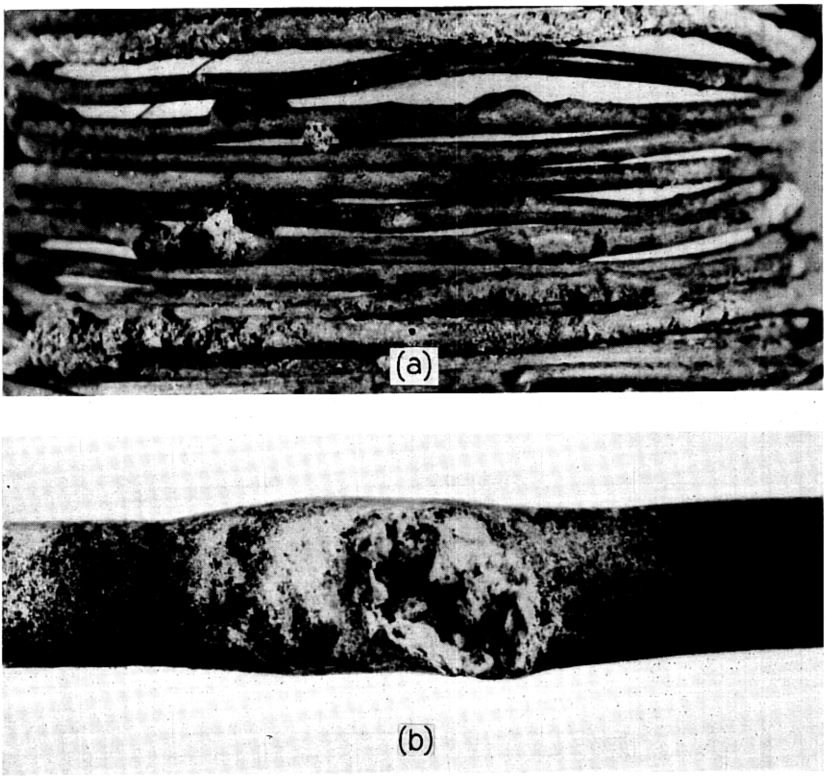


Fig. 1—(a) Blisters and surface deposits on a spool of SBR insulated wire #302A-1 after one year burial at Bainbridge. (b) Enlargement of a burst blister on this specimen.

pitting was found on the #305A-1 and #307A-1 samples and on the A-11 samples (without potential) #301, 304, 308, and 309. In general, the specimens under potential showed more degradation than those without potential and the degree of corrosion seemingly had little correlation with most of the rubber compounds tested.

### 2.1.2 *Discussion*

Corrosion has been defined as "a process by means of which metals undergo chemical reactions with nonmetallic elements in their environments producing chemical compounds which are either oxides or salts."[6] It has been well established that the process of corrosion is electrolytic in character.[7] A current flows between the anode and cathode areas and the amount of corrosion is proportional to the amount of current flow.

Discrete corrosion cells appear to have been created at the blisters, possibly due to discontinuities, pinholes, foreign inclusion, or any inhomogeneities which are certain to exist[8] and thus supply a point of potential difference.[7] The rubber coatings are permeable and water accumulates under a coating in the form of the blisters providing a wet environment for the creation of an oxygen concentration cell.[9,10] R. M. Burns and W. W. Bradley have warned that an accumulation of alkali, natural or as a corrosion product, in the cathodic areas of the electrolytic cells may lessen adhesion, give rise to blistering, and cause chemical disintegration in the structure of the coating.[8]

Figure 2a is an example of a typical blister on one of polychloroprene

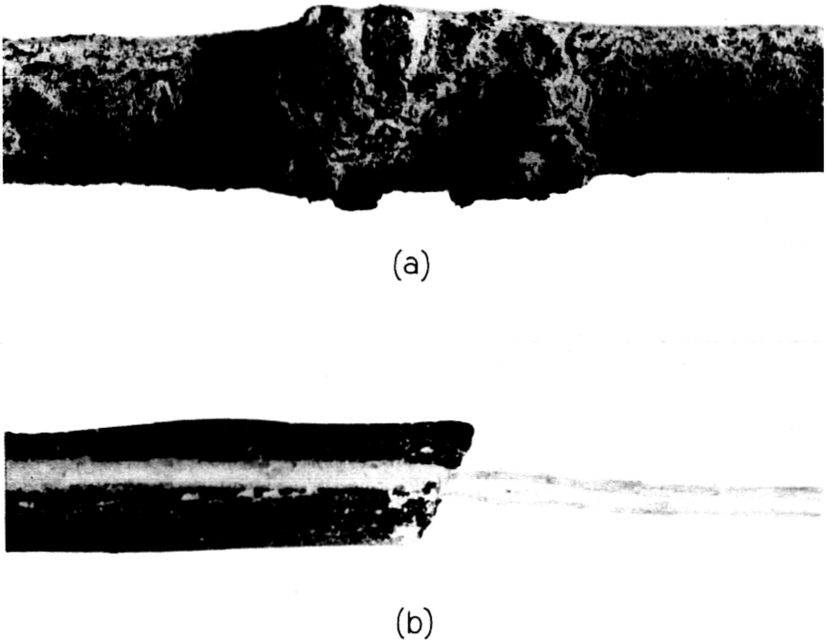


Fig. 2—(a) Typical blister on the polychloroprene jacketed aluminum wire #318A-1 after burial at Bainbridge for one year. (b) Cutaway section of one of these blisters revealing the severe corrosion of the aluminum conductor and the powdered aluminum oxide corrosion product.

jacketed aluminum wires (#318A-1) and Fig. 2b shows a cutaway section of one of these blisters which reveals the powdered aluminum oxide ($Al_2O_3$) corrosion product.[5] The conditions for corrosion of aluminum were so aggressive in some locations that the conductor was found to have completely corroded away leaving only the white oxide powder in its place after only a year's burial.

Aluminum is attacked by both alkalies and dilute acids (amphoteric).[7] With polychloroprene rubber, there is the possibility of the chlorination of the aluminum. Also, a high chloride content in the soil will destroy the protective oxide film and increase the conductivity of the electrolyte.[8] Additionally, the corrosion product on the surface influences the distribution of the attack, for beneath it the aluminum becomes anodic and corrodes.[11]

The loss in insulation resistance that occurred in this experiment is not necessarily a detriment in the case of the 15 jacket stocks, but it does mean that the 7 insulation compounds tested are virtually useless where soil burial is involved.

The loss in insulation resistance of buried insulated wire samples has been demonstrated before in other testing programs such as that of J. T. Blake, et al., and this loss was traced to soil fungi.[12,13] These microorganisms create micropores through the insulation which fill with water. The micropores occurred although the base hydrocarbon was stable. The fungi appear to consume the nutrient additives in the insulation, and the resultant tiny passages allow water to penetrate.

Also, the very poor performance demonstrated by the nonblack IIR insulation described above is similar to that reported by Blake, et al., for a white polychloroprene compound containing a hard clay filler.[13] They found this material to be more vulnerable to fungus attack than their black CR.

Because of the continuing trend of electrical and physical deterioration, further evaluations of the early rubber covered wires were discontinued after four years and the wires were removed from the test plots.

### 2.2 *Chemically Crosslinked Polyethylene Covered Wires*

Unlike the 1958 rubber covered wires, the crosslinked polyethylene wire specimens of 1959 and 1960 were left in the soil as planned. The black compounds showed no visible damage or loss in physical properties in the initial tests. Also, there seemed to be no indications of micropore formation. As can be seen in Table IV, these XPE compounds contained few additives and they did not contain plasticizers which are usually quite susceptible to microbial degradation. Interestingly, though, after eight years the buried specimens without black (#488) were extremely brittle and resembled nonblack specimens that had undergone severe ultraviolet radiation exposure. To confirm that no UV degradation has occurred, an infrared spectrum was run on the brittle jacket by the Attenuated Total Reflectance method and it revealed a significant carbonyl absorption band but an absence of any UV-formed vinyl bands.[14] However, the specimens had the characteristic, butyric acid-like, rancid odor of oxidized polyethylene. Also, the conductor beneath the deteriorated white jacket (#488) was found to be badly corroded while the conductors under the black jackets were in excellent condition.

In addition to not containing any carbon black, the white stock was the only one that contained calcium carbonate. This deterioration was unexpected since calcium carbonate is often listed as being inert or resistant and even regarded as a protective additive.

Embrittlement and corrosion were first noted on the eight-year white specimens retrieved from the Roswell burial site. This deterioration had not been observed on the previous samples removed after only four years exposure. Although no XPE specimens had been retrieved after eight years of burial at Bainbridge, a special visual inspection was made after a little more than ten years and the same brittle condition was found.

Fortunately, small particle size channel carbon black at a concentration of less than 2 percent is evidently a very effective protectant for chemically crosslinked polyethylene under soil burial conditions. It should be noted, however, that the #487 sample with a 100-part loading (<50 percent) of the coarser medium thermal (MT) black does have some holes and chew marks.

Included in Table IV are the results of compression tests* conducted on specimens of the compound after aging for two days at 100°C. These data are of interest because they reveal how well the nonblack compound #488 withstood this type of thermal aging and yet deteriorated badly in the soil. Similarly, the control specimens of this white #488 wire that have been shelf-aged for eleven years have remained unaffected both visually and physically. The contrast between the buried and shelf-aged, white, crosslinked polyethylene specimens and the seemingly unaffected buried black specimens can be seen in Fig. 3. Although the specimens were exposed straight and unstrained, they

---

* See Appendix A.

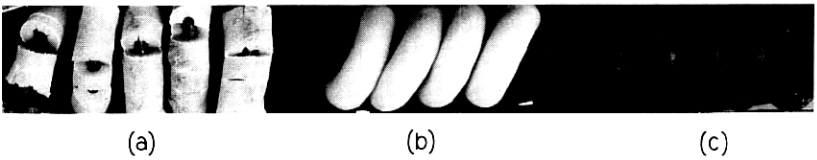


Fig. 3—Chemically crosslinked polyethylene covered wires. (a) Cracked and brittle white jacket compound #488 retrieved after eight years burial in the soil at Roswell. (b) Well-preserved white jacket compound #488 after 11 + years of shelf-aging in storage at Bell Laboratories, Murray Hill, N. J. (c) Apparently unaffected 2-percent black polyethylene compound #485 retrieved after eight years burial in the soil at Roswell.

were later wrapped around a mandrel to illustrate the extreme brittleness of the buried white specimen #488.

No other reports have been found of this difference in the soil burial aging of black and white chemically crosslinked polyethylene.

Both chemical and physical change can initiate brittle failures in polyethylenes.[15] For example, oxidation mechanisms are chemical, while stress cracking and thermal embrittlement are physical. They can act singly or in concert and it was difficult to identify the cause in the soil environment. Water was present and cracking seems to have occurred.

The addition of a chemical crosslinking agent permits the relatively inexpensive use of the combination of a low molecular weight grade of polyethylene and carbon black. The chemistry of free radical production and crosslinking by dicumyl peroxide has been described by E M. Dannenburg, et al.[16]

In this exposure experiment, two different peroxide vulcanizing agents were used to produce a 30-mil-thick jacket over 20-gauge copperweld conductors. It is now known that the 2,5-di-*t*-butylperoxy-2,5-dimethylhexane agent requires a higher temperature or slower extruder speed than the dicumyl peroxide agent. This difference is illustrated in Table IV with the Mooney Scorch Test* results for compounds #485 and #489. No allowance was made for this difference when these wires were jacketed and this probably explains the 30 percent increase in the physical test properties of the aged #489 specimens. An undercured vulcanizate often continues to cure with time and an increasing tensile strength is one indication of this.

Carbon black plays varied and important roles in the protection of

---

* Mooney Scorch Test—Mooney Shearing Disk Viscometer Test according to the procedure given in the 1967 Book of ASTM Standards, Part 28, D1646-63, ASTM, Phil., Pa., utilizing the small rotor at 2 rpm at 320°F.

polyolefins. It is effective as a light screen in protecting against photooxidation[17] and is used as a UV absorber in Bell System cables.[18] It also functions as an effective antioxidant.[19,20]

In general, the test program demonstrates the excellent resistance to degradation during soil burial of black chemically crosslinked polyethylene.

### 2.3 *Ethylene-Propylene Rubber Covered Wires*

Many specimens of the ethylene-propylene rubbers buried in 1965 are still in test. Coated wire specimens retrieved after four years of exposure in topsoil showed a considerable amount of insect damage, blistered jackets, and corroded conductors, but there was little or no damage in specimens buried in the subsoil. Although the subsoil sections appeared to be undamaged, there was a significant difference in the results of the physical tests conducted on specimens from the two exposure test sites (Table V). While the diene-terpolymer (EPDM) jacket specimens (#322) were apparently unaffected by the different soil and climate conditions, both the EPM (copolymer) insulation and EPM jacket formulations (#323 and #324) suffered a 50 percent greater loss in compression resistance after exposure in the acid soil than they did in the alkaline soil. This is the first rubber showing a difference in reaction to the soils of the two test sites.

## III. MOLDING COMPOUNDS

The Bell System utilizes a great number of molded rubber parts. This requires the development of and experience with many types of rubber compounds. In 1958, seven then currently used rubber compounds based on natural, styrene-butadiene, polychloroprene, nitrile-butadiene, and butyl rubber polymers were put into the soil burial program. The test specimens were prepared in the form of dumbbells cut with ASTM Die C from molded sheets as per ASTM D412-62T.[21]

Three additional compounds of rigid black-loaded chemically crosslinked polyethylene were later added. In 1960, the following materials were also added:

(*i*) A polyester urethane rubber with peroxide cure;
(*ii*) An ethylene-propylene rubber with peroxide cure;
(*iii*) A black-loaded polychloroprene containing no clay;
(*iv*) A black-loaded chlorosulfonated polyethylene.

Finally, in 1964, three more "improved" compounds were placed in the test. These consisted of two ethylene-propylene rubbers with and without clay and a black-loaded polyether urethane rubber.

A complete description of the formulation of all of these molded vulcanizates is given in Table II.

Measurements of physical properties of the original compounds were made immediately after molding. It was thus possible to compare the effects of soil burial and normal aging. The test data are presented in Table VII as tensile strength and percent elongation, while the retained percentage of these physical properties with increasing exposure time is graphically illustrated in Figs. 4 through 7. The measurements were made in accordance with ASTM D412-62T.[21]

It was unfortunate that precut dumbbells were used for testing the soil burial properties of the molding compounds because insects or rodents chewed the edges of the dumbbells. Some of these damaged specimens are shown in Figs. 8 and 9. It is now obvious that complete molded sheets should have been buried so that test specimens could have been cut from undamaged areas.

The exposure plot was sprayed with an insecticide (heptachlor) before the first samples were buried. It appears that the initial spray treatment was only effective for a few years. The damage became worse as the population became reestablished in succeeding years.

### 3.1 *Exposure Results*

The initial clay-loaded black polychloroprene #262 failed badly (50 percent loss in tensile strength) in less than one year due to water absorption. A second CR composition (#294) designed for soil burial with an easy processing channel (EPC) black loading and no clay showed greater resistance to deterioration. It retained its physical properties but during eight years of exposure it sustained much insect damage, particularly in New Mexico.

The compound showing the best performance in the first series of vulcanizates was #263—the high nitrile NBR with an EPC carbon black filler. Although there was some loss in elongation in New Mexico, the specimens were still in excellent condition when removed from both exposure sites after an eight-year burial. A loss in elongation was also found in the shelf-aged specimens so this did not probably result from the soil burial.

On the other hand, the nonblooming, heat resistant nitrile #264 lost about 20 to 25 percent of its original tensile strength while the elongation of the control remained fairly constant. It also had little insect damage.

## TABLE VII—MOLDING COMPOUNDS–PHYSICAL TEST RESULTS

| Compound | Site A-Georgia B-New Mexico | Original Tensile Strength psi | 8 yr Control Tensile Strength psi | 8 yr/6 inches Burial Tensile Strength psi | 8 yr/18 inches Burial Tensile Strength psi | Original Elongation % | 8 yr Control Elongation % | 8 yr/6 inches Burial Elongation % | 8 yr/18 inches Burial Elongation % |
|---|---|---|---|---|---|---|---|---|---|
| *260* Natural rubber | A | 3350 | 2490 | 1270 | 1460 | 490 | 310 | 160 | 170 |
| Black shock mount | B | 3350 | 2760 | 1310 | 1060 | 490 | 320 | 170 | 140 |
| *261* Styrene-butadine | A | 1225 | 900 | 930 | 1250 | 410 | 240 | 230 | 250 |
| Low comp. set | B | 1225 | 1325 | 1240 | 895 | 410 | 250 | 220 | 220 |
| *262* Polychloroprene-with clay | A | 2150 | 2080 | 900 | 1000 | 495 | 180 | 200 | 220 |
| Cover stock | B | 2150 | 1825 | 1520 | 1910 | 495 | 260 | 60 | 130 |
| *263* Nitrile-sulfur cure- | A | 2355 | 2210 | 2200 | 2450 | 460 | 330 | 300 | 340 |
| high shear | B | 2355 | 2750 | 2300 | 2340 | 460 | 210 | 160 | 160 |
| *264* Nitrile-heat resistant- | A | 3095 | 3180 | 2140 | 2500 | 320 | 240 | 180 | 220 |
| non-blooming | B | 3095 | 2750 | 2450 | 2260 | 320 | 240 | 210 | 200 |
| *265* Butyl-sulfur cure | A | 2460 | 2040 | 1800 | 2090 | 795 | 690 | 590 | 720 |
|  | B | 2460 | 2300 | 1340 | 1720 | 795 | 750 | 550 | 610 |
| *266* Butyl-quinoid cure | A | 1350 | 1010 | 950 | 930 | 650 | 600 | 450 | 430 |
| Hydrophone | B | 1350 | 1000 | 880 | 730 | 650 | 460 | 300 | 250 |
| *290* Polyethylene | A | 3310 | 3580 | 3440 | 3380 | no elongation measurements made |  |  | — |
| crosslinked-black | B | 3310 | 3700 | 3560 | 3570 |  |  |  |  |
| *291* Polyethylene | A | 2780 | 2790 | 2740 | 2710 | no elongation measurements made |  |  | — |
| crosslinked-black | B | 2780 | 2930 | 2780 | 2720 |  |  |  |  |
| *292* Polyethylene/SBR | A | 2750 | 2780 | 3130 | 3060 | 160 | 60 | 180 | 180 |
| crosslinked-black | B | 2750 | 2980 | 2600 | 3150 | 160 | 140 | 110 | 170 |
| *293* Ester Polyurethane | A | 2440 | (6 Yr) 2100 | (2 Yr) 190* | (2 Yr) 910* | 210 | 280 | (2 Yr) 60* | (2 Yr) 150* |
| black loaded-peroxide | B | 2440 | (6 Yr) 2150 | (2 Yr) 110* | (2 Yr) 330* | 190 | 260 | (2 Yr) 70* | (2 Yr) 110* |
| *294* Polychloroprene-no clay | A | 1690 | 1650 | 1510 | 1520 | 510 | 340 | 200 | 200 |
| -black loaded | B | 1650 | 1650 | 1200 | 1610 | 500 | 340 | 170 | 200 |
| *295* EPM-black loaded- | A | 1390 | 1500 | 960 | 1260 | 440 | 410 | 300 | 350 |
| peroxide cure | B | 920 | 920 | 920 | 820 | 550 | 540 | 500 | 450 |
| *296* Chlorosulfonated | A | 3210 | 3400 | 2140 | 3080 | 660 | 550 | 350 | 430 |
| polyethylene-litharge cure | B | 3080 | 3400 | 2960 | 2770 | 650 | 550 | 450 | 450 |
| *297* EPDM-no clay | 4 Yr A | 3050 | 2525 | 2610 | 2570 | 550 | 410 | 440 | 450 |
| low black-sulfur cure | 4 Yr B | 3160 | 2810 | 2530 | 2360 | 690 | 560 | 480 | 400 |
|  | 8 Yr B | 3160 | 2400 | 2100 | 2140 | 690 | 520 | 380 | 380 |
| *298* EPDM-clay loaded- | 4 Yr A | 1930 | 1600 | 1190 | 1610 | 800 | 620 | 580 | 680 |
| black-sulfur cure | 4 Yr B | 1600 | 1590 | 1500 | 1400 | 770 | 600 | 660 | 680 |
| *299* Ether polyurethane | 4 Yr A | 3400 | 3175 | 3160 | 3150 | 530 | 420 | 440 | 450 |
| black loaded-sulfur cure | 4 Yr B | 3120 | 3280 | 2820 | 2970 | 530 | 450 | 440 | 480 |

* Samples untestable (0) after 4 years exposure

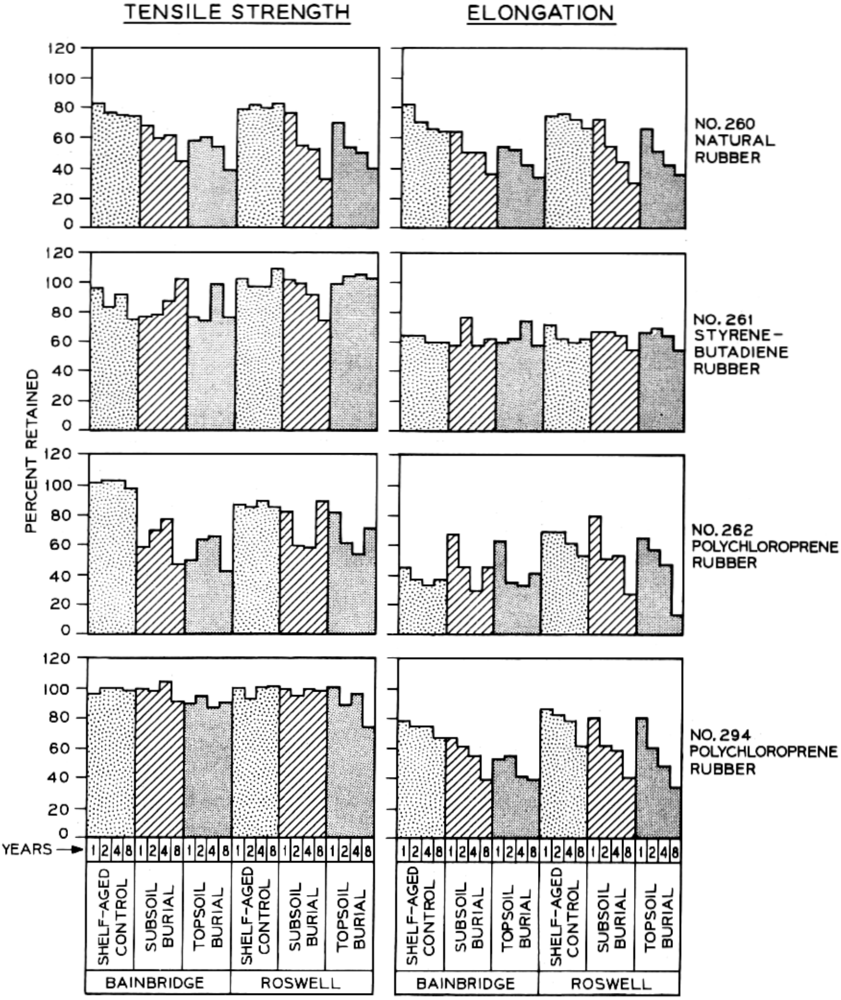


Fig. 4—Buried molding compounds (#260–262, 294)–percentage of retained tensile strength and elongation versus time of soil burial.

The IIR formulations #265 and 266 were designed for use as hydrophone diaphragms and involve different curing systems. The sulfur cure in #265 was found to corrode any contacting metal parts so a quinoid cure was provided in #266. Both butyls retained their physical properties fairly well (see Figure 5), but suffered considerable insect damage, particularly in New Mexico. Thus the test values vary greatly.

The EPM and CSM rubber stocks (#295, 296) appear to be better than many of the earlier vulcanizates but not as good as the sulfur cured nitrile #263. Also, the high-resilience NR shock mount compound #260 did not behave as well as the low-compression set SBR #261 which was only slightly affected.

As in the case of the vulcanized wire coatings, all of the black, chem-

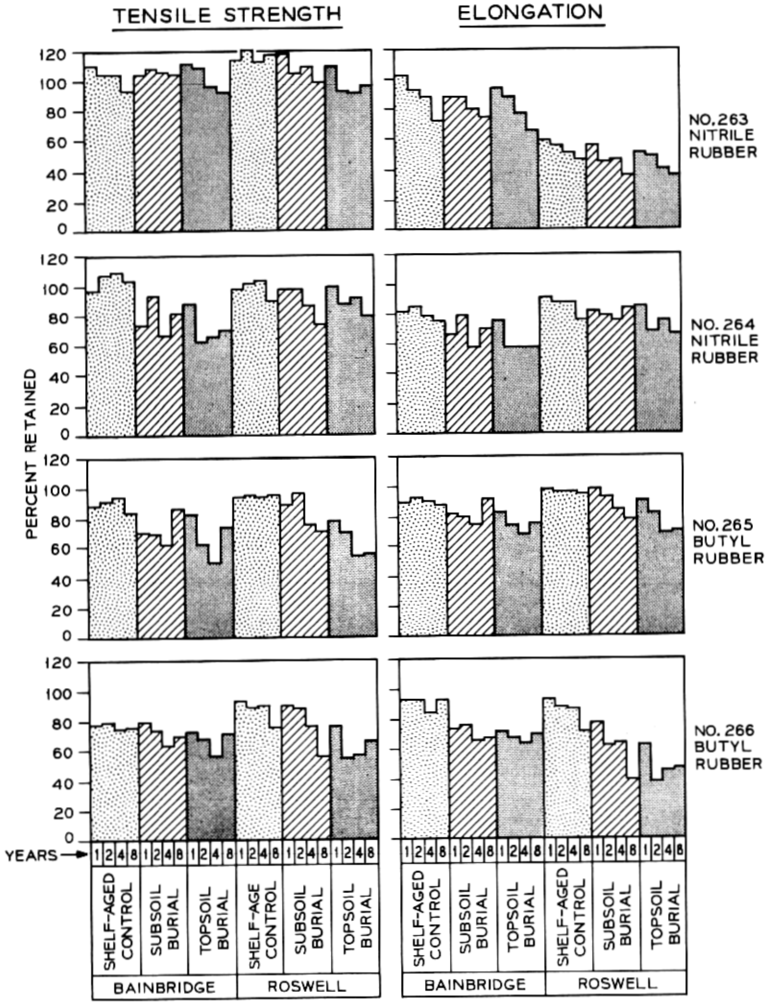


Fig. 5—Buried molding compounds (#263–266)–percentage of retained tensile strength and elongation versus time of soil burial.

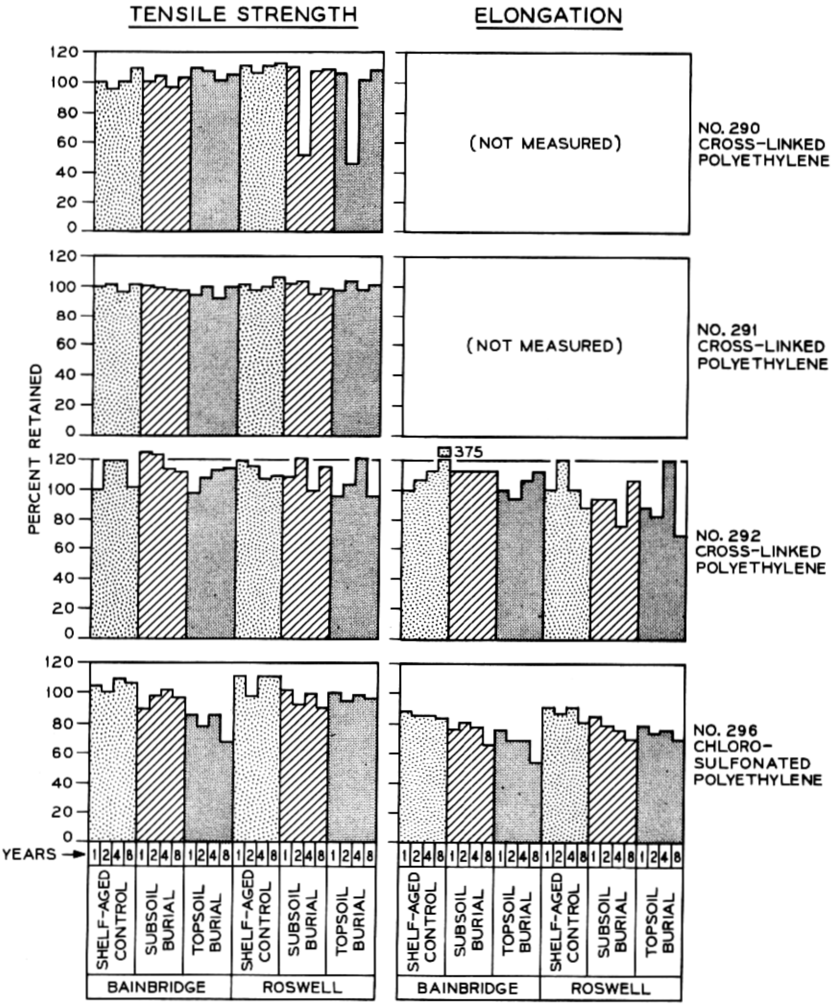
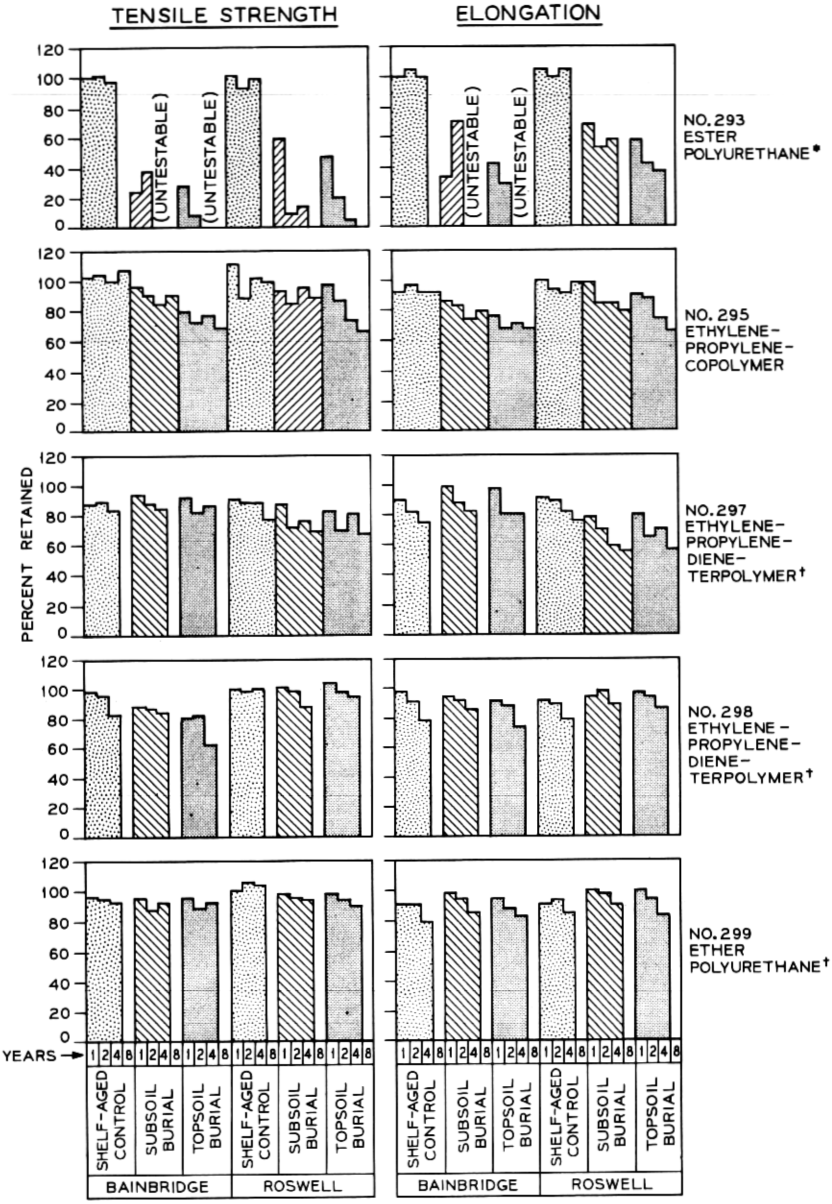


Fig. 6—Buried molding compounds (#290–292, 296)–percentage of retained tensile strength and elongation versus time of soil burial.

ically crosslinked polyethylene molded samples #290–292 appear to be unaffected by soil exposure and are being continued in the burial program.

This burial program has clearly demonstrated that the polyester urethane vulcanizate (#293) was biologically susceptible and subject to hydrolysis. This was evident after only one year of burial, for the #293



*-AFTER 4 YEARS ALL OF THE BURIED SPECIMENS COULD NOT BE TESTED

†-EIGHT YEAR DATA NOT YET AVAILABLE

Fig. 7—Buried molding compounds (#293, 295, 297–299)–percentage of retained tensile strength and elongation versus time of soil burial.

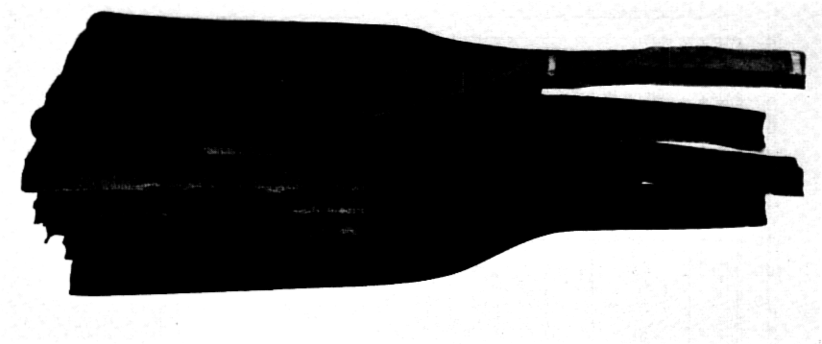


Fig. 8—Insect damaged edges of rubber dumbbell test specimens after four years burial at Roswell.

specimens already had deep cracks. Degradation was so severe that specimens could not be retrieved and tested after six years of exposure. In contrast, the polyether urethane compound (#299) was virtually unaffected by soil burial.

Figure 10 displays one more phenomenon involving the ester polyurethane sample that has occurred in the exposure test. The remains

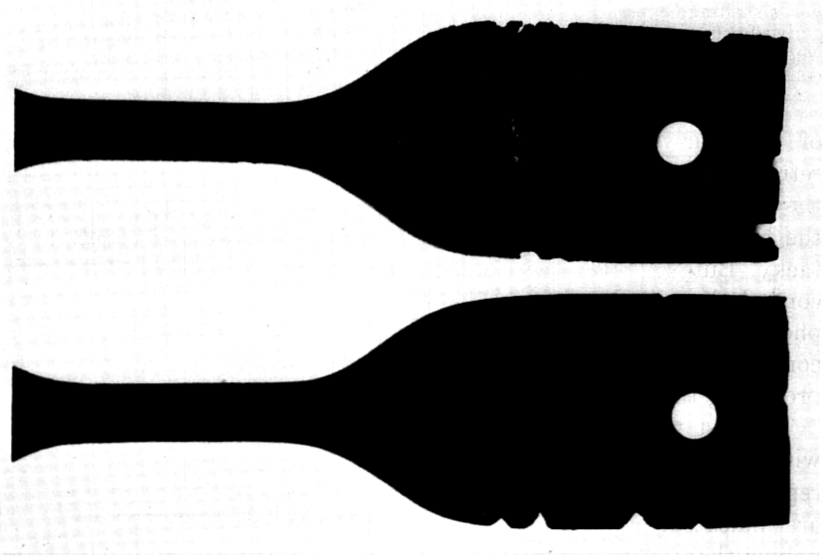


Fig. 9—Insect damaged specimens of butyl molding compounds #265 and #266 after four years burial at Roswell.

Fig. 10—A mixed pile of tested specimens of #293 polyester urethane from four years shelf-aging and four years burial at Roswell which have been in storage together for five years. Generally, the buried specimens were brittle and the shelf-aged were tacky, but where they have been in contact with each other, the shelf-aged controls were found to have liquified.

of the four-year shelf-aged control specimens and those that were retrieved after four years burial at Roswell were stored together after they had been tested. When examined after five years of storage, the burial specimens were found to be brittle and the controls to be tacky. But wherever they had been in contact with each other, the controls had liquified as is illustrated by the shiny wet areas in the photograph. In this same period of time, the remaining untested control specimens in this experiment are also getting moist and approaching this liquid state without contact with any other specimens.

The last three compounds in this program—two EPDMs with and without clay, #297, 298, and the sulfur-cured polyether urethane #299 represent four-year exposure data. Some losses in physical properties are indicated, particularly in elongation for #297 and #298. The #299 polyurethane also shows a very slight loss, as did the shelf-aged controls. None of the three suffered any insect damage.

To summarize the ranking of the better molding compounds for soil burial purposes, the black-loaded, chemically crosslinked polyethylenes are most resistant, followed in succession by the EPC black, high nitrile NBR #263, the black polychloroprene without clay #294, and finally the low-compression set SBR #261.

Since there has been little change during the first four years of exposure as contrasted with the earlier rubbers, with additional exposure time the polyether urethane compound #299 may also prove to be satisfactory for soil burial uses. But the polyester urethane elastomer should not be buried because its hydrolyzes badly. The 60-percent loss in tensile and elongation sustained by the natural rubbers makes them also a poor choice for use in the soil.

### 3.2 *Discussion*

A number of other reports on the effect of water on urethane vulcanizates appeared after the above samples were prepared. R. J. Athey[22] reported that water absorbed by a urethane vulcanizate produces two effects, a reversible plasticization and an irreversible degradation. Hydrolytic degradation of the polymer chain was found to cause a permanent reduction in the properties of the vulcanizate. The functional groups present on the chain, e.g., urethane, urea, or ester groups, are hydrolyzed, resulting in chain scission, loss of branching or crosslinking, and a decrease in molecular weight.

The susceptibility of the ester urethanes to degradation by water arises from the relative ease of hydrolysis of the ester group.[23] B. S. Biggs has reported on the ease of polyester hydrolysis. He found that hydrolysis is accelerated by high temperature and catalyzed by acids and alkalies.[24] Many polymers of this class are stable only when kept neutral and he felt that this has been the main barrier to their greater commercial utilization. W. Cooper, et al., also found that polyester urethanes are less resistant to dilute acid and confirmed that acidic exposure conditions will catalyze degradation.[23]

Practically the same pattern of deterioration was observed in a U. S. Government exposure test of these two types of urethane elastomers.[25] In this study, samples were exposed in the Canal Zone and Kentucky—environments of high humidity—and after one- to two-year service periods the polyester had become tar-like and unfit for further use, while the polyester vulcanizates were not adversely affected. Microbial attack was found along with the hydrolysis. This work also indicated that additions of a polycarbodiimide and pentachlorophenol retarded chain scission and microbial attack, respectively.

It should be noted that there are some "anti-oxidants" now available which are purported to improve the stability and retard the effects of hydrolysis on elastomeric vulcanizates containing ester groups.

Interestingly, a Russian study reported a nitrile rubber (butadiene-acrylonitrile copolymer), containing 15 percent acrylonitrile, tested with natural and butyl rubbers, that was affected by microorganisms, while the other synthetic rubbers in the program did not develop mold.[26] In this same study, all "carbon black vulcanizates" were found resistant to the action of microorganisms. The individual compound ingredients studied, nonblack fillers (kaolin and whiting) and carbon blacks, were also found to resist the action of microorganisms. There are many other ingredients in a typical rubber compound which individually or in combination could affect the resistance of a vulcanizate to degradation and a great deal more selective testing needs to be done.

Uncrosslinked polyethylene (PE) suffers a rapid loss of ductility and a sharp increase in brittle point with the incorporation of increasing loadings of carbon black; but the introduction of crosslinks allows considerable quantities of black to be added without sacrifice in brittle point.[16] In fact, yield point, ductility, elasticity, and tensile properties are improved, while problems of environmental stress cracking are minimized. The creation of the #290 molding compound with its 400 percent loading of medium thermal furnace (MT) black was only possible because of the peroxide vulcanization. Increased elongation or more elastic properties can be provided by including a rubbery polymer in the formulation as was done in compound #292.

## IV. ADHESION STUDIES

Many pieces of equipment in the Bell System require the permanent vulcanized bonding of molded rubber to metal as part of their construction. These composite articles involve a variety of different rubbers and metals. Since much of this equipment will be located underground in the future, samples were prepared for soil burial testing. These samples included a number of combinations of three different rubber compounds, three metals, and several proprietary bonding systems.

The test specimens consisted of rubber laminates composed of a piece of vulcanized rubber, approximately 1/8 inch thick by 1 inch by 3 inches with a 1/2-inch section on one end free of bonding agent. The rubber was bonded to the prepared surface of a similar sized sheet metal strip to provide a peel test specimen. These test specimens were mounted on polyethylene tubes in the conventional manner. A peel test supplies some rough, but valuable information despite its recent critics.[27]

Figure 11 illustrates the comparative pull test used in this program. If the bond is weak, an adhesion failure occurs and no rubber is left on the metal. If the rubber is weak, a cohesion failure occurs and it tears leaving a major portion of the rubber adhering to the metal. Strong bonds and strong rubbers are very difficult to pull apart.

Fig. 11—Hand-peel test used to determine the strength of rubber-to-metal bond. Specimen is #275-SBR compound M-206 bonded to stainless steel with the Chemlok 201 plus Chemlok 220 system—after retrieval from Bainbridge.

The three metals utilized in the construction of the samples were Type AA 2024-0 aluminum, Type SAE 1010-1015 cold-rolled steel, and Type AISI 430 stainless steel.

The three elastomeric vulcanizates tested were a styrene-butadiene rubber stock in seven metal-adhesive-rubber combinations, a polychloroprene rubber compound in six composites, and a natural rubber mixture which was used as part of two stainless steel laminates. The complete formulations are described in Table III.

Bonding agents are most often solutions or dispersions of specially compounded polymers in organic solvents. The bonding systems tested in this experiment included several adhesives designated commercially as "Ty-ply", made by the Marbon Chemical Division of Borg-Warner Corporation, and another series called "Chemlok" supplied by the Hughson Chemical Company, a division of Lord Manufacturing Company, Erie, Pennsylvania.

Although the cold-rolled steel and copper-bearing aluminum alloy are susceptible to significant corrosion, they were included in this study because of their ease of bonding and their convenience in checking the interfacial reactions. After six years of burial, these tests were discontinued.

Figure 12 shows a number of specimens retrieved after burial for one year in Bainbridge. The visibly corroded specimens in the photograph are aluminum and cold-rolled steel, while the shiny specimens are the #269 and #272 samples made with stainless steel. The A-1 specimens are from the 6-inch topsoil region and the A-6 items are from the subsoil.

The very severe corrosion of the untreated aluminum which occurred in the soil has destroyed the bonds of all five of the aluminum burial laminates shown in Fig. 13. In the polychloroprene aluminum specimens #279 (Fig. 14) the corrosion seems heavier at the rubber-metal interface than on the outside, indicating possible corrosion due to differential aeration and accelerated by chloride contaminants.[8]

Bacteria may have also been a cause for they can initiate or promote soil corrosion by producing corrosive substances, by the creation of a corrosive environment through establishment of oxygen concentration cells, or by their consumption of hydrogen which may affect corrosion by depolarizing cathodic reactions.[28]

Usually corrosion leads to bond failure, but among the retrieved specimens from Georgia there were two noteworthy exceptions. The laminates made with the Ty-Ply 3640 and with the combined Chemlok 201 and 220 adhesives maintained good bonds to the cold-rolled steel

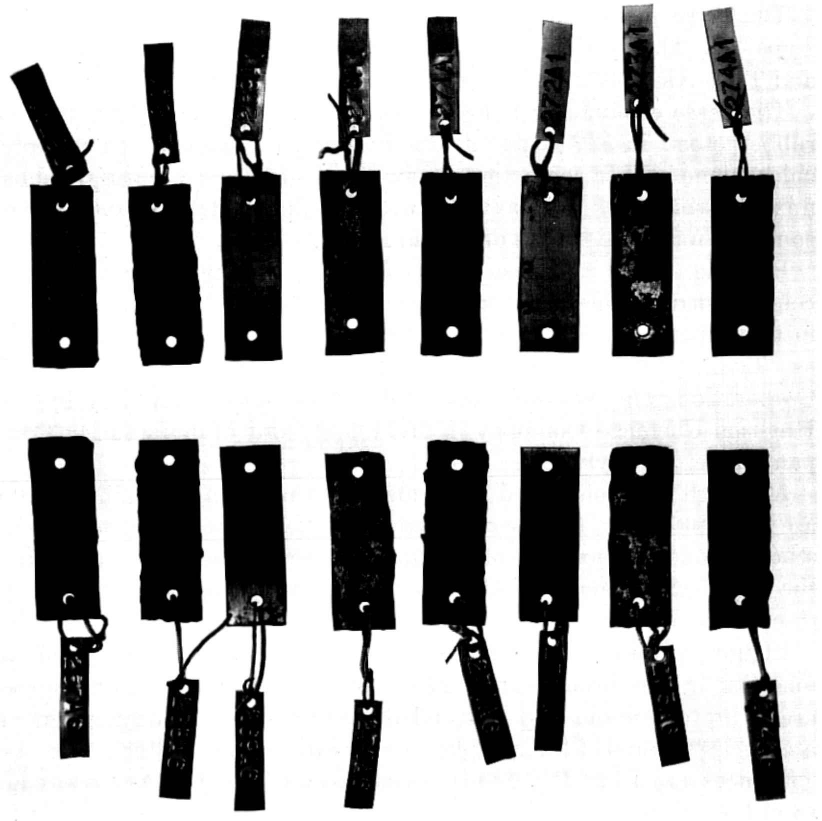


Fig. 12—Rubber-to-metal bonded laminate specimens after one year burial at Bainbridge. The A-1 specimens were buried in the topsoil, and the A-6 specimens were in the subsoil. Samples #269 and #272 are bonds to stainless steel while the corroded remainder are either cold-rolled steel or aluminum.

despite the corrosion. Duplicate specimens have, however, weakened in New Mexico and in general the damage in this alkaline soil seemed to be more severe than in the acidic Georgia soil, especially in the subsoil (A-6 specimens).

Specimens #280 were the only composites tested that involved polychloroprene bonded to cold-rolled steel. The bonding agent was Ty-Ply UP. On the #280 shelf-aged controls the bond remained strong but there was appreciable rust on the exposed metal surfaces of both control specimens. The bond of the buried specimens of this composite, however, was quite weak and had appreciable interfacial corrosion

Fig. 13—Specimens of the five rubber-to-aluminum bonded combinations retrieved from two depths after four years soil burial exposure at Roswell. All bonds have failed due to severe corrosion.

after only one year. It is possible that the CR rubber compound itself may contribute to the rusting of cold-rolled steel as indicated by similar laboratory tests with rubber molds of cold-rolled steel. In fact, polychloroprene has been reported to cause pitting through chromium platings on steel molds.[29] Similarly, plain steel molds are not usually used when working with other halogen-containing polymers such as PVC. In addition, the presence of carbon black was found to increase the galvanic current between adhesive and metal which causes a progressive destruction of the bond.[29]

Table VIII lists all of the components of the individual laminate samples and the results of eight years burial compared with shelf-aged controls. None of the rubber-to-metal bonded control specimens have suffered adhesion failures and nearly all are still in excellent condition.

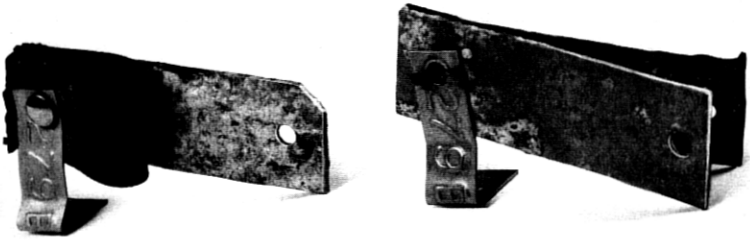


Fig. 14—Heavier corrosion at the interface of the polychloroprene-to-aluminum bond than on the exposed side of the metal strip.

TABLE VIII—RUBBER-TO-METAL BONDS–OBSERVATIONS AFTER SOIL BURIAL RETRIEVAL

| Sample & Site | Rubber | Metal | Adhesive System | Observations* |
|---|---|---|---|---|
| 267 A | CR | Alum. | Ty-Ply S | 4 yrs, rubber strong, severe corrosion, bond weak |
| B | | | | 6 yrs, little bond, heavy corrosion |
| 268 A | SBR | Steel | Ty-Ply 3640 | 4 yrs, rubber OK, heavy rust, bond excellent |
| B | | | | 6 yrs, heavy rust, weak bond and rubber |
| 269 A | CR | St. Steel | Ty-Ply S | 8 yrs, bond and rubber—excellent |
| B | | | | 8 yrs, bond and rubber—excellent |
| 270 A | CR | Alum. | Ty-Ply UP | 4 yrs, rubber strong, heavy corrosion, no bond |
| B | | | | 6 yrs, rubber strong, heavy corrosion, no bond |
| 271 A | SBR | Steel | Ty-Ply UP + Ty-Ply RC | 4 yrs, rubber weak, heavy rust, bond excellent |
| B | | | | 6 yrs, rubber tender, heavy rust, bond good |
| 272 A | NR | St. Steel | Ty-Ply UP + Ty-Ply RC | 8 yrs, bond good, rubber weakening |
| B | | | | 8 yrs, (no sample exposed) |
| 273 A | SBR | Alum. | Chemlok 201 + Chemlok 220 | 4 yrs, rubber good, some corrosion, bond fair |
| B | | | | 6 yrs, rubber good, heavy corrosion, no bond |
| 274 A | SBR | Steel | Chemlok 201 + Chemlok 220 | 4 yrs, rubber good, heavy rust, bond strong |
| B | | | | 6 yrs, rubber tender, heavy rust, bond fair |
| 275 A | SBR | St. Steel | Chemlok 201 + Chemlok 220 | 8 yrs, bond good, rubber weakening |
| B | | | | 8 yrs, bond fair, rubber strong |
| 276 A | SBR | Alum. | Chemlok 220 | 4 yrs, rubber weak, bad corrosion, no bond |
| B | | | | 6 yrs, rubber strong, heavy corrosion, no bond |
| 277 A | SBR | Steel | Chemlok 220 | 4 yrs, rubber weak, heavy rust, bond strong |
| B | | | | 6 yrs, rubber fair, heavy rust, bond fair |
| 278 A | NR | St. Steel | Chemlok 220 | 8 yrs, bond excellent, rubber weakening |
| B | | | | 8 yrs, bond and rubber good |
| 279 A | CR | Alum. | Chemlok 220 | 4 yrs, rubber strong, corrosion, bond weak |
| B | | | | 6 yrs, rubber strong, heavy rust, bond weak |
| 280 A | CR | Steel | Ty-Ply UP | 4 yrs, rubber strong, heavy rust, bond weak |
| B | | | | 6 yrs, rubber strong, heavy rust, little bond |
| 281 A | CR | St. Steel | Chemlok 220 | 8 yrs, bond and rubber—excellent |
| B | | | | 8 yrs, bond fair—rubber strong |

* The only 8-year samples still in burial test are Nos. 269, 272, 275, 278, 281; all others have been removed from test.
A—Bainbridge, Georgia B—Roswell, New Mexico St. Steel—Stainless Steel Alum.—Aluminum
SBR—Styrene-Butadiene NR—Natural CR—Chloroprene

The strength of the #270 control sample, consisting of a Ty-Ply UP bond of polychloroprene to aluminum, was significantly reduced.

The stainless steel laminates were the only ones that had not corroded after eight years of soil burial. When the test samples were prepared in 1958, stainless steel was a very difficult material to bond to rubber. It required some very careful preparation, skillful techniques, and luck. Today, however, with the newer bonding materials, stainless steel is no longer a problem to bond.

In the case of the SBR compound, only the combination Chemlok 201 and Chemlok 220 adhesive system gave a bond to stainless steel that was considered to be good enough to test and these specimens constituted the #275 series. They were bonded in January 1958 and, when examined in February 1960, the bonds in both the controls and the buried specimens seemed excellent.

Based on eight years of exposure, it is clear that if a strong bond is made to stainless steel initially, it will last in a soil medium for the life of the rubber.* In terms of the adhesion test, the polychloroprene bonds are giving spotty results and the natural rubber is getting quite weak. The SBR compound might have performed better had it contained an antioxidant. A number of other good rubber formulations for buried uses may be found among the series of rubber molding compounds described in Section III of this paper. New bonding systems are available and need to be tested for despite technological advances, adhesive compounding still remains primarily an art rather than a science.

All of the proprietary adhesive systems used in this program provided strong and lasting bonds as long as the metal did not corrode and the rubber did not deteriorate. Two specimens even maintained some adhesion to corroded metals.

## V. ACKNOWLEDGMENTS

An exposure program, covering a span of years such as this, inevitably results in changes in the identity of the participants. I would like to express my indebtedness, thanks, and appreciation to two grand old men—now retired—Eric L. Dias who did the initial wire coating work and Caleb M. Hill for the molding and bonding preparation. Their encouragement, support, and advice have been wonderful.

---

* See Appendix B.

## APPENDIX A

### *Compression Test*[30,31]

The Compression Testing Machine was developed at Bell Laboratories with the cooperation of the H. L. Scott Company. It was designed for the evaluation of rubber compounds in the form of insulation on solid wire and it measures the compressive force required to cause the conductor to cut through the insulation. In the test, a 2-inch length of insulated wire was compressed between a pair of steel blocks with parallel plane surfaces. As the rubber on the wire was compressed, it assumed an elliptical form. The layer of rubber between the conductor and the jaws became thinner as the pressure increased until rupture occurred. Compression resistance was automatically recorded as a numerical value of the load in pounds. This test apparatus and procedure have become a standard in the wire and cable industry.

A recent joint effort between R. F. Westover of Bell Laboratories and Scott Testers, Inc., has produced a modernization of the load weighing and recording systems for the Compression Machine manufactured by Scott. This improvement increased the reliability and accuracy of results, reduced testing time, and extended the application of the machine to the highly expanded or foam forms of insulation.

## APPENDIX B

### *Rubber-to-Stainless-Steel Bonds and Salt Water*[32]

William H. Lockwood has found that rubber-to-stainless-steel bonds will fail quickly in an environment containing salt water if the steel has not been properly treated initially. For with even the best of adhesives, the usual sand blasting of the steel prior to bonding is insufficient and leads to this failure. However, a hydrochloric acid treatment of the steel will result in long-lasting bonds.

## REFERENCES

1. DeVries, O., "The Destruction of the Hydrocarbon of Raw Rubber by Fungi," Chem. Abstr., *22* (Oct. 20, 1928), p. 4006.
2. Brown, Alfred E., "The Problem of Fungal Growth," Mod. Plastics, *23*, No. 8 (April 1946), pp. 189–256.
3. Greathouse, G. A., "Effect of Microorganisms on Rubber Degradation," Rubber Age, *63* (1948), pp. 337–338.
4. Ritzinger, B. B., "Fungus Resistance of Neoprene and Effect of Compounding and Use of Fungicides," Rubber Plastic Age, *40*, No. 10 (1959), pp. 1067–1069.
5. Alvino, W. M., Bell Laboratories, unpublished work (1962).
6. Burns, R. M., "Mechanisms of Corrosion Process," ASTM Bull., No. 126 (Jan. 1944), pp. 17–20.

7. Burns, R. M., "The Corrosion of Metals-I.," B.S.T.J., *15*, No. 1 (Jan. 1936), pp. 20–38.
8. Burns, R. M., and Bradley, W. W., *Protective Coatings for Metals*, Third Edition, ACS Mono. No. 163, New York: Reinhold Publishing Corp., 1967.
9. Vernon, W. H. J., "Second Experimental Report to the Atmospheric Corrosion Research Committee," Part I, Sec. VII, Trans. Faraday Soc., *23* (1927), pp. 159–165.
10. Burns, R. M., "The Corrosion of Metals-II.," B.S.T.J., *15*, No. 4 (Oct. 1936), pp. 603–625.
11. Keller, F., and Edwards, J. D., "The Behavior of Oxide Films on Aluminum," *Pittsburgh International Conference on Surface Reactions*, Pittsburgh, Pa.: Corrosion Publishing Co., 1948, pp. 202–212.
12. Blake, John T., Kitchen, Doland W., and Pratt, Orison S., "Failures of Rubber Insulation Caused by Soil Microorganisms," Trans. Amer. Inst. Elec. Eng., *69* part 2 (1950), pp. 748–755.
13. Blake, John T. Kitchen, Donald W., and Pratt, Orison S., "The Microbiological Deterioration of Rubber Insulation," Trans. Amer. Inst. Elec. Eng., *72* part 3 (1953), pp. 321–328.
14. Winslow, F. H., Matreyek, W., and Trozzolo, A. M., "Weathering of Polyethylene," Polym. Preprints, Amer. Chem. Soc., Div. Polym. Chem., *10*, No. 2 (Sept. 1969), pp. 1271–1280.
15. Heiss, J. H., and Lanza, V. L., "The Thermal Embrittlement of Stressed Polyethylene," Wire and Wire Products, *33*, No. 10 (Oct. 1958), pp. 1182–1187, 1285.
16. Dannenberg, E. M., Jordan, M. E., and Cole, H. M., "Peroxide Crosslinked Carbon Black Polyethylene Compositions," J. Poly. Sci., *31*, No. 122 (Aug. 1958), pp. 127–153.
17. Maibauer, A. E., and Myers, C. S., "Polyethylenes," Trans. Electrochem. Soc., *90* (1946), pp. 341–360.
18. Ambrose, John F., "Pigments in Polymer Materials," Bell Labs. Rec., *35* (July 1957), pp. 246–250.
19. Hawkins, W. L., Hansen, R. H., Matreyek, W., and Winslow, F. H., "The Effect of Carbon Black on Thermal Antioxidants for Polyethylene," J. Appl. Polymer Sci., *1* (1959), pp. 37–42.
20. Hawkins, W. L., Worthington, Mrs. M. A., and Winslow, F. H., "The Role of Carbon Black in the Thermal Oxidation of Polylefins," *Proc. Fourth Conf. on Carbon*, New York: Pergamon Press, 1960.
21. American Society For Testing and Materials, "Standard Methods of Tension Testing of Vulcanized Rubber," D412-62T *ASTM Standards* (1964), part 28.
22. Athey, Robert J., "Stability of Plastics," Rubber Age, *96*, No. 5 (Feb. 1965), pp. 705–712.
23. Cooper, W., Pearson, R. W., and Darke, S., "Isocyanate Reactions and the Structure of Polyurethane," Ind. Chemist, *36*, No. 421 (1960), pp. 121–126.
24. Biggs, B. S., "Deterioration of Organic Polymers," B.S.T.J., *30*, No. 2 (Oct. 1951), pp. 1078–1102.
25. Testroet, Frank B., "Performance of Urethane Vulcanizates in Environments of High Humidity," U. S. Gov. Res. Rep. *39*, 11, S26 (1964), AD-422028, Rock Island Arsenal Lab., Ill.: Rep. 63-2808, 1963, 38 pp.
26. Petnujova, A., and Zanova, V., "Influence of Composition on the Biological Degradation of Rubber Articles," Soviet Rubber Tech., *19*, No. 2 (July 1960), pp. 16–17.
27. Pickup, B., and Weatherstone, E., "Some Factors Influencing the Bonding of Rubber to Metal," J.I.R.I., *3*, No. 6 (Dec. 1969), pp. 254–261.
28. Lichtenstein, S., "Corrosion by Microbes, " Instrum. Contr. Syst., *41*, No. 2 (Feb. 1968), p. 99.
29. Buchen, S., *Rubber to Metal Bonding*, London: Crosby Lockwood and Sons Ltd., 1959, pp. 111–112.
30. Hippensteel, Claude L., "A New Mechanical Test for Rubber Insulation," Ind. Eng. Chem., *18* (April 1926), pp. 409–411.
31. Hippensteel, Claude L., "New Rubber Compression Testing Machine," Bell Lab. Rec., *5*, No. 5 (Jan. 1928), pp. 153–155.
32. Lockwood, William H., Bell Laboratories, unpublished work (1963).