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ENVIRONMENTAL EXPOSURE AND ACCELERATED TESTING OF  
RUBBER-TO-METAL VULCANIZED BONDED ASSEMBLIES

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**ENVIRONMENTAL EXPOSURE AND ACCELERATED  
TESTING OF RUBBER-TO-METAL VULCANIZED  
BONDED ASSEMBLIES**

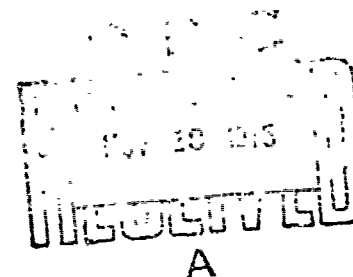
by  
**John A. Williams**

NOVEMBER 1974

**TECHNICAL REPORT**

**RESEARCH DIRECTORATE**

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**GENERAL THOMAS J. RODMAN LABORATORY  
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| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number)<br><br>The purpose of this work is to expose rubber-to-metal vulcanized bonded assemblies to environmental conditions experienced by weapon components in the field and to determine the effect of this exposure on the vulcanized bond. The purpose is also to duplicate these long term environmental effects with an accelerated laboratory test. Four different groups of rubber-to-metal vulcanized bonded assemblies were exposed outdoors in Panama and at Rock Island. Accelerated laboratory testing was conducted using heat and humidity. |                       |  |

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After one year exposure in Panama; the butadiene acrylonitrile rubber bonded assemblies showed a severe loss of 100 percent in bond strength; the EPDM bonded assemblies showed a 40 percent loss in bond strength and the SBR and neoprene bonded assemblies showed no loss in bond strength. Accelerated testing did show the same results as environmental exposure, but laboratory test periods (up to 56 days) are too long for a good accelerated test.

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## OBJECTIVE:

The purpose of this work was to determine the resistance of rubber-to-metal vulcanized bonds to long-term storage and environmental exposure, and to develop accelerated methods for use in predicting this resistance.

## BACKGROUND:

The most effective method of securing rubber-to-metal substrates with sufficient strength to withstand the shear and peel forces encountered in many weapons applications is that of bonding the rubber to the metal at the time that the rubber is being vulcanized. This is known as "vulcanized bonding". Examples of such bonds are the rubber coatings on the M60 machine gun components, the shock isolator and recoil adapter on the GAU 28/A Minigun, rubber pads for all tracked vehicles, shock mounts for numerous shipping containers, and many small components for weapon accessories such as the rubber stop on the target holding mechanism.

Personnel of the Research Directorate, GEN Thomas J. Rodman Laboratory, were recently informed that rubber-to-metal bonds on numerous weapon component spare parts have failed after a relatively short time in storage. Very little published data are available on the resistance of rubber-to-metal vulcanized bonds to long term storage or environmental exposure. These bond failures on replacement parts and the lack of data in this area demonstrate the need to develop the ability to predict the long term storage or outdoor life of vulcanized bonds.

The development of the ability to predict vulcanized bond life during long-term storage and to improve the resistance of these bonds to environmental exposure will lead to significant advances in the state of the art and will prove to be of unlimited benefit to the Army because of the wide range of weapon applications in which vulcanized bonding is essential. Longer storage life for these weapon components will result in a tremendous saving in cost to the Army by lower replacement rates for these spare parts.

## APPROACH:

Both steel and aluminum substrates were used in this study with surface preparation as prescribed in ASTM D429-73 <sup>1</sup>. Only bonding agents known to produce strong vulcanized bonds were employed. Four elastomers most commonly used in weapon components were chosen for the study. These elastomers are general-purpose rubbers butadiene/styrene and ethylene propylene terpolymer, and the oil resistant nitrile and chloroprene rubbers. Vulcanized bonded specimens were prepared for outdoor exposure at Panama and at Rock Island for periods of six months, one, two, and three years. Another set of specimens was stored indoors at Rock Island for the same time periods. Standard methods

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1. 1974 Annual Book of ASTM Standards, Part 37.

of laboratory exposure used to accelerate the environmental deterioration of rubber were screened to determine if these methods can be used to accelerate the deterioration of the bonded specimens. Methods B and C of ASTM D429-73 were used to measure the strength of the vulcanized bonds. Method B is a 90° peel test in which bond failure occurs between the rubber and the metal substrate in a peeling action and is measured in pounds per inch. Method C is a test in which the metal substrate is cone shaped and in which bond failure begins at the point of the cone and proceeds until completion. Test results from conical specimens are measured in pounds.

The disadvantage of the 90° peel test is that if the vulcanized bond has greater strength than that of the rubber, the rubber will tear before bond failure begins. With conical specimens the vulcanized bond will usually fail before the rubber tears, in which case, the true bond strength is given.

The type of failure that occurs during the evaluation of the bonds is indicated in the tables that follow. RC indicates failure that occurred at the rubber and cover coat interface; CP indicates that the failure took place at the cover coat and metal primer interface; M indicates that the failure was at the metal primer and metal interface; R indicates that the rubber has failed, but no real value is given for the vulcanized bond strength<sup>2</sup>.

Three hundred conical and three hundred 90° peel vulcanized bonded specimens were prepared from both steel and aluminum. The four elastomers mentioned earlier were used with bonding agents known to give the best original bond strength. These vulcanized bonded specimens were exposed outdoors at Panama and at Rock Island for periods of six months, one, two, and three years. Another set was placed in storage indoors at Rock Island for similar periods of time.

#### RESULTS AND DISCUSSION:

Formulations and physical properties of the elastomers used in the preparation of all vulcanized bonded specimens used in this study are shown in Table 1. These rubber compounds were formulated for weapon applications and not for ease of bonding. For example, butadiene/styrene based compound (S242) contains 5 parts/100 rhc UOP 88 antiozonant and Heliozone wax, ingredients which impart ozone resistance to the rubber, but which detract from bondability. Similarly the nitrile based rubber compound (N200) contains 25 parts/100 rhc of plasticizer even though the plasticizer makes the rubber more difficult to bond. The plasticizer is used to impart good flexibility at -40°F.

The results of long term exposure at the two sites are shown in Tables 2, 3, 4, and 5. As shown in Table 2 the butadiene/styrene (SBR) bonded assemblies exhibited no significant reduction in bond strength, even after one year exposure. The bond strength of the conical specimens increased; probably due to

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2. Peterson, "Rubber-to-Metal Bond Failures", Rubber Age, vol. 93, No. 6, p. 929, September 1963.



TABLE 1

## ELASTOMERS USED IN THE VULCANIZED BONDING OF RUBBER TO METAL

| <u>COMPOUNDING INGREDIENTS</u>  | <u>E57</u> | <u>Parts by Weight</u> |             | <u>R200</u> |
|---|------------|------------------------|-------------|-------------|
|   |            | <u>S242</u>            | <u>M115</u> |             |
| Nordel 1040 (EPDM)  | 100        |                        |             |             |
| Stereon 700 (SBR)   |            | 100                    |             |             |
| Neoprene WD (CR)  |            |                        | 100         |             |
| Paracril AJ (NBR)   |            |                        |             | 85          |
| Paracril B (NBR)  |            |                        |             | 15          |
| Philblack N550  | 45         |                        |             |             |
| Philblack N220  |            |                        | 40          | 75          |
| Philblack N110  |            | 50                     |             |             |
| HiSil 233   |            |                        |             | 20          |
| Necton 60   | 20         | 20                     |             |             |
| Tributoxyethyl Phosphate  |            |                        |             | 10          |
| Tricetyl Phosphate  |            |                        |             | 15          |
| Dioctyl Sebacate  |            |                        | 20          |             |
| UQP-88  |            | 5                      |             |             |
| Age Rite Resin D  |            | 2.5                    |             | 1           |
| Akroflex CD   |            |                        | 3           |             |
| Heliozone   |            | 1                      | 3           |             |
| Stearic Acid  | 1          |                        |             | 1           |
| Zinc Oxide  | 5          | 3                      | 5           | 5           |
| Magnesium Oxide   |            |                        | 4           |             |
| Sulfur  | 0.5        | 2                      |             |             |
| Altax   | 1          |                        |             | 3           |
| Santocure   |            | 0.8                    |             |             |
| Methyl Tuads  | 2          |                        |             | 1.5         |
| Ethyl Tuads   |            |                        |             | 1.5         |
| Na-22   |            |                        | 1           |             |
| <u>Physical Properties</u>  |            |                        |             |             |
| Tensile Strength, psi   | 1650       | 2220                   | 2760        | 2070        |
| Elongation, %   | 580        | 630                    | 340         | 320         |
| Hardness, Shore A   | 51         | 59                     | 63          | 68          |
| Low Temperature Flexibility,<br>ASTM D1043, Temp. where Young's<br>Modulus is 10,000 psi, °F. | -50        | -92                    | -52         | -48         |
| 70 HRS/212°F/ASTM #3 Oil:<br>Volume change, %   | -          | -                      | +45         | +23         |
| Compression Set, ASTM D395,<br>Method B, 22 hrs@ 158°F, %                                     | 14         | -                      | 11          | 10          |

increased crosslinking or aging of the rubber. Also the peel specimens have aged; but, because the specimens have failed within the rubber rather than at the bond, the true bond strength could not be ascertained.

The results of the exposure of EPDM vulcanized bonded assemblies are shown in Table 3. Here, the decrease in bond strength is clearly indicated. The steel 90° peel specimens decreased in strength only 7 percent after one year exposure indoors at Rock Island, but these peel specimens decreased 68 percent after one year exposure at Panama. The type of failure changed from rubber to rubber/cover coat interface to metal/metal primer interface as the exposure time and the severity of the site increased. Decrease in the bond strength of the conical specimens is not as great as the peel specimens. Metal-primer interface failure became apparent after one year exposure outdoors at Rock Island and this increased after one year exposure in Panama.

The results of the storage and environmental exposure of neoprene rubber-to-metal vulcanized bonded assemblies are shown in Table 4. Change in bond strength was not significant, but the conical specimens did show a change in the type of bond failure; rubber failure changing to rubber-cement interface failure.

The changes in bond strength and type of bond failure produced by the exposure of butadiene/acrylonitrile (NBR) rubber-to-metal vulcanized bonded assemblies at the two exposure sites are shown in Table 5. After exposure for one year outdoors at Rock Island, the steel 90° peel specimens showed a loss of 71 percent; and the aluminum specimens, a loss of 48 percent in bond strength. The original bond failures were rubber, that is, a stock break. The specimens, that had been exposed for one year outdoors at Rock Island also showed bond failures of rubber, but these specimens had only a very thin layer of rubber on the metal substrate surface. Although both failures were rubber failures, a change in the type of bond failure occurred. Peel specimens exposed for 6 months at Panama exhibited a loss in bond strength of 78 percent for steel and 34 percent for aluminum, with metal-primer interface failure increasing to 68 percent. Steel 90° peel specimens exposed one year at Panama separated at the metal-primer interface during the exposure period. Aluminum specimens showed 83 percent loss in bond strength and a metal-primer interface failure of 75 percent. Steel conical specimens showed a 54 percent and a 97 percent loss, respectively, in bond strength after 6 months and one year at Panama.

After data had been obtained showing the effect of long term exposure on bond strength, attempts were made to produce comparable changes in bond strengths by standard accelerated aging tests for rubber.

The results of heat aging on conical specimens of neoprene and SBR rubber bonded to steel are shown in Table 6. Aging at 212° F had no significant effect on neoprene specimens. The SBR specimens showed an increase in bond strength after aging at 212° F because of increased cross linking of the bond. These bonded assemblies were then aged at temperatures beyond the normal for these rubbers. Bond strengths remained good, and the rubber showed a normal reaction to these higher temperatures, with neoprene decreasing in strength and SBR increasing in strength.

TABLE 2

ENVIRONMENTAL EXPOSURE OF BUTADIENE/STYRENE  
RUBBER-TO-METAL VULCANIZED BONDED-ASSEMBLIES

|                          | <u>Method B, 90° peel, lb/in</u> |                 | <u>Method C, Conical, lb</u> |                 |
|--------------------------|----------------------------------|-----------------|------------------------------|-----------------|
|                          | <u>Substrate</u>                 |                 | <u>Substrate</u>             |                 |
|                          | <u>Steel</u>                     | <u>Aluminum</u> | <u>Steel</u>                 | <u>Aluminum</u> |
| Original                 | 157R                             | 159R            | 220 RC                       | 258 RC          |
| 6 Months, indoors at RI  | 165R                             | 166R            | 323 RC                       | 311 RC          |
| One year, indoors at RI  | 167R                             | 166R            | 352 RC                       | 347 RC          |
| 6 Months, outdoors at RI | 160R                             | 158R            | 326 RC                       | 346 RC          |
| One year, outdcors at RI | 141R                             | 135R            | 301 RC                       | 295 RC          |
| 6 Months at Panama       | 149R                             | 154R            | 297 RC                       | 298 RC          |
| One year at Panama       | 141R                             | 138R            | 314 RC                       | 337 RC          |

Primer: Thixon P-5

Cover Cement: Thixon CB-3

TABLE 3

ENVIRONMENTAL EXPOSURE OF ETHYLENE PROPYLENE TERPOLYMER  
RUBBER-TO-METAL VULCANIZED BONDED-ASSEMBLIES

|                          | <u>Method B, 90° peel, lb/in</u> |                 | <u>Method C, Conical, lb</u> |                 |
|--------------------------|----------------------------------|-----------------|------------------------------|-----------------|
|                          | <u>Substrate</u>                 |                 | <u>Substrate</u>             |                 |
|                          | <u>Steel</u>                     | <u>Aluminum</u> | <u>Steel</u>                 | <u>Aluminum</u> |
| Original                 | 137R                             | 126R            | 231 RC                       | 218 RC          |
| 6 Months, indoors at RI  | 116 R73 RC17                     | 118 R57 RC43    | 229 RC                       | 240 RC          |
| One year, indoors at RI  | 127 R80 RC20                     | 106 R28 RC72    | 245 RC                       | 243 RC          |
| 6 Months, outdoors at RI | 76RC                             | 79 RC75 R25     | 236 RC                       | 231 RC          |
| One year, outdoors at RI | 83 RC90 M10                      | 80 RC           | 246 RC77 M23                 | 221 RC97 M3     |
| 6 Months, at Panama      | 90 R58 M25 RC17                  | 75 RC85 R15     | 234 RC                       | 234 RC          |
| One year, at Panama      | 44 M57 R43                       | 75 RC77 R23     | 217 RC60 M40                 | 217 R M8        |

Primer: Thixon P-5

Cover Coat: Thixon AF1559

TABLE 4.

ENVIRONMENTAL EXPOSURE OF NEOPRENE RUBBER-TO-METAL  
VULCANIZED POND-ASSEMBLIES

|                          | <u>Method B, 90° peel, lb/in</u> |                 | <u>Method C, Conical, lb</u> |                 |
|--------------------------|----------------------------------|-----------------|------------------------------|-----------------|
|                          | <u>Substrate</u>                 |                 | <u>Substrate</u>             |                 |
|                          | <u>Steel</u>                     | <u>Aluminum</u> | <u>Steel</u>                 | <u>Aluminum</u> |
| Original                 | 82R                              | 100R            | 378 R90 RC10                 | 380 R90 RC10    |
| 6 Months, indoors at RI  | 82R                              | 94R             | 386 R93 RC7                  | 300 RC87 R13    |
| One year, indoors at RI  | 98R                              | 81R             | 409 R90 RC10                 | 372 R55 RC45    |
| 6 Months, outdoors at RI | 98R                              | 98R             | 386 R90 RC10                 | 329 R50 RC50    |
| One year, outdoors at RI | 68R                              | 75R             | 380 R95 RC5                  | 319 R57 RC43    |
| 6 Months, at Panama      | 89R                              | 91R             | 334 R67 RC33                 | 356 R73 RC27    |
| One year, at Panama      | 66R                              | 68R             | 404 R97 RC3                  | 327 R72 RC28    |

Primer: Chemlok 205

Cover Coat: Chemlok 233

TABLE 5

ENVIRONMENTAL EXPOSURE OF BUTADIENE/ACRYLONITRILE  
RUBBER-TO-METAL VULCANIZED BONDED-ASSEMBLIES

|                          | <u>Method B, 90° peel, lb/in</u> |                 | <u>Method C, Conical, lb</u> |                 |
|--------------------------|----------------------------------|-----------------|------------------------------|-----------------|
|                          | <u>Substrate</u>                 |                 | <u>Substrate</u>             |                 |
|                          | <u>Steel</u>                     | <u>Aluminum</u> | <u>Steel</u>                 | <u>Aluminum</u> |
| Original                 | 72R                              | 65R             | 360R                         | 359R            |
| 6 Months, indoors at RI  | 50R                              | 54R             | 348 R50 RC50                 | 349R            |
| One year, indoors at RI  | 52R                              | 59R             | 379R                         | 360R            |
| 6 Months, outdoors at RI | 53R                              | 53R             | 374R                         | 379R            |
| One year, outdoors at RI | 21 R98 M2                        | 34R             | 321 R83 M17                  | 342R            |
| 6 Months, at Panama      | 16 M68 R32                       | 43 M27 R73      | 165 M50 RC42 R8              | 343 M52 R48     |
| One year, at Panama      | Bond Failure M during exposure   | 11 M75 R25      | 10M                          | 263 M93 R7      |

Primer: Chemlok 205

Cover Coat: Chemlok 231

EPDM specimens aged up to 450°F are shown in Table 7. At 300°F, all bonding agents provided good retention of bond strength. The bond strength remained good until the temperatures were raised above the normal limitations of this rubber.

The results of the heat aging of butadiene/acrylonitrile rubber-to-metal vulcanized bonded-assemblies are shown in Table 8. At 212°F, the bond strength remained high; however, rubber failure occurred. Aging at temperatures above the normal for this nitrile rubber showed that the rubber aged and lost strength. At 300°F, the bonds began to fail, except those bonds prepared with Thixon AM-2.

The results of a hydrolysis test used to accelerate the bond failure of steel conical vulcanized bonded-assemblies are shown in Tables 9, 10, and 11. Control specimens were aged in an air oven at 185°F, while specimens from another set were aged over water in a closed container at 185°F.

Neoprene and SBR specimens showed no loss in bond strength after they had been exposed for 56 days to the high humidity of this test. Specimens bonded with Thixon D12809 showed a change in type of bond failure from 100 percent rubber cement to 100 percent metal-primer interface bond failure. Although bond strength did not change, continued exposure would have resulted in a rapid loss of bond strength.

The results of EPDM vulcanized bonded-assemblies that had been exposed to the accelerated hydrolysis test are shown in Table 10. No loss was noted in the bond strength of these specimens. In most cases, an increase in bond strength was noted; this was probably due to increased cross linking. Specimens bonded with Chemlok 236 showed a change in type of bond failure from rubber-cement to metal-primer interface. Although an increase in bond strength occurred, a rapid loss can be expected with continued exposure.

The results of the exposure of butadiene/acrylonitrile rubber-to-steel vulcanized bonded-assemblies to the accelerated hydrolytic stability test are shown in Table 11. After 28 days, a change in the type of bond failure began to occur. After an exposure of 56 days, total or complete change from rubber to metal-primer interface bond failure had occurred in all bonding agents evaluated. Loss in bond strength increased to 52 percent; but, with the change in type of bond failure, complete loss of bond strength would have been rapid with continued exposure.

The results of the tests conducted on bonded assemblies exposed to heat and humidity indicate that these conditions do accelerate bond failure and that this type of test may be useful in predicting the life of the assemblies. The test does have the disadvantage of being too lengthy, 56 days or more in duration.

The effect of ASTM#3 oil on neoprene and butadiene/acrylonitrile rubber vulcanized-bonded assemblies is shown in Table 12. Butadiene/acrylonitrile rubber vulcanized-bonded with Thixon D12809 showed losses of 13 and 42 percent when aged at 212°F, respectively. Metal-primer interface bond failure was noted. The other specimens showed no loss in bond strength or change in type

TABLE 6

HEAT AGING OF NEOPRENE AND BUTADIENE/STYRENE RUBBER-TO-STEEL  
VULCANIZED BONDED-ASSEMBLIESMethod C, Conical, 1b

|                  | <u>Neoprene</u>            |               | <u>SBR</u>           |                           |
|------------------|----------------------------|---------------|----------------------|---------------------------|
|                  | <u>Bonding Agent</u>       |               | <u>Bonding Agent</u> |                           |
|                  | Chemlok 205<br>Chemlok 233 | Thixon D12809 | Thixon NM-2          | Thixon P-5<br>Thixon CB-3 |
| Original         | 378 R90 RC10               | 182 RC        | 193 RC               | 220 RC                    |
| Aged 70hrs/212°F | 358R                       | 197 RC        | 178 RC               | 316 RC                    |
| Aged 70hrs/250°F | —                          | —             | —                    | 478 R                     |
| Aged 70hrs/300°F | 282R                       | 287R          | 275 R60 RC40         | 464 R                     |
| Aged 70hrs/350°F | 228R                       | 275R          | 183 R20 RC80         | 195 M20 R80               |



TABLE 7

HEAT AGING OF ETHYLENE PROPYLENE TERPOLYMER RUBBER-TO-STEEL  
VULCANIZED BONDED-ASSEMBLIES

Method C, Conical, 1b

Bonding Agents

|                   | Thixon P-5<br>Thixon AP1559<br>(1 Coat) | Thixon P-5<br>Thixon AP1559<br>(2 Coats) | Thixon D12809<br>Thixon AP1654<br>(2 Coats) | Chemlok 205<br>Chemlok 234B<br>(2 Coats) | Chemlok 205<br>Chemlok 236<br>(2 Coats) |
|-------------------|---|--|---|--|---|
| Original          | 231 RC                                  | 257 RC                                   | 257 RC                                      | 227 RC                                   | 148 RC                                  |
| Aged 70hrs/212°F  | 250 RC                                  | 275 RC                                   | 288 RC                                      | 296 RC                                   | 171 RC                                  |
| Aged 70hrs/300°F  | 427 RC                                  | 439 RC                                   | 473 RC                                      | 279 RC                                   | 316 RC                                  |
| Aged 70hrs/400°F  | 116 RC 20M80                            | 226 RC 70 M30                            | 298 RC 70 R30                               | 70 RC30 M70                              | 76 RC15 M85                             |
| Aged 70hrs/425°F  | 152 RC 60 M20R20                        | 215 RC 90 M10                            | 296 RC 85 R15                               | 81 RC30 M70                              | 78 RC15 M85                             |
| Aged one hr/450°F | Rubber too brittle<br>to test           | _____                                    | _____                                       | _____                                    | _____                                   |

TABLE 8

HEAT AGING OF BUTADIENE/ACRYLONITRILE RUBBER-TO-STEEL  
VULCANIZED BONDED-ASSEMBLIES

Method C, Conical, 1b

Bonding Agents

|                  | Chemlok 205<br>Chemlok 233 | Thixon D12809<br>Thixon AM-2 | Thixon D12809 | Thixon AM-2 |
|------------------|----------------------------|------------------------------|---------------|-------------|
| Original         | 362 R                      | 368 R                        | 255 R70 RC30  | 275 R       |
| Aged 70hrs/212°F | 370 R                      | 391 R                        | 390 R         | —           |
| Aged 70hrs/250°F | 300                        | —                            | 271 R         | —           |
| Aged 70hrs/300°F | 229 R35 M65                | 246 R60M20RC20               | 184 R35 M65   | 258 R       |
| Aged 70hrs/350°F | 287 R35 M55 RC10           | 162 M                        | —             | 305 R       |

TABLE 9

HYDROLYTIC STABILITY OF NEOPRENE AND BUTADIENE/STYRENE RUBBER-TO-STEEL  
VULCANIZED BONDED ASSEMBLIES

Method C, Conical, 1b

Neoprene

Bonding Agent

SBR  
Bonding Agent

|                               | <u>Chemlok 205</u><br><u>Chemlok 233</u> | <u>Thixon D12809</u> | <u>Thixon NM-2</u> | <u>Thixon P-5</u><br><u>Thixon CB-3</u> |
|-------------------------------|--|----------------------|--------------------|---|
| Original                      | 378 R90 RC10                             | 182 RC               | 193 RC             | 220 RC                                  |
| Aged 14 days/185°F/air        | 360 R                                    | —                    | —                  | 416 RC                                  |
| Aged 14 days/185°F/over water | 324 R                                    | —                    | —                  | 309 RC                                  |
| Aged 56 days/185°F/air        | 422 R95 RC5                              | 205 RC               | 205 RC             | 318 R                                   |
| Aged 56 days/185°F/over water | 353 R90 RC10                             | 193 M                | 197 RC             | 352 R                                   |

TABLE 10

HYDROLYTIC STABILITY OF ETHYLENE PROPYLENE TERPOLYMER  
RUBBER-TO-STEEL VULCANIZED BONDED-ASSEMBLIES

Methad C, Conical, 1b

Bonding Agents

|                               | Thixon P-5<br>Thixon AP 1559 | Thixon D12809<br>Thixon AP 1654<br>(2 Coats) | Chemlok 205<br>Chemlok 234B<br>(2 Coats) | Chemlok 205<br>Chemlok 236<br>(2 Coats) |
|-------------------------------|------------------------------|--|--|---|
| Original                      | 231 RC                       | 257 RC                                       | 227 RC                                   | 148 RC                                  |
| Aged 14 days/185°F/air        | 229 RC                       | ---  | ---                                      | ---                                     |
| Aged 14 days/185°F/over water | 252 RC                       | ---  | ---                                      | ---                                     |
| Aged 56 days/185°F/air        | 267 RC                       | 364 RC10 R90                                 | 333 RC57 R43                             | 293 RC70 R30                            |
| Aged 56 days/185°F/over water | 278 RC                       | 319 RC50 R50                                 | 216 RC                                   | 268 M                                   |

TABLE 11

HYDROLYTIC STABILITY OF BUTADIENE/ACRYLONITRILE  
RUBBER-TO-STEEL VULCANIZED BONDED-ASSEMBLIES

Method C, Conical 1b

Bonding Agents

|                               | Chemlok 205<br>Chemlok 233 | Thixon D12809 | Thixon D12809<br>Thixon AM-2 |
|-------------------------------|----------------------------|---------------|------------------------------|
| Original                      | 362 R                      | 355 R70 RC30  | 368 R                        |
| Aged 14 days/185°F/air        | 349 R                      | —             | —                            |
| Aged 14 days/185°F/over water | 288 R                      | —             | —                            |
| Aged 28 days/185°F/air        | 384 R                      | —             | —                            |
| Aged 28 days/185°F/over water | 287 RC70 R30               | —             | —                            |
| Aged 56 days/185°F/air        | 355 R                      | 392 R         | 342 R                        |
| Aged 56 days/185°F/over water | 172 M                      | 255 M         | 221 M                        |

TABLE 12

EFFECT OF OIL ON BUTADIENE/ACRYLONITRILE AND NEOPRENE  
RUBBER-TO-STEEL VULCANIZED BONDED ASSEMBLIES

Method C. Conical. 1b

| <u>Acrylonitrile Rubber<br/>Bonding Agents</u> | <u>Original</u> | <u>Aged 70hrs/212°/ASTM #3 Oil</u> | <u>Aged 70hrs/257°/ASTM #3 OIL</u> |
|--|-----------------|------------------------------------|------------------------------------|
| Thixon D12309                                  | 355 R70 RC30    | 310 R85 M15                        | 205 R75 M25                        |
| Thixon D12809                                  |                 |                                    |                                    |
| Thixon AM-2                                    | 368 R           | 346 R                              | 306 R                              |
| Thixon AM-2                                    | 375 R           | 339 R                              | 312 R                              |
| Chemlok 205                                    |                 |                                    |                                    |
| Chemlok 233                                    | 362 R           | 331 R                              | 304 R                              |
| Neoprene Rubber<br><u>Bonding Agents</u>       |                 |                                    |                                    |
| Thixon D12809                                  | 182 RC          | 121 M                              | 95 M                               |
| Thixon NM-2                                    | 193 RC          | 146 RC                             | 132 RC                             |
| Chemlok 205                                    |                 |                                    |                                    |
| Chemlok 233                                    | 378 R90 RC10    | 299 R98 RC2                        | 276 R98 RC2                        |

of bond failure. Neoprene specimens bonded with Thixon D12809 showed bond strength losses of 34 and 48 percent when aged at 212°F and 257°F, respectively. The type of bond failure changed to 100 percent metal-primer interface. Specimens bonded with Thixon NM-2 showed bond strength losses of 24 and 32 percent after aging at 212°F and 257°F, respectively. Specimens bonded with Chemlok 233 showed no sign of bond strength loss. All neoprene-bonded specimens showed signs of oil deposits on the metal substrate surface after test.

The oil immersion tests indicate that they are probably unsuitable for use in accelerating bond failure. The butadiene/acrylonitrile rubber-bonded specimens showed no loss in bond strength after immersion in ASTM #3 oil because of the inherent resistance of this elastomer to oil. The neoprene rubbers specimens, on the other hand, evidenced loss in bond strength because of the relatively poor resistance of the rubber to oil. This fact indicates that a different oil or even a fuel would probably have to be used with each type of rubber being tested. An accelerated test in which the immersion medium would have to be changed for each type of rubber specimen would be an undesirable test.

Toward the end of FY74, the standard methods of accelerating the breakdown of solid rubber such as exposure to hot air, humidity and oil apparently were completely unsuitable for accelerating the breakdown of rubber-to-metal vulcanized bonds. Through active participation in ASTM Subcommittee D-11.13 on adhesion, it was learned that commercial adhesive manufacturers are also seeking means of accelerating the degradation of vulcanized bonds. The methods being employed are based on the dynamic fatigue of bonded specimens. No entirely satisfactory fatigue tester has been developed to date. The Research Directorate personnel have come to the conclusion that perhaps a combination of heat, humidity, and dynamic flexing would provide the type of bond breakdown observed during long-term exposure. Accordingly, a dynamic test device was designed and built. This will be extensively used during the FY75 portion of this study. The test device rotates one end of a bonded conical specimen 45° to the left and 45° to the right of center; the other end of the specimen is firmly clamped. Either tension or compression may be applied to the bonded specimen while it is being flexed in torsion. Speed of rotation is variable, up to 300 cpm. Dynamic flexing will be used before and after exposure of bonded specimens to heat and humidity or, if this fails to accelerated bond breakdown, flexing will be completed in conjunction with heat and humidity.

#### CONCLUSIONS:

Environmental exposure data and hydrolytic stability test data obtained in this study can be compared to show that both have the same general effect on vulcanized bonded-assemblies. Neoprene and SBR specimens showed no loss in bond strength when subjected to the hydrolytic stability test or when exposed to the severe climatic conditions at Panama. EPDM and nitrile vulcanized bonded-specimens showed a change in the type of bond failure and a decrease in bond strength in both instances. The hydrolytic stability test is not as severe as the exposure test at Panama, but the pattern of decline of bond

strength was the same in both instances. Data obtained from accelerated testing can be used to enable one to select the bonding agent system that is suitable for the task required of the vulcanized bonded-assembly.

#### RECOMMENDATIONS:

Future work should include the development of dynamic methods of testing rubber-to-metal vulcanized bonded-assemblies. These dynamic methods should be used in conjunction with the accelerated test that involves heat and humidity.