Joint Technical Report

Validation of Novel Electroactive Polymers as Replacement of Hexavalent Chromium Pretreatments

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TABLE OF CONTENTS

TABLE OF CONTENTS		p. ii
LIST OF FIGURES		p. viii
LIST OF TABLES		p. xviii
LIST OF ACRONYMS		p. xxiv
ACKNOWLEDGEMENTS		p. xxvii
EXECUTIVE SUMMARY		p. xxviii
1.0	**INTRODUCTION**	p. 1
1.1	Background	p. 1
1.1.1	Synthesis of EAPs	p. 3
1.1.2	Corrosion Processes	p. 5
1.1.3	Corrosion Protection with EAPs	p. 7
1.2	Objective of the Demonstration	p. 10
1.3	Regulatory Drivers	p. 11
2.0	**DEMONSTRATION TECHNOLOGY**	p. 12
2.1	Technology Description	p. 12
2.2	Technology Development	p. 14
2.3	Advantages and Limitation of the Technology	p. 16
3.0	**PERFORMANCE OBJECTIVES**	p. 17
4.0	**SITE/PLATFORM DESCRIPTION**	p. 22
4.1	Test Platform/Facilities	p. 22
4.1.1	Test Platforms/Facilities at WPAFB	p. 22
4.1.2	Test Platforms/Facilities at ARL	p. 25
4.1.3	Test Platforms/Facilities at Fleet Readiness Centers (FRC)	p. 26
4.1.4	Test Platforms/Facilities at NASA- KSC	p. 28
4.2	Present Operations	p. 29
4.2.1	Present Operations at WPAFB	p. 29
4.2.2	Present Operations at ARL	p. 30
4.2.3	Present Operations at FRC	p. 30
4.2.4	Present Operations at NASA-KSC	p. 31
4.3	Site-Related Permits and Regulations	p. 31

5.0	**TEST DESIGN**	p. 32
5.1	Laboratory Testing	p. 33
5.1.1	Adhesion Testing	p. 33
5.1.1.1	Crosshatch Adhesion (MIL-PRF-23377)	p. 33
5.1.1.2	X-Cut Tape Adhesion (MIL-PRF-85285)	p. 36
5.1.1.3	Wet-Tape Adhesion (MIL-PRF-23377, MIL-PRF-85285)	p. 36
5.1.1.4	Pull-off Adhesion ASTM D 4541 PATTI	p. 36
5.1.2	Accelerated Weathering Tests	p. 38
5.1.2.1	Neutral Salt Spray Exposure (ASTM B 117) (MIL-DTL-81076, MIL-PRF-23377)	p. 39
5.1.2.2	Accelerated Corrosion Test (GM9540P)	p. 41
5.1.2.3	Xenon Arc Accelerated Weathering Test, Corrosion Resistance and Crosshatch Adhesion Test Series (ASTM G 155, ASTM B117, ASTM D 3359)	p. 45
5.1.2.4	Moist Sulfur Dioxide (SO_2) Tests	p. 45
5.1.2.5	Filiform Corrosion Resistance of Organic Coating Testing	p. 46
5.1.3	Pencil Hardness (MIL-PRF-23377, MIL-PRF-85285)	p. 46
5.1.4	Impact Flexibility Testing (MIL-PRF-23377, MIL-PRF-85285)	p. 46
5.1.5	Hydraulic, Lubricating Oil and JP-8 Fluid Resistance (MIL-PRF-83282, MIL-PRF-23699, MIL-DTL-83133, MIL-PRF-23377, MIL-PRF-85285)	p. 47
5.1.6	Repairability Testing (T.O. 1-1-8, Table 3-2; MIL-R-81294; MIL-PRF-25134 and TT-R-2918)	p. 47
5.2	Outdoor Exposure Testing	p. 50
5.2.1	Marine Outdoor Exposure Testing	p. 51
5.3	Toxicology Testing	p. 51
5.3.1	Acute Oral Toxicity-Up and Down Procedure (UDP) Standard Protocol Number 1010-02	p. 51
5.3.2	Acute Dermal Toxicity/LD_{50} in Rabbits Standard Protocol Number 1100-02	p. 52
5.3.3	Acute Eye Irritation in Rabbits Standard Protocol 1200-02	p. 52
5.3.4	Acute Dermal Irritation in Rabbits Standard Protocol 1130-02	p. 52
5.3.5	Delayed Contact Dermal Sensitization Test-Buehler Standard Protocol Number 1160-03	p. 53
5.3.6	Acute Inhalation Toxicity/LC_{50} in Rats Standard Protocol Number 1300-02	p. 54
5.4	Field Testing	p. 55
5.4.1	Field Testing at WPAFB	p. 55
5.4.2	Field Testing at ARL	p. 55
5.4.3	Field Testing at FRC	p. 55

6.0	**PERFORMANCE ASSESSMENT**	p. 56
6.1	Laboratory Testing	p. 56
6.1.1	**Laboratory Testing at WPAFB**	**p. 56**
6.1.1.1	BAM-PPV Laboratory Testing	p. 56
6.1.1.1.1	Solubility Studies	p. 56
6.1.1.1.1.1	HVLP Application and Panel Preparations	p. 58
6.1.1.1.1.2	Panel Preparation	p. 59
6.1.1.1.1.3	Solution Concentration and Coating Thickness Parameters	p. 60
6.1.1.1.1.4	Dwell and Drying Parameters	p. 65
6.1.1.1.1.5	General Application Parameters	p. 74
6.1.1.1.2	Adhesion Testing	p. 74
6.1.1.1.2.1	ASTM D 3359, Method B, Crosshatch Adhesion (MIL-PRF-23377)	p. 74
6.1.1.1.2.2	FTMS 141D, Method 6301, Wet-Tape Adhesion (MIL-PRF-23377, MIL-PRF-85285)	p. 79
6.1.1.1.2.3	ASTM D 4541 PATTI, Pull-off Adhesion	p. 82
6.1.1.1.3	Accelerated Weathering Tests	p. 86
6.1.1.1.3.1	Neutral Salt Spray Testing (ASTM B117)	p. 86
6.1.1.1.3.2	GM9540P Accelerated Corrosion Test	p. 100
6.1.1.1.3.3	Xenon-Arc Accelerated Weathering Test	p. 107
6.1.1.1.4	Additional Tests	p. 111
6.1.1.1.4.1	ASTM D 3363 Pencil Hardness (MIL-PRF-23377, MIL-PRF-85285)	p. 112
6.1.1.1.4.2	Hydraulic, Lubricating oil, and JP-8 Fluid Resistance (MIL-PRF-83282, MIL-L-23699, MIL-PRF-23377, MIL-PRF-85285)	p. 115
6.1.1.1.4.3	ASTM D 6905 GE Impact Flexibility Testing (MIL-PRF-23377, MIL-PRF-85285)	p. 122
6.1.1.1.4.4	Repairability Testing (T.O. 1-1-8, Table 3-2; MIL-PRF-81294; MIL-PRF-25134 and TT-R-2918)	p. 125
6.1.1.1.4.5	Storage and Stability Studies	p. 127
6.1.1.1.5	***Conclusions for BAM-PPV Laboratory Testing***	***p. 131***
6.1.1.2	**MEH-PPV Studies**	**p. 132**
6.1.1.2.1	Solubility Studies	p. 132
6.1.1.2.1.1	Panel Preparation	p. 133
6.1.1.2.1.2	Solution Concentration and Coating Thickness Studies	p. 134
6.1.1.2.1.3	Dwell and Cure Parameters	p. 136
6.1.1.2.1.4	General Application Parameters	p. 136
6.1.1.2.2	Adhesion Testing	p. 137
6.1.1.2.2.1	ASTM D 3359 Method B, Crosshatch Adhesion (MIL-PRF-23377)	p. 137

6.1.1.2.2.2	FTMS 141D Method 6301, Wet-Tape Adhesion (WTA) (MIL-PRF-23377, MIL-PRF-85285)	p. 139
6.1.1.2.2.3	ASTM D 4541 PATTI, Pull-off Adhesion	p. 141
6.1.1.2.3	Accelerated Weathering Tests	p. 142
6.1.1.2.3.1	Neutral Salt Spray Testing (ASTM B117)	p. 142
6.1.1.2.3.2	GM9540P Accelerated Corrosion Test	p. 147
6.1.1.2.3.3	Xenon-Arc Accelerated Weathering Test	p. 150
6.1.1.2.4	Additional Tests	p. 151
6.1.1.2.4.1	ASTM D 3363 Pencil Hardness (MIL-PRF-23377, MIL-PRF-85285)	p. 151
6.1.1.2.4.2	Hydraulic, Lubricating Oil, JP-8 Fluid Resistance (MIL-PRF-83282, MIL-PRF-23699, MIL-DTL-83133, MIL-PRF-23377, MIL-PRF-85285)	p. 153
6.1.1.2.4.3	ASTM D 6905 GE Impact Flexibility Testing (MIL-PRF-23377, MIL-PRF-85285)	p. 156
6.1.1.2.5	***Conclusions of MEH-PPV Laboratory Testing***	***p. 157***
6.1.2	**Laboratory Testing at ARL**	**p. 158**
6.1.2.1	BAM-PPV and MEH-PPV Laboratory Testing	p. 158
6.1.2.1.1	Adhesion Studies	p. 159
6.1.2.1.2	Neutral Salt Spray Testing (ASTM B 117) for BAM-PPV Pretreatment	p. 165
6.1.2.2	***Conclusions for BAM-PPV and MEH-PPV Laboratory Studies***	***p. 176***
6.1.3	**Laboratory Testing at NAWCAD/NAWCWD for BAM-PPV/MEH-PPV Pretreatment Coatings**	**p. 176**
6.1.3.1	Tape Adhesion Studies	p. 176
6.1.3.2	Electrical Contact Resistance Testing (ECR)	p. 193
6.1.3.3	Accelerated Weathering Tests	p. 196
6.1.3.3.1	SO_2 Corrosion Testing for BAM-PPV and MEH-PPV Pretreatment Coatings	p. 196
6.1.3.3.2	Neutral Salt Spray Testing (ASTM B 117) for BAM-PPV Pretreatment Coating	p. 208
6.1.3.3.2.1	Neutral Salt Spray Exposure Testing for BAM-PPV and MEH-PPV Pretreatments with Full Military Coatings	p. 209
6.1.3.3.3	Filiform Testing for BAM-PPV and MEH-PPV Pretreatment Coatings	p. 211
6.1.3.4	***Conclusions for BAM-PPV and MEH-PPV Laboratory Testing***	***p. 213***
6.2	**Outdoor Exposure Testing**	**p. 213**
6.2.1	Testing Performed at WPAFB	p. 213
6.2.1.1	Outdoor Exposure (ASTM D 1014) for BAM-PPV Coated Coupons	p. 213
6.2.1.1.1	***Conclusions for BAM-PPV Outdoor Exposure Testing***	***p. 216***
6.2.2	**Marine Outdoor Exposure Testing**	**p. 217**

6.2.2.1	Testing Conducted at NASA-KSC	p. 217
6.2.2.2	Corrosion Resistance Performance	p. 219
6.2.2.2.1	ASTM D 1654 - Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments	p. 219
6.2.2.2.2	ASTM D 610 - Standard Test Method for Evaluating Degree of Rusting on Painted Surfaces	p. 222
6.2.2.2.3	ASTM D 714 - Standard Test Method for Evaluating Degree of Blistering of Paints	p. 223
6.2.2.2.4	Aesthetic and Other Technical Parameters	p. 224
6.2.2.2.5	ASTM D 2244- Standard Practice for Calculation of Color Tolerances and Color Differences from Instrumentally Measured Color Coordinates	p. 224
6.2.2.2.6	ASTM D523-Standard Test Method for Specular Gloss	p. 225
6.2.2.2.7	Sample Photodocumentation	p. 226
6.2.2.3	***NASA-KSC Conclusions for Marine Outdoor Exposure Testing***	***p. 236***
6.3	**Toxicology Results**	**p. 237**
6.3.1	Acute Oral Toxicity-Up and Down Procedure (UDP)	p. 237
6.3.2	Acute Dermal Toxicity/LD_{50} in Rabbits	p. 237
6.3.3	Acute Dermal Irritation in Rabbits	p. 237
6.3.4	Acute Eye Irritation in Rabbits	p. 238
6.3.5	Delayed Contact Dermal Sensitization Test-Buehler Method	p. 238
6.3.6	Acute Inhalation Toxicity/LC_{50} in Rats	p. 239
6.3.7	***Conclusions for Toxicology Studies***	***p. 240***
6.4	**Field Tests for BAM-PPV Pretreatment Coating Systems**	**p. 241**
6.4.1	Field Tests for BAM-PPV Pretreatment Coating Conducted at WPAFB	p. 241
6.4.1.1	***Conclusions for Field Studies Conducted at WPAFB***	***p. 245***
6.4.2	Field Tests for BAM-PPV Pretreatment Coating Conducted at ARL	p. 245
6.4.2.1	***Conclusions for Field Studies Conducted at ARL***	***p. 248***
6.4.3	Field Tests for BAM-PPV Pretreatment Coating Conducted at FRC-NW	p. 248
6.4.3.1	***Conclusions for Field Studies Conducted at FRC-NW***	***p. 251***
7.0	**COST ASSESSMENT**	p. 251
7.1	Cost Model	p. 251
7.2	Cost Analysis and Comparison	p. 252
8.0	**IMPLEMENTATION ISSUES**	p. 253
8.1	Environmental Checklist	p. 253

8.2	Future Plans	p. 253
8.3	Regulatory Issues	p. 254
8.4	End-User/Original Equipment Manufacturer (OEM) Issues	p. 254
9.0	**REFERENCES**	p. 255
10.0	**LIST OF PAPERS/PRESENTATIONS**	p. 260
APPENDIX A:	**POINTS OF CONTACT**	p. 262
APPENDIX B:	**MATERIALS AND SYNTHETIC METHODS**	p. 263
Appendix B.1.0	Materials and General Analytical Methods	p. 263
Appendix B.1.1	Polymerization of Monomer and Characterization of BAM-PPV	p. 263
Appendix B.1.2	Improvement in the Synthesis of the Monomer (1,4-bis(chloromethyl)-2-(2-ethylhexyloxy)-5-methoxybenzene) to obtain MEH-PPV	p. 267
Appendix B.1.3	Synthesis of racemic 1-(2-ethylhexyloxy)-4-methoxybenzene (Structure 1)	p. 269
Appendix B.1.4	Synthesis of racemic 1,4-bis(chloromethyl)-2-(2-ethylhexyloxy)-5-methoxybenzene (Structure 2)	p. 270
Appendix B.1.5	Polymerization of 1,4-bis(chloromethyl)-2-(2-ethylhexyloxy)-5-methoxybenzene to obtain MEH-PPV	p. 272

LIST OF FIGURES

Figure 1: Four Forms of PANI p. 3
Figure 2: Protonic Acid Doping p. 4
Figure 3: Heterocyclic Structure p. 4
Figure 4: PPV Synthesis p. 5
Figure 5: BAM-PPV Structure p. 9
Figure 6: BAM-PPV on AA2024-T3 Panels at 336 hours Neutral Salt Spray (NSS) Exposure p. 9
Figure 7: BAM-PPV coated AA2024-T3 (> 1 micron) at 336 hours NSS p. 14
Figure 8: TCP coated AA2024-T3 at 336 hours NSS p. 14
Figure 9: Air Force C-5 Cargo Aircraft p. 24
Figure 10: C-5 Aircraft Rear Hatch Door p. 24
Figure 11: Army Bradley Vehicle with Headlight Cover p. 25
Figure 12: Naval Air Station, Whidbey Island p. 27
Figure 13: NASA Beach Corrosion Test Site p. 29
Figure 14: Flow Chart for EAP testing and Down-selection for Field Studies during Phases I and II p. 32
Figure 15: ASTM D 3359, Method B, Crosshatch Adhesion Rating Scale Description p. 34
Figure 16: ASTM D 3359, Method B, Crosshatch Adhesion Visual Standard Reference Image p. 35
Figure 17: Hydraulic Adhesion Test Equipment and Dolly configuration p. 38
Figure 18: Rating Scale from ASTM D 714 p. 44
Figure 19: ASTM D 3363 Pencil Hardness Rating System p. 46
Figure 20: Primary Coating System Application for Repairability Samples p. 49
Figure 21: Coating System Removal for Repairability Testing p. 50
Figure 22: Chemical Structures of Xylene with Ethylbenzene and of Oxsol-100 (Taken from Sigma-Aldrich) p. 57
Figure 23: Paint Booth used for Spray gun application of BAM-PPV at NAWCWD p. 59
Figure 24: Surface Profile of BAM-PPV Coating Applied at Approximate Thickness of 0.7 µm using Oxsol-100 as the Solvent. p. 61
Figure 25: Edge of BAM-PPV at Tear, Approximation of Thickness at 0.7 µm p. 61
Figure 26: Salt Spray Samples, 1 wt% BAM-PPV; 3 Cross-coats; AA2024-T3 Substrate p. 62
Figure 27: Salt Spray Samples, 1 wt% BAM-PPV; 3 Cross-coats; AA7075-T6 Substrate p. 62

Figure 28: 1 wt% BAM-PPV, Approximately 1 µm Thick, for Comparison with Figure 29 p. 64
Figure 29: 3 wt% BAM-PPV, Approximately 1 µm Thick, for Comparison with Figure 28 p. 65
Figure 30: Neutral Salt Spray Samples, 1 wt% BAM-PPV in Oxsol-100, 1-hour Dwell before HVLP Spray Application p. 67
Figure 31: Neutral Salt Spray Samples, 1 wt% BAM-PPV in Oxsol-100, 3-hour Dwell before HVLP Spray Application p. 67
Figure 32: SEM Micrograph, 1 wt% BAM-PPV in Oxsol-100, 1-hour Dwell p. 68
Figure 33: SEM Micrograph, 1 wt% BAM-PPV in Oxsol-100, 3-hour Dwell p. 68
Figure 34: Neutral Salt Spray Samples, 1 wt% BAM-PPV in Oxsol-100; 24-hour Room Temperature Cure and Ambient Relative Humidity p. 70
Figure 35: Neutral Salt Spray Samples, 1 wt% BAM-PPV in Oxsol-100; 16-hour Force Cure at 60 °C and Near-Zero Relative Humidity p. 70
Figure 36: Salt Spray Samples, 1 wt% BAM-PPV in *p*-Xylene, 24-hour Room Temperature Cure and Ambient Relative Humidity p. 71
Figure 37: Salt Spray Samples, 1 wt% BAM-PPV in *p*-Xylene; 16-hour Force Cure at 60 °C and Near-Zero Relative Humidity p. 71
Figure 38: SEM Micrograph, 1 wt% BAM-PPV in Oxsol-100, 24-hour Room Temperature Cure and Ambient Relative Humidity p. 72
Figure 39: SEM Micrograph, 1 wt% BAM-PPV in Oxsol-100, 16-hour Force Cure at 60 °C and Near-Zero Relative Humidity p. 72
Figure 40: SEM Micrograph, 1 wt% BAM-PPV in *p*-Xylene, 24-hour Room Temperature Cure and Ambient Relative Humidity p. 73
Figure 41: SEM Micrograph, 1 wt% BAM-PPV in *p*-Xylene, 16-hour Force Cure at 60 °C and Near-Zero Relative Humidity p. 73
Figure 42: BAM-PPV + MIL-PRF-23377 Chromated Primer; ASTM B 117 Results p. 87
Figure 43: BAM-PPV + MIL-PRF-23377 Chromated Primer + MIL-PRF-85285 APC Topcoat; ASTM B 117 Results p. 87
Figure 44: Alodine 1200S + MIL-PRF-23377 Chromated Primer; ASTM B 117 Results p. 88
Figure 45: Alodine 1200S + MIL-PRF-23377 Chromated Primer + MIL-PRF-85285 APC Topcoat; ASTM B 117 Results p. 88

Figure 46: PreKote + MIL-PRF-23377 Chromated Primer; ASTM B 117 Results p. 89
Figure 47: PreKote + MIL-PRF-23377 Chromated Primer + MIL-PRF-85285 APC Topcoat; ASTM B 117 Results p. 89
Figure 48: Neutral Salt Spray Results for Alodine 1200S after 336 hours of Exposure p. 90
Figure 49: Neutral Salt Spray Results for BAM-PPV after 336 hours of Exposure p. 91
Figure 50: Neutral Salt Spray Results for PreKote after 336 hours of Exposure p. 91
Figure 51: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A after 2000 hours of Exposure p. 93
Figure 52: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 after 2000 hours of Exposure p. 93
Figure 53: Neutral Salt Spray Results for Pretreatments Nonchromated Epoxy Primer Deft 02-GN-084 after 2000 hours of Exposure p. 94
Figure 54: Neutral Salt Spray Results for Pretreatments MIL-PRF-23377 Nonchromated Epoxy Primer Hentzen 16708TEP after 2000 hours of Exposure p. 94
Figure 55: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 2000 hours of Exposure p. 97
Figure 56: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 2000 hours of Exposure p. 97
Figure 57: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 2000 hours of Exposure p. 98
Figure 58: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 2000 hours of Exposure p. 98
Figure 59: Neutral Salt Spray Results for Pretreatments with Nonchromated Epoxy Primer Deft 02-GN-084 and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 2000 hours of Exposure p. 99
Figure 60: Neutral Salt Spray Results for Pretreatments with Nonchromated Epoxy Primer Deft 02-GN-084 and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 2000 hours of Exposure p. 99

Figure 61: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Hentzen 16708TEP and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 2000 hours of Exposure p. 100
Figure 62: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Hentzen 16708TEP and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 2000 hours of Exposure p. 100
Figure 63: GM9540P Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 40 Cycles of Exposure p. 104
Figure 64: GM9540P Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 40 Cycles of Exposure p. 104
Figure 65: GM9540P Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 40 Cycles of Exposure p. 105
Figure 66: GM9540P Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 40 Cycles of Exposure p. 105
Figure 67: GM9540P Results for Pretreatments with Nonchromated Epoxy Primer Deft 02-GN-084 and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 40 Cycles of Exposure p. 106
Figure 68: GM9540P Results for Pretreatments with Nonchromated Epoxy Primer Deft 02-GN-084 and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 40 Cycles of Exposure p. 106
Figure 69: GM9540P Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Hentzen 16708TEP and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 40 Cycles of Exposure p. 107
Figure 70: GM9540P Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Hentzen 16708TEP and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 40 Cycles of Exposure p. 107
Figure 71: Reparability Using Standard Coating (Left side) and Test Coating w/BAM-PPV (Right side) p. 127
Figure 72: Reparability Using CCC + Nonchromated system (Left side) and Test Coating w/BAM-PPV p. 127
Figure 73: Entire Panel Coated /Repaired with Test System (BAM-PPV + MIL-PRF-23377C+ MIL-PRF-85285 APC) p. 127

Figure 74: Repaired System (Left Side) with BAM-PPV p. 127
Figure 75: Repaired System (Right Side) with BAM-PPV p. 127
Figure 76: BAM-PPV (As Received) Performance after 1000 hours of Salt Spray p. 130
Figure 77: BAM-PPV (6 months in storage) Performance after 1000 hours of Salt Spray p. 131
Figure 78: MEH-PPV Structure p. 132
Figure 79: Neutral Salt Spray Samples, 1 wt% MEH-PPV in Oxsol-100 and p-Xylene 4-5 Days-Room Temperature Cure and Ambient Relative Humidity p. 135
Figure 80: Crosshatch Adhesion Samples, 1 wt% MEH-PPV in Oxsol-100 and *p*-Xylene 4-5 Days- Room Temperature Cure and Ambient Relative Humidity p. 136
Figure 81: Neutral Salt Spray Results for MEH-PPV Pretreatment after 336 hours of Exposure p. 142
Figure 82: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A after 2000 hours of Exposure p. 144
Figure 83: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 after 2000 hours of Exposure p. 144
Figure 84: Neutral Salt Spray Results for Pretreatments Nonchromated Epoxy Primer Deft 02-GN-084 after 2000 hours of Exposure p. 145
Figure 85: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A and MIL-PRF-85285 Topcoat Deft ELT 99-GY-001 after 2000 hours of Exposure p. 145
Figure 86: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 and MIL-PRF-85285 Topcoat Deft ELT 99-GY-001 after 2000 hours of Exposure p. 146
Figure 87: Neutral Salt Spray Results for Pretreatments with Nonchromated Epoxy Primer Deft 02-GN-084 and MIL-PRF-85285 Topcoat p. 146
Figure 88: GM9540P Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer and MIL-PRF-85285 Topcoat after 40 Cycles of Exposure p. 148
Figure 89: GM9540P Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer and MIL-PRF-85285 Topcoat after 40 Cycles of Exposure p. 149

Figure 90: GM9540P Results for Pretreatments With Nonchromated Epoxy Primer and MIL-PRF-85285 Topcoat after 40 Cycles of Exposure p. 149
Figure 91: Adhesive debonding p. 159
Figure 92: Cohesive debonding p. 159
Figure 93: Mixed mode debonding p. 159
Figure 94: Example of Multiple Failure Modes and Locations p. 159
Figure 95: BAM-PPV on grit blasted panel with MIL-DTL-53022 and MIL-P-53039 after 432 hours ASTM B 117 exposure p. 167
Figure 96: BAM-PPV on as received steel with MIL-DTL-53022 and MIL-PRF-85285 following 432 hours ASTM B 117 exposure p. 169
Figure 97: DoD-P-15328 on as received steel panel with MIL-DTL-53022 and MIL-DTL-64159 after 432 hours ASTM B 117 exposure p. 171
Figure 98: DoD-P-15328 on grit blasted steel panel with MIL-DTL-53022 and MIL-DTL-64159 after 432 hours ASTM B 117 exposure p. 171
Figure 99: Average 1-Day Wet Tape Adhesion Ratings for MEH-PPV on AA2024 p. 179
Figure 100: Test Panels after 1-Day WTA - MEH-PPV on AA2024 with MIL-PRF-23377C2 p. 179
Figure 101: Test Panels after 1-Day WTA- MEH-PPV on AA2024 with 02GN083 p. 179
Figure 102: Test Panels after 1-Day WTA MEH-PPV on AA2024 with 16708TEP p. 180
Figure 103: Test Panels after 1-Day WTA- MEH-PPV on AA2024 with 44GN098 p. 180
Figure 104: Test Panels after 1-Day WTA- MEH-PPV on AA2024 with TT-P-2756 p. 180
Figure 105: Average 1-Day Wet Tape Adhesion Ratings for MEH-PPV on AA6061 p. 181
Figure 106: Test Panels after 1-Day WTA - MEH-PPV on AA6061 with MIL-DTL-53022 p. 181
Figure 107: Test Panels after 1-Day WTA - MEH-PPV on AA6061 with MIL-DTL-53030 p. 181
Figure 108: Average 1-Day Wet Tape Adhesion Ratings for MEH-PPV on AA7075 p. 182
Figure 109: Test Panels after 1-Day WTA-MEH-PPV on AA7075 with MIL-PRF-23377C2 p. 182
Figure 110: Test Panels after 1-Day WTA-MEH-PPV on AA7075 with 02GN083 p. 182
Figure 111: Test Panels after 1-Day WTA- MEH-PPV on AA7075 with 16708TEP p. 183

Figure 112: Test Panels after 1-Day WTA-MEH-PPV on AA7075 with 44GN098 p. 183
Figure 113: Test Panels after 1-Day WTA-MEH-PPV on AA7075 with TT-P-2756 p. 183
Figure 114: Average 1-Day Wet Tape Adhesion Ratings for BAM-PPV on AA2024 p. 184
Figure 115: Test Panels after 1-Day WTA- BAM-PPV on AA2024 with MIL-PRF-23377C2 p. 184
Figure 116: Test Panels after 1-Day WTA- BAM-PPV on AA2024 with 02GN083 p. 184
Figure 117: Test Panels after 1-Day WTA-BAM-PPV on AA2024 with 16708TEP p. 185
Figure 118: Test Panels after 1-Day WTA-BAM-PPV on AA2024 with 44GN098 p. 185
Figure 119: Test Panels after 1-Day WTA- BAM-PPV on AA2024 with TT-P-2756 p. 185
Figure 120: Average 1-Day Wet Tape Adhesion Ratings for BAM-PPV on AA6061 p. 186
Figure 121: Test Panels after 1-Day WTA-BAM-PPV on AA6061 with MIL-DTL-53022 p. 186
Figure 122: Test Panels after 1-Day WTA - BAM-PPV on AA6061 with MIL-DTL-53030 p. 186
Figure 123: Average 1-Day Wet Tape Adhesion Ratings for BAM-PPV on AA7075 p. 187
Figure 124: Test Panels after 1-Day WTA-BAM-PPV on AA7075 with MIL-PRF-23377C2 p. 187
Figure 125: Test Panels after 1-Day WTA-BAM-PPV on AA7075 with 02GN083 p. 187
Figure 126: Test Panels after 1-Day WTA-BAM-PPV on AA7075 with 16708TEP p. 188
Figure 127: Test Panels after 1-Day WTA-BAM-PPV on AA7075 with 44GN098 p. 188
Figure 128: Test Panels after 1-Day WTA-BAM-PPV on AA7075 with TT-P-2756 p. 188
Figure 129: Average 1-Day Wet Tape Adhesion Ratings for BAM-PPV on 4130 p. 189
Figure 130: Test Panels after 1-Day WTA-BAM-PPV on 4130 with MIL-PRF-53039 Topcoat p. 189
Figure 131: Test Panels after 1-Day WTA-BAM-PPV on 4130 with MIL-PRF-85285 Topcoat p. 190
Figure 132: Test Panels after 1-Day WTA-BAM-PPV on 4130 with MIL-DTL-64159 Topcoat p. 190
Figure 133: Average 1-Day Wet Tape Adhesion Ratings for Test Panels with MIL-PRF-23377C2 p. 190
Figure 134: Average Wet Tape Adhesion Ratings for Test Panels with 02GN083 p. 191

Figure 135: Average 1-Day Wet Tape Adhesion Ratings for Test Panels with 16708TEP p. 191
Figure 136: Average 1-Day Wet Tape Adhesion Ratings for Test Panels with 44GN098 p. 192
Figure 137: Average 1-Day Wet Tape Adhesion Ratings for Test Panels with TT-P-2756 p. 192
Figure 138: ECR Results for BAM-PPV and MEH-PPV Test Panels p. 195
Figure 139: ECR Results for BAM-PPV Test Panels after 168hr NSF(NSS) p. 195
Figure 140: BAM-PPV on AA2024 with MIL-PRF-23377N after 500 hours SO_2 p. 197
Figure 141: BAM-PPV on AA2024 with MIL-PRF-85582C2 after 500 hours SO_2 p. 198
Figure 142: BAM-PPV on AA7075 with MIL-PRF-23377N after 500 hours SO_2 p. 198
Figure 143: BAM-PPV on AA2024 with MIL-PRF-23377C2 after 500 hours SO_2 p. 199
Figure 144: BAM-PPV on AA7075 with MIL-PRF-23377C2 after 500 hours SO_2 p. 199
Figure 145: BAM-PPV on AA2024 with MIL-PRF-85582N after 500 hours SO_2 p. 200
Figure 146: BAM-PPV on AA7075 with MIL-PRF-85582N after 500 hours SO_2 p. 200
Figure 147: BAM-PPV on AA7075 with MIL-PRF-85582C2 after 500 hours SO_2 p. 201
Figure 148: BAM-PPV on AA2024 with MIL-DTL-53022 after 500 hours SO_2 p. 201
Figure 149: BAM-PPV on AA7075 with MIL-DTL-53022 after 500 hours SO_2 p. 202
Figure 150: MEH-PPV on AA7075 with MIL-PRF-23377N after 500 hours SO_2 p. 203
Figure 151: MEH-PPV on AA7075 with MIL-PRF-85582N after 500 hours SO_2 p. 204
Figure 152: MEH-PPV on AA2024 with MIL-PRF-23377N after 500 hours SO_2 p. 204
Figure 153: MEH-PPV on AA7075 with MIL-PRF-85582C2 after 500 hours SO_2 p. 205
Figure 154: MEH-PPV on AA7075 with MIL-PRF-23377C2 after 500 hours SO_2 p. 205
Figure 155: MEH-PPV on AA2024 with MIL-PRF-23377C2 after 500 hours SO_2 p. 206
Figure 156: MEH-PPV on AA2024 with MIL-PRF-85582N after 500 hours SO_2 p. 206
Figure 157: MEH-PPV on AA2024 with MIL-DTL-53022 after 500 hours SO_2 p. 207

Figure 158: MEH-PPV on AA2024 with MIL-PRF-85582C2 after 500 hours SO_2 p. 207
Figure 159: MEH-PPV on AA7075 with MIL-DTL-53022 after 500 hours SO_2 p. 208
Figure 160: BAM-PPV at 0 hours p. 209
Figure 161: BAM-PPV at 336 hours p. 209
Figure 162: BAM-PPV at 840 hours p. 209
Figure 163: BAM-PPV at 1034 hours p. 209
Figure 164: Outdoor Weathering after 24 months, Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer and MIL-PRF-85285 Topcoat p. 214
Figure 165: Outdoor Weathering after 24 months, Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer and MIL-PRF-85285 Topcoat p. 215
Figure 166: NAWCWD Panels at the NASA Beach Corrosion Test Site p. 219
Figure 167: Initial-Rack 1 p. 227
Figure 168: Initial-Rack 2 p. 228
Figure 169: Initial-Rack 3 p. 229
Figure 170: Initial-Rack 4 p. 230
Figure 171: 12 Month Exposure-Rack 1 p. 231
Figure 172: 12 Month Exposure-Rack 2 p. 232
Figure 173: 12 Month Exposure-Rack 3 p. 233
Figure 174: 12 Month Exposure-Rack 4 p. 234
Figure 175: Deft 02-Y-40 Primer (Chromated epoxy primer MIL-PRF-23377C): Deft 99-GY-001 Topcoat (Polyurethane APC topcoat MIL-PRF-85285) p. 235
Figure 176: Deft 02-GN-083 Primer (Non chromate epoxy primer MIL-PRF-23377N): Deft 99-GY-001 Topcoat p. 235
Figure 177: Deft 02-GN-084 Primer (Non chromated epoxy primer MIL-PRF-23377N): Deft 99-GY-001 Topcoat p. 236
Figure 178: Deft 02-Y-40 Primer: Deft 99-GY-001 Topcoat p. 236
Figure 179: Deft 02-W-36 Primer: Deft 99-W-009 Topcoat p. 236
Figure 180: SEM Image of Ground BAM-PPV Powder p. 240
Figure 181: C-5 Rear Hatch Door Coated with 1wt. % BAM-PPV Pretreatment solution p. 242
Figure 182: C-5 Rear Hatch Door with Primer Coatings p. 243
Figure 183: C-5 Escape Hatch Door with Full Coating System p. 243
Figure 184: C-5 Test Door at 12 months (299.7 hours) p. 244
Figure 185: Headlight Cover Sand Blasted Prior to Coating with BAM-PPV p. 246
Figure 186: BAM-PPV Coated Headlight Cover p. 246

Figure 187: Headlight Cover Coated with MIL-PRF-53022 Primer and CARC Topcoat p. 247
Figure 188: Abrasion/Wear Damage after 1 Year Field Testing Headlight Cover p. 247
Figure 189: Abrasion/Wear Damage after 1 Year Field Testing (Backside View) p. 248
Figure 190: BAM-PPV coated bomb hoist prior to field testing (left) BAM-PPV coated with MIL-PRF-23377N and topcoated with MIL-PRF-85285 after 6 months of field testing (right) p. 249
Figure 191: BAM-PPV coated towbars prior to field testing (left) BAM-PPV coated with MIL-PRF-23377N and topcoated with MIL-PRF-85285 after 6 months of field testing (right) p. 249
Figure 192: BAM-PPV bomb hoist coated with MIL-PRF-23377N and topcoated with MIL-PRF-85285 after 1 year of field testing p. 250
Figure 193: BAM-PPV coated towbars with MIL-PRF-23377N and topcoated with MIL-PRF-85285 after 1 year of field testing p. 250
Figure 194: BAM-PPV Polymerization p. 264
Figure 195: FTIR spectrum of BAM-PPV film p. 265
Figure 196: Synthesis of 1,4-Bis(chloromethyl)-2-(2-ethylhexyloxy) -5-methoxybenzene (Structure 2) p. 268
Figure 197: MEH-PPV polymerization p. 273

LIST OF TABLES

Table 1: Best/Worst Performing BAM-PPV Pretreatment in Full Military Coating Systems p. xxix
Table 2: Structures of EAPs and Conductivities p. 2
Table 3: Estimated Compliance Costs for General Industry with New (CrVI) Guidelines p. 12
Table 4: Environmental Systems Allocation Database p. 16
Table 5: Performance Objectives for WP200527 ESTCP Project p. 18
Table 6: ASTM D 3359 Adhesion, Method A, Rating Scale p. 36
Table 7: PATTI Coating Failure Descriptions p. 37
Table 8: RXSSO Scribe Appearance Rating System p. 40
Table 9: RXSSO Undercutting Rating System p. 40
Table 10: Blistering Rating System (Modified from ASTM D 714) p. 40
Table 11: Rating of Failure at Scribe (Procedure A) Representative Mean Creepage from Scribe p. 42
Table 12: Rating Scale from ASTM D 610 p. 43
Table 13: Draize Scale for Erythema p. 53
Table 14: Draize Scale for Edema p. 53
Table 15: Scoring Code p. 54
Table 16: Solubility Data for BAM- PPV p. 58
Table 17: HVLP Spray Application Parameters, BAM-PPV p. 64
Table 18: ASTM D 3359 Crosshatch Adhesion (Pretreated Systems) p. 75
Table 19: ASTM D 3359 Crosshatch Adhesion (Primed Systems) p. 75
Table 20: ASTM D 3359 Crosshatch Adhesion (MIL-PRF-85285 Standard Topcoat) p. 76
Table 21: ASTM D 3359 Crosshatch Adhesion (MIL-PRF-85285 APC Topcoat) p. 77
Table 22: ASTM D 3359 Crosshatch Adhesion (MIL-PRF-85285 Topcoat) p. 78
Table 23: FTMS 141, ASTM D 3359 Wet Tape Adhesion (Primed Systems) p. 79
Table 24: FTMS 141, ASTM D 3359 Wet Tape Adhesion (MIL-PRF-85285 Standard Topcoat) p. 80
Table 25: FTMS 141, ASTM D 3359 Wet Tape Adhesion (MIL-PRF-85285 APC Topcoat) p. 81
Table 26: ASTM D 4541 Pull-Off Adhesion (Primed Systems) p. 83
Table 27: ASTM D 4541 Pull-Off Adhesion (MIL-PRF-85285 Standard Topcoat) p. 84
Table 28: ASTM D 4541 Pull-Off Adhesion (MIL-PRF-85285 APC Topcoat) p. 85
Table 29: ASTM B 117 Salt Spray Ratings (336 h) (Pretreated Systems) p. 90

Table 30: ASTM B 117 Salt Spray Ratings (2000 hours) (Primed Systems) p. 92
Table 31: ASTM B 117 Salt Spray Ratings (2000 hours) (MIL-PRF-85285 Standard Topcoat) p. 95
Table 32: ASTM B 117 Salt Spray Ratings (2000 hours) (MIL-PRF-85285 APC Topcoat) p. 96
Table 33: GM9540P Accelerated Corrosion (MIL-PRF-85285 Topcoat) p. 102
Table 34: GM9540P Accelerated Corrosion (MIL-PRF-85285 APC Topcoat) p. 103
Table 35: Xenon-Arc Exposure (MIL-PRF-85285 and APC Topcoats) p. 109
Table 36: Xenon-Arc and Salt Spray Exposure (MIL-PRF-85285 Standard Topcoat) (Alternating Every 500 hours, Total of 2000 hours Each) p. 110
Table 37: Xenon-Arc and Salt Spray Exposure (MIL-PRF-85285 APC Topcoat) (Alternating Every 500 hours, Total of 2000 hours Each) p. 111
Table 38: ASTM D 3363 Pencil Hardness (Primed Systems) p. 112
Table 39: ASTM D 3363 Pencil Hardness (MIL-PRF-85285 Topcoat) p. 113
Table 40: ASTM D 3363 Pencil Hardness (MIL-PRF-85285 APC Topcoat) p. 114
Table 41: MIL-L-23699 Lubricating Fluid Exposure (MIL-PRF-85285 Standard Topcoat) p. 116
Table 42: MIL-PRF-83282 Hydraulic Fluid Exposure (Primed Systems) p. 117
Table 43: MIL-PRF-83282 Hydraulic Fluid Exposure (MIL-PRF-85285 Standard Topcoat) p. 118
Table 44: MIL-PRF-83282 Hydraulic Fluid Exposure (MIL-PRF-85285 APC Topcoat) p. 119
Table 45: JP-8 Jet Fuel Exposure (MIL-PRF-85285 Standard Topcoat) p. 120
Table 46: JP-8 Jet Fuel Exposure (MIL-PRF-85285 APC Topcoat) p. 121
Table 47: ASTM D 6905 GE Impact (Primed Systems) p. 122
Table 48: ASTM D 6905 GE Impact (MIL-PRF-85285 Topcoat) p. 123
Table 49: ASTM D 6905 GE Impact (MIL-PRF-85285 APC Topcoat) p. 124
Table 50: Reparability Coating System Combinations for BAM-PPV Pretreatment Coating p. 125
Table 51: Comparative Criteria for Evaluation of Reparability Color Data p. 126
Table 52: Color Data, Reparability Testing of BAM-PPV Pretreatment Coating p. 126
Table 53: Storage Stability Laboratory Testing Performed at WPAFB p. 128
Table 54: Compiled Data for Storage Stability Testing, AA2024-T3 p. 129
Table 55: Solubility Data for MEH-PPV p. 133
Table 56: HVLP Spray Applications Parameters, MEH-PPV p. 137
Table 57: ASTM D 3359 Crosshatch Adhesion (MEH-PPV Pretreated System) on Various Substrates p. 137

Table 58: ASTM D 3359 Crosshatch Adhesion (MEH-PPV Primed Systems) p. 138
Table 59: ASTM D 3359 Crosshatch Adhesion (MEH-PPV Pretreatment with MIL-PRF-85285 Topcoated Systems) p. 139
Table 60: FTMS 141, ASTM D 3359 Wet Tape Adhesion (Primed Systems) p. 140
Table 61: FTMS 141, ASTM D 3359 Wet Tape Adhesion (MIL-PRF-85285 Topcoated Systems) p. 140
Table 62: ASTM D 4541 Pull-Off Adhesion (Primed Systems) p. 141
Table 63: ASTM D 4541 Pull-Off Adhesion (MIL-PRF-85285 Topcoated Systems) p. 141
Table 64: Neutral Salt Spray Ratings (336 hours) (MEH-PPV Pretreatment) p. 142
Table 65: Neutral Salt Spray Ratings (2000 hours) (MEH-PPV with Primer) p. 143
Table 66: Neutral Salt Spray Ratings (2000 hours) (MIL-PRF-85285 Topcoated Systems) p. 143
Table 67: GM9540P Accelerated Corrosion on 4130 Normalized Steel, Solvent-Cleaned Samples (MIL-PRF-85285 Topcoated Systems) p. 147
Table 68: GM9540P Accelerated Corrosion on 4130 Normalized Steel, Grit-Blasted Samples (MIL-PRF-85285 Topcoated Systems) p. 148
Table 69: Xenon Arc and Salt Spray Exposure (MIL-PRF-85285 Topcoated Systems) p. 150
Table 70: Xenon Arc Exposure (MIL-PRF-85285 Topcoated Systems) p. 151
Table 71: ASTM D 3363 Pencil Hardness (Primed Systems) p. 152
Table 72: ASTM D 3363 Pencil Hardness (MIL-PRF-85285 Topcoated Systems) p. 152
Table 73: MIL-PRF-23699 Lubricating Fluid Exposure (Primed Systems) p. 153
Table 74: MIL-PRF-23699 Lubricating Fluid Exposure (MIL-PRF-85285 Topcoated Systems) p. 154
Table 75: MIL-PRF-82382 Hydraulic Fluid Exposure (Primed Systems) p. 154
Table 76: MIL-PRF-83282 Hydraulic Fluid Exposure (MIL-PRF-85285 Topcoated Systems) p. 155
Table 77: MIL-DTL-83133 Jet Fuel Exposure (MIL-PRF-85285 Topcoated Systems) p. 156
Table 78: ASTM D 6905 GE Impact (Primed Systems) p. 156

Table 79: ASTM D 6905 GE Impact (MIL-PRF-85285 Topcoated Systems) p. 157
Table 80: Comparison of steel substrate surface preparation with MEH-PPV pretreatment p. 160
Table 81: Comparison of steel substrate surface preparation with BAM-PPV pretreatment p. 160
Table 82: Performance of BAM-PPV with Army primers p. 161
Table 83: Performance of MEH-PPV with Army primers p. 161
Table 84: Performance of BAM-PPV with Navy primers p. 161
Table 85: Performance of MEH-PPV with Navy primers p. 162
Table 86: Topcoat adhesion performance of BAM-PPV and MIL-DTL-53022 p. 162
Table 87: Topcoat adhesion performance of BAM-PPV over MIL-DTL-53030 p. 162
Table 88: Topcoat adhesion performance of BAM-PPV over MIL-PRF-23377N1 p. 162
Table 89: Topcoat adhesion performance of BAM-PPV over MIL-PRF-23377N2 p. 163
Table 90: Topcoat adhesion performance of MEH-PPV over MIL-DTL-53022 p. 163
Table 91: Topcoat adhesion performance of MEH-PPV over MIL-DTL-53030 p. 163
Table 92: Topcoat adhesion performance of MEH-PPV over MIL-PRF-23377N1 p. 163
Table 93: Topcoat adhesion performance of MEH-PPV over MIL-PRF-23377N2 p. 163
Table 94: Pretreatment performance using Army Primers p. 164
Table 95: Pretreatment performance using Navy primers p. 164
Table 96: Pretreatment Performance on Aluminum 6061 p. 165
Table 97: Pretreatment Performance on Aluminum 5083 p. 165
Table 98: ASTM D 1654 ratings for blasted steel with MIL-DTL-53039 topcoat p. 166
Table 99: ASTM D 1654 ratings for blasted steel with MIL-DTL-64159 topcoat p. 167
Table 100: ASTM D 1654 ratings for BAM-PPV pretreated systems with MIL-PRF-85285 topcoat p. 168
Table 101: ASTM D 1654 ratings for DoD-P-15328 on as received steel p. 170
Table 102: ASTM D 1654 ratings for DoD-P-15328 on as received steel p. 170
Table 103: ASTM D 1654 ratings for as received steel pretreated with BAM-PPV and topcoated with MIL-PRF-85285 not applied at ARL p. 172

Table 104: ASTM D 1654 ratings for AA 6061 with BAM-PPV pretreatment and MIL-DTL-53022 p. 173
Table 105: ASTM D 1654 ratings for AA 6061 with BAM-PPV pretreatment and MIL-DTL-53030 p. 173
Table 106: ASTM D 1654 ratings for AA 6061 with TCP and MIL-DTL-64159 topcoat p. 173
Table 107: ASTM D 1654 ratings for AA5083 with MIL-DTL-53022 primer p. 174
Table 108: ASTM D 1654 ratings for AA5083 with MIL-DTL-53030 primer p. 174
Table 109: ASTM D 1654 ratings for TCP with military primers and MIL-DTL-64159 topcoat p. 175
Table 110: BAM-PPV and MEH-PPV Coated with Various Primers p. 177
Table 111: BAM-PPV Pretreatment on High Strength 4130 Steel p. 177
Table 112: Tape Adhesion Test Matrix for Aluminum Alloy Panels p. 178
Table 113: Tape Adhesion Test Matrix for BAM-PPV Coated 4130 Steel Panels p. 178
Table 114: Average 1-Day Wet Tape Adhesion Results for Aluminum Test Panels with Various Primers p. 193
Table 115: 1-Day Wet Tape Adhesion Results for Steel Test Panels with Various Primers and Topcoats p. 193
Table 116: ECR Results for BAM-PPV and MEH-PPV Coated Test Panels p. 194
Table 117: Performance Rankings for BAM-PPV AA2024 and AA7075 Panels after 500 hours SO_2 p. 196
Table 118: Performance Rankings for MEH-PPV AA2024 and AA7075 Panels after 500 hours SO_2 p. 202
Table 119: Neutral Salt Fog Results for BAM-PPV and MEH-PPV Pretreatments with Chromium Primers and Topcoat p. 210
Table 120: Neutral Salt Fog Results for BAM-PPV and MEH-PPV Pretreatments with Non-Chromium Primer and Topcoat p. 210
Table 121: Neutral Salt Fog Results for BAM-PPV and MEH-PPV Pretreatments with Non-Chromium Self-Priming Topcoat p. 211
Table 122: Filiform Corrosion Results for BAM-PPV and MEH-PPV Pretreatments p. 212
Table 123: Outdoor Weathering Ratings (24 months) (MIL-PRF-85285 Topcoated Systems) at WPAFB p. 216
Table 124: Test Matrix for Samples Received from UDRI p. 218
Table 125: Summary of ASTM D 1654 Ratings-Corrosion from Scribe p. 221
Table 126: Summary of ASTM D 610 Ratings-Degree of Rusting p. 222
Table 127: Summary of ASTM D 714 Ratings-Degree of Blistering p. 223
Table 128: Average Delta E Color Calculations by Coating Type p. 225

Table 129: Buehler Method Test Results for BAM-PPV p. 239
Table 130: BAM-PPV and Cr(VI) controls on C-5 Cargo Plane Rear Hatch Door p. 242
Table 131: Assessment of BAM-PPV Field Test on C-5 Cargo Aircraft Rear Hatch Door p. 244
Table 132: Assessment of BAM-PPV Field Test on Headlight Cover of Bradley Vehicle p. 248
Table 133: Assessment of BAM-PPV Field Test on Bomb Hoists and Towbars p. 250
Table 134: Determination of Cost Drivers for EAP Field Testing p. 252
Table 135: Estimated Cost of Making BAM-PPV p. 252
Table 136: ^{1}H NMR (400 MHz, δ ppm, d_8-toluene) of BAM-PPV p. 265
Table 137: ^{13}C NMR (400 MHz, δ ppm, d_8-toluene) of BAM-PPV p. 266
Table 138: FTIR Peaks and Assignments for BAM-PPV Polymer Film p. 267
Table 139: ^{1}H NMR Analysis (400 MHz, δ ppm, $CDCl_3$) of Structure 1 p. 269
Table 140: ^{13}C NMR Analysis (400 MHz, δ ppm, $CDCl_3$) of Structure 1 p. 270
Table 141: ^{1}H NMR Analysis (400 MHz, δ ppm, $CDCl_3$) of Structure 2 p. 271
Table 142: ^{13}C NMR Analysis (400 MHz, δ ppm, $CDCl_3$) of Structure 2 p. 272

LIST OF ACRONYMS

AA	Aluminum alloy
AF	Air Force
AIMD	Aircraft Intermediate Maintenance Detachment
ARL	Army Research Laboratory
ASTM	American Standard Test Method
ATC	Aberdeen Test Center
ATR	Attenuated Total Reflectance
BAM-PPV	poly(2,5-bis(N-methyl-N-hexylamino)phenylene vinylene)
BCME	Bis(chloromethyl)ether
BIF	Blisters in field
CARC	Chemical Agent Resistant Coating
CCC	Chromate conversion coating
Cr(VI)	Hexavalent chromium
CRADA	Cooperative Research and Development Agreement
CTIO	Coatings Technology Integration Office
DI water	Deionized water
DMF	N,N-Dimethylformamide
DMSO	Dimethylsulfoxide
DOD	Department of Defense
DSC	Differential Scanning Calorimetry
EA	Elemental analysis
EAP	Electroactive Polymers
EB	Emeraldine base
EIS	Electrochemical impedance spectroscopy
ECR	Electrical contact resistance
ENM	Electrochemical Noise Methods
ES	Emeraldine salt
ESTCP	Environmental Security Technology Certification Program
EU	European Union
FEMA	Federal Emergency Management Agency
FRC	Fleet Readiness Center
FSE	Field Service Evaluation
FTMS	Federal Test Method Standard
FTIR	Fourier Transform Infrared Spectroscopy
GM9540P	General Motors Accelerated corrosion test
GPC	Gel permeation chromatography
HAPs	Hazardous Air Pollutants
HSS	High Strength Steel
HATE	Hydraulic Adhesion Test Equipment
HCl	Hydrochloric acid
HVLP	High Volume Low Pressure
JTP	Joint Test Protocol
JTR	Joint Technical Report
KCl	Potassium Chloride

K-*t*-OBu	Potassium–*tert*-butoxide
KSC	Kennedy Space Center
LANL	Los Alamos National Laboratory
LC_{50}	Lethal concentration for 50% of specimens
LD_{50}	Lethal dosage for 50% of specimens
MEH-PPV	poly[2(2-(2-ethylhexyloxy)-5-methoxy-*p*-phenylene vinylene]
MMAD	Mass Median Aerodynamic Diameter
Mp	Melting point
mΩ	Milliohms
NACE	National Association of Corrosion Engineers
NaCl	Sodium Chloride
NASA	National Aeronautics and Space Administration
NAS	Naval Air Station
NAWCAD	Naval Air Warfare Center Aircraft Division
NAWCWD	Naval Air Warfare Center Weapons Division
N/A	Not available
NIOSH	National Institute for Occupational Safety and Health
NSS	Neutral salt spray
NMR	Nuclear Magnetic Resonance spectroscopy
OEM	Original Equipment Manufacturer
Oxsol-100	4-chlorobenzotrifluoride or 1-chloro-4-(trifluoromethyl)benzene
ONR	Office of Naval Research
PA	Polyacetylene
PANI	Polyaniline
PATTI	Pneumatic adhesion tensile test instrument
%	Percent
PPV	Poly(*para*-phenylene vinylene)
PPy	Poly(pyrrole)
PT	Poly(thiophene)
rpm	revolutions per minute
RH	Relative humidity
rt	room temperature
REACH	Registration Evaluation Authorization and Restriction of Chemicals
RXSSO	Logistics Systems Support Branch Office
SAR	Search and Rescue
SEM	Scanning electron microscopy
SERDP	Strategic Environmental Research and Development Program
SBIR	Small Business Innovative Program
SSPC	Society for Protective Coatings
SVET	Scanning Vibrating Electrode Technique
SVHC	Substance of Very High Concern
SO_2	Sulfur dioxide corrosion test

TAD	Temporary Active Duty
TCP	Trivalent Chromium Pretreatment
THF	Tetrahydrofuran
TGA	Thermogravimetric analysis
TLC	Thin layer chromatography
UDP	Up and Don Procedure
UV	Ultra-violet
UDRI	University of Dayton Research Institute
VOC	Volatile Organic compounds
WPAFB	Wright Patterson Air Force Base
WTA	Wet Tape Adhesion
wt%	Weight percent
XPS	X-ray photoelectron spectroscopy

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Dr. Peter Zarras directed the overall project, synthesized polymer and coated coupons with poly(2,5-bis(N-methyl-N-hexylamino)phenylene vinylene) (BAM-PPV) solution. Ms. Cindy Webber and Ms. Nicole Anderson developed the test matrix for BAM-PPV and aided in the overall implementation of the test matrix for each of the services. Dr. Mathew Davis synthesized monomers for the polymerization of poly[2(2-(2-ethylhexyloxy)-5-methoxy-*p*-phenylene vinylene] (MEH-PPV) via an improved synthetic strategy. Ms. Cindy Webber and Ms. Nicole Anderson polymerized both BAM-PPV and MEH-PPV compounds as well as coating aluminum and steel coupons with each polymer.

Ms. Diane Buhrmaster managed the laboratory, outdoor exposure testing of BAM-PPV and MEH-PPV coated coupons at WPAFB. In addition Ms. Diane Buhrmaster coordinated field tests of BAM-PPV on the C-5 Cargo aircraft. These field tests lasted one-year with an overseas flight. Ms. Amy Fowler in cooperation with Mr. Andrew Schwartz tested BAM-PPV and MEH-PPV coated coupons at NAWCAD using adhesion and accelerated weathering tests. Mr. Christopher Miller at the ARL managed the adhesion and accelerated weathering testing on BAM-PPV coated coupons and field tested BAM-PPV on non-critical military hardware for one year. Mr. Christopher Mahendra coordinated the one-year field tests of BAM-PPV pretreatment on non-critical military hardware. Dr. Mark Kolody from NASA-KSC performed marine outdoor exposure testing of BAM-PPV. MB Research conducted all toxicology testing of BAM-PPV.

EXECUTIVE SUMMARY

Chromate conversion coatings (CCC) are applied via immersion or spraying onto both aluminum and steel substrates. These coatings provide both corrosion inhibition and adhesion promotion between the primer and the pretreatment. Several recent studies have shown that residual hexavalent chromium (Cr(VI)) in CCCs provides corrosion protection via a self-healing mechanism. However, Cr(VI) is a known carcinogen and is highly regulated by the Environmental Protection Agency (EPA) and Occupational Safety and Health Agency (OSHA).

A viable alternative to Cr(VI) coatings must meet or exceed the performance of Cr(VI). In addition these alternative coatings must be able to passivate the metal surface allowing the corrosion current to shift to the noble metal region. Several studies over the past decade have focused on electroactive polymers (EAPs) as corrosion preventive coating. These studies have focused almost exclusively on polyaniline (PANI) and have confirmed a passivation mechanism for corrosion inhibition. More recent studies have focused on PANI as a replacement for chromated primers. Several studies using polythiophenes and polypyrroles have been used to coat both ferrous and nonferrous substrates. Electrochemical studies were performed on these EAPs as primer replacements. Though, very little work has been done on alternative pretreatments using EAPs. Scientists at the NAWCWD have successfully synthesized a *para*-phenylene vinylene (PPV) derivative that can perform as well as CCC. This polymer can meet the military pretreatment requirements for alternatives to CCC. This compound poly(2,5-bis(N-methyl-N-hexylamino)phenylene vinylene, BAM-PPV has shown corrosion prevention in simulated seawater. Its corrosion prevention properties were also evaluated using accelerated weathering conditions to determine its effectiveness as an alternative pretreatment to CCC. These tests under a Strategic Environmental Research and Development Program (SERDP) Pollution Prevention (WP1148) from FY00-04 showed conclusive evidence that BAM-PPV was a promising pretreatment coating as an alternative to CCC's.

A four-year Environmental Security Technology Certification Program (ESTCP) (WP-200527) FY05-08 was undertaken to prove the viability of this new compound and to test other alternative pretreatment coatings as controls. Each service, WPAFB, ARL and NAWCWD and NAWCAD tested BAM-PPV as a pretreatment coating along with poly[2(2-(2-ethylhexyloxy)-5-methoxy-*p*-phenylene vinylene] (MEH-PPV), Trivalent chromium pretreatment (TCP) and PreKote. **Each service tested these pretreatment coatings according to their military coating specification as alternatives to CCC.**

Each service had specific testing done in order to evaluate the performance of the EAP pretreatment coating. All co-performers (WPAFB, ARL and NAWCAD) tested EAP pretreatment coatings according to their specific laboratory requirements prior to any field tests. After each laboratory tested the EAP pretreatment coating and it passed their performance requirements; the EAP (BAM-PPV or MEH-PPV) was then selected for field testing.

Therefore, it is beneficial to the reader to see that the testing performed by each organization is presented in specific sections in this report attributed to that organization. The table of contents lists the specific testing done by each organization with final summations to provide the reader with take-away bullets

describing the successes and failures of the EAP pretreatments with and without full military coatings.

The results are reported by each facility. The laboratory tests were conducted with and without full military coatings containing Cr(VI) or non-chromium coatings in accelerated weathering chambers, adhesion testing, fluid resistance and outdoor exposure testing. Table 1 provides the best/worst full military coating systems in neutral salt spray (NSS) testing chamber and adhesion tests.

Table 1: Best/Worst Performing BAM-PPV Pretreatment in Full Military Coating Systems

Substrate	Pretreatment	Primer[a]	Topcoat[b]	NSS[c]	Adhesion[d,e]
AA2024-T3	BAM-PPV	MIL-PRF-23377N	MIL-PRF-85285	2,000 hours (pass, scribed)	4B[b] (pass)
AA7075-T6	BAM-PPV	MIL-PRF-23377N	MIL-PRF-85285	2,000 hours (pass, scribed)	4B[b] (pass)
AA6061-T6	BAM-PPV	MIL-DTL-53022	MIL-DTL-53039	2,016 hours (pass, no-scribe)	1,185 psi[c] (adhesive failure)
AA5083	BAM-PPV	MIL-DTL-53030	MIL-DTL-53039	2,016 hours (pass, no-scribe)	824 psi[c] (adhesive failure)
AA2024-T3	BAM-PPV	MIL-PRF-23377N	MIL-PRF-85285	1,500 hours (fail, scribed)	3A (marginal)
AA7075-T6	BAM-PPV	MIL-PRF-23377N	MIL-PRF-85285	1,500 hours (fail, scribed)	3A (marginal)
4130 steel	BAM-PPV	MIL-PRF-23377N	MIL-PRF-85285	124 hours (fail, scribed)	1,853psi[c] (adhesive/cohesive)
4130 steel	BAM-PPV	MIL-DTL-53022	MIL-PRF-85285	X	1A (fail)

a: Non-chromated primers
b: Aircraft polyurethane topcoat (MIL-PRF-85285) and Army solvent-borne topcoat (MIL-PRF-53039)
c: NSS = Neutral salt spray testing (ASTM B117)
d: Adhesion testing (ASTM D 3359)
e: PATTI adhesion (pneumatic adhesion tensile test instrument, ASTM D 4541)

After extensive laboratory/outdoor exposure testing of these CCC alternatives, a candidate was selected for field testing by the three services. BAM-PPV and MEH-PPV were compared to CCC, TCP and Prekote performance in accelerated weathering chambers, fluid resistance, adhesion and outdoor exposure testing. A consensus was reached by the three services that BAM-PPV was a more robust pretreatment coating than MEH-PPV. These findings validate the performance of BAM-PPV as an alternative to CCC. The performance of BAM-PPV coated with chromated primers and topcoats exceeded the military requirements of 2,000 hours neutral salt spray exposure without evidence of corrosion, blistering or delamination. However, when BAM-PPV was coated with non-chromium primers, and topcoats, the performance in most cases was marginal or failure was evident, though, several non-chromium primers with BAM-PV as the

pretreatment and topcoats did provide adequate corrosion protection (see Table 1). These samples met the minimum requirement of 2,000 hours neutral salt spray exposure. BAM-PPV along with the best performing primers (Cr(VI) and non-chromium) with topcoats were selected for field testing by each service and field tested. The field tests were monitored for one-year by visual inspection.

Toxicology studies have also shown that BAM-PPV is a non-toxic material. There were no deaths or injuries to experimental laboratory animals administered doses of BAM-PPV for lethality, dermal and ocular sensitization.

Furthermore, outdoor exposure testing was conducted at WPAFB and NASA-KSC. The outdoor exposure testing performed by WPAFB using BAM-PPV as the pretreatment with primers (Cr(VI) and non-Cr(VI)) and topcoat showed comparable performance to a fully chromated system for 24 months of exposure. Similar testing at the NASA-KSC showed that BAM-PPV after 4-6 months of marine outdoor exposure could not match a fully chromated control system.

Each service conducted field testing on non-critical military hardware using BAM-PPV as the pretreatment coating with a primer and topcoat specific for that service. The Air Force (AF) and Army coatings incorporated BAM-PPV as the pretreatment coating in full military coatings. After one-year of field testing, BAM-PPV pretreatment coating with primer and topcoat showed similar performance to a fully chromated system. However, testing by the Navy on support equipment showed that BAM-PPV as the pretreatment coating did not give any improved corrosion or abrasion resistance as compared to the control. This may be due to improper surface preparation of the aluminum alloy prior to coating with BAM-PPV pretreatment.

Summation of field tests conducted by AF, Army and Navy:

- **AF field test on C-5 cargo plane aluminum rear hatch door using BAM-PPV as pretreatment with full military coating (non-chromium primer + topcoat) survived one-year field test with no visual evidence of corrosion, delamination or loss of protective properties.**

- **Army field test on aluminum flag holder for the Bradley vehicle using BAM-PPV as pretreatment with full military coating (non-chromium primer + topcoat) survived one-year field test with minor abrasion, minor loss of topcoat coating (BAM-PPV pretreatment coating intact) without any visual evidence of corrosion.**

- **Navy field test on aluminum support equipment using BAM-PPV as pretreatment with full military coating (non-chromium primer + topcoat) failed at six months of field tests, extensive delamination, cracking and loss of coating was evident.**

1.0 INTRODUCTION

1.1 Background

In order to adequately describe the new technology proposed and eventually demonstrated during this 4-year ESTCP program, a fundamental review of the specific polymers used during this program is warranted. The proposed technology employs electroactive polymers (EAPs), also referred to as conductive polymers (CPs), (both terms are used interchangeably) as alternatives to CCC pretreatment coatings. EAPs represent a new class of polymer materials referred to as "synthetic metals" that can be applied as coatings onto various metal substrates to provide corrosion protection. The historical background, various types of EAPs, corrosion history and current applications will be presented to help the reader get a fuller appreciation and understanding of this new technology.

Polymers were orignally thought of as electrical insulators, not electrical conductors. However, in the early 1960s Pohl, Katon and others synthesized and characterized polymers (some conjugated) with conductivities in the semiconductor range [1, 2]. The discovery that iodine-doped polyacetylene exhibited electrical conductivity many orders of magnitude higher than neutral polyacetylene shattered the belief that polymers were poor conductors. This discovery was made in 1976 by Hideki Shirakawa, Alan MacDiarmid and Alan Heeger. They received the 2000 Nobel Prize in Chemistry for their work [3]. The discovery of electrically conducting polymers triggered the development of a new multidisciplinary field known as "synthetic metals" [4].

Electroactive polymers (EAPs) are composed of conjugated chains containing π-electrons delocalized along the polymer backbone. In their neutral form, EAPs are semiconductive polymers that can be doped and converted into electrically conductive forms. The doping process can occur either by oxidative or reductive reactions, though oxidative reactions are more common. The conductivity is electronic in nature and does not involve concurrent ion migration in the solid polymer form. Some doping processes are reversible, with typical conductivities switching between those of insulators ($<10^{-10}$ S/ cm) to those of metals (10^{5} S/cm) [5].

EAPs comprise a broad range of materials which are characterized by conjugated repeat units and this conjugation is responsible for the unique electronic and optical properties of EAPs ranging from low oxidation potential to third order optical nonlinear properties. Researchers have exploited these unique materials to synthesize a variety of EAPs. These polymers exhibit a broad range of conductivities (10^{-4} to 10^{3} S/cm) in their doped (oxidized) states. There are several classes of EAPs: polyacetylenes, poly(*p*-phenylene)s, polyheterocycles, poly(phenylene vinylene)s, polyanilines and conjugated ladder polymers (Table 2).

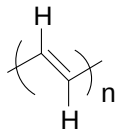
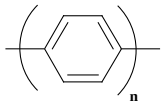
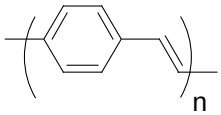
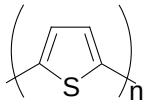
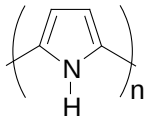
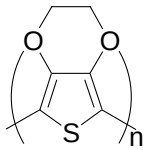
EAPs can be synthesized both chemically and electrochemically. There are three conducting states of EAPs:

- neutral (uncharged), where EAPs are insulators or semiconductors;
- oxidized (p-doped) where electrons are removed from the backbone;

- and the reduced (n-doped) (least common), where electrons are added to the backbone of the neutral polymer.

The ability to control the level of conductivity in polymers has given researchers the tools to tailor properties to specific needs and devices. Therefore, EAPs have potential applications in such diverse areas as actuators [6], supercapacitors/batteries [7], molecular electronics [8], electrochromic windows/displays [9], transistors [10], photovoltaics [11], bio- or chemical sensors [12] and corrosion protection [13].

Table 2: Structures of EAPs and Conductivities

Polymer	Structure	Conductivity [Doped form] (S/cm)
Polyacetylene (PA)		10^3-10^5
Poly(*para*-phenylene) (PPP)		10^2-10^3
Poly(*para*-phenylene vinylene) (PPV)		10^3-10^4
Poly(thiophene) (PT)		10^2
Poly(pyrrole) PPy		10^2-10^3
Poly(3,4-ethylenedioxythiophene) PEDOT		10^2

Corrosion protection using EAPs was first suggested by MacDiarmid in 1985 [14]. It has been observed that most EAPs can be electrochemically produced by anodic oxidation, enabling one to obtain a conducting film directly on a surface. This fact has led researchers into the field of anti-corrosives. EAPs can go from the insulating to the conducting state through several doping techniques such as (a) chemical doping by

charge transfer, (b) electrochemical doping, (c) doping of PANI by acid-base chemistry, (d) photodoping and (e) charge injection at a metal-semiconducting polymer interface. Depending on the doping technique used, this will have a significant influence as to the potential application of the EAP.

1.1.1 Synthesis of EAPs

In order to understand the chemistry of EAPs used in corrosion protection, it is necessary to start with the synthesis of EAPs. There are currently several classes of EAPs which can exhibit conductivities (in the doped state) ranging from those of semiconductors to those of metals. These EAPs comprise the polyacetylenes (PA), poly(*para*-phenylenes) (PPP), polyheterocycles such as polythiophenes (PT), poly(phenylenes vinylenes) (PPV), polyanilines (PANI) and conjugated ladder polymers. Of these, the polyheterocycles, PPVs and PANIs have been studied for corrosion protection [15]. The PPPs, PAs and ladder polymers have stability issues under ambient conditions or reduced processability which have limited their use.

The most widely studied of the EAPs for corrosion protection has has been PANI. PANI has several advantages over most current EAPs, including easy chemical and electrochemical polymerization of monomer. Doping and de-doping proceeds easily by treatment with standard aqueous acid and base and it is highly resistant to environmental degradation [16]. PANI is commonly prepared by polymerization of the aniline monomer using $(NH_4)_2S_2O_8$ in hydrogen chloride solution [17]. The polymer can also be prepared by electropolymerization of the monomer [18]. PANI is a difficult material to process under normal conditions. Several methods now exist for improving the processability and solubility of PANI. These methods include modification of the polymer backbone by introducing various functional groups such as alkoxy, amino, alkyl, aryl and sulfonyl, or by using novel acids such as camphor sulfonic acid to obtain a soluble form of the conducting version of PANI [19]. The PANI that is produced from both chemical and electrochemical processes can exist in four different oxidation states: leucoemeraldine (fully reduced state); pernigraniline (fully oxidized form), emeraldine base (intermediate form) and emeraldine salt (conductive form) (Figure 1) [20].

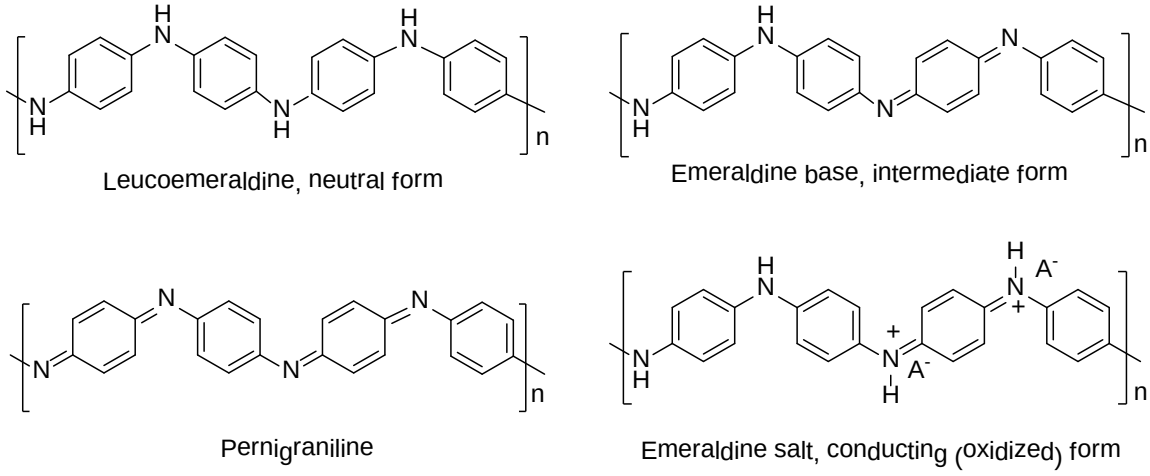


Figure 1: Four Forms of PANI

PANI is prepared as the emeraldine salt (ES) from both chemical and electrochemical polymerization techniques and then treated with base to give the emeraldine base (EB). This form (EB) is soluble in common organic solvents such as N, N-dimethylformamide, N,N-dimethylacetamide and 1-methyl-2-pyrrolidinone. Upon film formation or other casting technique, the emeraldine base can then be treated with acids (sometimes called protonic acid doping) to regenerate the conductive form, the emeraldine salt (Figure 2). The process to increase the electrical conductivity of PANI by protonation and decrease by deprotonation is sometimes referred to as compensation.

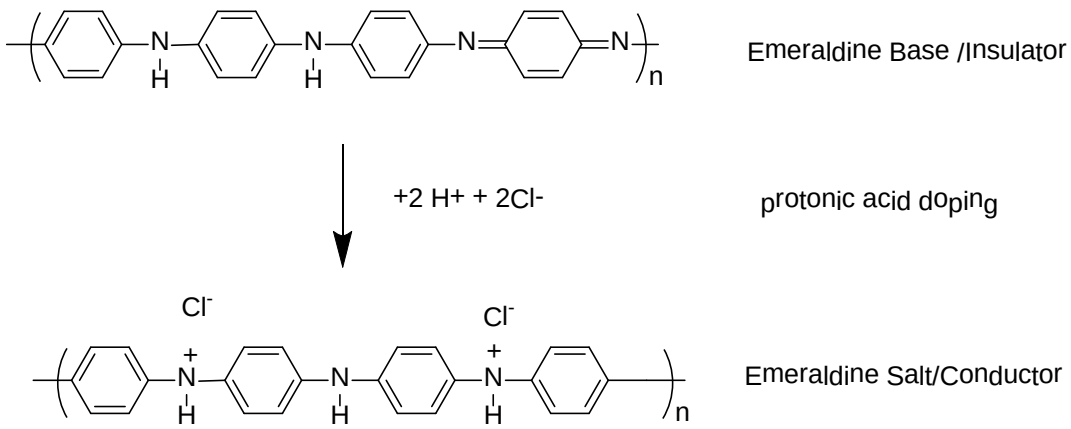


Figure 2: Protonic Acid Doping

The heterocyclic monomers represented in Figure 3 are a class of monomers that can be polymerized to form fully conjugated polymers [21]. The most common of these is pyrrole (X=NH), which has been recently investigated for corrosion protection.

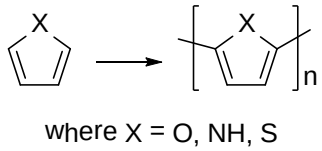


Figure 3: Heterocyclic Structure

The thiophenes (X=S) have also been studied extensively for their electronic, electro-optical and corrosion properties; whereas the furans (X=O) have not been studied as well due to their high oxidation potential (>1.7V vs. Ag/Ag+), which results in very poor quality materials. Polypyrrole (PPy) and polythiophene (PT) can be doped to give moderate to high conductivities. They have been polymerized using both chemical and electrochemical methods. The resulting polymers are electron-rich materials that are easily oxidized and are, therefore, stable in the oxidized form. Polypyrrole and polythiophene have received considerable attention due to their high stabilities and ease

of structural modification for improved processability. Both PPy and PT have been investigated for their corrosion properties [22].

Polypyrrole was prepared by the chemical oxidation of pyrrole using hydrogen peroxide and called "pyrrole black." Unmodified PPy and PT are both insoluble in common organic solvents and unprocessable. Chemical polymerization remains the simplest route, but electrochemical polymerization is the most important and versatile method, producing high quality films and high conductivities (10^2 S/cm). The co-polymerization of thiophenes includes chemical oxidative couplings of thiophenes, cross-coupling of Grignard reagents of dihalothiophenes, and electrochemical polymerization [23, 24]. PTs obtained from these methods are infusible and unprocessable. In order to improve solubility and processability of PTs, synthetic methods were developed to incorporate functional groups such as n-alkyl groups on the 3-position to improve processability without sacrificing electrical conductivity. The synthesis of poly(3-alkylthiophene)s (P3ATs) has been accomplished by Kumada cross-coupling [22], and oxidative polymerization [25]. Regiospecific synthetic techniques have been developed to obtain regioregular P3ATs [26].

Poly(phenylene vinylene)s (PPV) was first synthesized in 1960 by McDonald and Campbell [27]. Efforts at improving the solubility and processability of PPVs has lead to the development of several precursor routes. The most widely used precursor route is that developed by Wessling and Zimmerman (Figure 4) [28]. This process involves the synthesis of the bis-sulfonium salt of 1,4-bis(chloromethyl)benzene, followed by sodium hydroxide elimination and polymerization at low temperature to give an aqueous solution of a precursor polymer. The soluble precursor polymer can be processed into films, foams or fibers and converted to PPV by thermal elimination. There are several alternative approaches to Wessling and Zimmerman's sulfonium precursor methods, including the Gilch route [29] and Heck polymerization [30]. These alternative routes are similar in utility to the Wessling and Zimmerman route. As with PANI and polyheterocycles, PPVs have also been modified to improve solubility and processability [21].

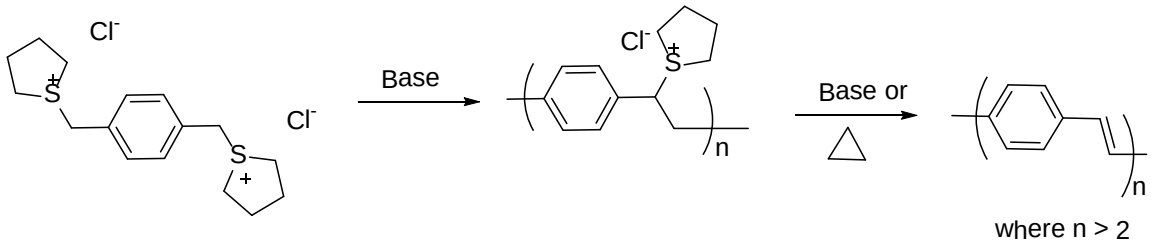


Figure 4: PPV Synthesis

1.1.2 Corrosion Processes

Along with a fundamental knowledge of the different types of EAPs, their synthesis and doping processes, an understanding of the corrosion process affecting metals is needed. Corrosion is the destructive result of chemical reactions that occur between a metal or a metal alloy and its environment [31, 32]. Corrosion impacts many aspects of our daily lives, and various estimates put the costs to the US economy between 100-300

billion dollars annually. The most striking features of the corrosion process are the immense variety of conditions under which it occurs and the large number of forms in which it appears [33]. Corrosion affects all structural materials and infrastructure of society to cause in many cases grave economic consequences or life-threatening situations. Infrastructure items that can be significantly damaged and eventually destroyed by corrosion include pipelines, bridges, automobiles, storage tanks, airplanes and ships (both military and commercial). The most common environments for corrosion to occur are in natural waters, atmospheric moisture, rain and man-made solutions (such as storage tanks) [34]. The ionic conductivity of an aqueous corrosive environment creates electrochemical reactions [35]. These reactions are strongly influenced by the surface potential and acidity or basicity of the environment which allows electrochemical reactions to take place on a metal surface. This changes the metal from the metallic state to a non-metallic state. The products of corrosion can be dissolved species or solid corrosion products. No matter what the scenario, the energy of the system is lowered as the metal converts to a lower-energy form. The most studied example of this phenomenon is the rusting of iron. The metal (iron) is converted into the corrosion product, the non-metallic form (rust or iron oxides). The difficulty in preventing corrosion from occurring is both a scientific and engineering problem. Corrosion science is the study of the electrochemical processes and metallurgical processes that occur during corrosion in various environments. Corrosion engineering, on the other hand, is the design of methods and materials to prevent corrosion. An understanding of both disciplines is essential to prevent corrosion from affecting infrastructure. There are generally several forms of corrosion that affect infrastructure. These corrosion forms are classified as (i) uniform or general corrosion, (ii) galvanic corrosion, (iii) pitting corrosion, (iv) environmentally-induced cracking, (v) hydrogen damage, (vi) intergranular corrosion, (vii) dealloying and (viii) erosion corrosion [36]. There are several corrosion-preventing barriers: anodic oxides, organic coatings, ceramics, inorganic coatings, phosphate and other conversion coatings [37]. These coatings provide a barrier that resists penetration by aggressive environmental constituents. The goal of these coatings is to prevent the cathodic reaction from taking place beneath the coating (Equation 1).

Equation 1 $$2\,H_2O + O_2 + 4e^- \rightarrow 4OH^-$$

These coatings work well, but over time the barrier coating can fail due to prolonged exposure to the environment. The organic coatings can develop what is called "under coating corrosion" which initiates from weak spots and develops into blisters and filiform threads leading to corrosion failure [38]. The organic protective coating (barrier coating) fails by separation at the coating/substrate interface [39]. The separation process is known as *delamination*. In time, delamination results in under film corrosion and eventual loss of the barrier properties of the coating. The corrosion-induced delamination occurs as a direct consequence of the electrochemical mechanism of corrosion [35]. The anodic (electron generating) and the cathodic (electron-consuming) reactions occur at the sites where delamination has occurred. The kinetic barriers are, at a minimum, due to the high electrical conductivity of the metal. This provides an easy pathway for electrons

between the anodic and cathodic sites. Metal pretreatments have been used to extend the lifetimes of barrier coatings. These pretreatments are normally composed of phosphates, chromates and oxides, which contain a variety of metal cations. These processes are well established industrially and prolong the lifetimes of barrier coatings through better adhesion of the coatings onto the metal substrate. The pretreatments and primers that are used for corrosion protection-specifically chromate conversion coatings (CCC) and chromate containing primers-have come under increased scrutiny and regulation from governmental agencies. The hexavalent chromium used in CCC and primers is a known human carcinogen [40] and is highly regulated [41]. Alternative approaches are needed to replace barrier coatings that give limited lifetimes for corrosion protection. Ideally, the material should not only provide the benefits of barrier coatings but also passivate the metal, allowing the corrosion current to shift to the noble metal region.

1.1.3 Corrosion Protection with EAPs

Over the past 20 years, published evidence that an EAP, specifically polyaniline, could inhibit corrosion has come from Mengoli [42], DeBerry [43], and Ahmand and MacDiarmid [44]. These results showed that PANI electrodeposited on passivated steel in a strong acid environment enhanced corrosion protection of the metal [15]. The most extensively studied of the EAPs for corrosion prevention has been PANI [45]. At the Los Alamos National Laboratory (LANL) and the John F. Kennedy Space Center (KSC), researchers demonstrated that doped PANI coatings inhibited corrosion of carbon steel [46]. Their approach was based on earlier work suggesting that the interfacial contact between the metal and a doped EAP would generate an electric field that would restrict the flow of electrons from the metal to an outside oxidizing species, thus preventing and/or reducing corrosion [47]. The LANL-KSC tests were conducted in a 3.5wt % NaCl/0.1 M HCl environment using ca. 0.005 cm thick films of PANI doped with *p*-toluenesulfonic acid on carbon steel. The PANI was covered with an epoxy topcoat. The PANI/epoxy coating performed significantly better than the epoxy topcoat alone. These initial results were used by researchers at LANL-KSC to develop EAP coatings to resist the corrosive effects of acid vapor generated during space shuttle launches. The ground support equipment and structures at the KSC were susceptible to the severe environmental conditions. The environment around the launch site consists of marine, severe solar, and intermittant high levels of acid vapors (hydrochloric acid), as well as elevated temperatures.

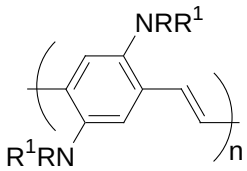
There have been numerous reports in the literature regarding the use of EAPs in retarding corrosion on steel alloys [48]. Elsenbaumer showed that PANI coated mild steel samples exposed to artificial brine and dilute HCl exhibited several times more corrosion protection than an epoxy barrier paint when scratched to expose precise areas of bare metal [49]. Their results showed that the enhanced corrosion protection was a result of a passivating iron oxide layer formed even for the exposed bare steel metal. Wessling has demonstrated through several experimental techniques (including scanning electrn microscopy (SEM) and X-ray photoelectron spectroscoy (XPS)) that dispersed PANI (CORRPASSIV^{TM}) containing paints can induce the passivation effect as a corrosion prevention primer [50]. Wessling proposed a mechanism to explain the corrosion protection of EAPs in which the protection of steel by PANI is attributed to the formation

of a passive layer of metal oxide. Benicewicz's group has also investigated polymers containing oligoaniline side groups that showed high electroactivity similar to PANI [51].

There is still debate over whether the emeraldine salt (ES) or emeraldine base (EB) form of PANI provides the best corrosion protection [52]. Several reports have shown that the EB form of PANI can perform as well as, or better than the ES form of PANI. Epstein et al showed that the EB form of PANI coated onto cold rolled steel and iron samples inhibited corrosion when exposed to humidity chambers from one to seven days [52]. XPS depth profiling showed anodic protection for their samples. In addition to the polyaniline, oligomers of aniline have been used to retard against corrosion and to provide better solubilty for casting films onto metal substrates [53]. Wei et.al. have demonstrated that aniline oligomers, particulary the trimers, are effective anti-corrosion materials [54]. While there is general agreement that PANI performs well in retarding corrosion on carbon steel, the mechanism for this process is still under investigation. There have been several techniques used both to test EAP coatings for corrosion resistance and to evaluate the mechanism. The normal evaluation of such coatings is accelerated weathering tests in a laboratory setting to examine the likely performance of a coating. These tests mimic outdoor exposure to determine the effects of acid, neutral and/or alkaline spray (ASTM B117), sulfur dioxide spray and ultra-violet light exposure. The tests compare the physical appearance of the coating to a known standard coating. Additional tests used to evaluate the corrosion inhibiting properties of a coating is electrochemical impedance spectroscopy (EIS) [55]. This ac technique measures adhesion and barrier properties of coatings and can give mechanistic data that traditional dc techniques cannot provide. Several hypotheses are suggested for the mechanism of corrosion protection using EAPs, specifically PANI: (1) PANI contributes to the formation of an electric field at the metal surface, restricting the flow of electrons from metal to oxidant, (2) PANI forms a dense, strong adherent, low-porosity film similar to a barrier coating and (3) PANI causes the formation of protective layers of metal oxides on a metal surface [15]. While PANI has been extensively studied for corrosion control, its major drawback is its pH dependence. It works very well in acidic environments due to the formation of the conductive ES form, but at higher pH (>7), the non-conductive EB is formed (see Figure 1), and the material no longer provides adequate protection against corrosion. Thus, its use in a marine environment (pH ~8) is limited. In marine applications, pH-stable EAPs must be employed. Several have been investigated. Double-stranded PANI has been coated onto AA 2024-T3 Al alloys and immersion tests in simulated seawater showed improved corrosion resistance as compared to control epoxy coating [56].

Recently, an amino PPV derivative, poly(2,5-bis(N-methyl-N-hexylmethylamino) phenylene vinylene) (BAM-PPV) (Figure 5), was shown to adhere to AA2024-T3 in an immersion test using simulated seawater (pH ~8) and to retard corrosion [57-63]. Constant current (galvanostatic) and constant potential (potentiostatic) method were used to investigate the corrosion protection of BAM-PPV on aluminum alloy plates. Quantitative evidence was obtained from these results to show corrosion inhibition was obtained without the loss of adhesion and no pH dependency was observed. These tests were run against control samples (non-coated Al alloys) and the coated coupons out performed the non-coated coupons. Visual inspection of the coupons showed significant pitting in the non-coated as compared to the coated (BAM-PPV) samples. Further study

showed that BAM-PPV could adhere onto AA2024-T3 substrates and meet the minimum neutral salt fog exposure requirement of 336 hours as an alternative to CCC. The BAM-PPV coated AA2024-T3 coupon (unscribed) was able to meet the 336 hours of exposure to neutral salt spray (NSS) exposure without evidence of blistering, delamination of the coating, discoloration or corrosion (Figure 6) [64,65].



where:

R= methyl and R^1=hexyl

Figure 5: BAM-PPV Structure

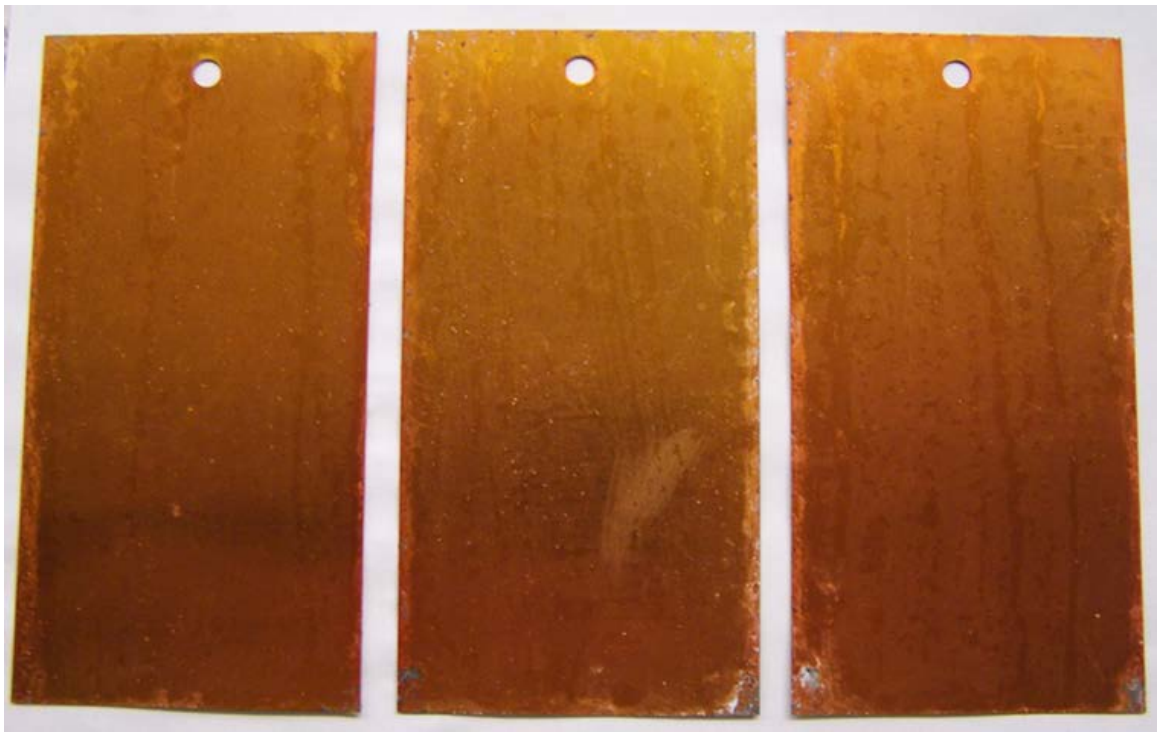


Figure 6: BAM-PPV on AA2024-T3 Panels at 336 hours
Neutral Salt Spray (NSS) Exposure

Several additional EAPs have been studied for their corrosion protective properties. Polypyrrole (PPy) has recently been examined for its anti-corrosion properties [66]. PPy normally has poor solubility and mechanical properties, preventing its use in corrosion studies. Recent advances in improving its solubility and mechanical properties have been accomplished by adding functional groups to the pyrrole ring [67]. Corrosion

studies based on poly(3-octyl pyrrole) [68] have shown that these polymers can inhibit and delay the onset of corrosion for AA 2024-T3. Poly (3-methylthiophene) was electropolymerized onto 430 stainless steel, and showed corrosion protection in both air and nitrogen saturated 1N sulfuric acid solutions [69]. Polythiophene has been electrodeposited onto mild steel surfaces to provide corrosion protection in 3.5 % aqueous sodium chloride solution as determined by polarization and EIS analysis. Enhanced corrosion protection was found with a polyaniline-layered montmorillonite (clay) nanocomposite system as compared to polyaniline [70]. The experiments were carried out in 5 wt% NaCl and showed better performance than conventional PANI by electrochemical measurements. Lignosulfonic acid-doped polyaniline (Ligno-PANI) has been investigated for its corrosion properties [71]. Ligno-PANI has shown improved processability as compared to conventional PANI. It is water dispersible and soluble in organic solvents such as DMSO and THF. The improved processability and reduced cost for this material makes it highly attractive for corrosion protection. Corrosion prevention has been demonstrated for Ligno-PANI and is found to be significant.

1.2 Objective(s) of the Demonstration

The objective(s) of this completed demonstration project were as follows:

- **conduct laboratory tests by co-performers to validate EAPs (BAM-PPV and/or MEH-PPV) pretreatment coating corrosion performance as compared to CCCs, Trivalent chromium pretreatment (TCP) and Prekote as a pretreatment coating only and in full military coating (primer + topcoat),**
- **demonstrate an effective, environmentally benign, repairable coating using EAPs (BAM-PPV and/or MEH-PPV) as a replacement for CCCs,**
- **validate performance with non-chromated primers and topcoats on aluminum alloys against known ASTM testing standards,**
- **validate performance on steel with non-chromated primers and topcoats against known ASTM testing standards,**
- **perform outdoor and marine outdoor exposure testing on BAM-PPVand MEH-PPV in full military coatings on aluminum and steel coupons for one-year exposure,**
- **determine toxicity of BAM-PPV and/or MEH-PPV using accredited toxicology laboratory,**
- **down-select best performing EAP (BAM-PPV or MEH-PPV or both) pretreatment coating with non-chromium primer and topcoat for one-year field testing,**
- **demonstrate via field tests using BAM-PPV and/or MEH-PPV pretreatment coatings on non-critical military hardware showing equal to or superior performance as compared to CCC pretreatment full military coatings.**

The Occupational Safety and Health Agency (OSHA) has published a final standard for occupational exposure to Cr(VI) in the February 28, 2006, Federal Register. The standard covers occupational exposure to Cr(VI) due to the known carcinogenic

nature of this compound and it is highly regulated by the Environmental Protection Agency (EPA) and OSHA [40, 41]. OSHA has determined based upon the best scientific evidence, that at the current permissible exposure limit (PEL) for Cr(VI), workers face a significant risk to their health. Therefore, a final rule establishes a permissible exposure limit (PEL) of 25 micrograms of Cr(VI) per cubic meter of air for aerospace painting (25 $\mu g/m^3$) as an 8-hour time weighted average (TWA) for all Cr(VI) compounds in the paint hangar. This ruling was based on extensive consideration of all comments and evidence submitted during this process. However, the PEL for the maintenance worker is still $5\mu g/m^3$. This standard must be met during painting by using protective personnel equipment (PPE).

1.3 Regulatory Drivers

As a result of the new Cr(VI) OSHA regulations, these new standards will now cover occupational exposure to Cr(VI) for workers during an 8-hour TWA period. In order for the DOD to comply with these regulations there will be an increase in disposal costs for Cr(VI) waste paint as a hazardous material. The costs associated for industry/government to comply with the new federal Cr(VI) PEL are summarized in Table 3. The estimates shown below are considered general costs in complying with the 5 $\mu g/m^3$ requirement. Each of the affected weapons systems field-tested during this ESTCP demonstration program will have to implement this new guideline. Current alternative Cr(VI)-free primers have been developed by each service and the EAP coating under this ESTCP study does not contain Cr(VI). This allows for easy compliance with the new military Cr(VI)-free coating, whereby EAPs are not considered hazardous materials. Therefore, the DOD would not be required to comply with expensive disposal costs.

As of January 24, 2013, the National Institute for Occupational Safety and Health (NIOSH) had posted a document entitled "Criteria for a Recommended Standard: Occupational Exposure to Hexavalent Chromium." NIOSH reviewed the critical health effect studies of hexavalent chromium compounds and updated its assessment of the potential health effects of occupational exposure to Cr(VI) compounds. The recommendation for the new PEL is 0.20 $\mu g/m^3$ for a workplace environment [41]. This recommendation by NIOSH was advisory only; NIOSH cannot issue a regulatory directive.

The European Union (EU) Registration, Evaluation, Authorization and Restriction of Chemicals (REACH) regulations (EC 1907/2006) were adopted in December 2006, and came into force June 2007. REACH was introduced because many thousands of chemicals are used in the EU, some in very large quantities and with increased risks to human health and to the environment. Cr(VI) is one of the many substances that are highly regulated by the EU and is currently a substance of very high concern (SVHC).

Table 3: Estimated Compliance Costs for General Industry with New Cr(VI) Guidelines

Cost Category	10 μg/m^3	5 μg/m^3	1 μg/m^3	0.5 μg/m^3	0.25 μg/m^3
Engineering Controls	$10,652,864	$14,475,735	$26,474,262	$52,467,526	$82,207,372
Initial Exposure Assessment	$15,250,335	$15,250,335	$15,250,335	$15,250,335	$15,250,335
Periodic Monitoring	$24,605,517	$30,034,467	$60,305,070	$126,687,514	$161,729,092
Respirators	$29,448,797	$29,448,798	$35,361,768	$40,773,063	$56,907,196
Medical Surveillance	$21,211,591	$13,230,302	$67,359,536	$62,126,618	$79,073,671
Communication of Hazards	$21,211,591	$21,193,263	$21,881,436	$21,889,546	$21,892,346

2.0 DEMONSTRATION TECHNOLOGY

2.1 Technology Description

As was extensively described in the Introduction Section 1.1, published evidence has shown that an EAP, such as polyaniline (PANI) could inhibit corrosion. These early investigations confirming the corrosion protection of EAPS on steel substrates were published by Mengoli [42], DeBerry [43] and Ahmand and MacDiarmid [14, 44]. Their results showed that PANI electrodeposited on passivated steel in a strong acid environment enhanced the corrosion protection of the metal [15, 45]. PANI has been the most extensively studied of the EAPs for corrosion prevention [45].

As a brief review, the Los Alamos National Laboratory (LANL) and KSC, investigators have demonstrated that doped PANI coatings inhibited corrosion on carbon steel [46]. Their approach was based on earlier work suggesting that the interfacial contact between the metal and a doped EAP would generate an electric field that would restrict the flow of electrons from the metal to an outside oxidizing species, thus preventing and/or reducing corrosion [47]. The LANL-KSC tests were conducted in a 3.5wt % NaCl/0.1 M HCl environment using 0.005 cm thick films of PANI doped with *p*-toluenesulfonic acid on carbon steel. The PANI primer was coated with an epoxy topcoat. The PANI/epoxy coating performed significantly better than the epoxy topcoat alone. These initial results were used by researchers at LANL-KSC to develop EAP coatings to resist the corrosive effects of acid vapor generated during space shuttle launches due to the harsh operating environment present during lauches. The ground support equipment and structures at the KSC were susceptible to these severe environmental conditions. Marine outdoor exposure testing over 28 months have shown that PANI/epoxy coatings

on carbon steel can survive a severe corrosive environment providing acceptable corrosion protection.

However, an examination of EAPs as an alternative to CCC pretreatments has not been thoroughly studied. The NAWCWD has investigated BAM-PPV as an alternative to CCC due to PPV's known redox properties and tertiary (3^0) amine functionalities capable of corrosion inhibition with adhesive properties. BAM-PPV has shown strong adhesion to AA2024-T3 alloy in an simulated seawater immersion test (pH~8). Its ability to inhibit corrosion was demonstrated [55-65]. Constant current (galvanostatic) and constant potential (potentiostatic) methods were used to investigate the corrosion protection of BAM-PPV on AA2024-T3 coupons. Quantitative evidence was obtained from these results which showed that corrosion inhibition was obtained without the loss of adhesion and no pH dependency was observed. These tests were run against control samples (non-coated AA2024-T3 coupon) and the BAM-PPV coated coupons outperformed the non-coated coupons. Visual inspection of the coupons showed significant pitting in the non-coated as compared to the coated (BAM-PPV) samples.

Prior to initiation of the completed ESTCP project WP 200527, a SERDP program WP 1148 was successfully completed over four years during FY00-04 and EAP polymers can repeatedly meet the minimum requirement of 336 hours of corrosion inhibition as a viable alternative to CCC. The accelerated weathering testing on both BAM-PPV and MEH-PPV proved that they can meet and exceed this military requirement. The EAP coated panels were compared to TCP (Metalast TCP-HF) and CCC (Alodine 1200S) coated AA 2024-T3 coupons. Figures 7 and 8 show the BAM-PPV coating compared to TCP coating. The military specification (MIL-DTL-81706, "Chemical Conversion Materials for Coating Aluminum and Aluminum Alloys, October 2004) for an alternative to CCC on aluminum alloys requires that the alternative pretreatment pass exposure to neutral salt spray (NSS) for 336 hours without blistering, delamination, corrosion or significant discoloration. All of these criteria have been met for BAM-PPV during the SERDP/ONR program. BAM-PPV polymers can be incorporated into military coatings and in most cases can perform as well as the CCC and TCP based military coating with non-chrome primers and topcoats. In general the alternatives to fully chromated systems do not meet the minimum requirement for neutral salt fog. However, the BAM-PPV pretreatment film is a potentially viable alternative to the currently approved pretreatment (TCP) coating system. BAM-PPV does not contain any heavy metals in the system (e.g. chromium: hexavalent or trivalent). This represents a significant improvement over heavy metal based coatings (e.g. TCP) that contain chromium. Trivalent chromium is not considered a carcinogen but with potential restrictions on the future use of chromium (in any form), a fully chromium-free coating will be more attractive to military installations as they seek to comply with future regulations. The best performing BAM-PPV coated with primer and topcoat onto AA 2024-T3 coupons were used as the basis for transition to the completed ESTCP program.

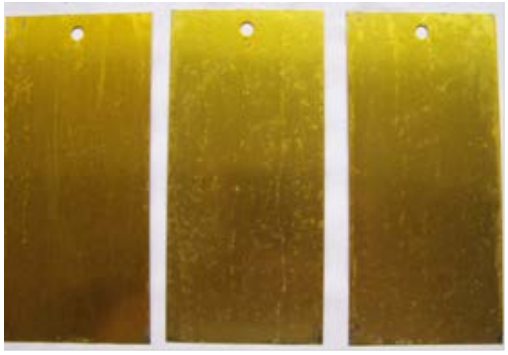


Figure 7: BAM-PPV coated AA 2024-T3 (>1 micron) at 336 hours NSS

Figure 8. TCP coated AA 2024-T3 at 336 hours NSS

2.2 Technology Development

The aerospace industry and DOD currently enhance the corrosion resistance and paint adhesion performance of aluminum alloys with hexavalent-chromium (Cr(VI)) based pretreatments. These pretreatments are known as CCC and are used to treat aluminum alloys such as 7075-T6, 7075-T3 and 2024-T3. Chromate conversion coatings are applied by either immersion, spray or wipe techniques that produce a thin coating that is inexpensive and extremely robust.

However, Cr(VI) has been identified as a health threat, and is a known carcinogen. Due to its toxicity, it is currently highly regulated. The current introduction of new, lower OSHA permissible exposure limits (PEL) has reduced the levels of Cr(VI) allowed to be discharged into the industrial environment. Therefore, chromate-free coatings are needed that exhibit equal or superior corrosion protection, while reducing or eliminating volatile organic compounds (VOCs) and hazardous air pollutants (HAPs).

Electroactive polymers (EAPs) coatings have demonstrated corrosion protection even when the coating is scratched and exposed to aqueous salts and hydrochloric acid. EAP based polymer coatings are robust materials that are environmentally benign. A comprehensive study of EAP coatings, including synthesis, scale-up, benign coating formulations, testing in accelerated weathering chambers and an examination of their corrosion protective mechanisms has been investigated from previous studies. These novel EAPs are now potential candidates to replace CCC. In addition, these new EAP coatings have been incorporated into military coating systems using non-chromium primers and topcoats. **Thus, EAP pretreatment coatings can provide the DOD community a replacement for CCC containing military coating systems**.

The objectives of this former SERDP program (WP 1148) have been completed successfully. The synthesis, scale-up and characterization of new monomers based on a bis-amino derivative of poly-*p*-phenylene vinylene (PPV) called poly(2,5-bis(N-methyl-N-hexylamino)phenylene vinylene, (BAM-PPV) and oligomers of polyaniline have been completed. These polymers have been thoroughly characterized using advanced spectroscopic and analytical methods. The details regarding the EAP synthesis has been published in the literature for duplication including potential industrial use. BAM-PPV has been processed using a variety of conditions including both high VOC and zero-VOC processing conditions. Initial studies focused on xylenes as a processing solvent with good quality films being produced. Further studies using *d*-limonene, a commercial food

and cosmetic additive (environmentally friendly and zero HAP) as the processing solvent also produced high quality films.

BAM-PPV pretreatment coating on AA2024-T3 processed from both of these solvents passed 336 hours neutral salt fog exposure. This test is required for new pretreatment coatings on aluminum alloys. Additional pretreatments were also studied including trivalent chromium pretreatment (TCP). BAM-PPV coatings in several cases exceeded the 336 hour requirement. BAM-PPV has also been incorporated into full military coating systems that include non-chromium primers and topcoats. These coatings have been tested against known controls such as chromium primers and TCP pretreatment with non-chromium primer and topcoat. BAM-PPV performed as well as the TCP pretreatment with non-chromium primer and topcoat. However, in both cases, each coating did not pass the 2000 hours neutral salt fog exposure test. When BAM-PPV was used with chromium primer and topcoat, this coating system lasted over 4000 hours in a neutral salt spray chamber. Additionally, BAM-PPV has been incorporated into a zero-VOC powder formulation and has matched a commercial polyester powder resin (control) performance in neutral salt fog exposure. Both the BAM-PPV powder coating and the control failed at 1500 hours in neutral salt fog exposure.

BAM-PPV coatings were investigated in several ways to help elucidate the mechanism of corrosion protection. Studies by electrochemical noise methods (ENM,) scanning vibrating electrode technique (SVET) and X-ray photeoelectron spectroscopy (XPS) showed evidence that BAM-PPV provides more than simply barrier protection to corrosive environments. The mechanism includes barrier protection and may passivate the metal surface during exposure to corrosive environments. This evidence is demonstrated by spontaneous oxidation/doping, rendering the polymer sufficiently conductive to mediate electron transfer from the metal/polymer interface to polymer/solution interface. The corresponding electronic or electrochemical interaction between the polymer and the metal may provide evidence for the passivation of the metal.

The results (summarized below) demonstrate that the SERDP WP 1148 program has achieved the required performance objectives [55-65, 72-77]:

- **The new EAP polymeric materials that contain no heavy metals (Cd, Cr, Ni, Zn, Cu, etc.),**
- **BAM-PPV was successfully scaled up to the kilogram quantity and the synthesis was improved for potential industrial production,**
- **New EAP (BAM-PPV) polymers were processed to produce thin films on aluminum and steel substrates,**
- **Solution spraying produced uniform, non-porous, dense films that provided the minimum barrier protection for corrosive environments,**
- **EAPs were coated onto various substrates using benign coating processes,**
- **BAM-PPV powder can be coated onto aluminum and steel substrates using a variety of coatings processes. These processes included solvent based and environmentally friendly solvents. Additionally, zero-VOC powder coatings incorporating BAM-PPV as the corrosion inhibitor were prepared. BAM-PPV was solvent sprayed onto aluminum alloys and non-chromium primers**

or chromium primers and topcoats added using standard DOD equipment currently used at NAWCAD,

- **BAM-PPV has successfully passed the 336 hours neutral salt fog exposure test as a viable alternative to CCC,**
- **BAM-PPV has shown both barrier capability and may passivate the metal surface during exposure to a corrosive environment via electrochemical/spectroscopic studies, and**
- **BAM-PPV was transitioned into a demonstration/validation program (ESTCP).**

In conclusion, the program has resulted in the acquisition of both basic and applied knowledge regarding synthesis, scale-up, coating processing and performance of EAP polymers used in corrosive environments. The application of this knowledge has enabled the transition of this technology to potential fleet-wide use under the ESTCP program (FY05-08).

2.3 Advantages and Limitations of the Technology

The advantages of using BAM-PPV as a new pretreatment coating will allow the elimination of Cr(VI) from the pretreatment coating process for metal alloys. Data available from FY06 shows CCC usage generating 5,182 lbs of hazardous waste (Table 4). The data presented in Table 4 is from the Environmental Systems Allocation Database. The use of BAM-PPV will eliminate this hazardous waste stream. The current alternative, TCP, while non-carcinogenic is a chromium based material. This material in the near future may be regulated by both EPA and OSHA due to its chromium based formulation.

Table 4: Environmental Systems Allocation Database

MIL-DTL-81706 CHEMICAL CONVERSION MATERIALS FOR COATING ALUMINUM AND ALUMINUM ALLOYS	
2006 Material Usage	
O&I Level Maintenance Usage:	1,907 lbs
Depot Level Maintenance Usage:	3,275 lbs
Total Usage:	**5,182 lbs**

Another advantage of this technology is that current paint equipment can be used. No special equipment would be necessary. One limitation of this technology could be the dilute solutions used. BAM-PPV has been prepared as a 1 weight % solution in solvents, such as Oxsol-100, xylenes, or limonene. Oxsol-100 is the most promising because it is a VOC-exempt solvent. Another aspect that has been rigorously tested is its full shelf life. The studies have shown that the shelf life of BAM-PPV is very long (years). It can be stored for long periods of time without degradation. As a solution it has a shelf-life of several months (6-10 months). In powder form it is stable for years ($\geq$ 4 years). Both NAWCWD and WPAFB have examined the stability and processability of BAM-PPV

powder and solution. The data supports the idea that BAM-PPV is stable and can be processed from powder after years of standing in a closed container.

3.0 PERFORMANCE OBJECTIVES

The December 2000 Joint Test Protocol (JTP) developed by NAWCAD has been used as a guide for CCC replacements to evaluate the effectiveness of the EAP pretreatments [78]. Quantitative and qualitative data was collected and used to evaluate the EAP coating system's ability to replace the CCC corrosion/adhesion performance, as well as to identify environmental benefits, cost savings, and improvements to mission readiness. Examples of performance objectives for this WP 200527 ESTCP project is included in Table 5 and acceptance/rejection criteria for these performance objectives are described in the table. Further discussion is also presented in this section describing the objectives.

Table 5: Performance Objectives for WP 200527 ESTCP Project

Type of Performance Objective	Primary Performance Criteria	Expected Performance (Metric)	Actual Performance Objective Met?
(1) Reduce Cr(VI) in coating/Quantitative	Reduce the use of chrome in a coating system	Eliminate the need for chrome in a pretreatment	Yes
(2) Corrosion Resistance in NSS/Quantitative	Corrosion Resistance: Neutral Salt Spray (NSS) on Unpainted Substrate with Pretreatment	336 hours with no visible sign of corrosion; lightening of coating is acceptable	Yes
(3) Corrosion Resistance on Scribed Panels/Quantitative	Corrosion Resistance NSS on Scribed, Painted Substrate	2,000 hours with no evidence of corrosion (minor surface corrosion in scribe permissible).	Pass with Chromated primer: 4000 hours Pass with Non-chrome primer: 2000 hours
(4) SO_2 Corrosion Resistance/Quantitative	SO_2 Salt Fog on Scribed, Painted Substrates	500 hours with no evidence of corrosion (minor surface corrosion in scribe permissible).	No, all panels failed
(5) Cyclic Corrosion Resistance/Qualitative	Cyclic Corrosion Test on Scribed, Painted Substrates	Equivalent or improved performance compared to controls.	Several panels showed acceptable performance
(6) Filiform Corrosion Resistance/Quantitative	Filiform Corrosion Resistance	All filaments < 1/4"; Majority <1/8".	No, all panels failed
(7) Electrical Contact Resistance (ECR) of EAP coated panels/Quantitative	Electrical Conductivity of Unpainted, Pretreated Substrates	<5 milliohms/square-inch as coated. <10 milliohms/square-inch after 168 hours neutral salt fog exposure.	Several coupons pass
(8) Marine outdoor exposure testing Qualitative/Quantitative	Marine Atmospheric Outdoor Test (Beach Test) Exposure on Scribed, Painted Substrates	Equivalent or improved performance compared to chromate controls	All panels failed at 6 months, except Cr(VI) controls
(9) Outdoor exposure testing Qualitative/Quantitative	Outdoor Exposure Tests	Equivalent or improved performance compared to chromate controls	BAM-PPV coated panels survived 24 months
(10) Wet Tape Adhesion (WTA)/Quantitative	Wet Tape Adhesion and Water Resistance of Painted Substrates	Rating of 4A or 5A.	Pass
(11) Dry Tape Adhesion Quantitative	Dry Tape Adhesion of Painted Substrates	Loss of two or more complete primer squares shall constitute failure (<3B).	Pass
(12) Adhesion Testing during field studies/Qualitative	In-Service Paint Adhesion	Equivalent or improved paint adhesion compared to chromate controls	BAM-PPV coated hardware passed AF and Army field testing, BAM-PPV coated hardware failed Navy field testing
(13) Corrosion testing during field studies/Qualitative	In-Service Corrosion	Equivalent or improved performance compared to chromate controls	BAM-PPV coated hardware passed AF and Army field testing, BAM-PPV coated hardware failed Navy field testing

The details for objectives listed in Table 4 can be found in Sections 5.1-5.4.3 laboratory testing. A brief overview is presented below with

- explanation of the objective,
- metric used to assess whether objective was met and
- criteria used to determine success.

Objective Number 1-Reduce Cr(VI) in Coating Systems

By eliminating Cr(VI) from the pretreatment coating one can reduce the amount of Cr(VI) released as hazardous waste during coating removal processes. Success for this objective is to test and quantify the CCC replacement coating and determine via accelerated weathering tests if is meets the minimum requirements set forth in the NAWCAD December 2000 JTP [78].

Objective Number 2-Corrosion Resistance in NSS Testing

In order to have a CCC replacement, the alternative pretreatment must meet a minimum of 336 hours NSS exposure testing. There can be no discoloration, corrosion, blistering or delamination of the coating in order to pass this test.

Objective Number 3-Corrosion Resistance on Scribed Panels in NSS Testing

In order to have a full Cr(VI) replacement, the alternative pretreatment + primer must meet a minimum of 2,000 hours NSS exposure testing. There can be no corrosion, blistering or delamination of the scribed coating in order to pass this test.

Objective Number 4-SO_2 Corrosion Resistance

For Navy applications only, to have a full Cr(VI) replacement, the alternative pretreatment + primer + topcoat must meet a minimum of 500 hours SO_2 exposure testing. This test is not required for the AF or Army. There can be minor surface corrosion in the scribe without loss of adhesion or delamination of the full military coating system.

Objective Number 5-Cyclic Corrosion Resistance

In order to have a full Cr(VI) replacement, the alternative pretreatment + primer + topcoat must meet or exceed Cr(VI) full military coating for a 40 cycle test period. This test is required for the AF and Navy. There can be minor surface corrosion in the scribe without loss of adhesion, blistering, rusting or delamination of the full military coating system.

Objective Number 6-Filiform Corrosion Resistance

For Navy applications only, to have a full Cr(VI) replacement, the alternative pretreatment + primer + topcoat must meet a minimum of 1,000 hours filiform corrosion exposure testing. This test is not required for the AF or Army. The number of filiform thread-like extension protruding from the scribe determines whether the panel passes or fails.

Objective Number 7-Electrical Contact resistance (ECR) of Alternative Pretreatment Coatings

This test is required for Navy pretreatment coatings as alternatives to CCCs. The test measures the ability of a pretreatment surface to maintain a conductive surface as prepared and after exposure to NSS (168 hours only). The values are obtained in milliohms (mΩ) and no corrosion is evident on the panel after 168 hours exposure to NSS.

Objective Number 8-Marine Outdoor Exposure Test

Marine outdoor exposure testing is required by the Navy. This test provides conclusive evidence regarding a full military's coating performance in a harsh environment that Navy military hardware is exposed to. Fully Cr(VI) military coatings are used as controls and the alternative coating systems are measured for a one-year study. Their performance is evaluated in terms of corrosion, blistering, delamination, rusting as compared to the Cr(VI) controls.

Objective Number 9- Outdoor Exposure Testing

The outdoor exposure testing is required for AF field testing of new non-chromium pretreatments. Prior to any field testing on non-critical hardware and post laboratory testing, outdoor exposure testing on a new pretreatment coating + non-chromium primer + topcoat vs. fully Cr(VI) coating system is exposure to outdoor conditions located at WPAFB. Success of new coating systems must show equal or better performance after one-year of testing and compared to a fully Cr(VI) system.

Objective Number 10-Wet Tape Adhesion Testing

This test is required of all co-performers (WPAFB, ARL, NAWCAD/NAWCWD) to determine inter-coat adhesion of an organic coating immersed in DI water for short periods of time (24 hours). A rating system of 0A-5A is assigned to each coating system after testing three replicates. A number rating of >3A is required for the coating to pass this test.

Objective Number 11-Dry Tape Adhesion Testing

This test is required of all co-performers (WPAFB, ARL, NAWCAD/NAWCWD) to determine adhesion between the substrate (Al and/or steel), pretreatment, primer and topcoat interfaces. A rating system of 0B-5B is assigned to each coating system after testing three replicates. A number rating of >3B is required for the coating to pass this test.

Objective Number 12-Adhesion Testing During Field Studies

During field testing, all co-performers (WPAFB, ARL, NAWCAD/NAWCWD) must examine via visual inspection the adhesion of the pretreatment + primer + topcoat on the non-critical military hardware. Adhesion of the coating onto these systems is visually examined periodically and recorded over a one-year field study.

Objective Number 13-Corrosion Testing During Field Studies

During field testing, all co-performers (WPAFB, ARL, NAWCAD/NAWCWD) must examine via visual inspection corrosion of the pretreatment + primer + topcoat on the non-critical military hardware. Corrosion inspection of the coating on the non-critical military hardware is visually examined periodically and recorded over a one-year field study

4.0 SITE/PLATFORM DESCRIPTION

During Phase I of the ESTCP Program laboratory tests were conducted by WPAFB, ARL and NAWCWD/NAWCAD to evaluate BAM-PPV and/or MEH-PPV as a pretreatment coating without any primer and topcoat and incorporated into a full military coating system (primer + topcoat). After laboratory evaluations the best performing system (full military coating) were then field tested by each service on non-critical military hardware.

4.1 Test Platforms/Facilities

The demonstration platforms that were selected during Phase II were based on each service analyzing the laboratory and outdoor exposure testing and determining a suitable platform for testing the BAM-PPV and/or MEH-PPV candidates. Thus, after full completion of the laboratory accelerated weathering, outdoor exposure and toxicology testing, a down-selection of the best performing EAP pretreatment coating with Cr(VI)-free primers and topcoats was applied to non-critical military hardware for field testing.

4.1.1 Test Platforms/Facilities at WPAFB

The platform for field testing the BAM-PPV pretreatment was determined by WPAFB. The WPAFB testing facility provided an excellent opportunity to test the BAM-PPV material on the C-5 cargo plane. The unique characteristics and history of the 445th Aircraft Wing at WPAFB provides first rate testing facilities and performance evaluations on new alternative pretreatment coatings to CCC. The 445th Airlift Wing, located at WPAFB is an operation wing of the Air Force Reserve. It maintains 11 operational C-5 Galaxy aircraft and has previously maintained other cargo and fighter aircraft. The 445th Airlift Wing is composed of an Aerospace Medicine Squadron, Aeromedical Staging Squadron and three attached groups. These three groups are:

- the 445th Maintenance Group, which consists of the Aircraft Maintenance Squadron, Maintenance Squadron, and Maintenance Operations Flight;
- the 445th Operations Group, which consists of the 89th Airlift Squadron, Aeromedical Evacuation Squadron, Airlift Control Flight, and Operations Support Squadron; and
- the 445th Mission Support Group, which consists of the 87th Aerial Port Squadron, Civil Engineer Squadron, Communications Flight, Logistics Readiness Squadron, Mission Support Flight, Security Forces Squadron, and Services Flight all of which fall directly under the wing commander.

The C-5 is a long-range troop and cargo transport built by Lockheed-Georgia Company. The aircraft is powered by four turbofan jet engines, each capable of producing 43,000 pounds of thrust. The C-5 Galaxy provides the Air Force's strategic airlift capability. Since reactivating at WPAFB, the 445th Airlift Wing has been active in providing airlift of troops and supplies around the globe and has provided operational

support to almost every contingency the Air Force has undertaken. One of the wing's operations was the re-supply of the scientific teams located at McMurdo Station, Antarctica, in conjunction with the 452nd Air Mobility Wing, March Air Reserve Base, CA. The wing has been active in the wake of the terrorist attacks of Sept. 11, 2001 by providing emergency airlift of supplies, medical teams and FEMA personnel to McGuire Air Force Base, N.J. This was done in order to assist with operations involving the World Trade Center collapse. The wing was also an active participant in Operation Enduring Freedom by being the first wing to fly Taliban and al-Qaeda detainees to Guantanamo Bay Naval Station, Cuba. Since the initial detainee flights, the 445th Airlift Wing delivered roughly half of the detainees housed at the Guantanamo Bay facility. The wing also served in the honorable mission of evacuating wounded personnel from the battlefield to regional treatment facilities and bringing our fallen service members home for burial.

The selection of the C-5 cargo plane (Figure 9) was chosen as an appropriate military aircraft for field testing the BAM-PPV pretreatment coating as an alternative to CCC's for several reason which are listed below:

- **the size of the rear-hatch door (Figure 10) which will provide adequate surface area such that multiple coating systems can be tested in patches side-by-side for immediate performance comparison,**
- **removable and if a serious coating failure is noted, the door can be removed and replaced easily with minimal interference using typical repair operations,**
- **C-5 maintenance group is located at WPAFB and is immediately accessible to AFRL Materials and Manufacturing Directorate personnel, so periodic checks and performance feedback is readily available,**
- **flight performance of the system will be ascertained from this test bed which is immediately comparable to many other aircraft performance requirements, and**
- **BAM-PPV coating in several laboratory test systems showed comparable performance to the chromate controls.**

Figure 9: Air Force C-5 Cargo Aircraft

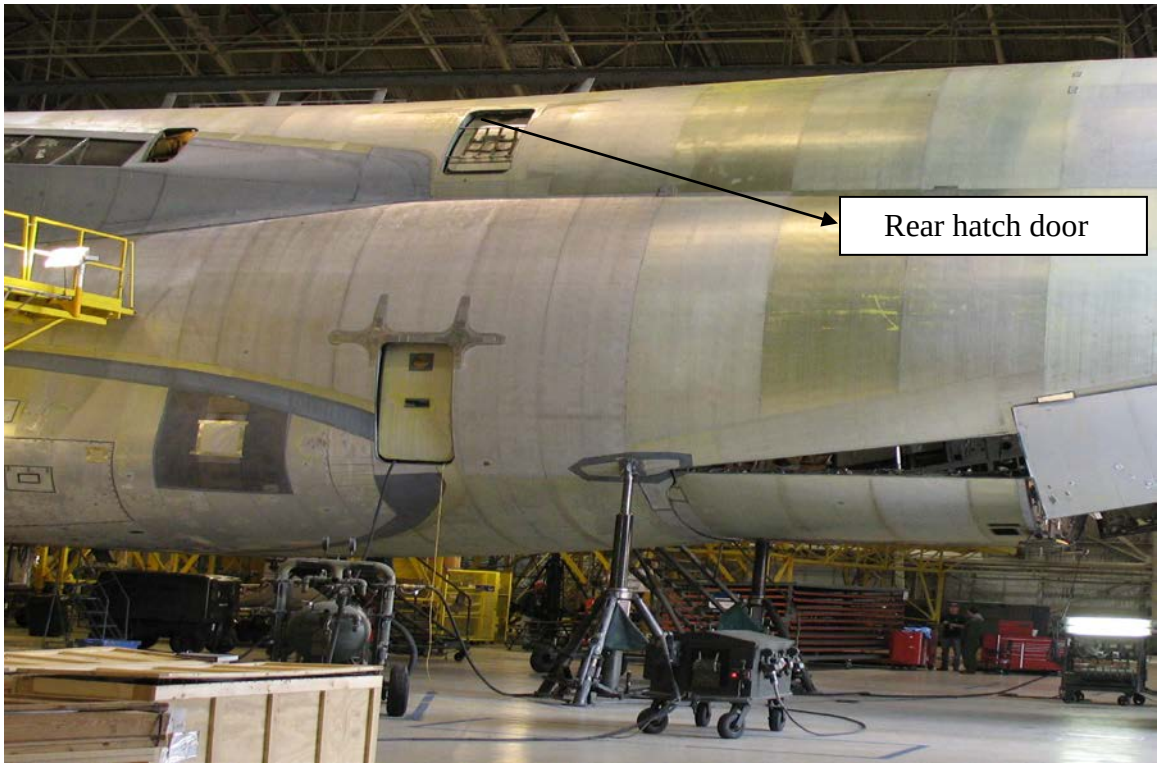


Figure 10: C-5 Aircraft Rear Hatch Door

4.1.2 Test Platforms/Facilities at ARL

The ARL has chosen the Bradley vehicle as the non-critical military hardware for field demonstration of the BAM-PPV pretreatment coating. The Bradley vehicle used only non-critical parts that were coated for the field demonstration. The headlight cover (Figure 11) of the Bradley vehicle was selected for field demonstration of the BAM-PPV compound. Several Bradley vehicles are available at ARL, Maryland and the headlight cover will provide enough area to test the new coating in various environmental conditions. The reasons for selecting the headlight cover of the Bradley vehicle are as follows:

- **Bradley Fighting Vehicle is an ideal Army platform for assessing the performance of the BAM-PPV,**
- **headlight cover is constructed of an aluminum alloy that provides the EAP a fair approximation of Army inventory, and the**
- **location of this vehicle at the Aberdeen Test Center (ATC) provides evaluators with several critical advantages over other locations.**

  - ✓ Firstly, because the vehicle is assigned to the nearby test track, it receives a more aggressive exposure than would be expected from the natural environment of central Maryland,
  - ✓ The Test Track is used to test complete systems in an accelerated environment that approximates the GM 9540 cyclic accelerated corrosion test,
  - ✓ Proximity to the Chesapeake Bay raises the night time humidity but ameliorates temperature extremes, and
  - ✓ The ATC is a mere 1.5 miles from the personnel whom would be performing the periodic evaluations.

This allows for closer monitoring of the EAP's coating performance.

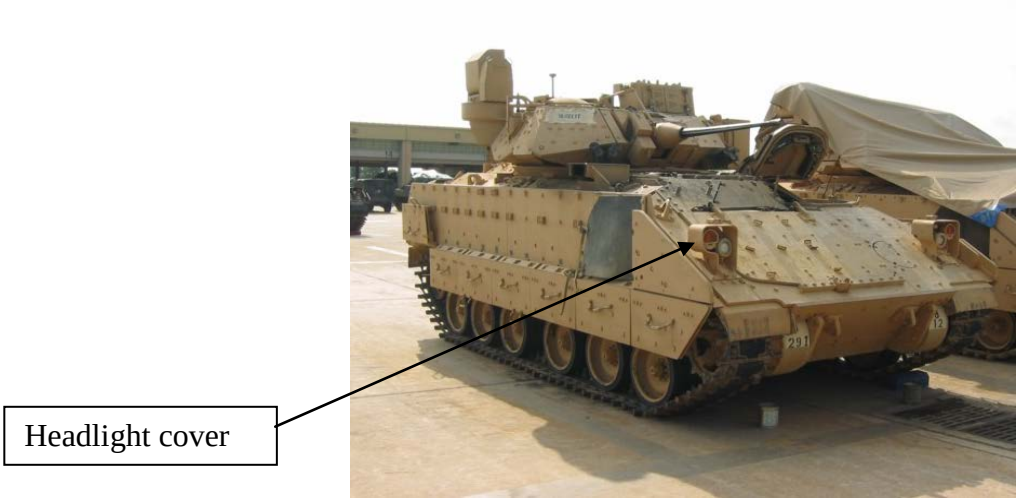


Figure 11: Army Bradley Vehicle with headlight cover

The ATC Test Track will provide a good opportunity to subject EAP coated material to an aggressive environment with the advantage that evaluations take place at regular intervals. Army systems evaluated at the test track are driven around the facility every business day for a period of three weeks. Equipment is subjected to humidity exposure over the weekends. After that period of exposure, called a Phase, the system is thoroughly evaluated and repaired (if necessary) before returning to the test track for the next Phase of exposure. There are weight loss coupons mounted on the pull vehicle to track the aggressiveness of the environment. In this way, it is hoped that design flaws are exposed that would not arise in the various static exposures. ATC has a fleet of vehicles available to either mount equipment onto or tow articles of interest.

4.1.3 Test Platform/Facilities at Fleet Readiness Centers (FRC)

In cooperation with NAWCAD, the NAVAIR-Lakehurst facility will demonstrate EAP coatings (BAM-PPV) as replacements for CCC on support equipment. The demonstration locations for the Navy will be Fleet Readiness Center Northwest (FRC-NW) at Naval Air Station Whidbey Island, WA and Fleet Readiness Center Southeast (FRC-SE) at Naval Air Station Jacksonville, FL. Whidbey Island serves as maintenance and repair facility for naval ground support equipment. Whidbey Island has substantial expertise in the evaluation of new coating systems under other ESTCP and related evaluations. Service items from each location that are used within the demanding and corrosive environment of U.S. Navy submarines and aircraft carriers present some of the largest opportunities for the evaluation of new coating systems. The components selected for the 12 month field service evaluations will be identified by Navy officials, but are likely to include the following items: the Navy's towbars and bomb hoists. These items were recommended based upon these reasons:

- **high likelihood of Field Service Evaluation (FSE) success,**
- **material quantities reduced/eliminated (for chromium-based pretreatments),**
- **potential reduction in labor costs required for EAP implementation,**
- **VOC, HAP, and hazardous waste reduction related to processing each component, and**
- **demonstration effort was confined to the Naval Air Station Whidbey Island and the Naval Air Station Jacksonville to insure real-world processing, testing and potential implementation pathways of new EAP materials.**

The Aircraft Intermediate Maintenance Detachment (AIMD), operating as a part of the larger Fleet Readiness Center Northwest (FRC-NW) at Whidbey Island, is the premier Intermediate Maintenance Activity in the Pacific Fleet and has sustained one of the highest ready-for-issue rates in the Navy. Located in building 2547, AIMD has a staff of 452 permanently-assigned Sailors, 13 Marines, 30 civilian personnel, 68 Temporary Active Duty (TAD) from NAS Whidbey commands, 241 Sea Operational Detachment personnel supporting all carrier requirements and 237 Expeditionary Logistics Unit personnel supporting forward-deployed EA-6B squadrons worldwide. Naval Air Station Whidbey Island encompasses over 7,000 acres of land and 60 buildings within Oak Harbor, Washington. The Whidbey Island AIMD provides intermediate maintenance

support to 14 deployable EA-6B squadrons, five P-3 squadrons, 11 aircraft carriers, one C-9 squadron, the station Search and Rescue (SAR) component, and various Northwest Regional activities. In addition, the sea component provides afloat intermediate level support that includes repairing avionics, airframes, power plants, and life support systems for embarking EA-6B squadrons via 25-man team detachments on board 11 aircraft carriers.

AIMD schedules over 100,000 maintenance actions each year in support of NAS Whidbey Island-based aircraft, deployed aircraft carriers, and various other naval activities in the Pacific Northwest Region (Figure 12). Roughly 40,000 aircraft parts are inducted of which 34,000 are repaired and returned to service, while the rest are referred for depot level repair or scrapped. There are 68 work centers that log over 1,000,000 man-hours of aviation maintenance annually. AIMD also staffs and manages the Support Equipment Rework Facility at Naval Air Station Everett in support of Pacific Northwest carriers. In addition to permanently assigned technicians, Navy and Marine Corps Reservists receive mobilization training and contribute to the production effort during drill weekends.

Figure 12: Naval Air Station, Whidbey Island

Naval Air Station (NAS) Jacksonville, is a multi-mission base hosting more than 100 tenant commands, is the third largest naval installation in the United States. Jacksonville has a large population of active duty military, dependents and retired military personnel in the greater Jacksonville area. NAS Jacksonville performs Depot maintenance on the P-3 Orion, T-2 Buckeye, F/A-18 Hornet, S-3 Viking and A-7 Corsair. In addition, NADEP Jacksonville is the Navy's premier engine facility and reworks jet engines and over 36,000 components and avionics. This installation serves as the host for the Patrol Wing Eleven, the southern component of the Atlantic Fleet P-3C force, and VP-30, the Fleet Replacement Squadron for that aircraft. This Squadron prepares and trains U.S. and foreign pilots, air crew and maintenance personnel for further operational assignments. The installation also hosts Sea Control Wing Atlantic and its five squadrons

of S-3B aircraft, and Helicopter Antisubmarine Wing Atlantic, and five squadrons of SH-60/HH-60 helicopters.

Fleet Readiness Center Southeast is one of eight Fleet Readiness Centers commissioned by the Navy to perform in-depth overhaul, repair and modification of aircraft, engines, aeronautical components, surveillance, and countermeasure systems. FRC Southeast is the largest industrial employer in Northeast Florida and Southeast Georgia, and the largest tenant command aboard Naval Air Station Jacksonville, Florida. The Airframe Teams at FRC Southeast are responsible for maintaining the structural and mechanical condition of aircraft and performing maintenance, repair and overhaul (MRO) operations on the F/A-18 A-F Hornet, the SH-60 Seahawk, the P-3 Orion, and the EA-6B Prowler. As one of the most modern, state-of-the-art engine facilities in the Navy, the FRC-SE Propulsion Facility has a full range of capabilities to support MRO at every level of complexity on aircraft engines, subsystems, components, and related accessories. The Facility performs complete disassembly of aircraft engine modules to the component level; repairs, refurbishes, manufacturing, and replaces work and damaged parts; and reassembles and tests engines to meet or exceed specifications, keeping the warfighters in mission ready operating condition. The biggest impact of CCC's is in the area of worker exposure. Testing and evaluation of the EAP coating may reduce worker exposure to toxic materials and resultant frequent personnel monitoring. Additionally, with its proximity to several ecologically sensitive areas and tribal areas, CNRNW continues to search for new process changes in order to reduce the quantity and toxicity of the hazardous waste it generates. The reduction of CCC usage will significantly reduce both the quantity and toxicity of hazardous waste throughout these FRCs as a result of maintenance and repair operations.

The selection of the non-critical military hardware components was based on laboratory studies conducted at NAWCWD and NAWCAD. From these laboratory studies, towbars and bomb hoists were selected rather than non-critical military aircraft components due to the poor performance of BAM-PPV in SO_2 salt fog and filiform corrosion studies.

4.1.4 Test Platform/Facilities at NASA/Kennedy Space Center (KSC)

The NASA Beach Corrosion Test Facility (Figure 13) was chosen for marine outdoor exposure testing for several reasons:

- **site's harsh marine conditions**
- **high solar,**
- **high humidity,**
- **salt spray,**
- **rain, and**
- **wind.**

These conditions provide an ideal environment to measure the robustness of new coatings systems in an environment that can mimic real world sea environments. This facility is located approximately 150 feet from the Atlantic Ocean, and is approximately 1 mile south of the Space Shuttle launch sites. The facility includes an atmospheric

exposure site, flowing seawater immersion tanks with spray-down capabilities, cathodic protection compatibility tank, weather station, on-site electrochemistry laboratory, monitoring station, and sample preparation and processing area. The beachfront laboratory has the capability to provide real-time data access to monitor corrosion experiments and surrounding weather conditions.

Figure 13: NASA Beach Corrosion Test Site

4.2 Present Operations

For each facility listed below are the current operations associated with the military hardware, coating processes and methods to monitor the new EAP coating during field testing.

4.2.1 Present Operations at WPAFB

WPAFB houses the AFRL Materials and Manufacturing Directorate and the 445th Airlift Wing. Current laboratory operations for the AF have been performed by the Coatings Technology Integration Office (CTIO). CTIO serves as the Air Force's central resource for aircraft coating systems and their applications. CTIO primary objectives are pollution prevention and improved coating system performance through the integration and transition of environmentally acceptable materials and processes for aircraft refinishing. The facility holds a climate-controlled paint booth that can be controlled to allow paint application and curing over the full range of environmental conditions likely to be found in the field. The equipment brings climate-related problems (e.g. film build,

sagging) into the lab for controlled study and resolution. Adjacent to the large booth (10 feet x 14 feet x 9 feet) is an equally large climate-controlled prep area. This combination permits us to coat samples that range from very small to large sections (8 feet by 4 feet and larger). Equipment is available to immerse samples in alkaline cleaners, acid etches, and conversion coatings (either Cr(VI) or Cr(VI)-free). The equipment accommodates both hand- and spray-applied surface pretreatments. University of Dayton Research Institute (UDRI) contractor support for the CTIO, operate the facility and their organization is accredited to ISO 17025 and SAE AS5505 for the application, testing, and performance characterization of organic coatings. CTIO has developed a working relationship with the C-5 Systems Group and the 445th Airlift Wing to solve de-paint, coating application and coating performance issues.

The 445th Airlift Wing provided the rear hatch door to CTIO for this program. This door was re-attached to the aircraft once it is coated by CTIO staff and returned to the 445th Airlift Wing. The door was coated with four coating systems during a 72 hour window and returned to the 445th Airlift Wing. This cure window allowed the polyurethane topcoat, conforming to MIL-PRF-85285, to cure to the appropriate level per T.O. 1-1-8, Application and Removal of Organic Coatings, Aerospace and Non-Aerospace Equipment, paragraph 6.12.8. The 445th Aircraft Wing provided 8 man-hours to remove and replace a door once it was coated. The aircraft was returned to service shortly after that.

4.2.2 Present Operations at ARL

The ARL at APG has the capability to apply most of the coatings and pretreatments included in the chemical agent resistant coatings (CARC) application specification to smaller parts and test coupons. ATC has similar capabilities with the added ability to apply these coatings *in-situ* to systems. The removed components should pose no problem for either facility. Both components had a blasted surface with BAM-PPV pretreatment substituted for a conversion coating. This was followed by a standard CARC coating of MIL-P-53022 primer and MIL-DTL-64159 waterborne polyurethane in a color to match the vehicle. The inspections were on a phase schedule that coincides with that of the vehicle to which the parts are affixed. This was once every three weeks which depended on the mechanical stability of the vehicle.

4.2.3 Present Operations at FRC

At the Fleet Readiness Center Northwest (FRC-NW)/Naval Air Station Whidbey Island and Intermediate Maintenance Facility (IMF)/Naval Base Kitsap at Bangor, the substrate pretreatment for non-flight support components typically includes a conversion coating. CCC's are used for both zinc-plated parts as well as aluminum components, in order to prevent corrosion. For aluminum components, a high-solids epoxy primer coating would be applied to this (based on MIL-PRF-23377, MIL-P-53022, or MIL-P-53030), followed by a polyurethane topcoat (based on MIL-PRF-85285). Coating pretreatments must serve the following corrosion prevention functions: passivation of base aluminum; action as a barrier against moisture, oxygen, and other corrosive agents; electrochemical insulation; and protection against mechanical erosion. In addition,

conversion coatings must provide other essential interfacial properties to complement the next coating layers. This includes an effective and continuous bonding site; chemical stability during the service life of coated products; insolubility, imperviousness and flexibility. The ability to provide a wettable subsurface for paint application and maintaining adhesive integrity is also required.

Regular maintenance is typically performed at the squadron level at the FRC-NW. The regular maintenance cycle calls for inspections every 364 days. Only when there is the need for a major repair do the components actually get sent back to FRC-NW. Upon receipt of the equipment, the coating system would be stripped prior to repair. Once the component has been repaired and the coating system refurbished, FRC-NW would send the component off as needed. It may go the supply shelf, or it may be returned to the aircraft in order to go back into service.

As part of the U.S. EPAs Aerospace Residual Risk Effort (AeroRR), the use of Cr(VI) has been determined to be one of the principal contributors to unacceptably high risk levels. The EPA is using the Human Exposure Model (HEM-3) to provide a more detailed assessment, through examination of all HAP emission data from select NAVAIR activities including NAS Whidbey Island. Specifically, HAP emissions from aerospace and rework operations are under consideration and testing/evaluation of new EAP pretreatment coatings will allow for compliance with current and future Cr(VI) regulations.

4.2.4 Present Operations at NASA-KSC

The NASA Beach Corrosion Test Facility has continued to operate for several decades as the premier marine outdoor exposure testing facility. The operating environment consisting of harsh marine conditions: solar, high humidity, salt spray, rain and wind make it an ideal operating environment to monitor the effects of marine corrosion on various surfaces. These conditions provide an ideal location to measure the robustness of new coatings systems that can mimic real world sea environments. As a result of its unique location and facilities, current operations monitoring corrosion on numerous panels supplied by various agencies is ongoing. The professionals/technicians are skilled in inspection of corrosion and their interpretations are well documented by this group and facility. The ability to obtain real-time data and to monitor corrosion experiments under harsh weather conditions is a noted feature of this facility. It continues to offer operating partnerships with industry, private and government organizations to monitor corrosion in an outdoor marine environment.

4.3 Site-Related Permits and Regulations

For each facility, no site-related permits or regulations allowing for field testing or marine outdoor exposure testing was required. The only basis for testing BAM-PPV coated panels on non-critical military components or marine outdoor exposure was the ability of the EAP to meet minimum corrosion/adhesion requirements set down in the Joint Test Protocol (JTP) December 2000 [78]. Additional requirements were met with toxicology testing by MB Research Laboratory that showed BAM-PPV is a non-toxic, non-hazardous material suited for field testing by military personnel.

5.0 TEST DESIGN

During Phase I, laboratory experiments by each of the co-performers was undertaken to determine the corrosion/adhesion properties of BAM-PPV and MEH-PPV as a pretreatment coating. Also BAM-PPV and MEH-PPV were incorporated into full military coatings and subjected to a variety of accelerated weathering and adhesion tests. In addition marine outdoor and outdoor exposure tests were performed at NASA/KSC and WPAFB respectively. Toxicology tests were also investigated to determine lethal doses on experimental animals using BAM-PPV powder. The best performing military coating systems using BAM-PPV as the pretreatment were down selected for fields testing during Phase II for this completed ESTCP program. The flow chart (Figure 14) shown below provides a review of the overall execution of Phases I and II.

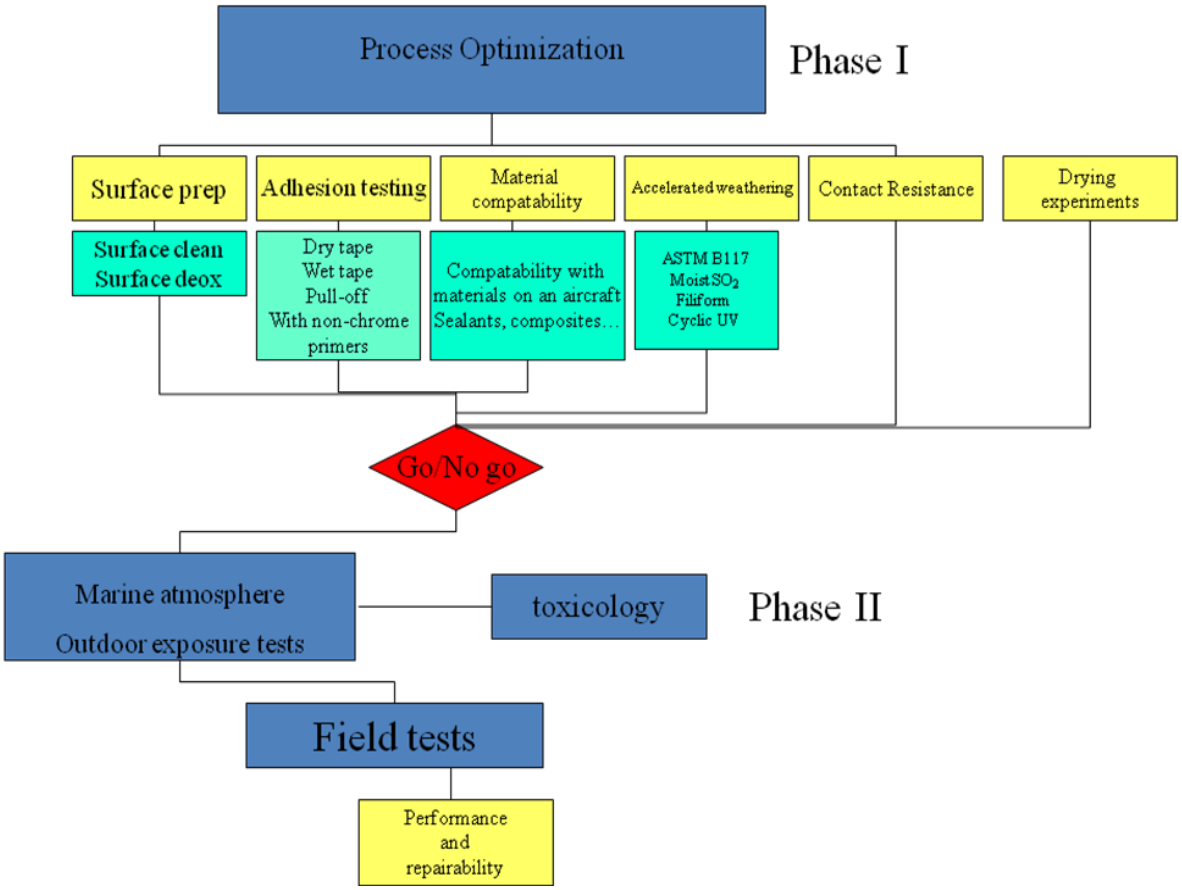


Figure 14: Flow Chart for EAP testing and Down-selection for Field Studies During Phases I and II

5.1 Laboratory Testing

No JTP was developed for this ESTCP program; rather, the JTP prepared by NAWCAD, December 13, 2000 was used as the guideline for testing the EAP pretreatment coatings [78]. The following sections describe the laboratory tests performed by each of the co-performers to evaluate the EAPs (BAM-PPV and MEH-PPV) for this study.

5.1.1 Adhesion Testing Methods

The following sections document the specific adhesion tests that were used to determine the adhesive properties of BAM-PPV and MEH-PPV pretreatments with and without full military coatings.

5.1.1.1 Crosshatch Adhesion (MIL-PRF-23377)

Crosshatch adhesion testing was performed to determine the adhesion between the substrate, pretreatment, primer, and topcoat interfaces [79]. This test was performed on (*i*) pretreated, (*ii*) primed and (*iii*) topcoated systems for AA2024-T3 aluminum and 4130 normalized steel substrates. UDRI/CTIO Laboratory Procedure CLG-LP-008, *Tape Test Adhesion*, in accordance with ASTM D 3359, *Standard Test Methods for Measuring Adhesion by Tape Test*, was used for guidance to run the test. Guidance for sample evaluation was taken from MIL-DTL-81706, MIL-PRF-23377, and MIL-PRF-85285, *Performance Specification, Coating: Polyurethane, Aircraft and Support Equipment*. Samples with pretreatment-only or pretreatment and primer only (no topcoat) were tested using the 1-millimeter spacing crosshatch blade. Topcoated samples were tested with the 2-millimeter spacing crosshatch blade. There were three replicates for each coating system on aluminum samples and one test performed per sample. However, because of limited materials, steel samples only had two replicates per coating system, so there were two tests performed per sample for a total of four readings on each coating system on steel. The ratings provide only general information concerning the overall adhesion performance of the system. The description of the rating scale, as taken from ASTM D 3359, is given in Figure 15 and the visual standard is given in Figure 16.

5B	The edges of the cuts are completely smooth; none of the squares of the lattice is detached.
4B	Small flakes of the coating are detached at intersections; less than 5 % of the area is affected.
3B	Small flakes of the coating are detached along edges and at intersections of cuts. The area affected is 5 to 15 % of the lattice.
2B	The coating has flaked along the edges and on parts of the squares. The area affected is 15 to 35 % of the lattice.
1B	The coating has flaked along the edges of cuts in large ribbons and whole squares have detached. The area affected is 35 to 65 % of the lattice.
0B	Flaking and detachment worse than Grade 1.

Figure 15: ASTM D 3359, Method B, Crosshatch Adhesion Rating Scale Description

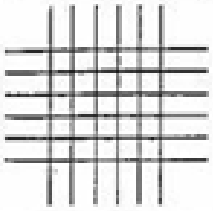
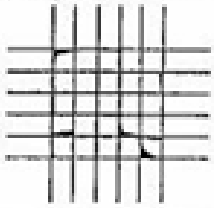
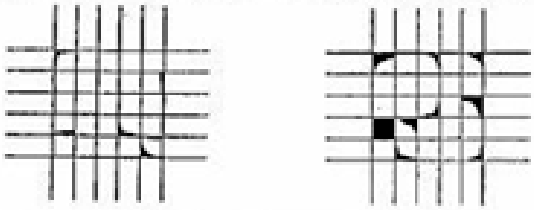
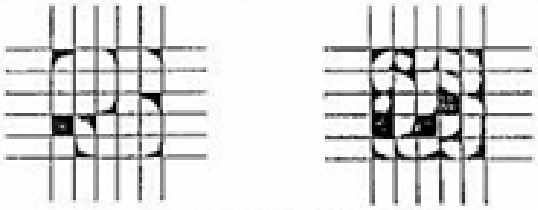
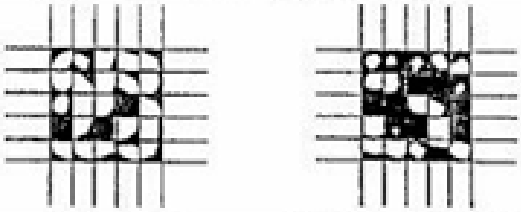
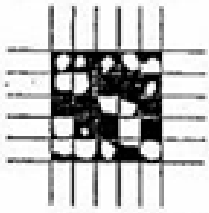
CLASSIFICATION OF ADHESION TEST RESULTS		
CLASSIFICATION	PERCENT AREA REMOVED	SURFACE OF CROSS-CUT AREA FROM WHICH FLAKING HAS OCCURRED FOR SIX PARALLEL CUTS AND ADHESION RANGE BY PERCENT
5B	0% None	
4B	Less than 5%	
3B	5 - 15%	
2B	15 - 35%	
1B	35 - 65%	
0B	Greater than 65%	

Figure 16: ASTM D 3359, Method B, Crosshatch Adhesion Visual Standard Reference Image

5.1.1.2 X-Cut Tape Adhesion (MIL-PRF-85285)

X-cut tape adhesion testing was performed according to ASTM D 3359, Method A, to determine the adhesion between the substrate, pretreatment, primer, and topcoat interfaces when the coating is greater than 5 thousandths of an inch in thickness [79]. This test was performed on repairability samples (sized 12 x 12 inches) following 2000 hours of total UVB exposure. UDRI/CTIO Laboratory Procedure CLG-LP-008 was used for guidance to run the test and to evaluate the samples. There were three replicates for each coating system, and two tests were performed per sample; one on the right side of the panel and one of the left side.

5.1.1.3 Wet-Tape Adhesion (MIL-PRF-23377, MIL-PRF-85285)

The wet-tape adhesion test is specified in MIL-PRF-23377 and MIL-PRF-85285. It is designed to measure inter-coat adhesion of an organic coating immersed in water for a short period of time. This test was performed on (*i*) primed and (*ii*) topcoated systems for AA2024-T3 substrates only. UDRI/CTIO Laboratory Procedure CLG-LP-033, Wet Tape Adhesion, conformed to Federal Test Method Standard (FTMS) 141D, *Paint, Varnish, Lacquer and Related Materials: Methods of Inspection, Sampling, and Testing*, Method 6301, *Adhesion (Wet) Tape Test*, These methods were used for guidance to run the test and to evaluate the samples (see Table 6). There were three replicates per coating system.

Table 6: ASTM D 3359 Adhesion, Method A, Rating Scale

5A	No peeling or removal of coating
4A	Trace peeling or removal along incisions or intersections
3A	Jagged removal along incisions up to 1/16 inch on either side
2A	Jagged removal along most of incisions up to 1/8 inch
1A	Removal from most of the inscribed area
0A	Removal beyond the inscribed area

5.1.1.4 Pull-Off Adhesion ASTM D 4541 PATTI

The pneumatic adhesion tensile test instrument (PATTI) pull-off test is designed to give specific information concerning both the inter-coat adhesion and the intra-coat cohesion of organic coating systems [80]. This test was performed on (*i*) primed and (*ii*) topcoated systems for AA2024-T3 substrates only. UDRI/CTIO Laboratory Procedure CLG-LP-046, Tensile Adhesion, in accordance with ASTM D 4541, *Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers*, was used for guidance to run the test and to evaluate the samples. Samples were milled with a 0.5 inch inner-diameter ring prior to testing. Samples were cleaned with methanol prior to pull-stub application rather than more aggressive solvents, to prevent adverse effects to the EAP prior to testing. Light abrasion with 240-grit sandpaper was used on primed and topcoated samples. There were three samples from each coating system, and those

samples were cut in half to produce a total of 6 replicates per coating system for this test. The reported data are numeric and are the result of the calculated pull-off strength of either the coating layers that are removed or the maximum of the adhesive. For coating systems with multiple coatings (i.e., primer followed by topcoat), a description of the nature of the coating failure is included with the reported numerical value of the pull-off. A guideline to those descriptions is given in Table 7.

Table 7: PATTI Coating Failure Descriptions

Notation	Description	Failure Mode
T/T	Topcoat on pull stub and panel surface	Topcoat–Topcoat Cohesion
T/P	Topcoat on pull stub and primer on panel surface	Topcoat–Primer Adhesion
P/P	Primer on pull stub and on panel surface	Primer–Primer Cohesion
P/S	Primer on pull stub and no visible coating on panel surface (includes failures at the conversion coating, if visible)	Primer–Substrate Adhesion
T/E	Topcoat on panel and epoxy either on panel or on stub (Epoxy failure only – no coating failure noted)	Topcoat–Epoxy Adhesion
P/E	Primer on panel and epoxy either on panel or on stub (Epoxy failure only – no coating failure noted)	Primer–Epoxy Adhesion
tension that a surface area can bear and remains intact / Pretreat	Primer on the stub and pretreatment on the panel (Specific reference to BAM-PPV which is readily visible)	Primer–Pretreatment Adhesion
Pretreat / Pretreat	Pretreatment on the stub and on the panel (Specific reference to BAM-PPV which is readily visible)	Pretreatment–Pretreatment Cohesion

Pull-off adhesion testing of BAM-PPV coatings by ARL was measured to assess the effects of pretreatment or surface preparation at the substrate. These tests were performed in accordance with ASTM D 4541. An Elcometer Model 108 Hydraulic Adhesion Test Equipment (HATE) (Figure 17) was used for this procedure. In addition to being a more quantitative test method, pull-off adhesion is also less prone to human elements in testing such as variations in pressure applied during scribing as well as interpretation and perception of results. For the pull-off adhesion test, a loading fixture commonly referred to as a "dolly" is secured normal to the coating surface using an adhesive. The adhesive used was cyanoacrylate. After allowing the adhesive to cure for 24 hours at 25 ^{o}C in ambient conditions, the attached dolly was inserted into the test apparatus. The load applied by the apparatus was gradually increased and monitored on the gauge until a plug of coating was detached. The failure value (in psi) was recorded and the failure mode was characterized. For pull-off data to be valid, the specimen substrate must be of sufficient thickness to ensure that the coaxial load applied during the

removal stage does not distort the substrate material and cause a bulging or "trampoline effect." When a thin specimen is used, the resultant bulge causes the coating to radially peel away outwards from the center instead of being uniformly pulled away in pure tension and thus results in significantly lower readings than for identically prepared specimens with greater substrate thickness. At 0.25 inches, all of the metallic panels evaluated in the test matrix had adequate thickness for valid pull-off test results. Measurements for each coating system and substrate were obtained by taking 16 measurements on each of the two panels within each set for a total of 32 replicates. Any failure measurements due to coating separation between the topcoat surface and the cyanoacrylate adhesive were rejected.

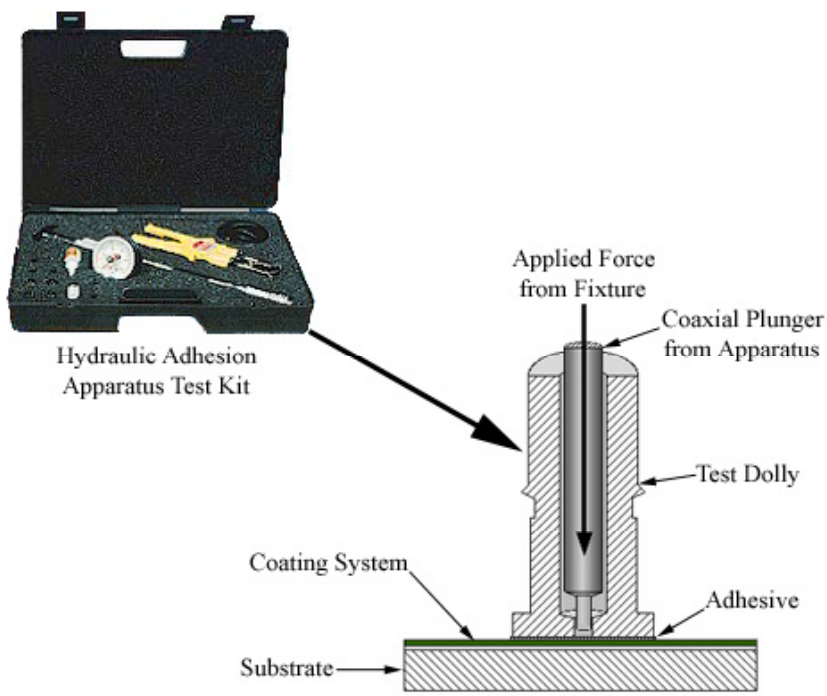


Figure 17: Hydraulic Adhesion Test Equipment and Dolly configuration

5.1.2 Acccelerated Weathering Tests

Accelerated weathering testing often referred to as "laboratory testing" are used to predict corrosion behavior of applied coatings onto various substrates prior to actual field testing. These types of tests are used as a screening mechanism to down-select the best performing coating system prior to field testing and service implementation. Accelerated weathering tests include various kinds of cabinet-controlled and autoclave-controlled environments. These tests are appropriate for quality control, materials selection, material and environmental combinations and determining the mechanisms of corrosion inhibition.

5.1.2.1 Neutral Salt Spray Exposure (MIL-DTL-81706, MIL-PRF-23377)

Neutral salt spray (NSS) exposure testing was performed to evaluate the ability of the coating systems to withstand a 5-weight percent sodium chloride solution, pH-adjusted to a range of 6.5 – 7.2 [81]. This test was performed on

- (*i*) pretreatment-only,
- (*ii*) primed, and
- (*iii*) topcoated systems on AA2024-T3 aluminum substrates.

UDRI/CTIO Laboratory Procedure CLG-LP-019, *Salt Fog Corrosion*, in accordance with ASTM B 117, *Standard Practice for Operating Salt Spray (Fog) Apparatus*, was used for guidance to run the test. All samples subject to salt spray exposure were photographed before and after the test to document the coating performance. There were three replicates per coating system. The guidance for sample evaluation was taken from MIL-DTL-81706, *Chemical Conversion Materials for Coating Aluminum and Aluminum Alloys*, and MIL-PRF-23377, P*erformance Specification, Primer Coatings: Epoxy, High Solids*. Samples that were pretreated-only were exposed for 336 hours and checked for blistering or signs of corrosion. Discoloration was not cause to reject pretreatment-only samples. Samples that were primed or primed and topcoated were scribed with an X-scribe (full size), exposed for 2000 hours, and checked for blistering, loss of adhesion, undercutting, pitting and corrosion build-up in the scribe.

The rating system that was used included all pretreatments. The criteria for pass or fail followed MIL-DTL-81706 which states that, "following 336 hours of salt spray exposure, there will be no evidence of corrosion when compared to an unexposed sample with the naked eye." Because the comparison is relative and there is no ratings scale, the rating for pretreatments is a description of the corrosion present after 336 hours.

The rating systems for primed and topcoated samples exposed to neutral salt spray are given in Tables 8-10 and are derived from ASTM methods and historical practices for military specification interpretations. **The AF has used these rating systems based on a comparative scale for corrosion data. Specifically WPAFB Logistics Systems Support Branch Office (RXSSO) has used this rating system for decades due to its easy interpretation and uniformity. This reporting has been followed by additional laboratories to make presentation and interpretation of data more easily understood by non-corrosion scientists and engineers. The rating numbers are referenced in this order:**

- **1st number = appearance,**
- **2nd number = undercutting and**
- **3rd number = blistering.**

Ratings are listed along with an interpretation rating of "acceptable", "low", or "poor". An interpretation rating of "acceptable" is minor corrosion no greater than 2. An interpretation rating of "low" is minor corrosion no greater than 2 in combination with undercutting no greater than 1 or blistering equal to 1. An interpretation rating of "poor"

is any combination of ratings for corrosion, undercutting, or blistering that is greater than the previous two mentioned. For example, a rating of 2 0 0 is considered "acceptable" performance and is comparable to control data. A rating of 2 0 1 is considered "low" performance because it is slightly less robust than the control data. A rating of 2 3 0 or 2 2 1 is considered "poor" performance because it is much less robust than the control data and does not provide comparable performance.

Table 8: RXSSO Scribe Appearance Rating System

Rating	Evaluation
0	Bright and clean
1	Staining no corrosion build up
2	Minor corrosion build up
3	Moderate corrosion build up
4	Major corrosion build up
5	Severe corrosion buildup

Table 9: RXSSO Undercutting Rating System

Rating	Evaluation
0	No lifting of coating
1	Lifting or loss of adhesion up to 1/16 in. (2 mm)
2	Lifting or loss of adhesion up to 1/8 in. (3 mm)
3	Lifting or loss of adhesion up to 1/4 in. (6 mm)
4	Lifting or loss of adhesion up to 1/2 in. (13 mm)
5	Lifting or loss of adhesion beyond 1/2 in. (>13 mm)

Table 10: Blistering Rating System (Modified from ASTM D 714)

Blister Size			Blister Frequency	
ASTM D 714 Rating	**CTIO Rating Scale**	**Evaluation**	**Rating**	**Evaluation**
10	0	None	F	Few
8	1	Very small	M	Medium
6	2	Small	MD	Medium Dense
4	3	Medium	D	Dense
2	4	Large		
0	5	Delamination		

5.1.2.2 Accelerated Corrosion Test (GM9540P)

GM9540P is an accelerated corrosion test developed by General Motors that was designed for the evaluation of assemblies and components [82]. Steel substrates often perform poorly when scribed and placed in ASTM B 117 salt spray exposure because of the nature of that accelerated test. Because the GM9540P, *Accelerated Corrosion Test*, was designed for steel substrates, this test has provided good results for the anticipated corrosion behavior of organic coating systems. This test was performed on (*i*) primed and (*ii*) topcoated systems for 4130 normalized (4130N) steel substrates only. Half of the samples were grit-blasted with 120-grit aluminum oxide prior to application of the pretreatment, and the other half of the samples were solvent-cleaned with a combination of mineral spirits wipes, Brulin 815GD detergent cleaner immersion, and methyl ethyl ketone wipes to remove surface debris prior to application of the pretreatment (no abrasive cleaning). This provided two substrate systems denoted with "grit-blasted" and "solvent-cleaned." One representative sample from each coating system was photographed at the initial point, prior to exposure to GM9540P for reference. Primed and topcoated samples received a single scribe line down the center of the panel (not an "X"), and were exposed for 40 cycles (5 cycles per week, with samples left untouched over weekends). One representative sample from each coating system was photographed at the beginning of the test, weekly for the duration of the test, and at the end of the test. There were three replicates per coating system.

For these systems, sample corrosion was rated based upon SSPC/NACE guidelines which recommend ASTM D 1654, *Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments*, for corrosion undercutting at the scribe, ASTM D 610, *Standard Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces*, for corrosion of steel in the field of the sample, and ASTM D 714, *Standard Test Method for Evaluating Degree of Blistering of Paints*, for blistering in the field of the sample. The rating scales for these ASTM standards are given in Tables 11 and 12 and Figure 18.

Table 11: Rating of Failure at Scribe (Procedure A)
Representative Mean Creepage from Scribe

Millimeters	Inches (approximate)	Rating Number
Zero	0	10
Over 0. to 0.5	0 to $\frac{1}{64}$	9
Over 0.5 to 1.0	$\frac{1}{64}$ to $\frac{1}{32}$	8
Over 1.0 to 2.0	$\frac{1}{32}$ to $\frac{1}{16}$	7
Over 2.0 to 3.0	$\frac{1}{16}$ to $\frac{1}{8}$	6
Over 3.0 to 5.0	$\frac{1}{8}$ to $\frac{3}{16}$	5
Over 5.0 to 7.0	$\frac{3}{16}$ to $\frac{1}{4}$	4
Over 7.0 to 10.0	$\frac{1}{4}$ to $\frac{3}{8}$	3
Over 10.0 to 13.0	$\frac{3}{8}$ to $\frac{1}{2}$	2
Over 13.0 to 16.0	$\frac{1}{2}$ to $\frac{5}{8}$	1
Over 16.0 to more	$\frac{5}{8}$ to more	0

Table 12: Rating Scale from ASTM D 610

Rust Grade	Percent (%) of Surface Rusted	Spots (S) (Visual)	General (G) (Visual)	Pinpoint (P) (Visual)
10	Less than or equal to 0.01 %		None	
9	Greater than 0.01 % and up to 0.03 %	9-S	9-G	9-P
8	Greater than 0.03 % and up to 0.1%	8-S	8-G	8-P
7	Greater than 0.1% and up to 0.3%	7-S	7-G	7-P
6	Greater than 0.3% and up to 1.0%	6-S	6-G	6-P
5	Greater than 1.0% and up to 3.0%	5-S	5-G	5-P
4	Greater than 3.0% and up to 10.0%	4-S	4-G	4-P
3	Greater than 10.0% and up to 16.0 %	3-S	3-G	3-P
2	Greater than 16.0% and up to 33.0%	2-S	2-G	2-P
1	Greater than 33.0% and up to 50.0%	1-S	1-G	1-P
0	Greater than 50.0%		None	

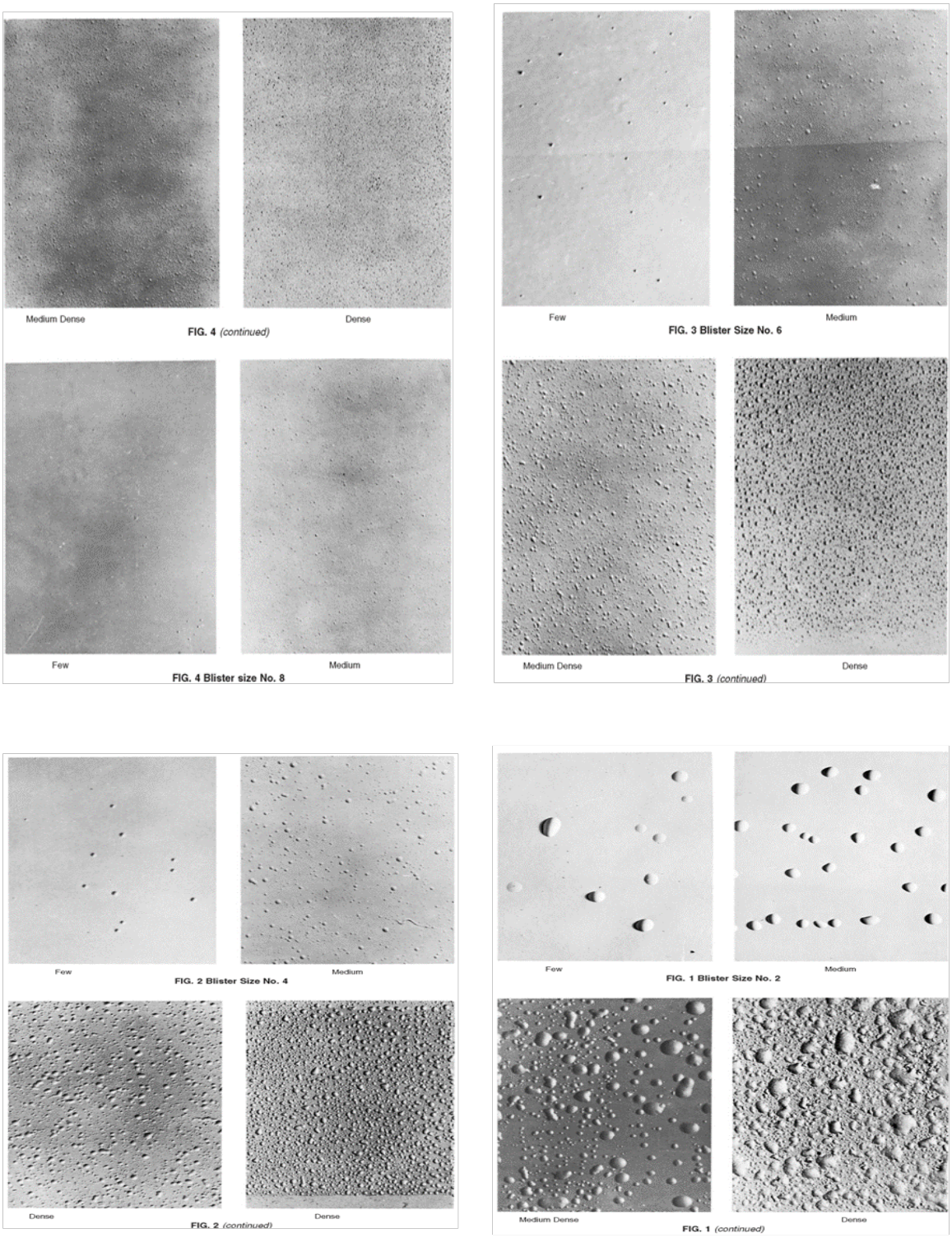


Figure 18: Rating Scale from ASTM D 714

5.1.2.3 Xenon Arc Accelerated Weathering Test, Corrosion Resistance, and Crosshatch Adhesion Test Series (ASTM G 155, ASTM B 117, ASTM D 3359)

Accelerated weathering of organic coatings by exposure to xenon arc light and a water spray provides information about the degradation of the coating matrix [83]. Following weathering, corrosion resistance may be decreased if the coating matrix has been degraded sufficiently to permit the passage of a corrosive liquid such as a 5-weight percent salt spray solution to the substrate/pretreatment interface. Additionally, inter-coat adhesion problems may become a factor following weathering if the coating matrix has degraded sufficiently to permit passage of the water in between coating layers. To provide comparative data for outdoor weathering, a test series was performed in which samples were shuffled between xenon arc exposure and salt spray exposure.

This test was performed on topcoated systems for AA2024-T3 substrates only. UDRI/CTIO Laboratory Procedures CLG-LP-036, *Xenon Arc Accelerated Weathering Test*, CLG-LP-019, and CLG-LP-008 were used for guidance to run the test. These procedures conform to ASTM G 155, *Standard Practice for Operating Xenon Arc Light Apparatus for Exposure of Non-Metallic Materials*. Both ASTM B 117 and ASTM D 3359 were used for guidance in sample evaluation. Half of the samples received an X-scribe on the lower 3 inches of the panel and were taped on all edges. Initial color and gloss measurements were taken. These samples were placed first into xenon arc exposure for 500 hours, and color and gloss measurements were taken. Samples were then exposed to salt spray for 500 hours. This represented one full rotation. Samples were continually rotated in this fashion until there was a total of 2000 hours of xenon arc exposure and 2000 hours of ASTM B 117 exposure. Samples were photographed at specific intervals to provide a visual record of sample performance. The remaining samples were taped on the edges only and received no scribe. These were also alternated between xenon arc exposure and salt spray exposure. At the completion of 1000 hours of exposure in both xenon arc and salt spray, these samples were cut in half and one half was tested for adhesion following ASTM D 3359, Method B, using the 2-millimeter spacing crosshatch blade. Remaining cut samples were returned for rotations between xenon arc and salt spray for another 1000 hours in each exposure area. At the end of testing, cut samples were again tested for adhesion following ASTM D 3359, Method B, using the 2-millimeter spacing crosshatch blade. This data is meant for comparison with outdoor exposure testing.

5.1.2.4 Moist Sulfur Dioxide (SO_2) Tests

Corrosion test panels were subjected to a 5% salt spray test with a periodic introduction of SO_2 gas directly into the chamber in accordance with ASTM G 85 Annex 4 [84]. The SO_2 gas was emitted into the test chamber for 1 hour every six hours (four times a day). The significant surface of the test panels was inclined 6º from the vertical. All test panels were exposed for 500 hours and rated in accordance with ASTM D 1654.

5.1.2.5 Filiform Corrosion Resistance of Organic Coatings Testing

This test measures the ability of a coating system to protect the substrate against the formation of filiform corrosion [85]. An “X” incision is scribed through the coating so that the smaller angle of the "X" is 30° to 45°, making sure that the coating has been scribed all the way through and into the substrate. The scribe must have a 45° bevel, and each line of the "X" should be approximately 4 inches long. The scribed coupons are placed in a desiccator containing 12 N hydrochloric acid (HCl) for one hour at 24 ± 3°C (75 ± 5°F). Within five minutes of removal from the desiccator, the coupon is placed in a humidity cabinet maintained at 40 ± 1.7°C (104 ± 3°F) and 80 ± 5 percent RH for 1,000 hours. Measure the length of any thread-like filaments at the end of the test duration and rating of pass/fail with MJ = majority of filiform and MN = minority of filiform present along scribe lines.

5.1.3 Pencil Hardness (MIL-PRF-23377, MIL-PRF-85285)

Pencil hardness testing was performed in conjunction with crosshatch adhesion testing to provide baseline data and comparative data for use with JP-8 fluid immersion testing as described by MIL-PRF-23377 and MIL-PRF-85285 [86]. This test was performed on (*i*) primed and (*ii*) topcoated systems for AA2024-T3 substrates only. UDRI/CTIO Laboratory Procedure CLG-LP-005, Pencil Hardness of Coatings, in accordance with ASTM D 3363, *Standard Test Method for Film Hardness by Pencil Test*, was used for guidance to run the test and to evaluate the samples. There were three replicates for each coating system, and the AA2024-T3 panels used in the adhesion testing outlined in Section 5.0 were the same panels used in this test. The rating system from ASTM D 3363 is given in Figure 19.

$$\underbrace{6B-5B-4B-3B-2B-B-HB-F-H-2H-3H-4H-5H-6H}_{\text{Softer} \qquad\qquad\qquad\qquad \text{Harder}}$$

Figure 19: ASTM D 3363 Pencil Hardness Rating System

5.1.4 Impact Flexibility Testing (MIL-PRF-23377, MIL-PRF-85285)

One method of testing the flexibility of organic coatings is by using the GE Impact test instrument [87]. This test was performed on (*i*) primed and (*ii*) topcoated systems for AA2024 substrates only. UDRI/CTIO Laboratory Procedure CLG-LP-016, *GE Impact Flexibility Test*, in accordance with ASTM D 6905, *Standard Test Method for Impact Flexibility of Organic Coatings*, and MIL-PRF-85285 were used for guidance to run the test and to evaluate the flexibility of the samples. There were three replicates per coating system. Impact flexibility was tested by using a GE Model 172 reverse impact tester. Both ends of the indenter were dropped onto the test sample in reverse impact mode. The raised indentations produced on the sample were examined for signs of cracking and coating failure. The flexibility of the coating was determined by a visual examination of the coating at a magnification of 10X.

5.1.5 Hydraulic, Lubricating Oil, and JP-8 Fluid Resistance (MIL-PRF-83282, MIL-PRF-23699, MIL-DTL-83133, MIL-PRF-23377, MIL-PRF-85285)

Fluid resistance testing is performed to determine what effect can be expected by common aerospace fluids that come in contact with organic coatings. Hydraulic fluid and lubricating oil resistance tests were performed on

- (*i*) primed
- (*ii*) topcoated systems for AA2024-T3 substrates only.

JP-8 fluid resistance testing was performed on topcoated systems for AA2024-T3 substrates only. UDRI/CTIO Laboratory Procedures called out in CLG-LP-023, *Fluid Immersion—Hydraulic Fluid*, CLG-LP-024, *Fluid Immersion—Jet Fuel*, and CLG-LP-025, *Fluid Immersion—Lubricating Oil*, and conforming to MIL-PRF-23377 and MIL-PRF-85285, were used as the guidance documents to run the test and to evaluate the samples. The fluids were:

- MIL-PRF-83282, *Performance Specification, Hydraulic Fluid, Fire Resistant, Synthetic Hydrocarbon Base, Metric, NATO Code Number H-537*, at 66 ± 3°C,

- MIL-PRF-23699, *Performance Specification, Lubricating Oil, Aircraft Turbine Engine, Synthetic Base, NATO Code Number O-156*, at 121°C, and

- MIL-DTL-83133, *Detail Specification, Turbine Fuel, Aviation, Kerosene Type*, JP-8 jet fuel (without the +100 additive) at room temperature.

Initial crosshatch and pencil hardness values were taken from replicates exposed to the appropriate temperatures for the hydraulic fluid and lubricating oil tests and there were three replicates per coating system per test fluid.

5.1.6 Repairability Testing (T.O. 1-1-8, Table 3-2; MIL-R-81294; MIL-PRF-25134; and TT-R-2918)

Repairability testing focused on the behavior of the EAP when applied as a repair coating to an existing system. Guidance for this test was taken from T.O. 1-1-8, *Technical Manual: Application and Removal of Organic Coatings, Aerospace and Non-Aerospace Equipment*, MIL-R-81294, *Military Specification Remover, Paint, Epoxy, Polysulfide, and Polyurethane Systems*, MIL-PRF-25134, *Performance Specification Remover, Paint and Lacquer, Solvent Type*, and TT-R-2918, *Federal Specification Remover, Paint, No Hazardous Air Pollutants* (HAP). The assumption was made that the standard system found on many USAF aircraft is the pretreatment conforming to MIL-DTL-81706 or MIL-C-5541, *Detail Specification Chemical Conversion Coatings on Aluminum and Aluminum Alloys*; chromated epoxy primer conforming to MIL-PRF-23377; and polyurethane topcoat conforming to MIL-PRF-85285. This coating system was targeted for repair or replacement with non-chromated coatings in this test program

by changing either the pretreatment or both the pretreatment and the primer in the coating system. One alternative coating system was BAM-PPV pretreatment, replacing chromate conversion coatings in the standard system.

Another alternative coating system was MEH-PPV pretreatment, replacing chromate conversion coatings in the standard system. A third alternative was BAM-PPV pretreatment and the MIL-PRF-23377 qualified non-chromated epoxy primer Deft Inc. 02-GN-083 (Deft 02-GN-083) replacing chromate conversion coatings and MIL-PRF-23377 chromated primer in the standard system. A final system was MEH-PPV pretreatment and the MIL-PRF-23377 qualified non-chromated primer Deft 02-GN-083 replacing chromate conversion coatings and MIL-PRF-23377 chromated primer in the standard system. All of the alternative coating systems were topcoated with Deft Inc. Defthane ELT 99-GY-001 color 36173 (Deft ELT 99-GY-001), qualified to MIL-PRF-85285. These systems were applied to AA2024-T3 bare 0.032-inch substrate sized 12 x 12 inches, cured for 14-days at standard conditions of 77°F and 50% relative humidity, and subject to selective removal and recoat procedures as described in this section. The goal was to identify coating system combinations for repairability of current systems with the alternative coating systems and of an alternative system with itself.

Finite accelerated weathering cabinet space, each 12 x 12 inch sample had two "primary" systems applied on 6 x 12 inch portions of the panel (Figure 20).

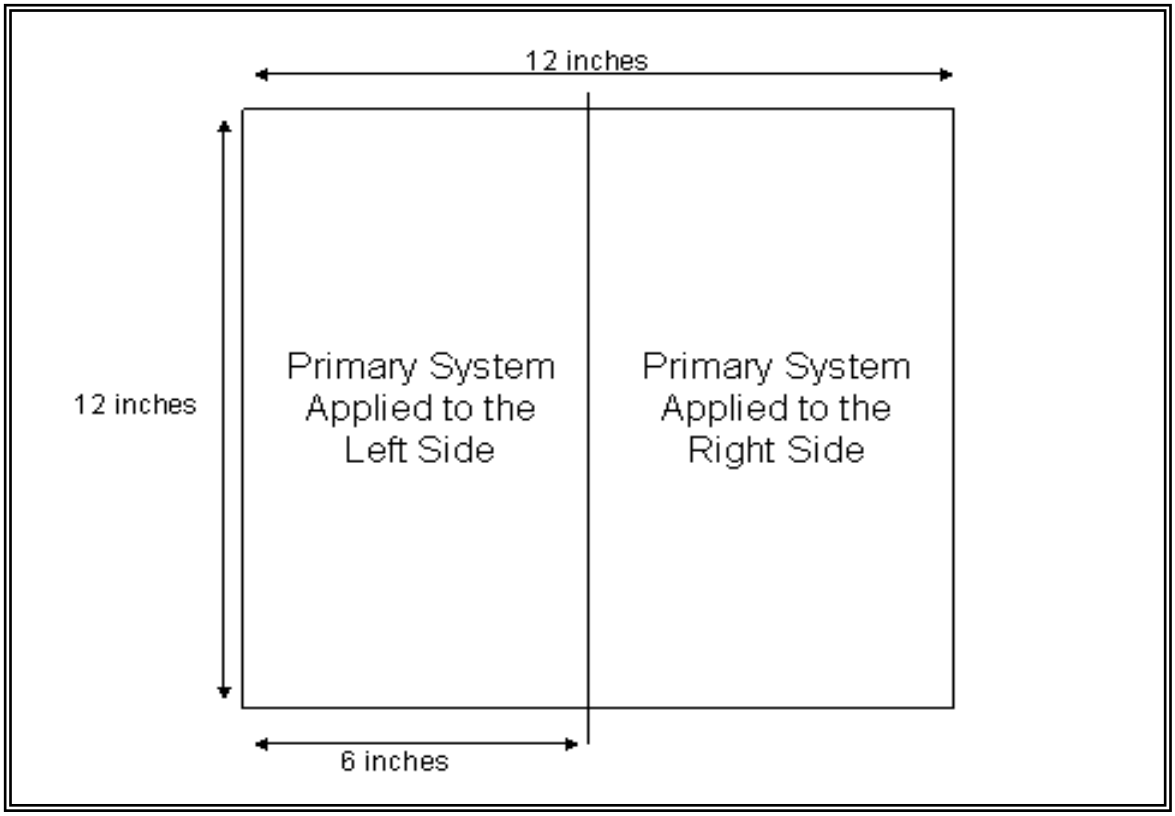


Figure 20: Primary Coating System Application for Repairability Samples

Color and gloss data was taken on the samples prior to testing (initial point). Samples were then exposed to accelerated weathering through UVB exposure for 1000 hours, and color and gloss data was taken (1000-hour exposure point). Each sample was sanded down using an orbital sander to remove the coating in layers so that the final result was bare substrate exposed in one band, primer exposed in one band, and topcoat exposed in the other band (approximately in thirds; Figure 21). Samples were wiped clean with 1-chloro-4-(trifluoromethyl)benzene (Oxsol-100) and then methyl ethyl ketone. The systems were applied in a stack-up, with care taken to apply pretreatment only to the bare substrate portions of the sample followed by primer and topcoat application over the entire sample. Samples were left to cure for 14 days at standard laboratory conditions, and color and gloss data was taken (1000-hour post-repair point). Samples were returned to UVB exposure for another 1000 hours, and color and gloss data was taken (2000-hour exposure point). Samples were tested for pencil hardness, ASTM D 3363 and adhesion following ASTM D 3359, Method A.

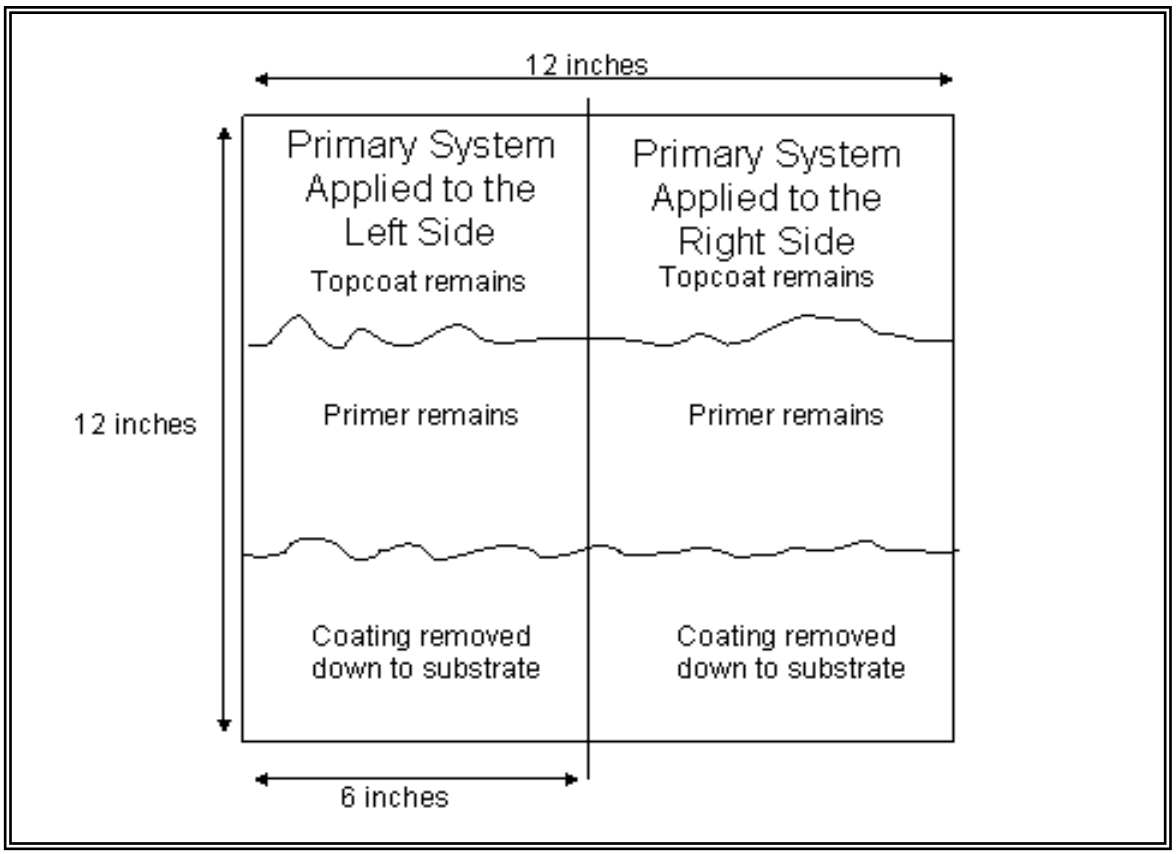


Figure 21: Coating System Removal for Repairability Testing

5.2 Outdoor Exposure Testing

Because accelerated testing can only give an estimate as to the corrosion resistance, degradation and adhesion characteristics of a coating system, outdoor weathering is included in a battery of exposure tests to determine the effect natural weather patterns and environmental exposure will have on a coating system [88]. These data along with accelerated corrosion and weathering data provide more detail to the anticipated performance of the EAP coating system prior to field testing. This test was performed on topcoated systems for AA2024-T3 substrates only. ASTM D 1014, *Standard Practice for Conducting Exterior Exposure Tests of Paints and Coatings on Metal Substrates*, was used for guidance to run the test and to evaluate the samples. Samples were scribed with an X-scribe (full size) prior to exposure. To prevent loss of the panel number during exposure, a label was applied to the backs of each sample prior to exposure. There were three replicates per coating system. Samples were rated and photographed every month for the first six months and then every three months thereafter until the end of the test.

5.2.1 Marine Outdoor Exposure Testing

The NASA Beach Corrosion Test Facility was used as the site for marine outdoor exposure testing. Full military X-scribe (full size) coatings are exposed to solar, high humidity, salt spray, rain and wind. The beachfront laboratory has the capability to provide real-time data access to monitor corrosion experiments and surrounding weather conditions. Typical evaluations are similar to laboratory methods and the list is provided below following ASTM methods:

- Corrosion from the Scribed Surface (ASTM 1654)
- Degree of Rusting on Painted Steel Surface (ASTM D 610)
- Degree of Blistering of Paints (ASTM D 714)
- Color in CIE Lab format (ASTM D 2244)
- Gloss (ASTM D 523)
- Overview photo-documentation of the samples.

5.3 Toxicology Testing

Toxicology testing was performed on laboratory animals using BAM-PPV powder as the unknown material to determine its potential lethality in the workplace. Since most inhalation and skin absorption are the normal routes by which workplace chemicals enter the body, several tests were performed using BAM-PPV. The most relevant from the occupational exposure viewpoint are the inhalation and skin application tests. However, the most frequently performed lethality study is the oral LD_{50}. This test and similar tests are performed because giving chemicals to animals by mouth is much easier and less expensive than other techniques. The results of these oral, dermal and inhalation studies are important for drugs, food poisonings and accidental domestic poisonings. Oral occupational poisonings might occur by contamination of food or cigarettes from unwashed hands and by accidental swallowing. Therefore, the following tests were performed on BAM-PPV powder to determine its potential toxicity in a workplace environment.

5.3.1 Acute Oral Toxicity-Up and Down Procedure (UDP) Standard Protocol Number 1010-02

This test was used to determine the potential for toxicity of the test article (BAM-PPV) when administered orally [89]. The dosing will be for one animal (rat) at 2000 mg/kg. If the first animal dies, conduct the main test to determine the LD_{50} (lethal dosage for 50% of the population). If the first animal dosed at 2000mg/kg survives, 4 animals will be sequentially dosed (a total of 5 animals are tested). If three animals die, the limit test is terminated and the main test is performed. The limit LD_{50} calculation is based on the following outcome: the LD_{50} is less than 2000 mg/kg when three or more animals die or the LD_{50} is greater than 2000 mg/kg when three or more animals survive.

5.3.2 Acute Dermal Toxicity/LD_{50} In Rabbits Standard Protocol Number 1100-02

This test determined the potential for toxicity of the test article (BAM-PPV) when applied dermally [90]. The test article was administered dermally to the prepared site on the dorsal area of the trunk of each animal (rabbit). The test limit of 2000 mg/kg of body weight at three dose levels was administered. Doses are selected in an attempt to achieve a range of toxic effects and mortality rates and to produce a dose-response curve and permit an acceptable estimation of the medium lethal dose. For pesticide products, dose levels may be selected in correlation with the OPP toxicity categories. In this case, determination of an LD_{50} may not be necessary. A confirming group of 5 rabbits of the opposite sex will be dosed at the LD_{50} to determine if one sex is more sensitive to the test article (BAM-PPV). If the LD_{50} in the opposite sex indicates increased sensitivity, an additional LD_{50} in that sex will be considered.

5.3.3 Acute Eye Irritation in Rabbits Standard Protocol 1200-02

This test determined the irritant or corrosive effects, if any, of a test article (BAM-PPV) when instilled into the rabbit's eye [91]. The rabbit is the system of choice because it has been shown to be sensitive to the irritant and corrosive effects of a variety of chemicals and it is a standard animal model for ocular irritation studies. The test article (BAM-PPV) was instilled into the conjunctival sac of the rabbit's eye. A solid sample was ground to a fine dust and dosed as a 0.1 mL equivalent, not to exceed 100 mg. The weight of 0.1 mL equivalent was recorded and the treated eye of each animal was examined and scored for irritation of the cornea, iris and conjunctiva at 1, 24, 48 and 72 hours post-dose using a hand-held source of illumination.

5.3.4 Acute Dermal Irritation in Rabbits Standard Protocol 1130-02

This test did determine the irritant or corrosive effects (if any) of a test article (BAM-PPV) when applied dermally [92]. The rabbit is the system of choice because it has been shown to be sensitive to the irritant and corrosive effects of a variety of chemicals and is a standard animal model for acute dermal irritation studies. The day prior to the test article application (BAM-PPV), the fur was clipped from the dorsal area of the trunk of each animal. The clipped area was at least 10 x 10 cm and remained intact. Solids were moistened slightly with water or other suitable vehicle to form a pasty consistency to ensure good contact with the skin. The amount of liquid used to moisten the test article (BAM-PPV) was documented. If considered necessary, solids may be pulverized. The dose will be based on the dry weight of the test article (BAM-PPV). The test article (BAM-PPV) was covered with a four-layered 2.5 x 2.5 cm surgical gauze patch. The test article (BAM-PPV) was then applied to the prepared site under the gauze patch and gentle pressure applied. The patch was secured with non-irritating tape and then scored for dermal irritation at 60 minutes after patch removal, and again at 24, 48, 72 hours after patch removal. Erythema and edema was scored according to the Draize Scale shown in the following Tables 13 and 14. The skin was evaluated for ulceration and necrosis, or any evidence of tissue destruction. Additional signs of dermal irritation were also described.

Table 13: Draize Scale for Erythema

No erythema	0
Very slight erythema (barely perceptible)	1
Well-defined erythema	2
Moderate to severe erythema	3
Severe erythema (beet redness) to slight eschar formation (injuries in depth)	4

Table 14: Draize Scale for Edema

No edema	0
Very slight edema (barley perceptible)	1
Slight edema (edges of area well-defined by definite raising)	2
Moderate edema (raised approximately 1 millimeter (mm))	3
Severe edema (raised >1 mm and extending beyond area of exposure)	4

5.3.5 Delayed Contact Dermal Sensitization Test-Buehler Standard Protocol Number 1160-03

This test determines the potential of a product (BAM-PPV) to promote skin sensitization reactions after repeated applications [93]. Contact dermal sensitization is an immunological process where the host animal, through repeated skin exposure, acquires a specific sensitivity (reactivity) to the substance (in this case BAM-PPV). In the Buehler guinea pig model, contact dermal sensitivity is manifested as increased erythema. The guinea pig is the system of choice because it is a standard animal model for the dermal sensitization studies and is the species preferred by the EPA. The day prior to the test article application (BAM-PPV), the dorsal area of each screen animal (20 animals per test

article and positive control groups for statistically relevant data) was clipped free of hair with an electric clipper. The clipped area was approximately 10 x 10 cm. If a solid is used at 100% concentration, it was moistened with 0.1mL of DI water in order to facilitate contact with the test site. The test article (BAM-PPV) was applied to each site under a 25mm Hilltop Chamber with or without a cotton pad, depending upon the nature of the test article or the sponsor's instructions. The use of the cotton pad was documented in the study file. The treated sites of each animal was examined and scored at 24 and 48 hours following patch removal for both the induction and the challenge phases. Redness was also evaluated using the following scoring code found in Table 15. The duration of the sensitization phase was 31 days unless extended for re-challenge at the request of the sponsor (NAWCWD) (not requested).

Table 15: Scoring Code

No reaction	0
Very faint erythema, usually non-confluent	0.5
Faint erythema, usually confluent	1
Moderate erythema	2
Strong erythema, with or without edema	3

5.3.6 Acute Inhalation Toxicity/LC_{50} in Rats Standard Protocol Number 1300-02

The objective of this test was to provide information on the health effects which may arise from the short term exposure by the inhalation route [94]. The rat is the species of choice because it has been shown to be sensitive to toxic effects of a variety of chemicals, is a standard animal model for inhalation toxicity tests and is the referred species specified in the regulatory requirements. The exposure to the test article (BAM-PPV) will be via inhalation, an exposure period of 4 hours at 2.0 mg/L with Mass Median Aerodynamic Diameter (MMAD) particle size of between 1 and 4 micrometers or where this is not possible due to physical or chemical properties of the test substance, the maximum attainable concentration with the smallest particle size. If zero or one animal dies during the limit test and the exposure concentration of the test article (BAM-PPV) is greater than either 2.0mg/L or the maximum attainable concentration, no LC_{50} will be conducted in order to avoid the unnecessary use of laboratory animals. If two or more animals die following the limit exposure, an LC_{50} may be conducted with sponsor authorization. At least three exposure concentrations will be used and spaced appropriately to produce test groups with a range of toxic effects and mortality rates. The more sensitive sex will be determined by the limit test, and they will be used in groups of 5 per sex. The data should be sufficient to produce a dose-responsive curve and permit an acceptable estimation of the median lethal concentration. Test animals (rats) will be

monitored at hourly intervals during the exposure period and one-hour post-exposure for toxicity, pharmacological and behavioral effects. Beginning with the day immediately following the day of exposure to the test article (BAM-PPV), animals will be observed once daily for 14 days. The observations will include, but will not be limited to, evaluation of the skin and fur, eyes and mucous membranes, respiratory and circulatory effects, autonomic effects such as salivation, central nervous system effects including tremors and convulsions, changes in the level of activity, gait and posture, reactivity to handling or sensory stimuli, altered strength and stereotypes or bizarre behavior. All animals will be observed twice daily (AM & PM) for mortality. **This test was not performed, see page 240!**

5.4 Field Testing

Each service field tested the best performing full military coating down-selected from Phase I for **a one-year field** test during Phase II.

5.4.1 Field Testing at WPAFB

The test components (C-5 cargo rear hatch door) were monitored for 12 months in field service conditions as determined by C-5 Systems Group and mission requirements. Because of the proximity of the 445th Airlift Wing to AFRL/RX operations, the preparation of the test articles was performed with direct assistance from AFRL. The C-5 rear hatch door (see Figure 10) was removed from the aircraft and transported to the CTIO facility a short distance away and coated there by AFRL personnel. The door was re-affixed to an operational aircraft and monitored for adhesion behavior, corrosion resistance and color retention.

5.4.2 Field Testing at ARL

This specific piece of hardware (headlight cover on Bradley Vehicle) was selected due to its non-critical military hardware status and ease of visual inspection and removal during maintenance cycles. The Bradley vehicle headlight cover was sand blasted prior to coating to achieve the appropriate surface roughness. Solutions were then applied onto the substrate using HVLP spray equipment, coated with MIL-DTL-53022 non-Cr(VI) primer and top coated with MIL-DTL-64159 (waterborne chemical agent resistant coating,(CARC) polyurethane topcoat). The vehicle underwent field testing on the ARL outdoor weathering test tract in Maryland and exposed to rain, snow, sleet, sun, wind, coastal moisture and humidity. Visual inspections for corrosion, adhesion, color change, gloss, chalking, cracking, peeling and alligatoring were performed every three months during the one-year field test experiment.

5.4.3 Field Testing at FRC

Bomb hoists and towbars were pretreated via HVLP spray gun with appropriate EAP solution, sprayed via HVLP and then primed (MIL-PRF-23377N) and topcoated with MIL-PRF-85285. The parts were put into service and monitored monthly via visual

inspection for corrosion, chipping, abrasion and delamination of coating during normal operations.

6.0 Performance Assessment

6.1 Laboratory Testing

All laboratory testing was carried out independently by each organization. The following labs: WPAFB, NAWCWD/NAWCAD and ARL in accordance with their specific testing are listed independently to allow the reader full understanding of what tests were conducted. The best-performing EAP system was down-selected for field testing and evaluation. The tests described in Sections 5.1-5.2.1 determined the effectiveness of BAM-PPV and/or MEH-PPV for corrosion inhibition and adhesion. Critical points are highlighted in bold type, tables or bullets to help the reader identify key elements of the test and the results.

6.1.1 Laboratory Testing at WPAFB

The results from WPAFB laboratory testing of BAM-PPV and MEH-PPV are found in sections 6.1.1 – 6.1.1.2.4.3. Bulletized summations for WPAFB laboratory testing are found in sections 6.1.1.1.5 and 6.1.1.2.5 respectively. These extensive studies have proven that BAM-PPV is a more processable, robust, stable compound than MEH-PPV and was therefore selected for field testing.

6.1.1.1 BAM-PPV Laboratory Testing

The following sections will describe in detail the tests performed on BAM-PPV solutions, films, coatings and its performance relative to control samples.

6.1.1.1.1 Solubility Studies

Because of the chemical structure of BAM-PPV (Figure 5) and the ability of xylenes to dissolve the material, both the chemical structure and the physical property data for various solvents were used to determine which materials might dissolve the BAM-PPV and generate a viable coating. The solvents and their solubility characteristics from the literature are given in Table 16. The solutions were left overnight to determine the long-term solubility behavior of the material.

For most of the solutions, the BAM-PPV did not dissolve well enough to remain in solution after 24 hours and, generally, showed phase separation between “soluble” BAM-PPV and solvent. These solvents were removed from further testing. More successful solutions included the use of *p*-xylene, Oxsol-100, or several of the solvent blends of Oxsol-100 and either acetone or methyl acetate. However, with the solvent blends, the BAM-PPV partially solidified and could not be made to re-dissolve upon addition of fresh solvent, even when heat and stirring were applied. This behavior suggested that either the solidified form of BAM-PPV did not retain all of the properties of the powder form as supplied or that the BAM-PPV solidified material contained impurities from either the solvent or from the powder as-supplied, such that the material

was partially resistant to solvent. This behavior was also seen with the Oxsol-100 mixture, but to a lesser extent and, although this was a concern, it was not considered detrimental enough to eliminate Oxsol-100 from further testing. Those solutions that demonstrated initial potential were tested (see Table 16), both in the heated state and at ambient temperature, for the generation of a film using a #3 draw-down bar, which generated the thinnest film possible using this technique. The films were ranked visually for the consistency of the coating produced and the evaporation rates of the solvents, both of which were criteria for the potential application of a film using high volume low pressure (HVLP). Those systems that demonstrated best performance were applied later with an airbrush to determine spray-ability and for comparison with the film generated by draw-down for visual consistency of the film.

From this testing, BAM-PPV in *p*-xylene mixed at room temperature for 2–4 hours produced the most continuous draw-down film and, because of the thin nature of the coating, evaporated rapidly enough to prevent running and sagging when applied by the airbrush. This system, then, was identified as the non-VOC-compliant baseline material to which all other VOC-compliant mixtures could be compared. The Oxsol-100, mixed at 60–100 °C for 2–4 hours produced a similar film when applied at room temperature, both with the draw-down bar and with the airbrush, though it produced very fine particles of undissolved material during spray-out. Because these two systems demonstrated the best overall performance, they were down-selected for additional testing. The chemical structures for both solvents are given in Figure 22. Because no additional solubility testing was performed once a system was identified that could be tested using HVLP application, no further data was available on the solubility of BAM-PPV in the various solvents or on the "clumping and agglomerative" behavior of the BAM-PPV in Oxsol-100 over time. Solubility concerns such as these can be investigated further as the project progresses into other phases of product development.

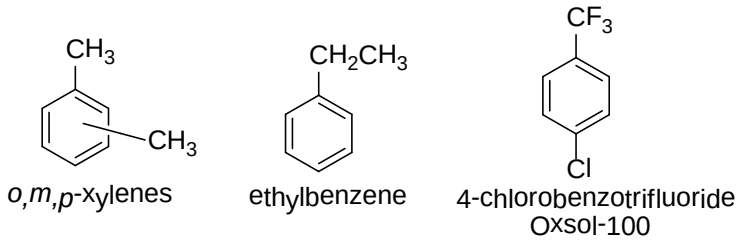


Figure 22: Chemical Structures of xylenes, ethylbenzene and Oxsol-100

Table 16: Solubility Data for BAM- PPV

Solvent	CAS Number	Evaporation Rate (compared to butyl acetate)	Mixing Time (hours)	Heated (Yes / No)	Draw-Down Film – Visual Description
m-xylene	108-38-3	0.71	4	No	discontinuous
p-xylene	106-42-3	0.60	4	No	clean film, good consistency
α-chloro-*p*-xylene	95-72-7	~ 0.80	4	No	undissolved matl
1-chloro-4 (trifluoromethyl)benzene (aka. Oxsol-100)	98-56-6	0.90	2 to 4	Yes	clean, continuous
Solvent Blend 50/50 methyl acetate/ Oxsol-100	79-20-9 98-56-6	N/A	24	Yes	solvent flash-off too fast / polymerized in vial
Solvent Blend 70/30 acetone/ Oxsol-100	67-64-1 98-56-6	N/A	24	Yes	solvent flash-off too fast / polymerized in vial
Solvent Blend 30/70 acetone/ Oxsol-100	67-64-1 98-56-6	N/A	24	Yes	solvent flash-off too fast / polymerized in vial
cyclopentanone	67-64-1	no data	24	No	undissolved matl
tetrahydrofuran	109-99-9	no data	24	No	undissolved matl
toluene	108-88-3	2.24	1 to 2	Yes	no solubility
N-methyl-2-pyrolidinone	872-50-4	no data	1 to 2	Yes	no solubility
Solvent Blend 75/25 xylenes/ ethyl benzene	1330-20-7 100-41-4	~ 0.80	1 to 2	Yes	clean film, some undissolved matl
acetone	67-64-1	5.6	1 to 2	No	no solubility

6.1.1.1.1.1 HVLP Application and Panel Preparations

From the extensive solubility testing, two solvents were chosen for solubilizing BAM-PPV and MEH-PPV. The BAM-PPV and MEH-PPV solutions were coated onto various substrates using HVLP spray. The coated coupons were tested for application, process, adhesion and corrosion resistance testing. These two solvents were *p*-xylene and

Oxsol-100, the former being considered the control system and the latter being used as the VOC-exempt solvent system for aerospace applications.

During the course of these laboratory studies, all co-performers (AF, Army, and NAWCWD/NAWCAD) have demonstrated application of BAM-PPV solution on various metal substrates using either an air brush or HVLP spray gun without any need for a clean room. Figure 23 shows the paint booth only (no spray gun). The coating process at NAWCWD is operator friendly without requiring extensive training of personnel and high levels of cleanliness. The HVLP spray gun and booth are available at all DOD depots and laboratories specializing in coatings evaluations.

Figure 23: Paint Booth used for Spray gun application of BAM-PPV at NAWCWD

6.1.1.1.1.2 Panel Preparation

The testing centered on identifying the ideal concentration (weight percent) of BAM-PPV in solvent and the proper dwell time for that material to create a usable coating. Several additional application parameters were investigated to generate the optimal coating thickness and the required cure process, all of which would generate a viable coating with good adhesion and that can withstand 1 week of ASTM B 117 neutral salt spray exposure for aluminum substrates and 5 cycles of GM9540P for steel. Testing was performed on several combinations of these variables, with down-selection steps to determine which variables were of primary importance and which were negligible. The BAM-PPV material was provided by NAWCWD in small batches (between 2 and 10 grams available per batch). Because of this, samples were prepared as material became available rather than in one large test matrix. To simplify description of these various

tests, the data are summarized and discussed in the following subsections and not necessarily in chronological order of the experiments performed. .

6.1.1.1.1.3 Solution Concentration and Coating Thickness Parameters

From the literature and discussions with NAWCWD, the lower limit for the coating thickness was estimated to be 0.8–1 µm. Because no previous application data was available for process parameters using HVLP paint gun applications, the production of sufficiently thick coatings without application defects (such as fish eyes, sagging, etc.) was the first parameter for testing. Samples that were successfully produced on substrates were then tested for crosshatch adhesion and salt spray resistance to determine next steps in the development process. Initial experiments used 1 wt% BAM-PPV in Oxsol-100 to produce coatings on AA2024-T3, AA7075-T6, and 4130N steel using HVLP spray application and 1, 2, or 3 cross-coats. HVLP paint application settings are given in Table 17. These samples were tested for crosshatch adhesion and ASTM B 117 salt spray resistance to determine the ability of the coating to remain adhered to various substrates as well as to provide corrosion resistance. SEM micrographs were taken of the coated aluminum samples to collect surface morphology data, and the micrographs were analyzed using ImageProPlus image analysis software to determine the approximate coating thickness of the samples. Because of the very thin nature of the coatings, accurate thickness of the thinnest coatings was difficult to obtain. Approximate values for the thin coatings (1 cross-coat) gave a thickness of less than 0.2 µm, and for the thicker coatings (3 cross-coats) values were between 0.5 and 0.7 µm (Figures 24 and 25). The crosshatch adhesion data demonstrated that there was no measurable difference in the adhesion characteristics of the three thicknesses for any of the substrates and that the material demonstrated excellent adhesion in all cases (all samples rated 5B in ASTM D 3359 using a 1-mm grid). However, neutral salt spray and GM9540P testing determined that the thickness of the material was the key factor to its corrosion resistance properties and that those samples that were thicker (i.e., 3 cross-coats in this case) lasted longer in salt spray. Additionally, none of these samples demonstrated sufficient corrosion resistance, and all were corroded severely after 48 hours (Figures 26 and 27).

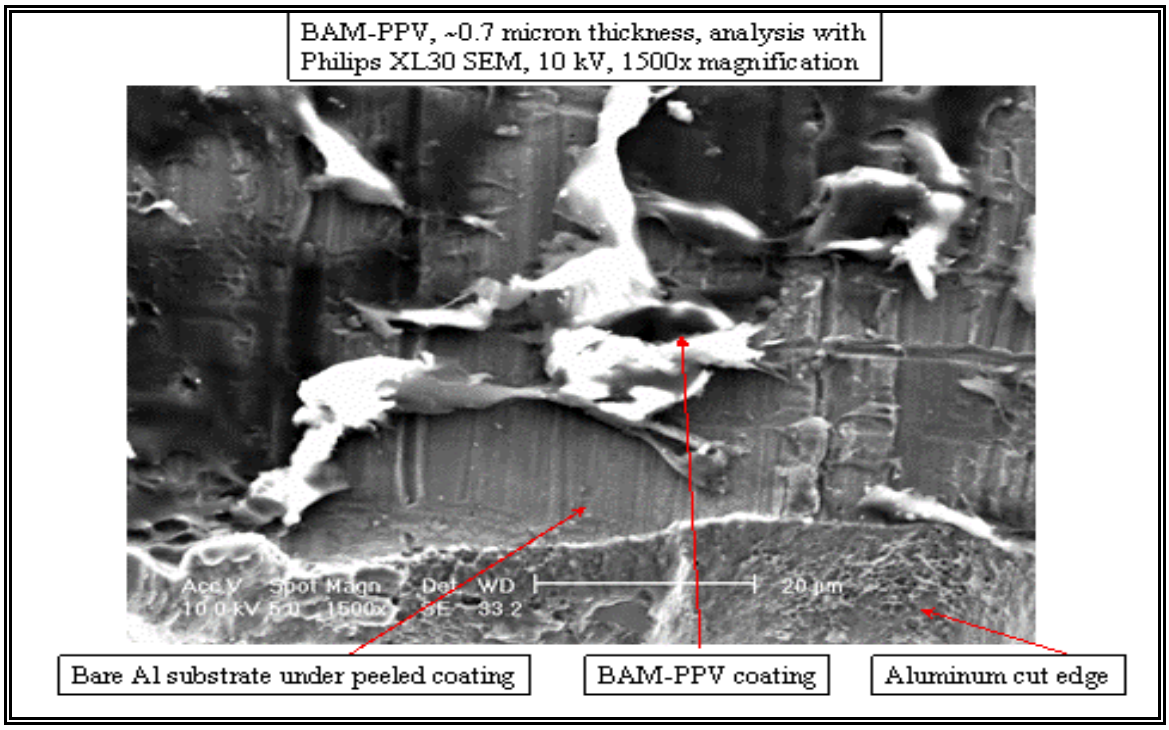


Figure 24: Surface Profile of BAM-PPV Coating Applied at Approximate Thickness of 0.7 µm using Oxsol-100 as the Solvent

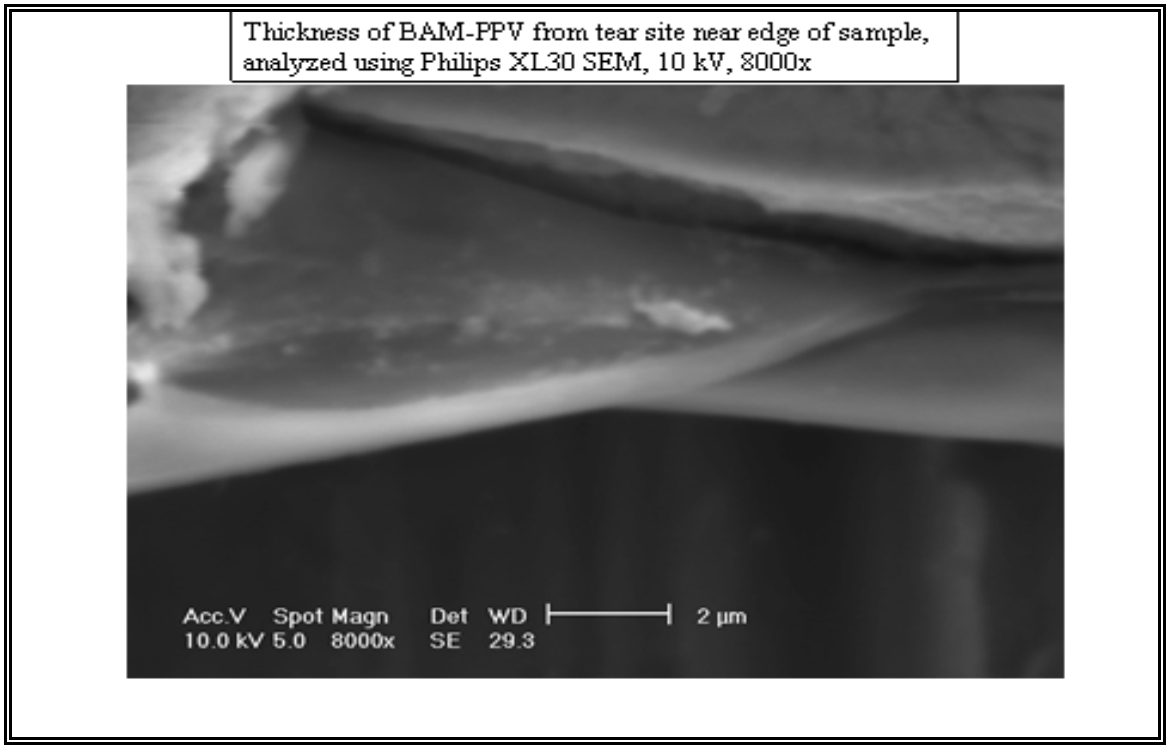


Figure 25: Edge of BAM-PPV at Tear, Approximation of Thickness at 0.7 µm

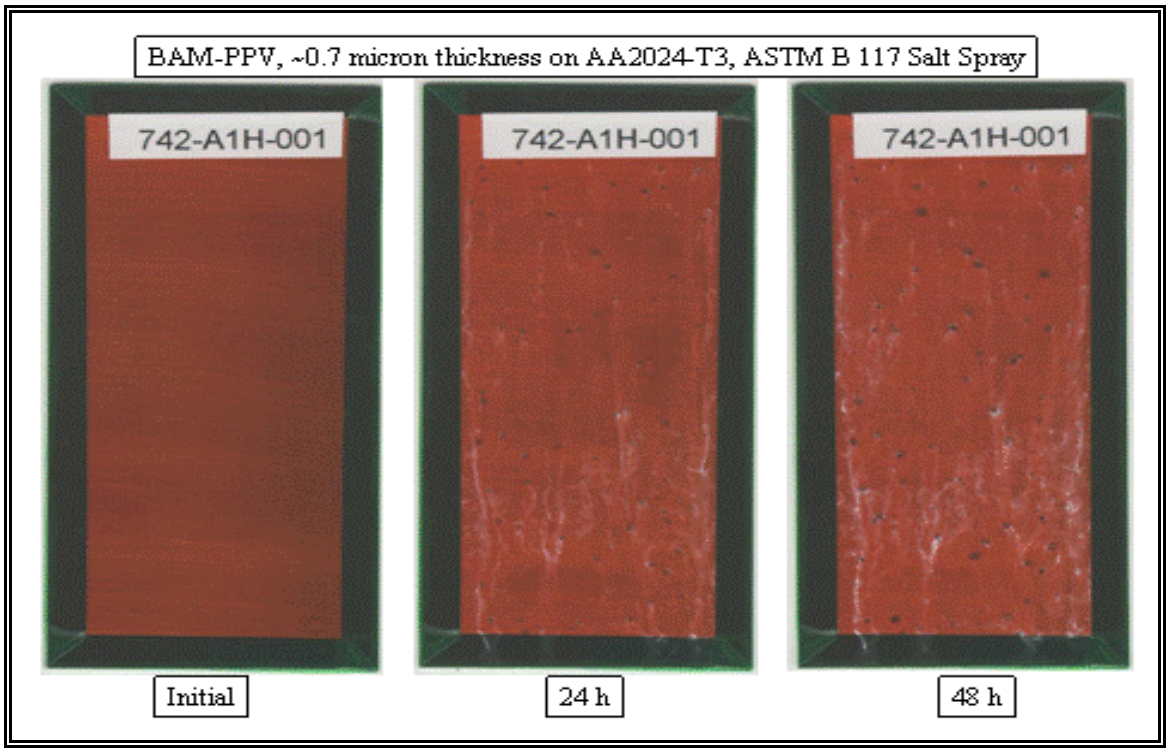


Figure 26: Salt Spray Samples, 1 wt% BAM-PPV; 3 Cross-coats; AA2024-T3 Substrate.

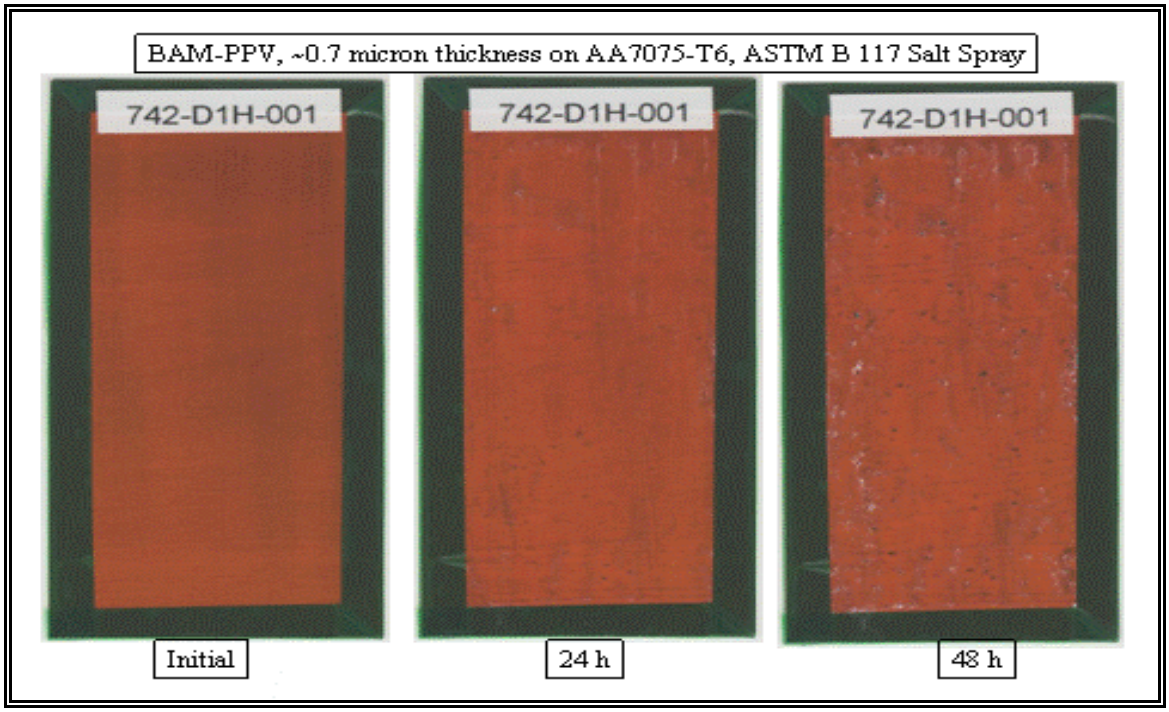


Figure 27: Salt Spray Samples, 1 wt% BAM-PPV; 3 Cross-coats; AA7075-T6 Substrate.

Continued evaluations of EAP coating thicknesses had verified that a thicker coating, in the range of 1.5–2 microns, was necessary for corrosion resistance. To that end, additional experimentation with 1 wt% and 3 wt% solutions of BAM-PPV in either Oxsol-100 or *p*-xylene were performed to determine if coating thickness could be increased by increasing the concentration of BAM-PPV rather than by increasing the number of cross-coat applications. These coatings were applied to AA2024-T3 only, using the HVLP application process and approximately 3–6 cross-coats to generate the various films. Review of the process notes from HVLP spray application identified that a large quantity of the BAM-PPV material was caught in the filter prior to application despite that the material appeared to be thoroughly dissolved prior to application. When the approximate weight of this material was taken into consideration, the approximate concentration of the 3 wt% as-mixed solutions was actually 1–1.5 wt% after losses. Analysis of SEM micrographs of these samples confirmed that there was little difference in the thickness of the samples (Figures 28 and 29), and they ranged approximately 0.8–1.5 µm in thickness. Again, samples were tested for adhesion and corrosion resistance. The data showed that the crosshatch adhesion characteristics were not affected by the varied concentration of the material or by the poor solubility of the 3 wt% (~1.5 wt% actual, calculated) BAM-PPV coatings (5B rating for ASTM D 3359, 1-mm grid). Also, salt spray resistance was not appreciably different, suggesting that the slightly increased concentration of the material did not have much effect on the various coating thicknesses. Combined analysis of these data confirmed that there was no benefit to adhesion or corrosion resistance by increasing the BAM-PPV concentration above 1 wt% and that coating thickness could not be increased by increasing the solution concentration and holding the number of cross-coats constant. Final thickness experimentation was performed using 1 wt% BAM-PPV in Oxsol-100 applied at 9 cross-coats and tested for crosshatch adhesion and salt spray. Again, samples were prepared using the HVLP spray application parameters listed in Table 17. SEM micrographs showed that the samples had an approximate thickness of 1.8–2 µm. The adhesion data did not show any change and continued to confirm that the material had good adhesion to the substrate. Corrosion resistance was significantly improved with the thicker BAM-PPV films. Corrosion resistance was demonstrated using ASTM B117 testing for one week on 1.8-2.0 µm BAM-PPV film coupons.

Table 17: HVLP Spray Application Parameters, BAM-PPV

Line PSI:	15
Fluid Setting	1.5
Hose inner diameter	3/8
Hose length	30
HVLP Gun	GTI
Needle Size	413
Cap Size	100
Cap PSI	<10
Application Notes	Material applied with a 5 min flash-off period between each 2–3 cross-coats

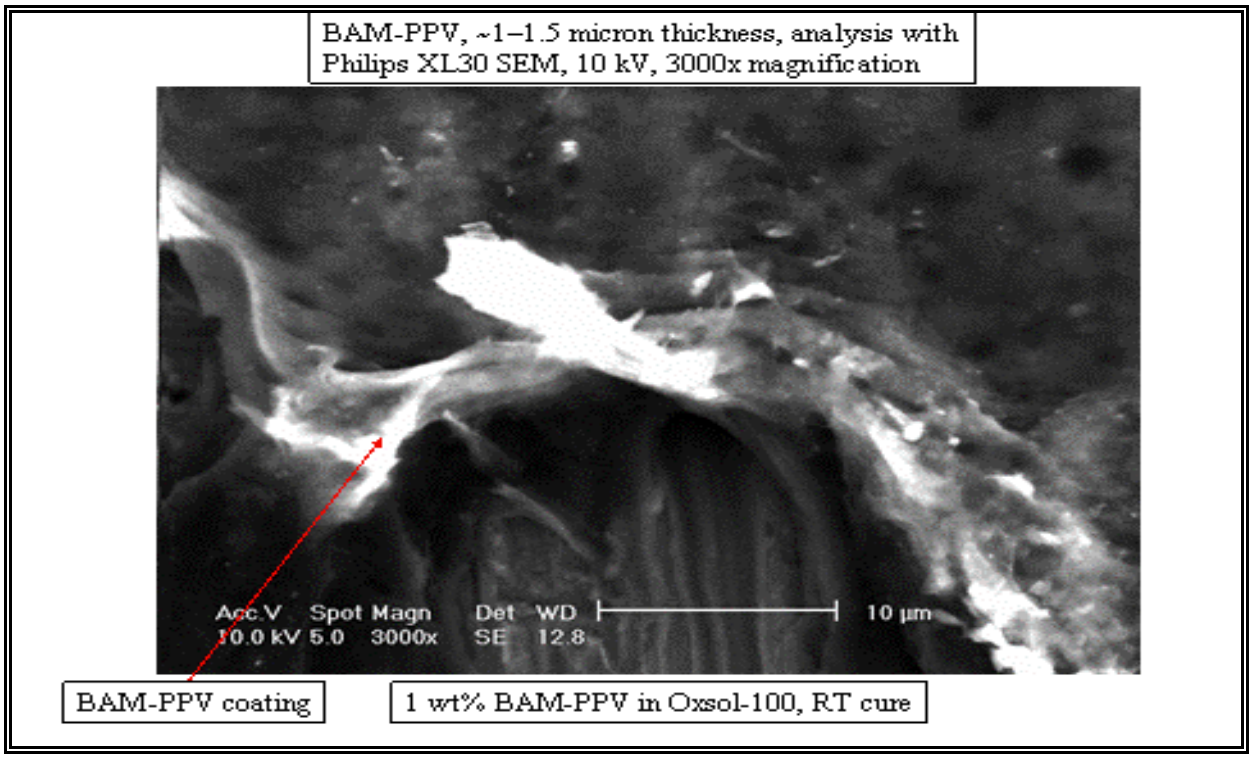


Figure 28: 1 wt% BAM-PPV, Approximately 1 µm Thick, for Comparison with Figure 29

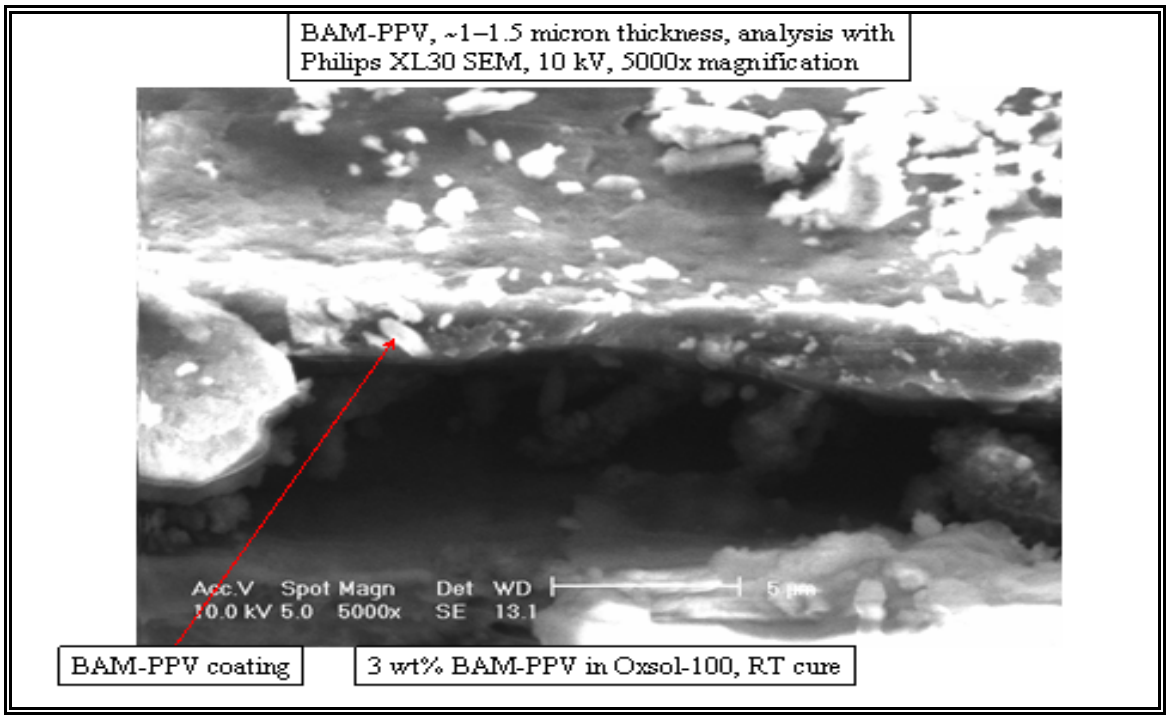


Figure 29: 3 wt% BAM-PPV, Approximately 1 µm Thick, for Comparison with Figure 28

6.1.1.1.1.4 Dwell and Drying Parameters

The thickness rather than solution concentration was identified as the primary factor for salt spray resistance of BAM-PPV coatings, additional testing was performed to determine what effect the dwell time and curing process would have on coating thickness. The goal was to determine which of these parameters was of primary importance for the production of viable coatings as determined through crosshatch adhesion and corrosion resistance testing. The solubility testing with Oxsol-100 as the solvent, determined that BAM-PPV dissolved best with applied heat set to approximately 100–150 °C for 60 min on a stir plate set to 300 rpm. This process produced a nearly clear dark-orange solution. Once this was obtained, the heat was removed but the stirring was maintained while the material returned to ambient temperature. This step marked the beginning of dwell time for the coating. For these experiments, the dwell time was identified as the length of time between the initial identification that the material is in solution to the time that the material is applied to the panel. Generally, the material was continuously agitated during dwell. Solubility and coating thickness work with BAM-PPV also identified that the material was likely to gel and become "more viscous" during the dwell process, particularly when the material had been left to sit for up to 8 hours or more. What was not yet identified was the relationship between the dwell time and the application window for BAM-PPV or the effect that dwell time would have on the coating morphology, conductive behavior, thickness and ultimately on adhesion and corrosion resistance. When it is presumed that heat applied during the mixing process has an effect on the morphology of the applied coating and, therefore, on the corrosion

resistance, it is also possible that heat applied during the curing process would have an effect on the same. To address these parameters, several experiments were performed in conjunction with the coating thickness testing already discussed to determine what effect dwell time and cure process had on the coating thickness, adhesion and corrosion resistance.

Several samples of 1 wt% BAM-PPV in Oxsol-100 were prepared, with varied dwell time of either 1 or 3 hours. Following the dwell time, samples were HVLP spray-applied onto AA2024-T3 panels that were cleaned. Once the material had been applied to the panels, a cure process of either 16 h at 60 °C and close to zero humidity or between 24 hours at ambient temperature and humidity was applied to the panels. Following cure, samples were tested in crosshatch adhesion and ASTM B 117 salt spray resistance for 1 week (168 hours) and 2 weeks (336 hours) of exposure. Sample morphology was analyzed using scanning electron microscopy (SEM). There was no measurable difference in crosshatch adhesion between the different dwell times for the two systems. All samples demonstrated good adhesion (5B rating for ASTM D 3359, 1-mm grid). However, the corrosion resistance of the 1 hour dwell samples was lower than that of the 3-hour dwell samples (Figures 30 and 31). Even though the "1-hour dwell" system showed minimal corrosion after 336 hours, blistering and corrosion was present along with discoloration by 168 hours. This was not the case for the "3-hour dwell" system. Instead, this system showed very minimal discoloration after 168 hours and showed only minor discoloration and on blister after 336 hours. SEM analysis verified that there was a difference in the morphology between the two systems. The 1-hour dwell system showed charge build-up from the beam, often significant of a material behaving more as an insulator and less as a conductor (Figures 32 and 33). Additionally, the 1-hour dwell system appeared to have "peaks and valleys" of BAM-PPV material on the coating surface, providing local differences in thickness. By comparison, the 3-hour dwell system showed a continuous coating without the "peaks and valleys" or the charge-up that was seen in the 1-hour dwell system. The coating thicknesses were approximately the same, however, as analyzed at the cut edge. This suggested that the peaks were BAM-PPV material that had not formed into a cohesive matrix during the cure process, which ultimately may have caused the material to behave more as an insulator and less as a conductive coating. This behavior may have had an effect on the overall corrosion resistance demonstrated between the two systems.

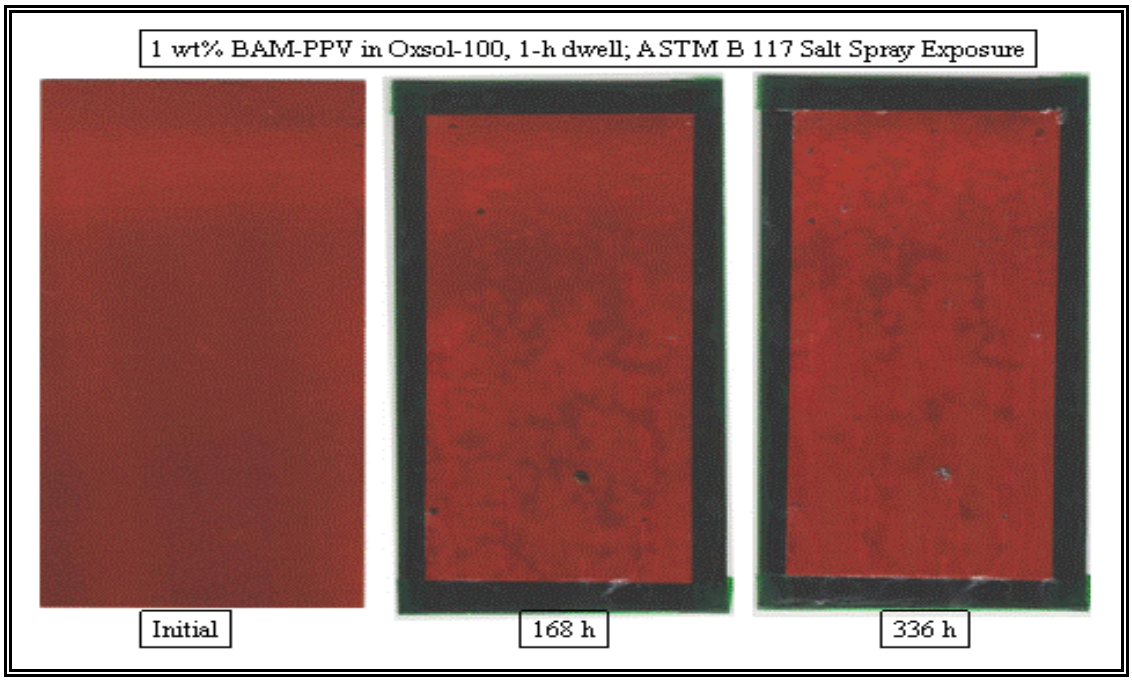


Figure 30: Neutral Salt Spray Samples, 1 wt% BAM-PPV in Oxsol-100, 1-hour Dwell before HVLP Spray Application

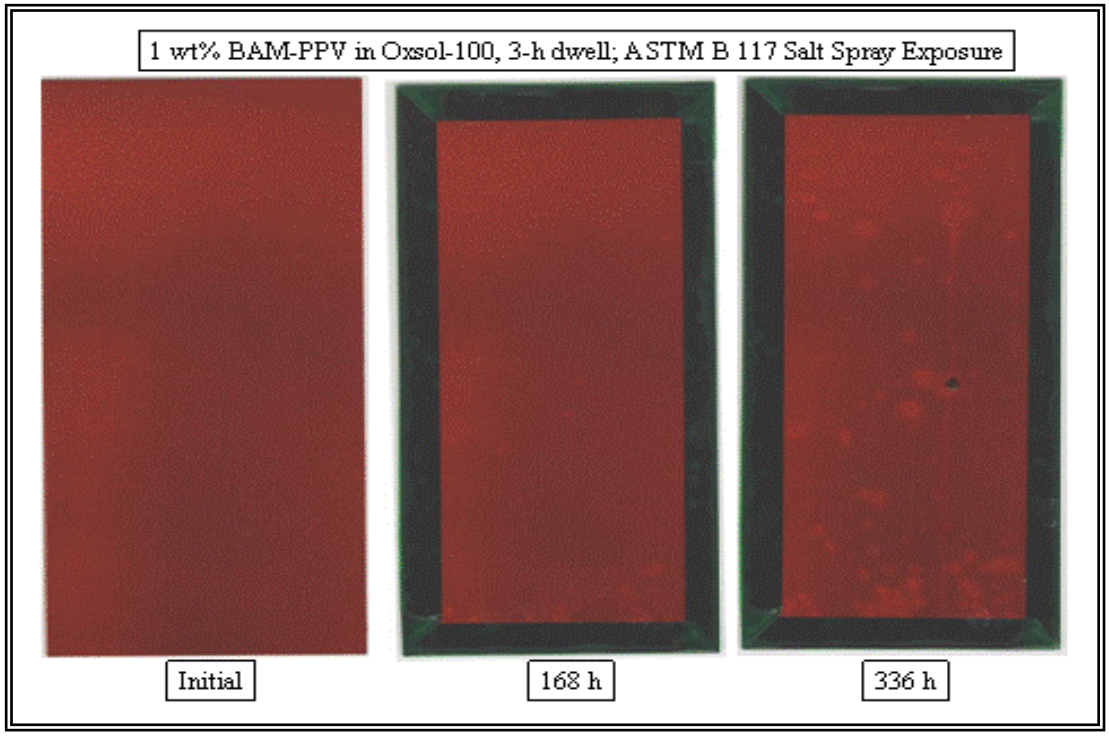


Figure 31: Neutral Salt Spray Samples, 1 wt% BAM-PPV in Oxsol-100, 3-hour Dwell before HVLP Spray Application

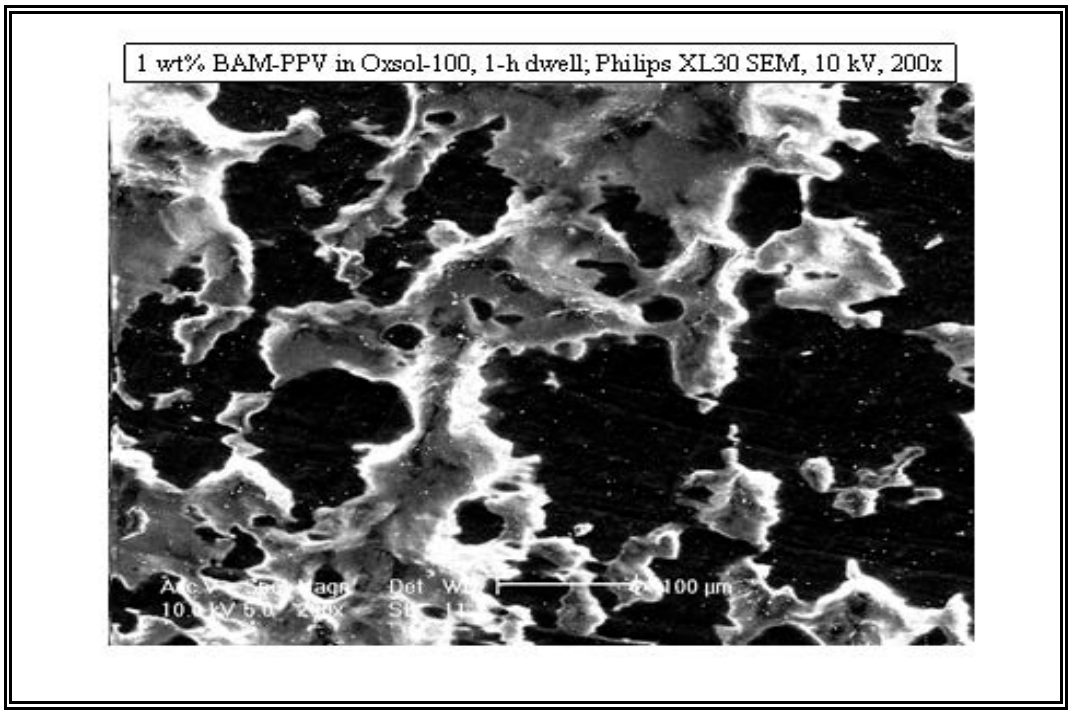


Figure 32: SEM Micrograph, 1 wt% BAM-PPV in Oxsol-100, 1-hour Dwell

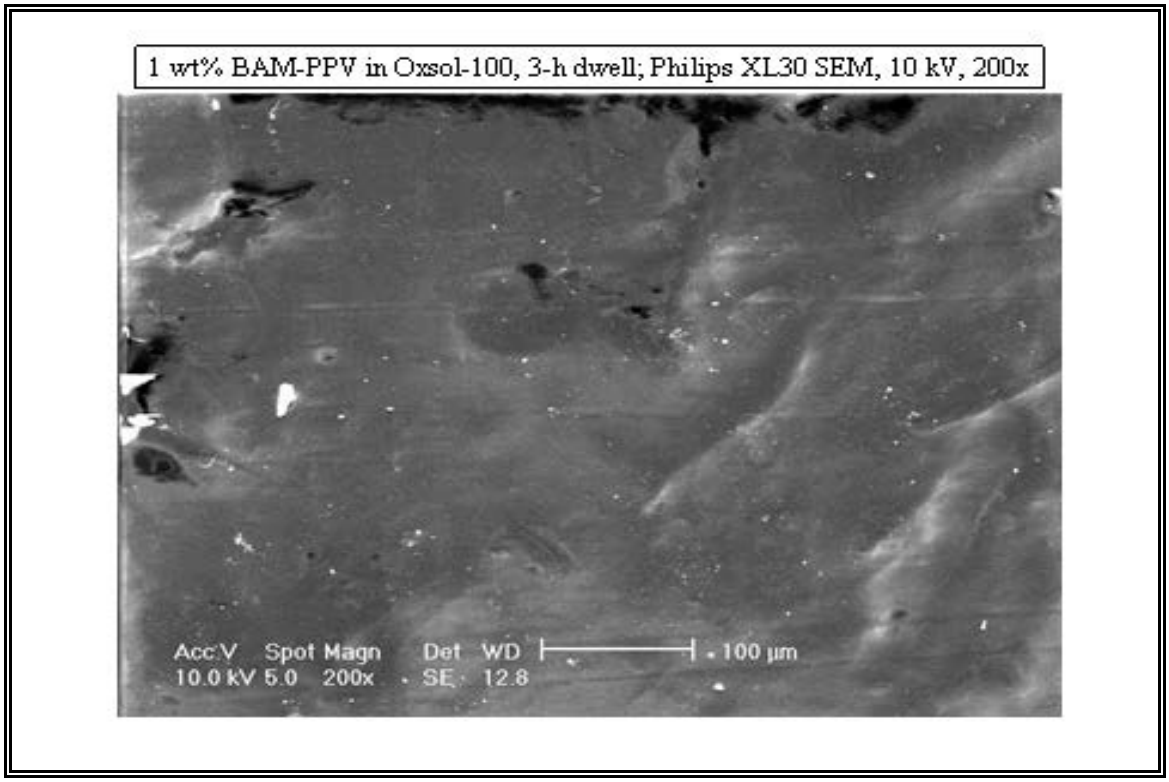


Figure 33: SEM Micrograph, 1 wt% BAM-PPV in Oxsol-100, 3-hour Dwell

The cure parameter tests were also performed on 1 wt% BAM-PPV in Oxsol-100 with changes only in the curing process and the dwell-time was held constant at 3-hour prior to HVLP spray application. Solutions of 1 wt% BAM-PPV in *p*-xylene were prepared also and HVLP spray-applied for baseline comparison of the BAM-PPV behavior. For both sets of samples, HVLP spray application was approximately 6 cross-coats. The cure parameters were chosen based upon work with BAM-PPV in xylenes at China Lake. One cure parameter was that the sample was cured for 24 hours at room temperature and ambient relative humidity. For the parameter, the sample was force-cured for 16 hours at 60 °C and zero relative humidity. These samples were also tested for adhesion characteristics and corrosion resistance. Again, there was no difference in the adhesion behavior between the two solvent systems and two cure processes, and all samples demonstrated good adhesion (5B rating for ASTM D 3359, 1-mm grid). Corrosion resistance was comparable between the two solvent systems, with neither system performing measurably better than the other. Both systems showed discoloration and slight blistering after 168 hours of ASTM B 117 salt spray exposure, with a few more blisters on the Oxsol-100 system cured at room temperature versus the *p*-xylene system cured at room temperature. Both systems (Oxsol-100 or *p*-xylene) showed staining, discoloration, and blistering following 168 hours of exposure to salt spray (Figures 34-37). With the behavior of the two systems very similar, morphology and thickness was presumed to be similar as well. Coating thickness was verified using ImageProPlus software for analysis of SEM micrographs and, again, the coatings were approximately between 1 and 1.5 µm in thickness. The morphology of the two systems was very similar, and both films were reasonably continuous (Figures 38-41). It verified, then, that the corrosion resistance was not affected by the cure process and that a room temperature cure could be used and generate a viable coating.

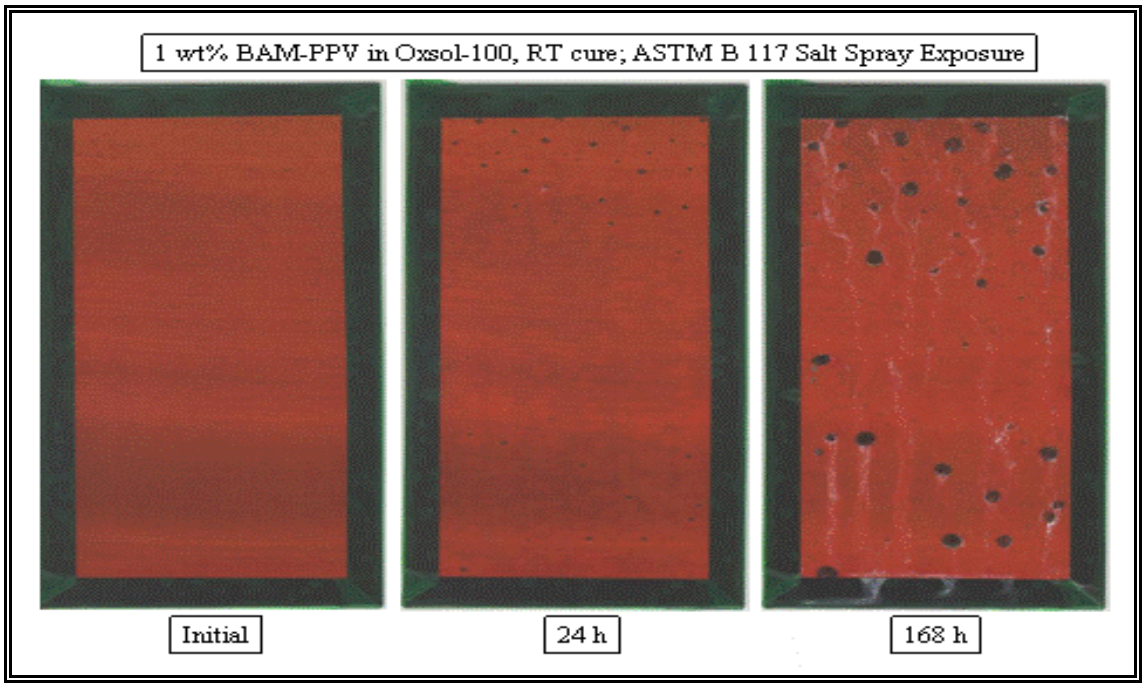


Figure 34: Neutral Salt Spray Samples, 1 wt% BAM-PPV in Oxsol-100; 24-hour Room Temperature Cure and Ambient Relative Humidity

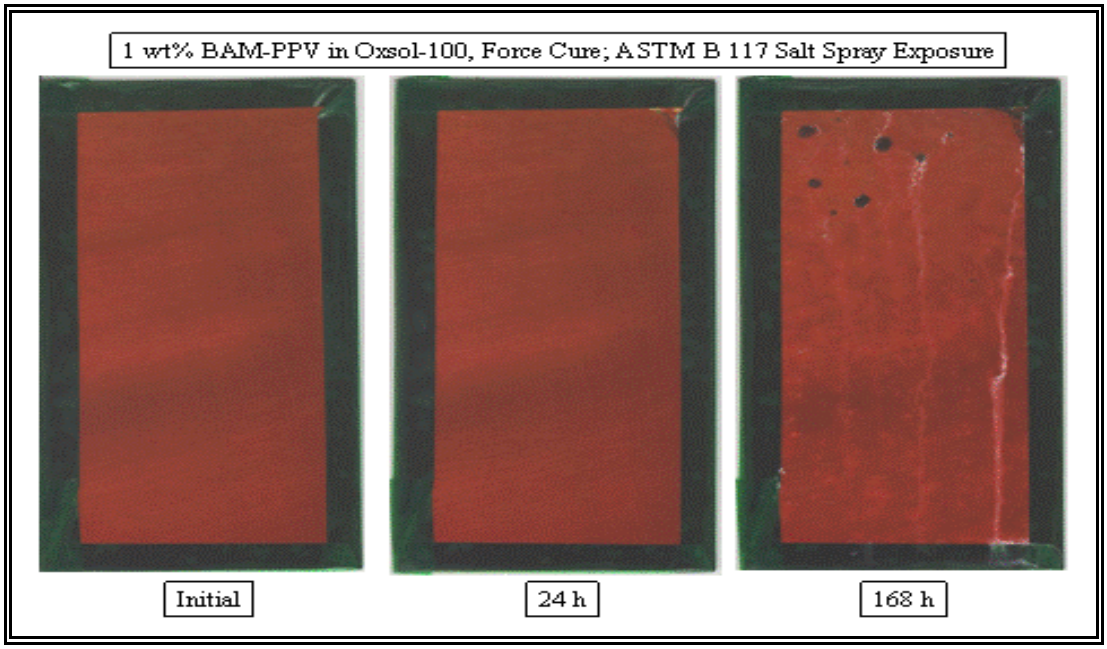


Figure 35: Neutral Salt Spray Samples, 1 wt% BAM-PPV in Oxsol-100; 16-hour Force Cure at 60 °C and Near-Zero Relative Humidity

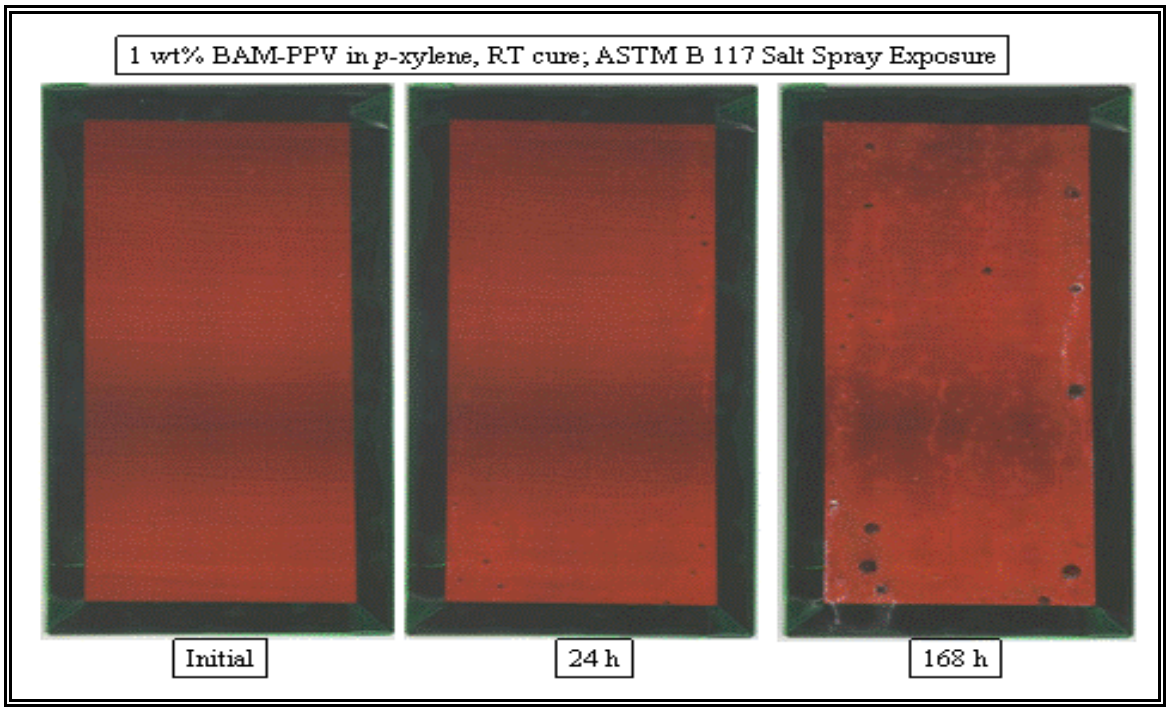


Figure 36: Salt Spray Samples, 1 wt% BAM-PPV in p-Xylene, 24-hour Room Temperature Cure and Ambient Relative Humidity

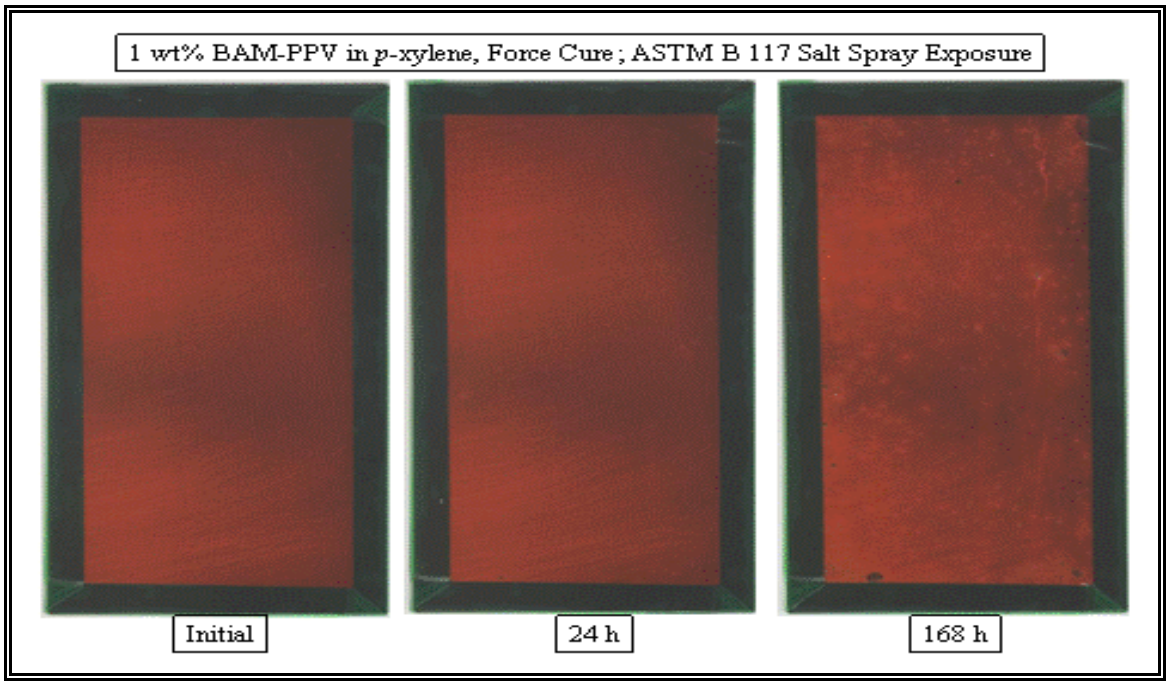


Figure 37: Salt Spray Samples, 1 wt% BAM-PPV in p-Xylene; 16-hour Force Cure at 60 °C and Near-Zero Relative Humidity

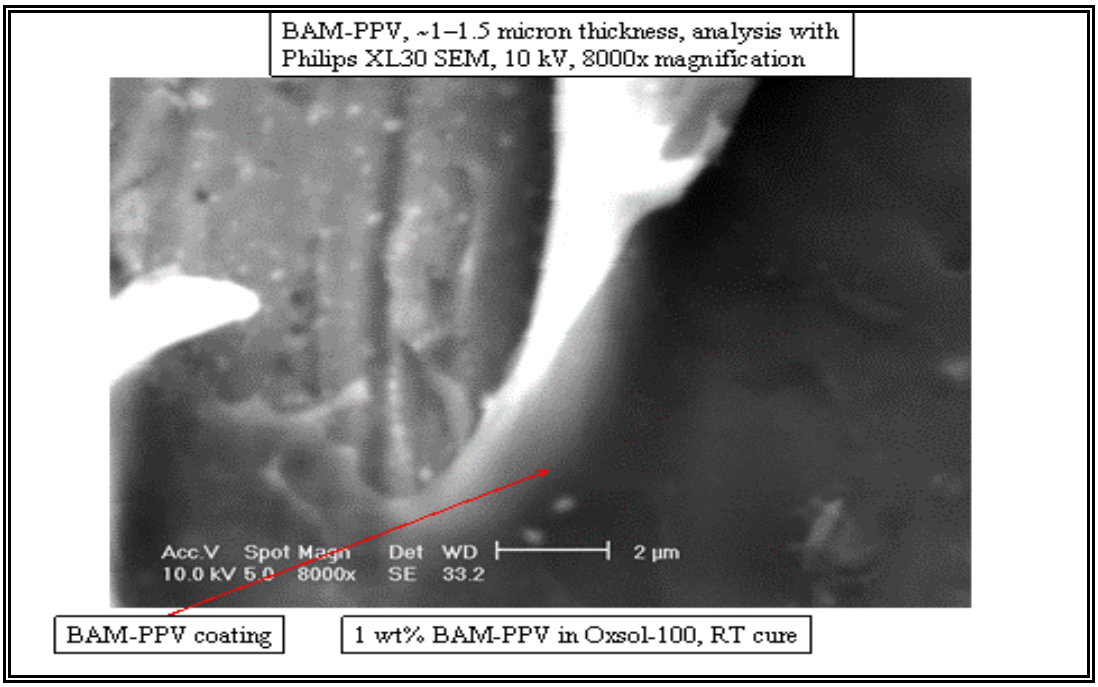


Figure 38: SEM Micrograph, 1 wt% BAM-PPV in Oxsol-100, 24-hour Room Temperature Cure and Ambient Relative Humidity

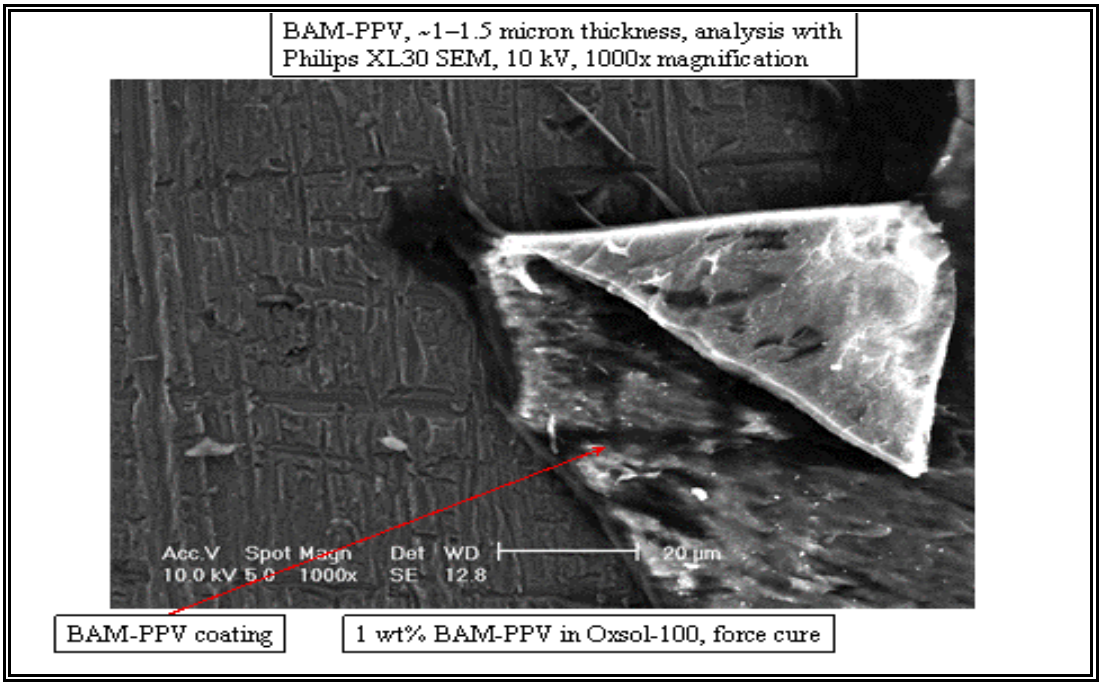


Figure 39: SEM Micrograph, 1 wt% BAM-PPV in Oxsol-100, 16-hour Force Cure at 60 °C and Near-Zero Relative Humidity

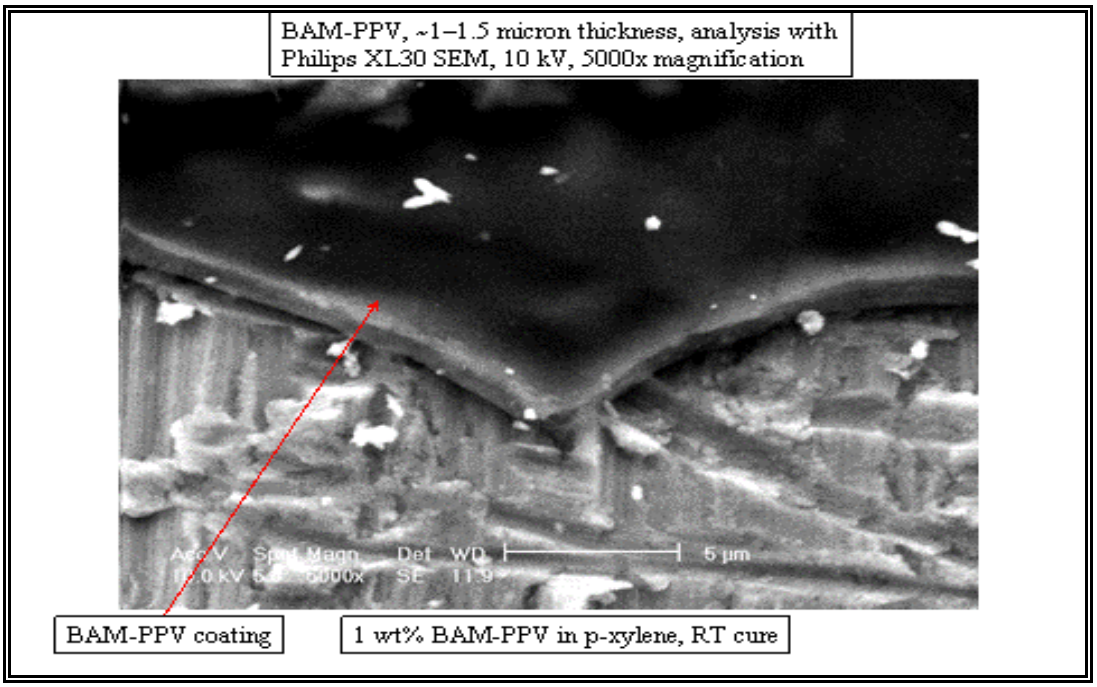


Figure 40: SEM Micrograph, 1 wt% BAM-PPV in *p*-Xylene, 24-hour Room Temperature Cure and Ambient Relative Humidity

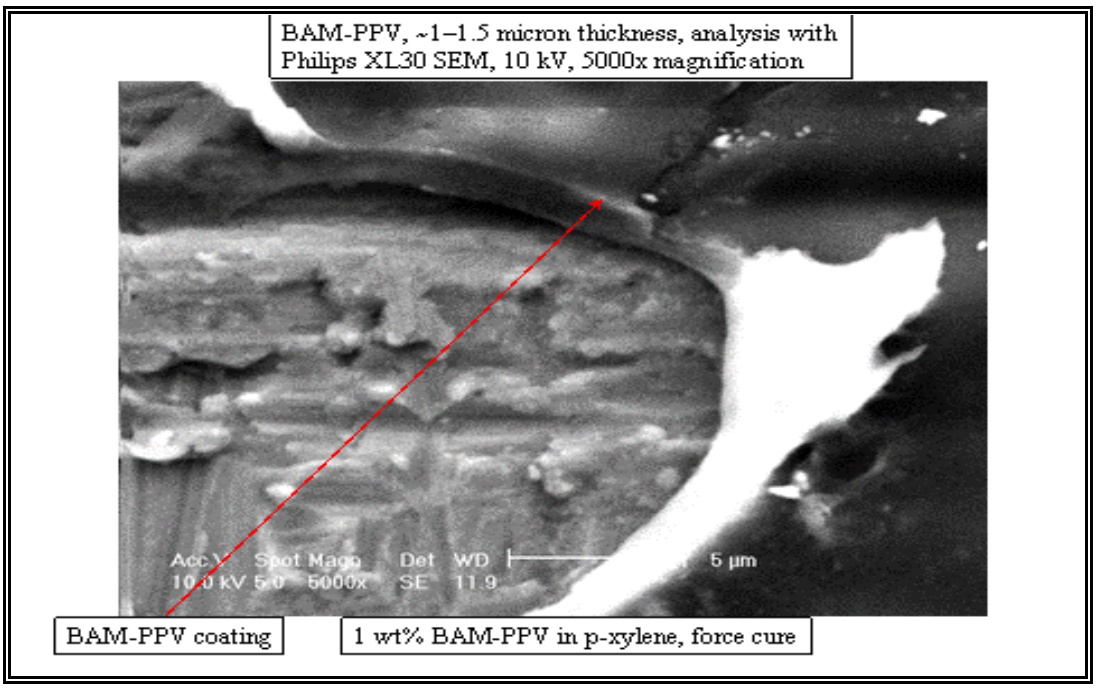


Figure 41: SEM Micrograph, 1 wt% BAM-PPV in *p*-Xylene, 16-hour Force Cure at 60 °C and Near-Zero Relative Humidity

6.1.1.1.1.5 General Application Parameters

The trial application processes for thickness, dwell and cure testing using the HVLP spray application process produces "wastes." A minimum amount of 25 mL of solution with each fill. Also it has been determined that approximately 200 mL of solution would cover approximately 36 ft^2 with single-passes. The coating could be applied in multiple cross-coats on near-vertical surfaces with 5 min flash-off period after every 2–3 cross-coats. Again, general HVLP settings are given in Table 17. The coating thickness and dwell time data show that the BAM-PPV coating was most viable when it was permitted a minimum 3-hour dwell period prior to application and when it was HVLP spray-applied to be thicker - approximately 9 cross-coats - to obtain a thickness of about 2 µm. With this thickness and this number of cross-coats required the set-to-touch time for the BAM-PPV coating was very important since the material would need to be ready to be coated with primer as soon as possible. Drying time experiments on samples demonstrated that the material was certainly set-to-touch well within 30 minutes of the final cross-coat application of the BAM-PPV material dissolved in Oxsol-100.

6.1.1.1.2 Adhesion Testing

The following sections describe the adhesion testing performed on BAM-PPV pretreatment coatings. The BAM-PVV pretreatment coating was tested in various combinations with primers and topcoats. Both qualitative and quantitative adhesion testing was performed on BAM-PPV coated coupons.

6.1.1.1.2.1 ASTM D 3359, Method B, Crosshatch Adhesion (MIL-PRF-23377)

This test follows ASTM D 3359 and is defined by ASTM as an implied adhesion test method which is "used to assess coating performance under actual service conditions" and provides "insight into the approximate, relative level of adhesion" of the coating system. This test provides only general information concerning the overall adhesion performance of the system. The description of the rating scale, as taken from ASTM D 3359 and the visual standard, is given in Figures 15 and 16. The data are comparative in nature, meaning that the behavior of the control system is often used as a metric for the rating of the remaining systems. This test was performed on (*i*) pretreatment-only, (*ii*) primed, and (*iii*) topcoated systems. The data are reported for all primed and topcoated systems but only for select pretreatment-only panels (i.e., one cannot rate the adhesion of a thin coatings, such as PreKote, to the substrate when the coating cannot be identified visually without the aid of high magnification microscopy such as scanning electron microscopes). Historical behavior of the control system has been ratings of 4B to 5B. Therefore, coatings which demonstrate this level of adhesion are given a "pass" rating. The data for BAM-PPV coated coupons are given in Tables 18-22. The control system is highlighted in **green**. Coating systems which demonstrated acceptable performance are highlighted in **blue**. Almost all of the tested coated systems demonstrated acceptable adhesion behavior.

Table 18: ASTM D 3359 Crosshatch Adhesion (Pretreated Systems)				
AA2024-T3 Data			**AA7075-T6 Data**	
Pretreatment Only	**Rating**		**Pretreatment Only**	**Rating**
BAM-PPV	**pass 5B**		**BAM-PPV**	**pass 5B**
AA5086-H116 Data			**AA6061-T6 Data**	
BAM-PPV	**pass 5B**		**BAM-PPV**	**pass 5B**

Table 19: ASTM D 3359 Crosshatch Adhesion (Primed Systems)		
Coating System (No Topcoat)	**AA2024-T3 Data**	**AA7075-T6 Data**
Pretreatment + Primer	**Rating**	**Rating**
Alodine 1200S Deft 02-Y-40 chromated epoxy	**control 5B**	**control 4B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	**pass 5B**	**pass 5B**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	**pass 5B**	**pass 5B**
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy	**pass 4B**	**pass 4B**
BAM-PPV + Deft 02-Y-40A □chromate epoxy	**pass 5B**	**pass 5B**
BAM-PPV + Deft 02-GN-083 nonchromated epoxy	**pass 4B**	**pass 5B**
BAM-PPV + Deft 02-GN-084 nonchromated epoxy	fail 3B	fail 3B
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy	fail 3B	fail 3B
PreKote + Deft 02-Y-40A □chromate epoxy	**pass 5B**	**pass 5B**
PreKote + Deft 02-GN-083 nonchromated epoxy	**pass 5B**	**pass 5B**
PreKote + Deft 02-GN-084 nonchromated epoxy	**pass 5B**	**pass 5B**
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy	**pass 5B**	**pass 5B**

Table 20: ASTM D 3359 Crosshatch Adhesion (MIL-PRF-85285 Standard Topcoat)		
Coating System	**AA2024-T3 Data**	**AA7075-T6 Data**
Pretreatment + Primer + Topcoat	**Rating**	**Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control 4B**	**control 4B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5B**	**pass 5B**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5B**	**pass 5B**
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4B**	**pass 4B**
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4B**	**pass 4B**
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4B**	**pass 4B**
BAM-PPV + Deft 02-GN-084 nonchromated epoxy+ Deft 03-GY-321 (85285)	**pass 4B**	**pass 4B**
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4B**	**pass 4B**
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4B**	**pass 4B**
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4B**	**pass 5B**
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4B**	**pass 5B**
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4B**	**pass 4B**

Table 21: ASTM D 3359 Crosshatch Adhesion (MIL-PRF-85285 APC Topcoat)		
Coating System	**AA2024-T3 Data**	**AA7075-T6 Data**
Pretreatment + Primer + Topcoat	**Rating**	**Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	**control 4B**	**control 4B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 5B**	**pass 5B**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 5B**	**pass 5B**
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 4B**	**pass 4B**
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 4B**	**pass 4B**
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 4B**	fail 3B
BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 4B**	fail 3B
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 4B**	fail 3B
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 4B**	**pass 4B**
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 5B**	**pass 5B**
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 5B**	**pass 5B**
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 4B**	**pass 4B**

Table 22: ASTM D 3359 Crosshatch Adhesion (MIL-PRF-85285 Topcoat)		
Coating System	**4130 Solvent-Cleaned Data**	**4130 Media-Blasted Data**
Pretreatment + Primer + Topcoat	**Rating**	**Rating**
Cadmium Plated + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control 4B**	**control 4B**
Cadmium Plated + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5B**	**pass 4B**
Cadmium Plated + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5B**	**pass 4B**
Cadmium Plated + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4B**	**pass 4B**
BAM-PPV+ Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4B**	**pass 4B**
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5B**	**pass 5B**
BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5B**	**pass 5B**
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4B**	**pass 4B**
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4B**	**pass 4B**
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5B**	**pass 5B**
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5B**	**pass 4B**
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4B**	**pass 4B**

6.1.1.1.2.2 FTMS 141D, Method 6301, Wet-Tape Adhesion (MIL-PRF-23377, MIL-PRF-85285)

This test is outlined in the Federal Test Method Standard (FTMS) 141 as an inter-coat and surface adhesion test for organic systems immersed in water. The rating system from ASTM D 3359, Method A, is used to determine a pass/fail condition for the coating system. However, the rating does not distinguish between inter-coat, intra-coat, and substrate interface failure modes. Systems that receive a 4A or higher rating are designated a "pass" and are highlighted in blue and failure (≤3A) highlighted in red. The data are given in Tables 23-25. The control system is highlighted in **green**.

Table 23: FTMS 141, ASTM D 3359 Wet Tape Adhesion (Primed Systems)

Coating System (No Topcoat)	AA2024-T3 Data
Pretreatment + Primer	**Rating**
Alodine 1200S Deft 02-Y-40 chromated epoxy	**control 5A**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	**pass 5A**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	**pass 5A**
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy	**pass 5A**
BAM-PPV + Deft 02-Y-40A □chromate epoxy	**pass 5A**
BAM-PPV + Deft 02-GN-083 nonchromated epoxy	**pass 5A**
BAM-PPV + Deft 02-GN-084 nonchromated epoxy	**pass 5A**
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy	fail 0A
PreKote + Deft 02-Y-40A □chromate epoxy	**pass 5A**
PreKote + Deft 02-GN-083 nonchromated epoxy	**pass 5A**
PreKote + Deft 02-GN-084 nonchromated epoxy	**pass 5A**
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy	**pass 5A**

Table 24: FTMS 141, ASTM D 3359 Wet Tape Adhesion (MIL-PRF-85285 Standard Topcoat)	
Coating System	**AA2024-T3 Data**
Pretreatment + Primer + Topcoat	**Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control 5A**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5A**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5A**
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5A**
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4A**
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5A**
BAM-PPV + Deft 02-GN-084 nonchromated epoxy+ Deft 03-GY-321 (85285)	**pass 5A**
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	fail 0A
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5A**
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5A**
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 5A**
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 4A**

Table 25: FTMS 141, ASTM D 3359 Wet Tape Adhesion (MIL-PRF-85285 APC Topcoat)	
Coating System	**AA2024-T3 Data**
Pretreatment + Primer + Topcoat	**Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	**control 4A**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 5A**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 5A**
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 5A**
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 4A**
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 5A**
BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 5A**
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-0001 polyurethane (APC)	**pass 5A**
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 5A**
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 5A**
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 4A**
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 4A**

6.1.1.1.2.3 ASTM D 4541 PATTI, Pull-Off Adhesion

This test is defined by ASTM as a measure of the direct normal applied force required to remove one or more layers from a coating system as a measure of adhesion strength. The raw data are the psi-gauge values from the apparatus, and these values are converted to direct pull-off tensile strength (psi) using the following equation (Equation 2).

Equation 2 pull-off tensile strength (psi) = [(ab)-c]/d

Where;

a = contact area of the gasket in the F-8 piston = 8in^2
b = burst pressure from gauge (psig)
c = piston constant for F-8 = 1.068 lbs
d = contact area of the pull stub = 0.196 in^2

There are limitations to interpretation of the data that was generated from these studies. Those limitations can be summarized from the scope of the method as defined by ASTM and quoted here "1.0 Scope 1.1. This test method covers a procedure for evaluating the pull-off strength (commonly referred to as adhesion) of a coating on rigid substrates such as metal, concrete or wood. The test determines either the greatest perpendicular force (in tension) that a surface area can bear before a plug of material is detached, or whether the surface remains intact at a prescribed force (pass/fail). Failure will occur along the weakest plane within the system comprised of the test fixture, adhesive, coating system, and substrate, and will be exposed by the fracture surface. This test method maximizes tensile stress as compared to the shear stress applied by other methods, such as scratch or knife adhesion, and results may not be comparable" [77]. The following criteria were applied to the data to determine outliers, suspect data and confidence intervals. ASTM recommends that a statistical analysis for outliers is performed on all data sets to reject unreliable numerical pull-off values. Although the data are numerical and quantitative, the failure modes are qualitative. Therefore, ASTM recommends that the numerical value for the pull-off be disregarded when more than 50% of the failure mode was the result of adhesive failure. Final guidance on the interpretation of the data is given concerning the precision data, which is instrument and laboratory dependent. The instrument used for this test was the Type IV self-alignment adhesion tester as defined in ASTM D 4541. The precision of this test method, as defined by ASTM, is that three replicates of any test specimen must not differ by more than 29% to provide a confidence interval of 95% for that data. The resultant data sets for each coating system are given in Tables 26-28. The assignment of pass/fail for a coating system is largely based upon consideration of the average pull-off value and failure mode in comparison to that of the control; therefore, data interpretations are comparative in nature. This test was performed on (*i*) primed and (*ii*) topcoated systems. Most of the systems tested demonstrated acceptable adhesion behavior in this test. No system ratings for pass/fail are assigned.

Table 26: ASTM D 4541 Pull-Off Adhesion (Primed Systems)		
Coating System (No Topcoat)	**AA2024-T3 Data**	**Failure Mode**
Pretreatment + Primer	**Rating**	**Rating**
Alodine 1200S Deft 02-Y-40 chromated epoxy	**control 1253**	**P / P**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	1522	P / P
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	1630	P / P
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy	1419	P / P
BAM-PPV + Deft 02-Y-40A □chromate epoxy	1414	P / P
BAM-PPV + Deft 02-GN-083 nonchromated epoxy	1635	Pretreat / Pretreat
BAM-PPV + Deft 02-GN-084 nonchromated epoxy	939	Pretreat / Pretreat
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy	880	Pretreat / Pretreat
PreKote + Deft 02-Y-40A □chromate epoxy	1191	P / P
PreKote + Deft 02-GN-083 nonchromated epoxy	1303	P / P
PreKote + Deft 02-GN-084 nonchromated epoxy	1293	P / S
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy	1187	P / P

Table 27: ASTM D 4541 Pull-Off Adhesion (MIL-PRF-85285 Standard Topcoat)		
Coating System	**AA2024-T3 Data**	**Failure Mode**
Pretreatment + Primer + Topcoat	**Rating**	**Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control 1768**	**P / P**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	1877	T / P
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	1815	P / P
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	1973	P / P
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	1122	P / P
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	830	Pretreat / Pretreat
BAM-PPV + Deft 02-GN-084 nonchromated epoxy+ Deft 03-GY-321 (85285)	1191	Pretreat / Pretreat
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	1029	Pretreat / Pretreat
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	2258	P / P
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	654	P / P
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	589	P / P
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	1297	P / P

Table 28: ASTM D 4541 Pull-Off Adhesion (MIL-PRF-85285 APC Topcoat)		
Coating System	**AA2024-T3 Data**	**Failure Mode**
Pretreatment + Primer + Topcoat	**Rating**	**Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	**control 1729**	**P / P**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1841	P / P
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1851	P / P
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1718	P / P
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	1221	P / S
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	765	Pretreat / Pretreat
BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1141	Pretreat / Pretreat
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	962	P / Pretreat
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	1700	P / P
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1054	P / P
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	2279	P / P
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1638	P / P

6.1.1.1.3 Accelerated Weathering Tests

The following sections describe BAM-PPV pretreatment coated coupons using various accelerated weathering testing methods and its performance relative to control coupons.

6.1.1.1.3.1 Neutral Salt Spray Testing (ASTM B 117)

The previous sections have described WPAFB success at optimizing the BAM-PPV compound for coating application onto aluminum substrates. Samples of BAM-PPV coatings on AA2024-T3 have been applied successfully using standard spray equipment. The next steps determined what differences in corrosion and adhesion properties BAM-PPV pretreatment coating demonstrated when a primer and topcoat were applied to generate a full military coating system. Samples were prepared using BAM-PPV in Oxsol-100 as the pretreatment and then coated with MIL-PRF-23377 chromated epoxy primer (Deft 02-Y-40A) and MIL-PRF-85285 APC polyurethane topcoat (Deft 99-GY-001). Samples were tested for crosshatch adhesion characteristics using ASTM D 3359 and for corrosion resistance after 2000 hours of exposure to ASTM B 117 neutral salt fog spray. Samples were then compared to the performance of Alodine 1200S (chromate conversion coating, (CCC)) and PreKote (current non-chrome pretreatment) with the same primer and topcoat system. BAM-PPV was allowed a 1-hour dwell prior to application of the material using HVLP (application settings are given in Table 17). BAM-PPV were set-to-touch after 30 minutes but samples were not coated with primer until the following work day, giving these materials about 16 hours between pretreatment and primer application. Topcoat was applied 4 hours after primer application. Samples were left to cure at room temperature and ambient relative humidity (approximately 75 °F and 50% RH) for 14 days prior to testing. Neutral salt fog spray testing demonstrated that the BAM-PPV coating system behaved comparably and showed slightly better performance than the PreKote system. All three of these non-chrome pretreatment systems demonstrated minor corrosion in the scribe, minor build-up after 2000 hours, and minor undercutting at the intersection of the scribe. Visual aesthetics were better with the BAM-PPV systems than with the PreKote system, with the latter system showing more staining and the appearance of slightly more build-up of corrosion in the scribe after 2000 hours. The Alodine 1200S coating system (standard chromate conversion coating) showed only very minor corrosion in the scribe for the primer-only system but demonstrated typical corrosion build-up in the scribe with the APC topcoat, a phenomena that is well-documented in CTIO lab testing of this system. In comparison between the four complete systems, the BAM-PPV performed satisfactorily as compared to the PreKote system performance and slightly underperformed as compared to the Alodine 1200S system. Photographs of the systems from the initial point through 2000 hours of exposure are given in Figures 42-47. Adhesion testing of these four systems was performed using the 1-mm grid for primer-only systems and the 2-mm grid on the topcoated systems. Data consistently showed that for all pretreatments, when primer was applied, the adhesion rating was 5 (no loss of adhesion), whereas when topcoat was applied, the adhesion rating was 4 (minor loss of adhesion). This result was consistent across the board with all of the pretreatment samples and with samples run in triplicate.

The data suggests that once primer is applied, the BAM-PPV did not demonstrate a debit on the adhesion to the substrate (AA2024-T3).

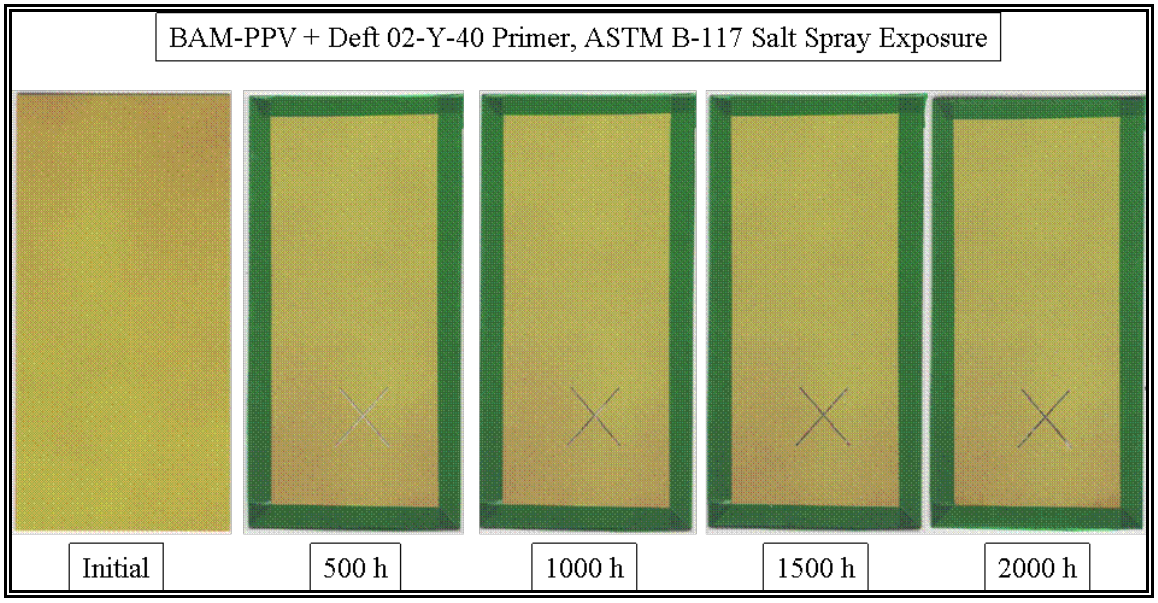


Figure 42: BAM-PPV + MIL-PRF-23377 Chromated Primer; ASTM B 117 Results

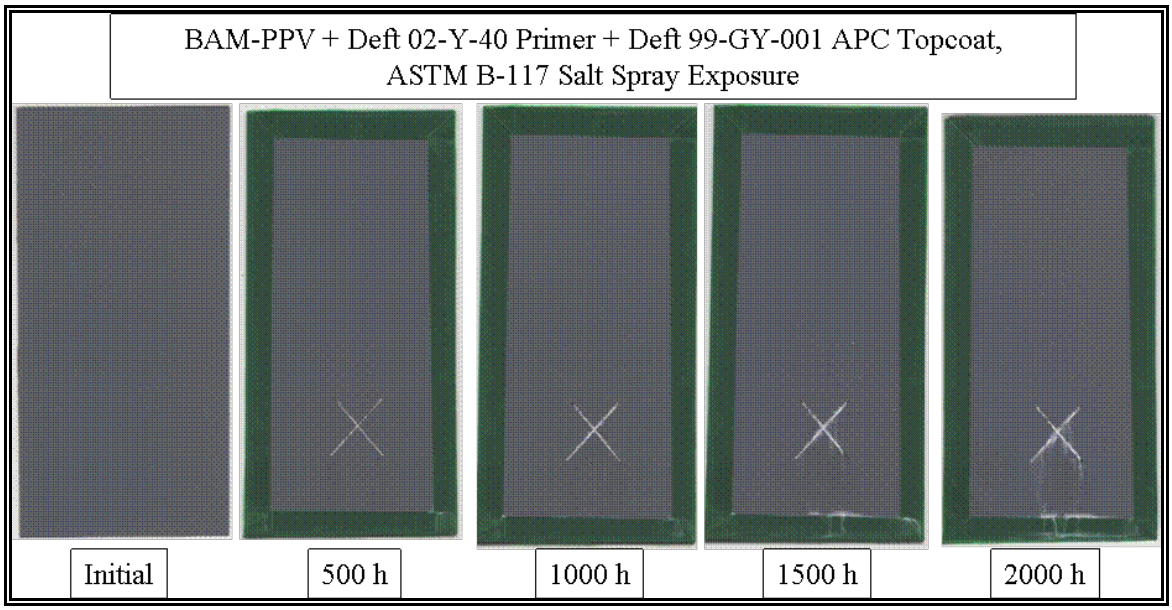


Figure 43: BAM-PPV + MIL-PRF-23377 Chromated Primer + MIL-PRF-85285 APC Topcoat; ASTM B 117 Results

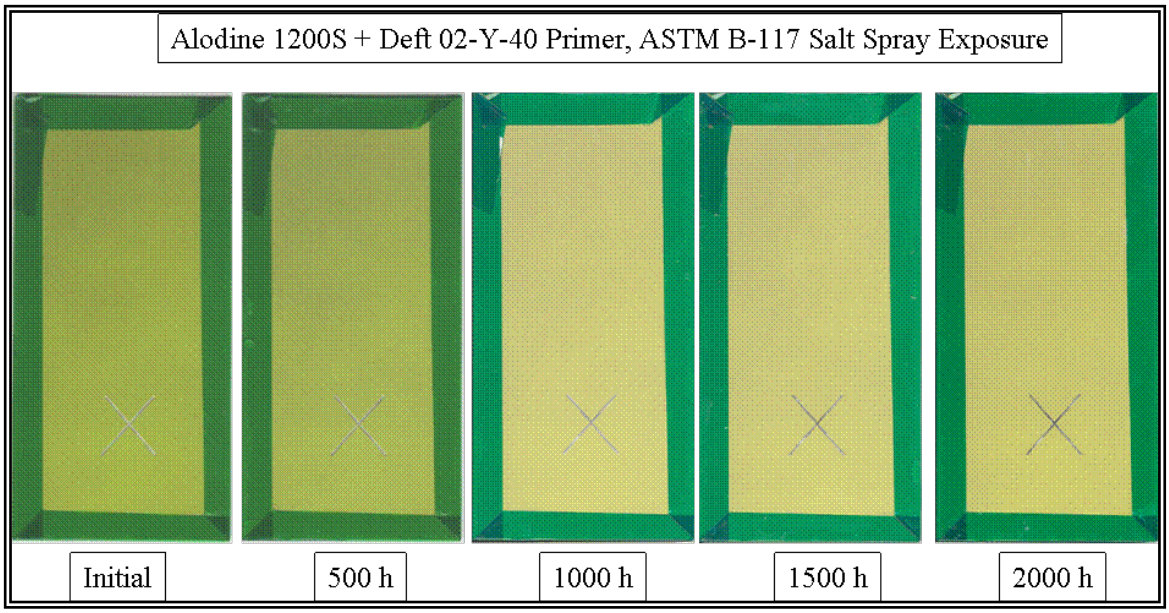


Figure 44: Alodine 1200S + MIL-PRF-23377 Chromated Primer; ASTM B 117 Results

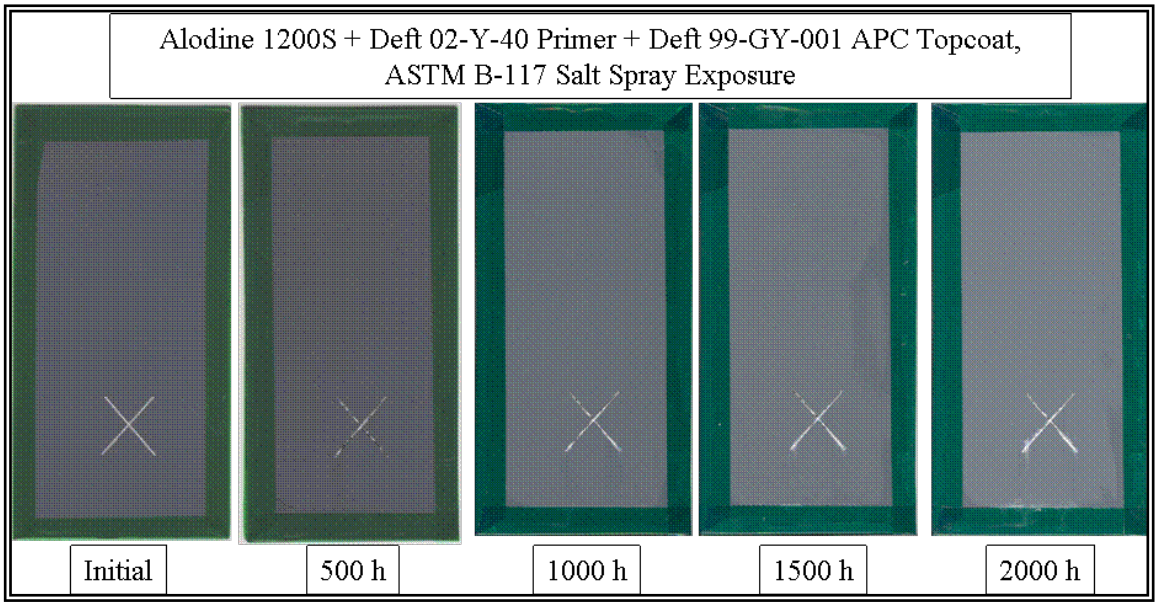


Figure 45: Alodine 1200S + MIL-PRF-23377 Chromated Primer + MIL-PRF-85285 APC Topcoat; ASTM B 117 Results

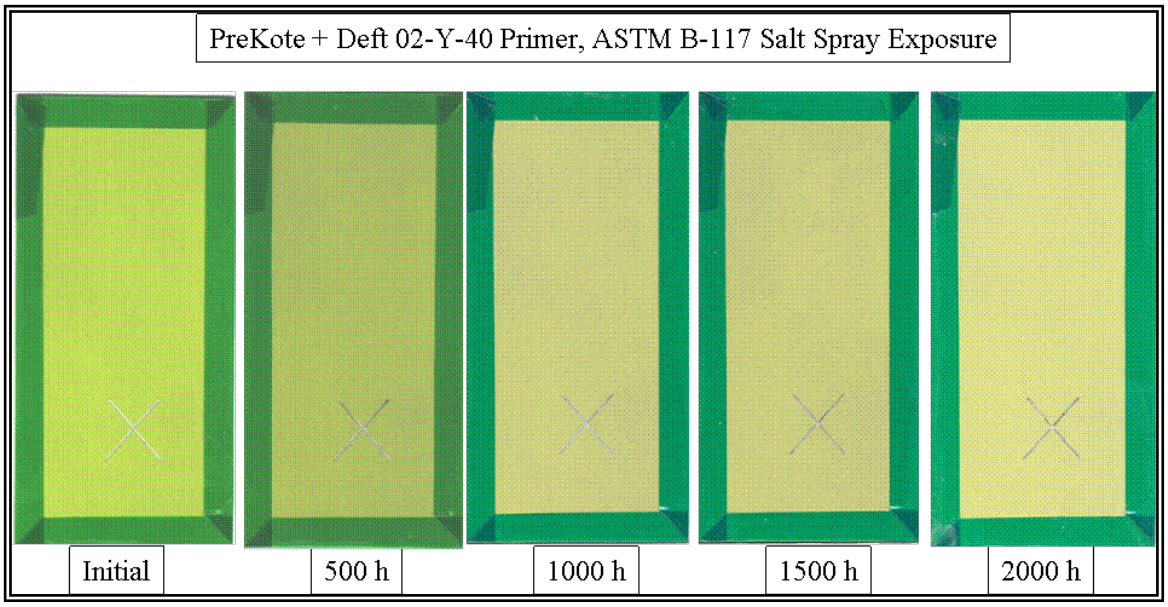


Figure 46: PreKote + MIL-PRF-23377 Chromated Primer; ASTM B 117 Results

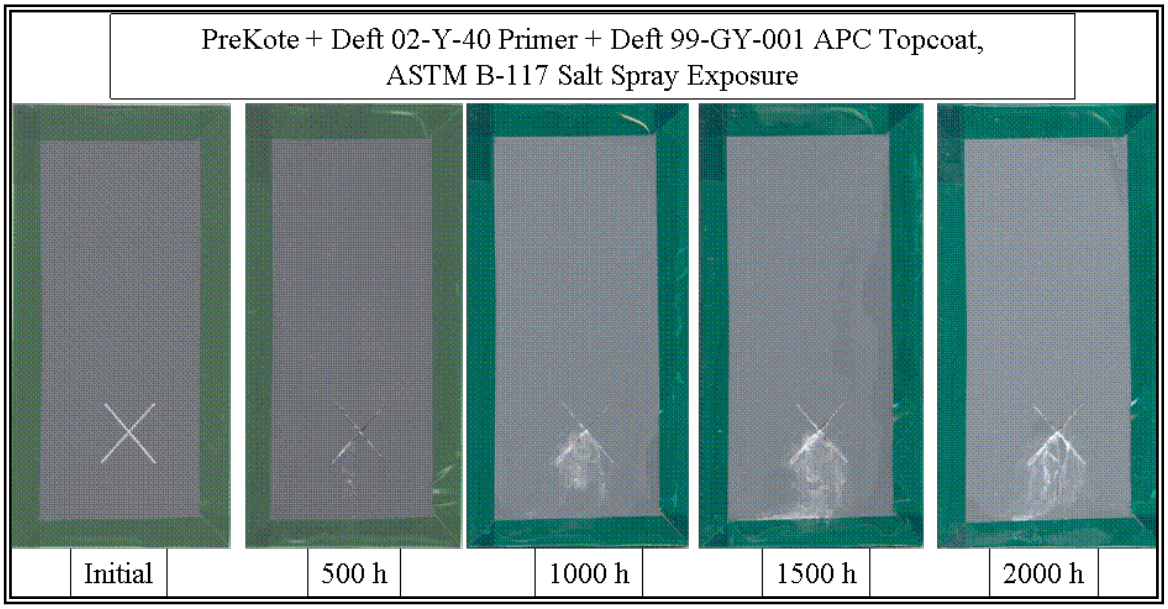


Figure 47: PreKote + MIL-PRF-23377 Chromated Primer + MIL-PRF-85285 APC Topcoat; ASTM B 117 Results

Continued testing through Phase I and II on BAM-PPV samples were evaluated after 2000 hours of exposure to neutral salt fog spray. After examination of the panels, they were assigned either pass, low and poor. These ratings were based on comparison to controls. Coating systems with no more than minor corrosion in the scribe (rating 0 – 2) received a "pass" rating. Systems with little undercutting (rating 0 – 2) received a "low" rating. Systems with greater amounts of corrosion in the scribe and undercutting, or those with blistering received a "poor" rating. Pretreatment-only systems received "pass/fail" ratings based upon their performance per MIL-DTL-81706. The data are given in Tables 29-32. The control system is highlighted in **green** for easy reference.

Coating systems with acceptable performance are highlighted in **blue**. Comparative photographs of the coating systems performance in ASTM B 117 are given in Figures 48-62.

Table 29: ASTM B 117 Salt Spray Ratings (336 h)
(Pretreated Systems)

AA2024-T3 Data			**AA7075-T6 Data**	
Pretreatment Only	**Rating**		**Pretreatment Only**	**Rating**
Alodine	**control**		**Alodine**	**control**
BAM-PPV	fail		BAM-PPV	fail
PreKote	fail		PreKote	fail
AA5086-H116 Data			**AA6061-T6 Data**	
Alodine	**control**		**Alodine**	**control**
BAM-PPV	**pass**		**BAM-PPV**	**pass**
PreKote	fail		PreKote	fail

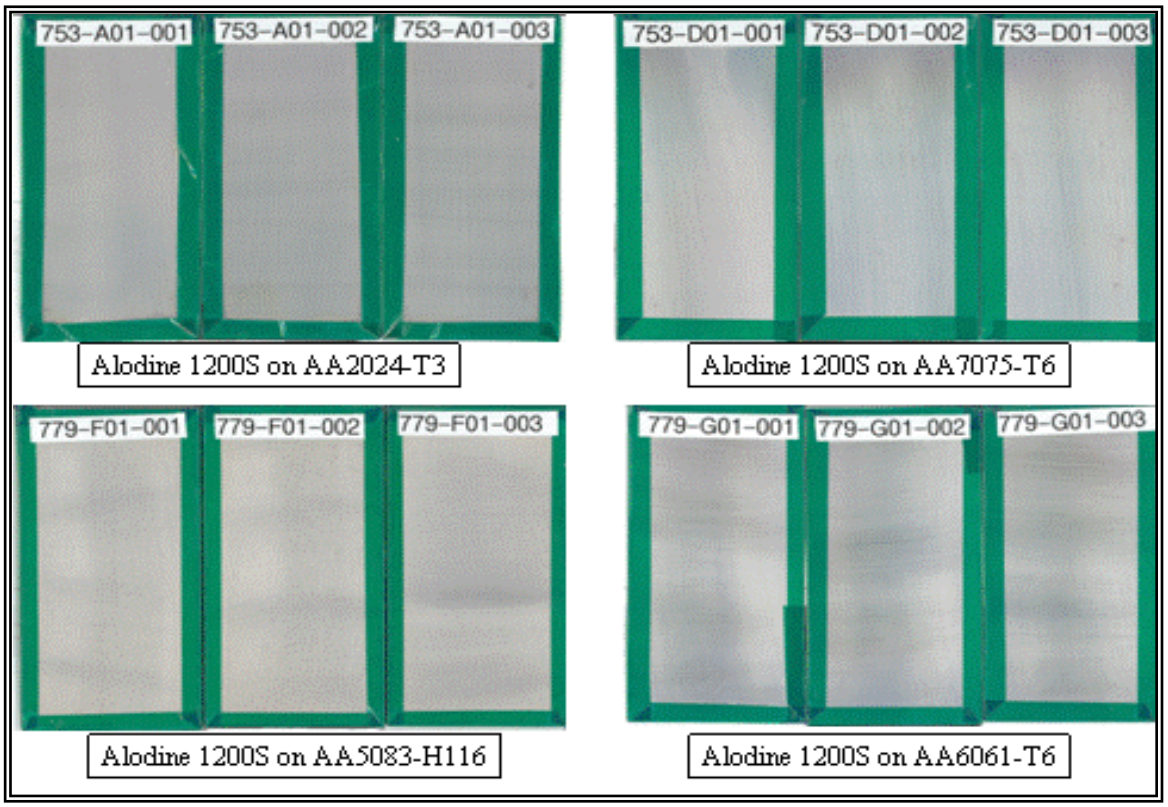


Figure 48: Neutral Salt Spray Results for Alodine 1200S after 336 hours of Exposure

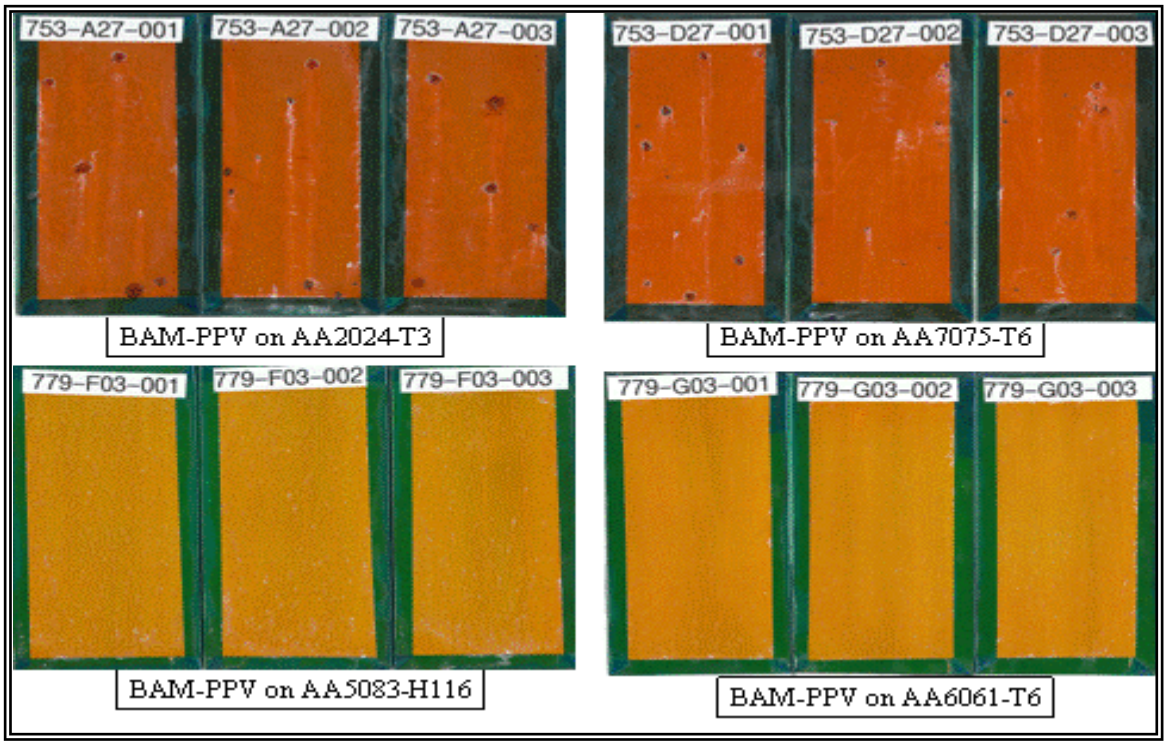


Figure 49: Neutral Salt Spray Results for BAM-PPV after 336 hours of Exposure.

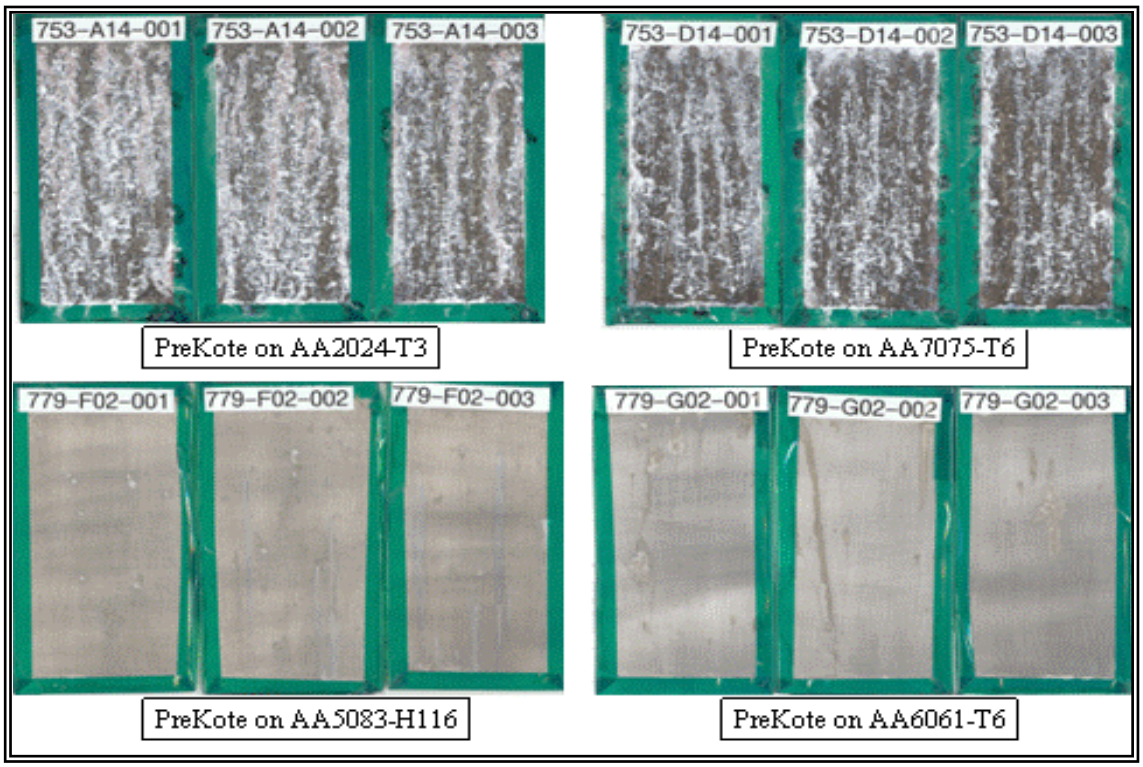


Figure 50: Neutral Salt Spray Results for PreKote after 336 hours of Exposure

Table 30: ASTM B 117 Salt Spray Ratings (2000 hours) (Primed Systems)		
Coating System (No Topcoat)	**AA2024-T3 Data**	**AA7075-T6 Data**
Pretreatment + Primer	**Rating**	**Rating**
Alodine 1200S Deft 02-Y-40 chromated epoxy	**control 1 0 0**	**control 0 0 0**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	**pass 2 0 0**	**pass 1 0 0**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	**pass 1 0 0**	low 1 1 0
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy	**pass 1 0 0**	**pass 1 0 0**
BAM-PPV + Deft 02-Y-40A □chromate epoxy	**pass 1 0 0**	**pass 1 0 0**
BAM-PPV + Deft 02-GN-083 nonchromated epoxy	**pass 2 0 0**	low 2 2 0
BAM-PPV + Deft 02-GN-084 nonchromated epoxy	**pass 2 0 0**	low 2 2 0
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy	low 2 1 0	poor 2 3 0
PreKote + Deft 02-Y-40A □chromate epoxy	**pass 1 0 0**	**pass 1 0 0**
PreKote + Deft 02-GN-083 nonchromated epoxy	low 2 2 0	poor 2 3 0
PreKote + Deft 02-GN-084 nonchromated epoxy	poor 2 3 0	low 2 2 0
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy	poor 2 1 2	low 2 2 0

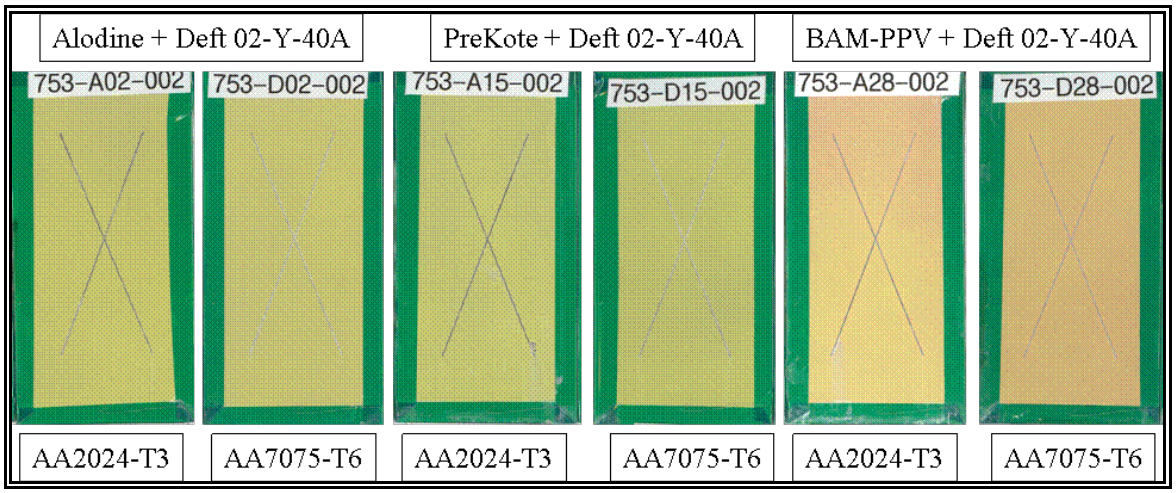


Figure 51: Salt Spray Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A after 2000 hours of Exposure

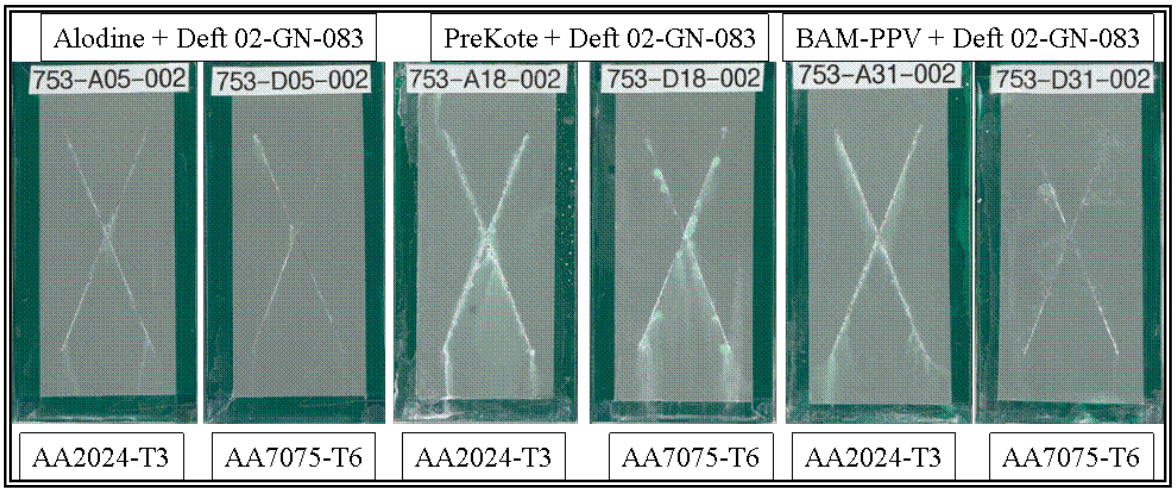


Figure 52: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 after 2000 hours of Exposure

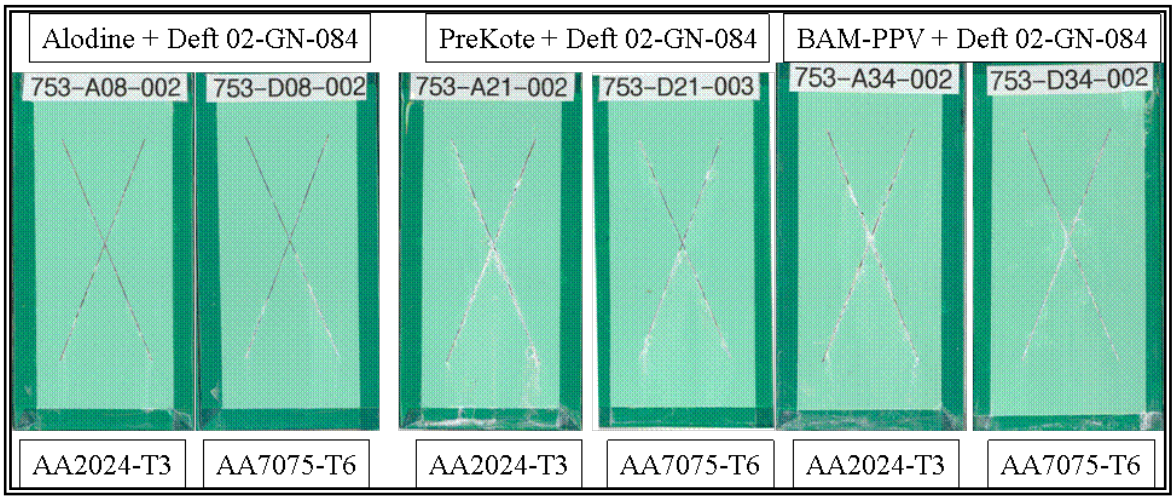


Figure 53: Neutral Salt Spray Results for Pretreatments MIL-PRF-23377N Nonchromated Epoxy Primer Deft 02-GN-084 after 2000 hours of Exposure

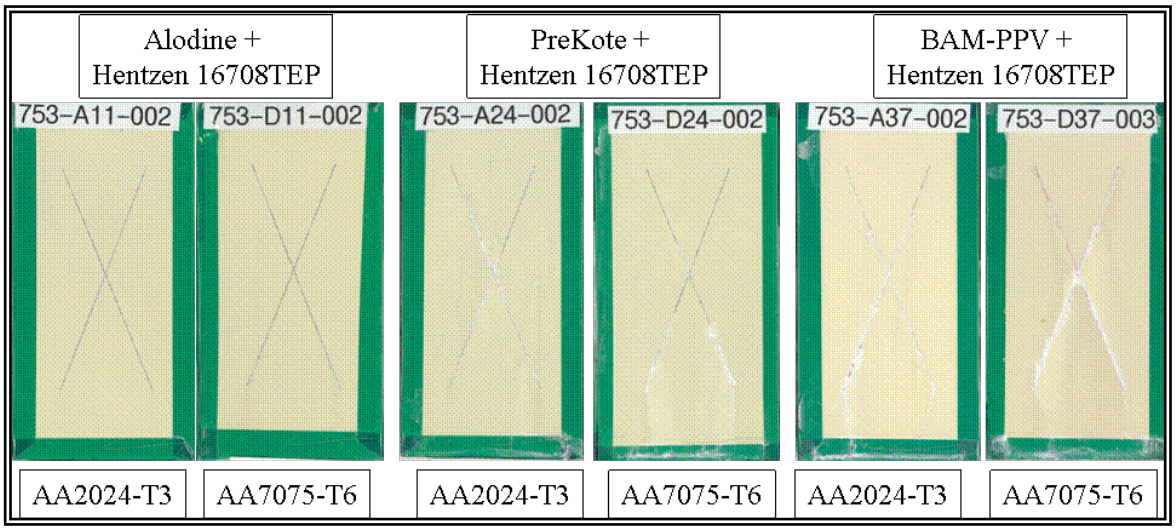


Figure 54: Neutral Salt Spray Results for Pretreatments MIL-PRF-23377 Nonchromated Epoxy Primer Hentzen 16708TEP after 2000 hours of Exposure

Table 31: ASTM B 117 Salt Spray Ratings (2000 hours) (MIL-PRF-85285 Standard Topcoat)		
Coating System	**AA2024-T3 Data**	**AA7075-T6 Data**
Pretreatment + Primer + Topcoat	**Rating**	**Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control 1 0 0**	**control 1 0 0**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 1 0 0**	**pass 1 0 0**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 2 0 0**	**pass 1 0 0**
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 2 0 0**	low 1 1 0
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 1 0 0**	low 1 1 0
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**pass 1 0 0**	**pass 1 0 0**
BAM-PPV + Deft 02-GN-084 nonchromated epoxy+ Deft 03-GY-321 (85285)	pass 2 0 0	poor 2 5 0
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	low 2 2 0	low 2 2 0
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	pass 1 0 0	poor 1 4 0
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	low 1 1 0	low 1 1 0
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	poor 2 3 0	low 1 2 0
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	low 1 1 0	low 1 2 0

Table 32: ASTM B 117 Salt Spray Ratings (2000 hours) (MIL-PRF-85285 APC Topcoat)		
Coating System	**AA2024-T3 Data**	**AA7075-T6 Data**
Pretreatment + Primer + Topcoat	**Rating**	**Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	**control 2 0 0**	**control 1 0 0**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 2 0 0**	**pass 1 0 0**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 2 0 0**	low 1 1 0
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 2 0 0**	low 1 1 0
BAM-PPV + Deft 02-Y-40A □hromate epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 1 0 0**	low 1 1 0
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	low 1 2 0	low 1 1 0
BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	low 1 2 0	poor 2 5 0
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	low 2 2 0	low 2 2 0
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	**pass 1 0 0**	**pass 1 0 0**
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	poor 2 3 0	low 1 1 0
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	poor 2 3 0	low 1 2 0
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	low 1 1 0	low 1 1 0

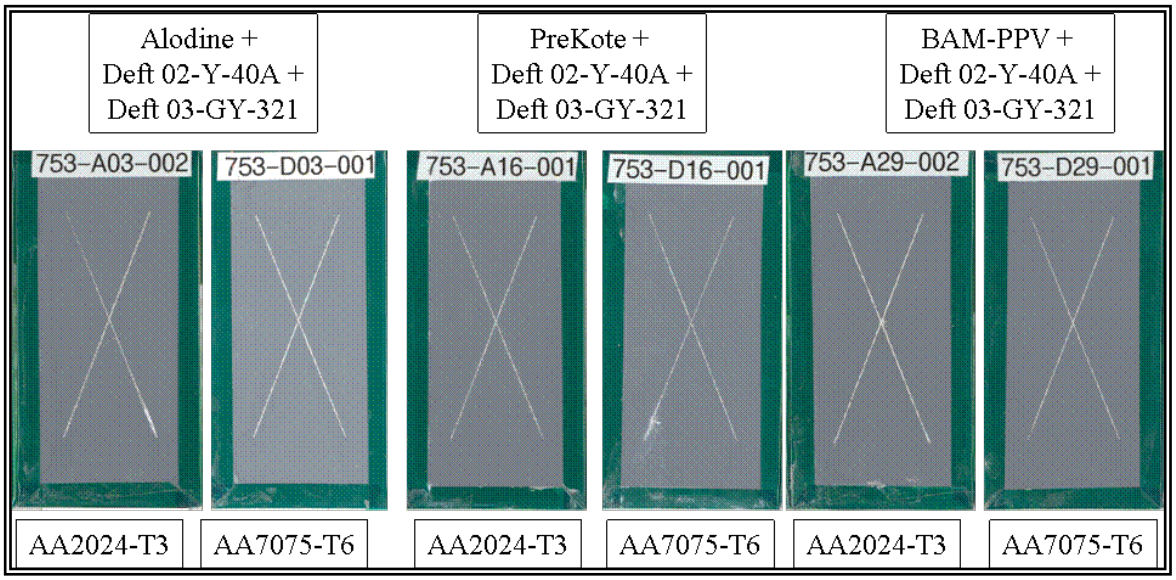


Figure 55: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 2000 hours of Exposure

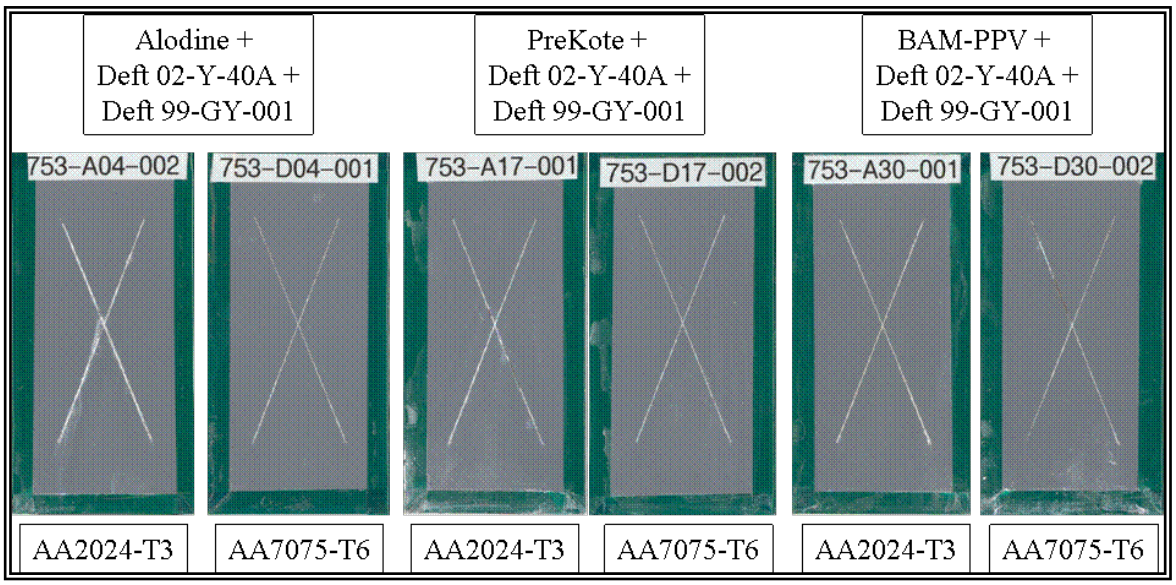


Figure 56: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 2000 hours of Exposure

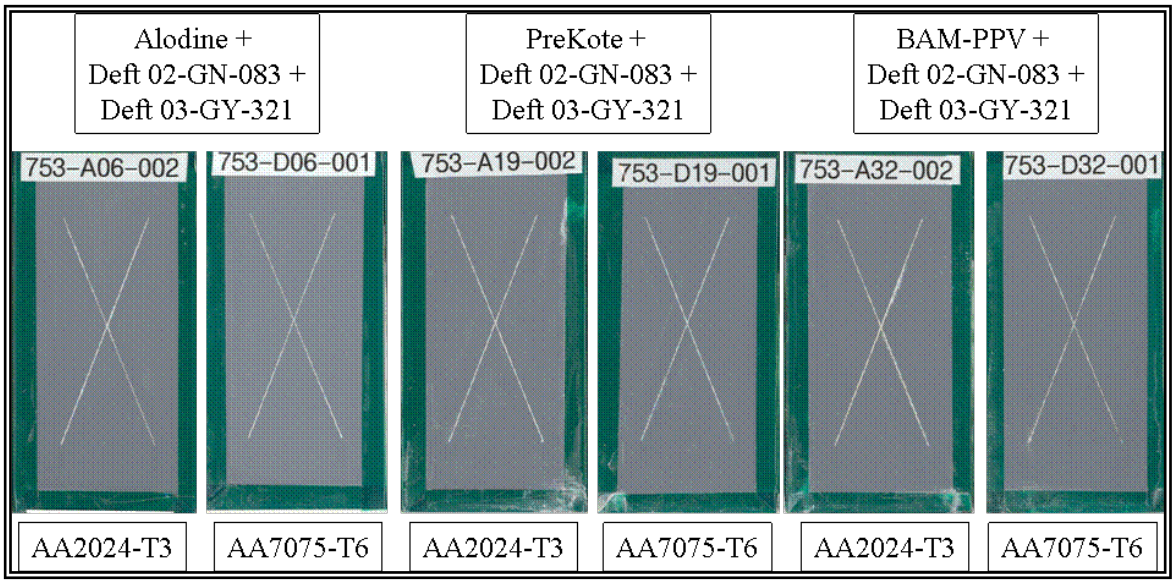


Figure 57: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 2000 hours of Exposure

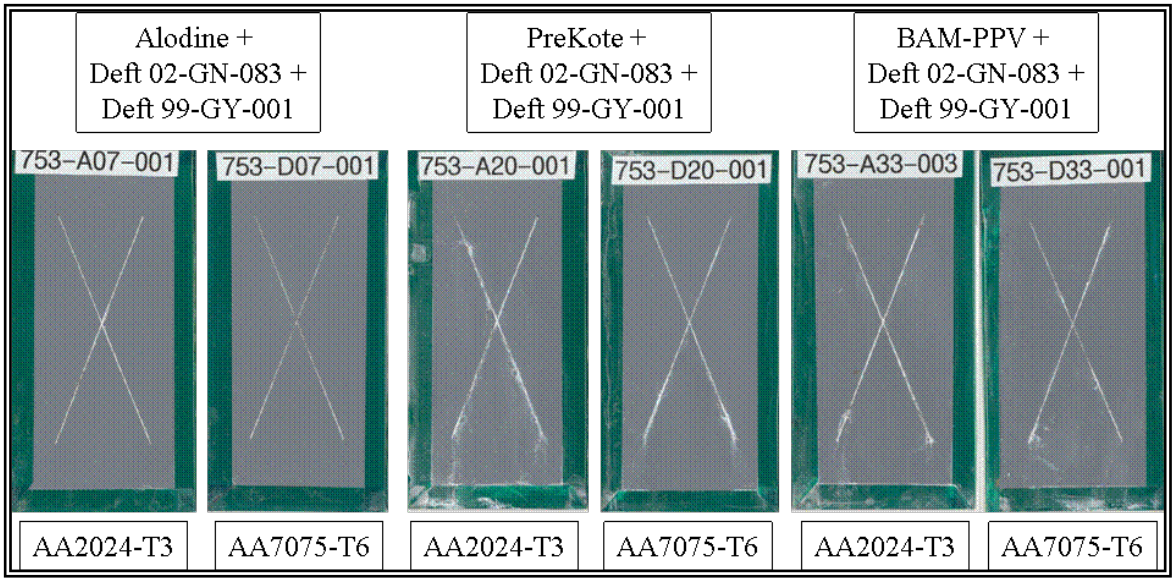


Figure 58: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 2000 hours of Exposure

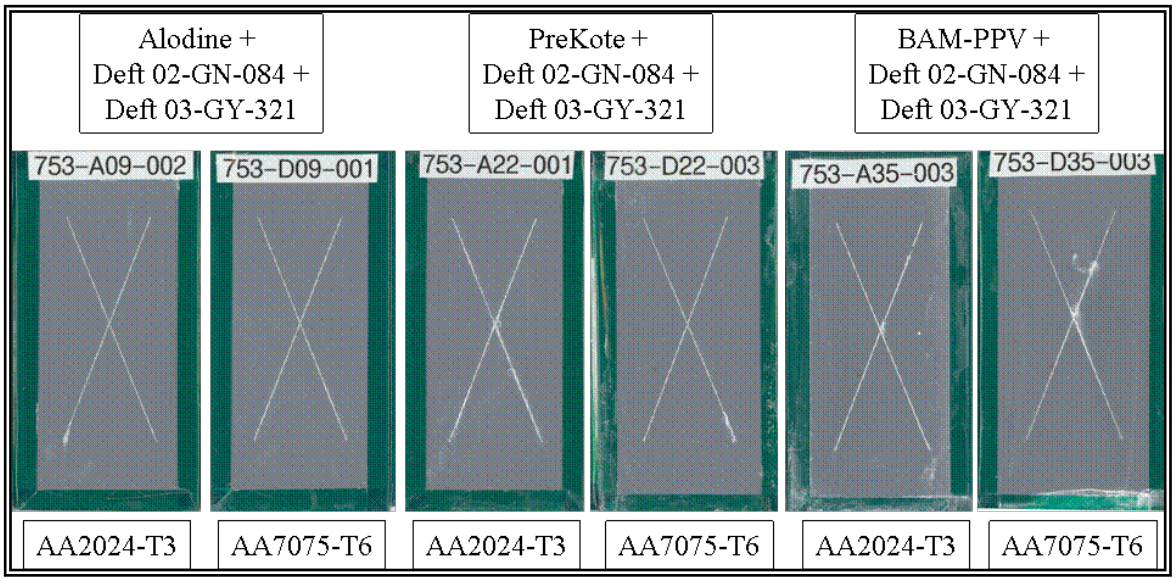


Figure 59: Neutral Salt Spray Results for Pretreatments with Nonchromated Epoxy Primer Deft 02-GN-084 and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 2000 hours of Exposure

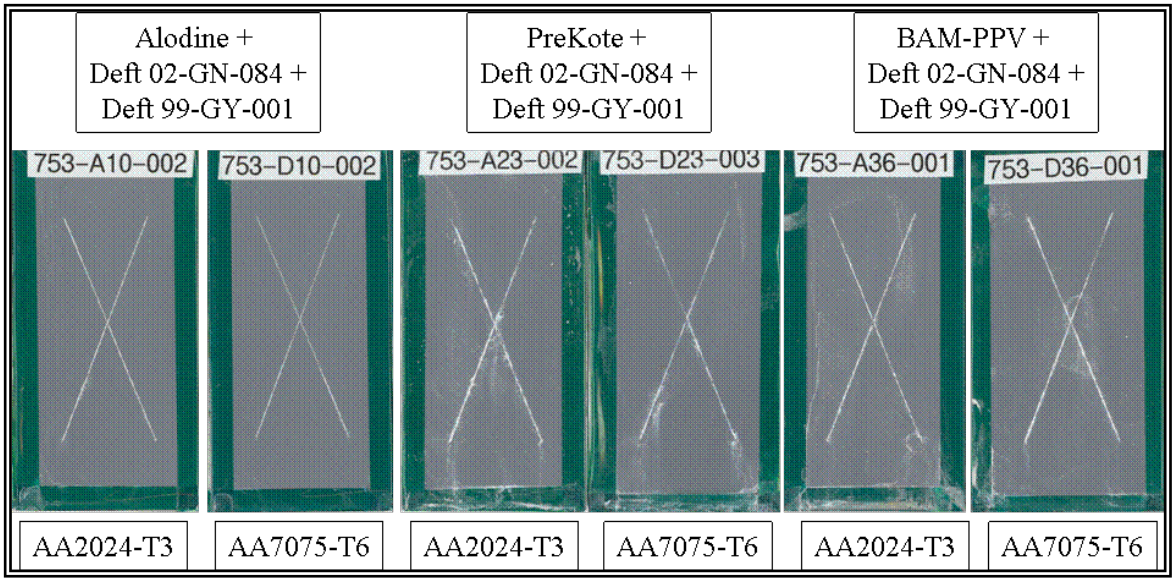


Figure 60: Neutral Salt Spray Results for Pretreatments with Nonchromated Epoxy Primer Deft 02-GN-084 and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 2000 hours of Exposure

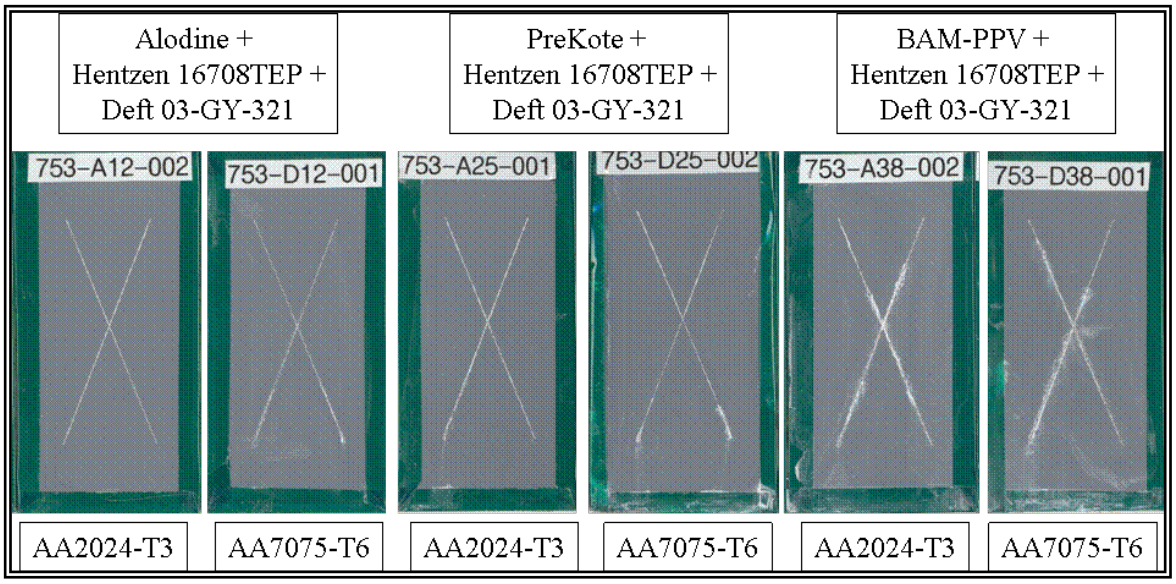


Figure 61: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Hentzen 16708TEP and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 2000 hours of Exposure

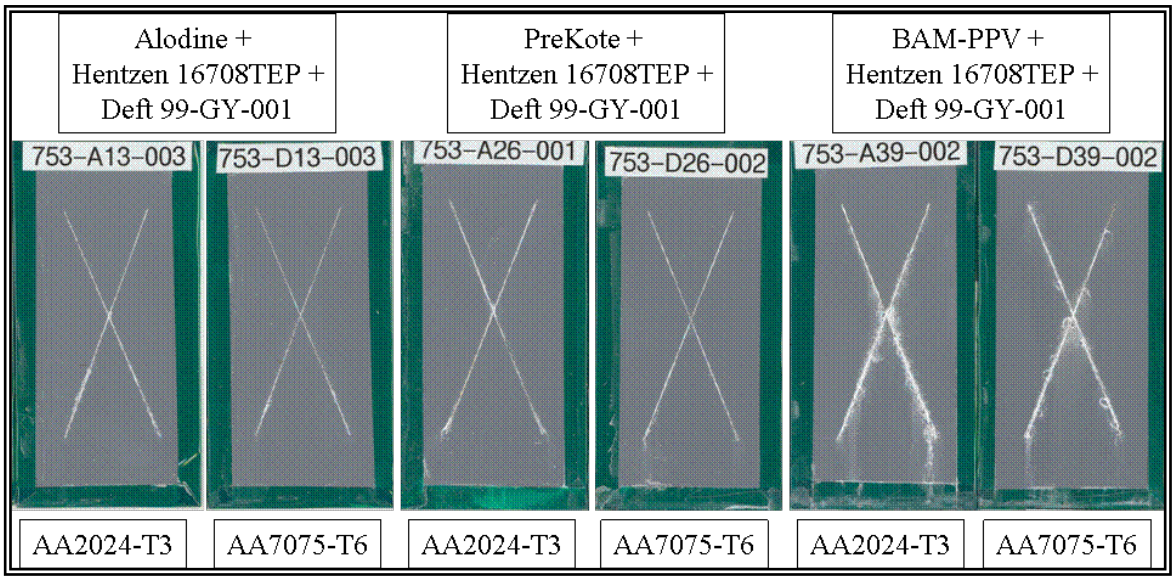


Figure 62: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Hentzen 16708TEP and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 2000 hours of Exposure

6.1.1.1.3.2 GM9540P Accelerated Corrosion Test

The GM9540P is an accelerated corrosion test developed by General Motors that was designed for the evaluation of assemblies and components. The procedure followed by CTIO is an adaptation of that procedure in that it follows the exposure cycles but does not follow specimen design or require mass loss data analysis. This method is the preferred method for accelerated corrosion testing of steel substrates and has provided a

better comparison to real-world corrosion phenomena than ASTM G 85 prohesion has provided, historically. Typically, accelerated corrosion test performance is not rated by the brightness of the scribe or the character of the scribe corrosion since only a very small amount of Fe_2O_3 and FeOOH is necessary to generate visible discoloration which often leads to erroneous "fail" ratings assigned for scribe brightness and failure of the coating. For these systems, samples were rated based upon SSPC/NACE guidelines which recommend ASTM D 714 for blistering of the substrate and ASTM D 610 for corrosion of steel. In general, coating systems that receive ratings which are similar to those assigned to the control system will receive an overall "acceptable" rating. The data are given in Tables 33 and 34. The control system is highlighted in **green**. Systems with overall acceptable performance are highlighted in **blue**. Photographs of coating system behavior in GM9540P exposure are given in Figures 63-70. Ratings of "acceptable", "low", and "poor" were assigned based upon comparative performance with the control. Any systems with ratings of 9S or better in accordance with ASTM D 610 and minor blistering (0 – 2) in accordance with the CTIO scale based upon ASTM D 714 received an "acceptable" rating. Any systems with greater corrosion than 9S or blistering of "3" received a "low" rating and with blistering greater than "3" received a "poor" rating.

BAM-PPV was examined as a replacement for both Cd and Prekote-plated substrates to determine the corrosion effectiveness of BAM-PPV. Both Cd and Prekote pretreatments were used as standards to measure the effectiveness of the NAWCWD alternative pretreatment coatings in the GM 9540P accelerated weathering tests on steel substrates. Typically steel substrates used Cd plating as the method for corrosion protection. Therefore, BAM-PPV was compared to this standard treatment.

Table 33: GM9540P Accelerated Corrosion (MIL-PRF-85285 Topcoat)		
Coating System	**4130 Solvent Cleaned Data**	**4130 Grit-Blasted Data**
Pretreatment + Primer + Topcoat	**Rating**	**Rating**
Cadmium Plated + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control 9S / 0**	**control 9S / 0**
Cadmium Plated + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 1	acceptable 9S / 1
Cadmium Plated + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 1	acceptable 9S / 1
Cadmium Plated + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 1	acceptable 9S / 1
BAM-PPV+ Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 1	acceptable 9S / 2
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 1	acceptable 9S / 2
BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	poor 9S / 5	acceptable 9S / 1
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 1	acceptable 9S / 2
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 2	low 9S / 3
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	poor 9S / 4	low 9S / 3
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	poor 9S / 5	acceptable 9S / 2
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	poor 9S / 4	low 9S / 3

Table 34: GM9540P Accelerated Corrosion (MIL-PRF-85285 APC Topcoat)		
Coating System	**4130 Solvent Cleaned Data**	**4130 Grit-Blasted Data**
Pretreatment + Primer + Topcoat	**Rating**	**Rating**
Cadmium Plated + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control 9S / 1**	**control 9S / 0**
Cadmium Plated + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 1	acceptable 9S / 0
Cadmium Plated + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 1	acceptable 9S / 1
Cadmium Plated + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 1	acceptable 9S / 1
BAM-PPV+ Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 2	acceptable 9S / 1
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	low 9S / 3	acceptable 9S / 1
BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 2	acceptable 9S / 1
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 1	acceptable 9S / 1
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 1	acceptable 9S / 2
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 2	acceptable 9S / 2
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	acceptable 9S / 2	acceptable 9S / 1
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	poor 9S / 4	acceptable 9S / 1

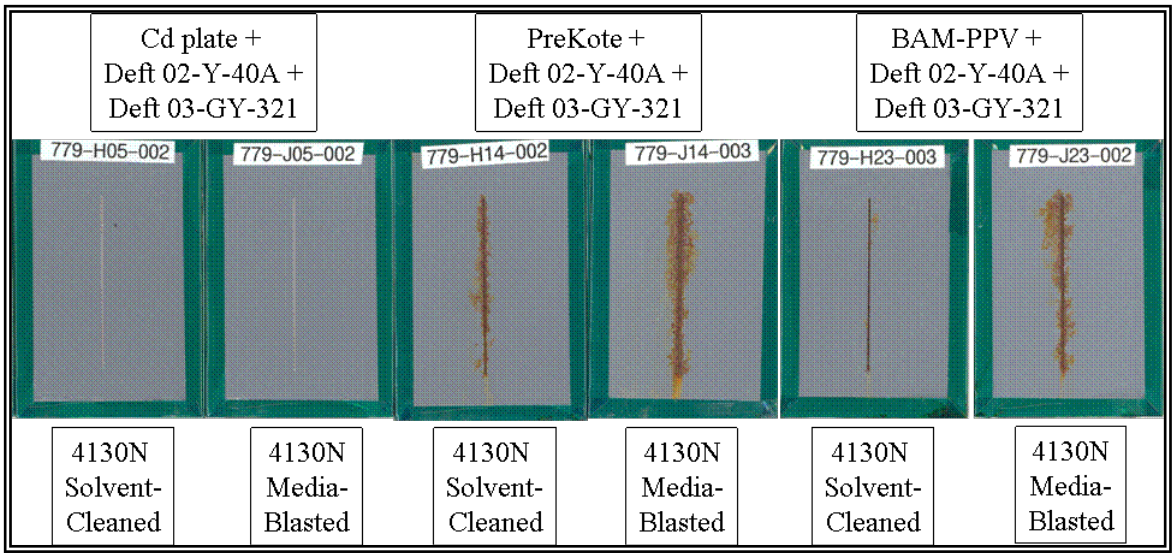


Figure 63: GM9540P Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 40 Cycles of Exposure

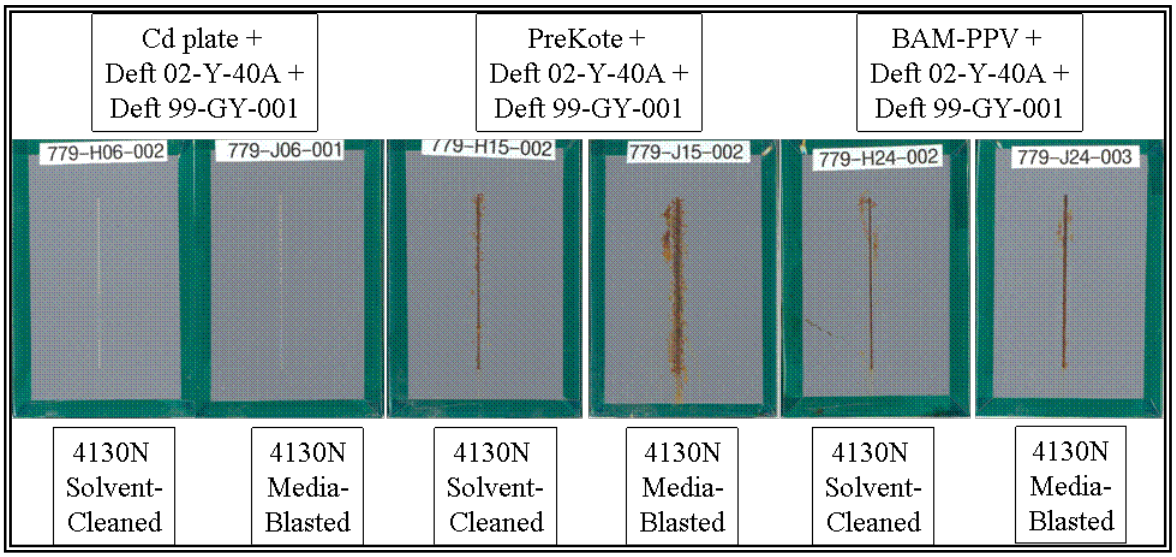


Figure 64: GM9540P Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 40 Cycles of Exposure

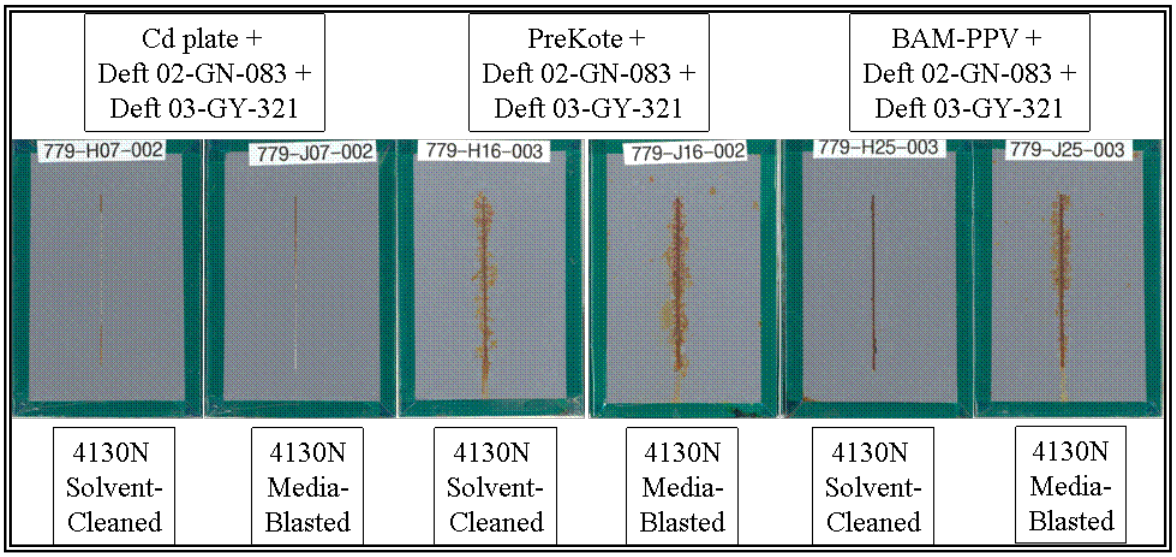


Figure 65: GM9540P Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 40 Cycles of Exposure

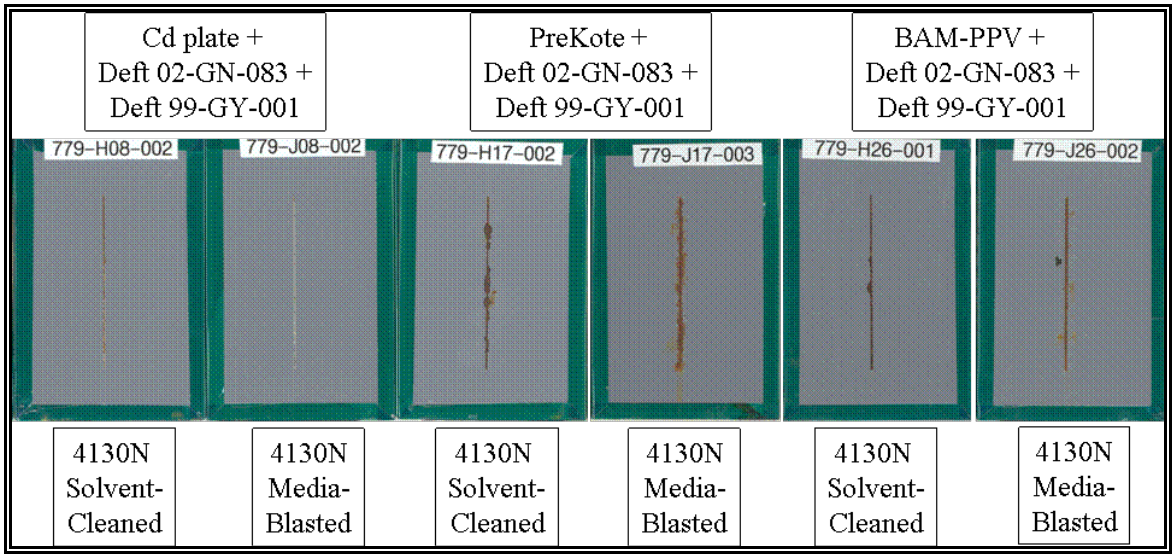


Figure 66: GM9540P Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 40 Cycles of Exposure

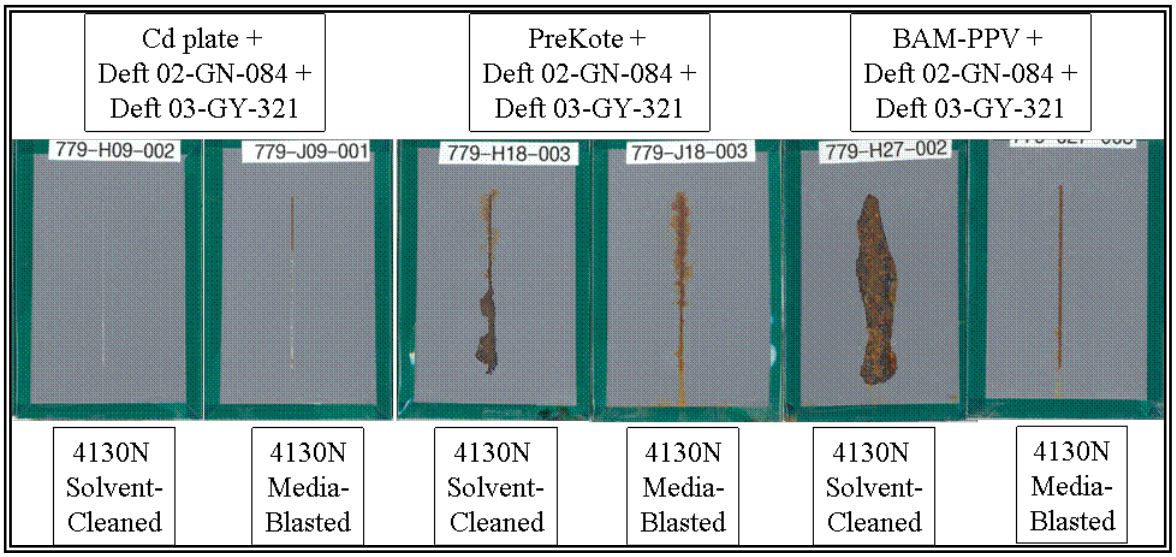


Figure 67: GM9540P Results for Pretreatments with Nonchromated Epoxy Primer Deft 02-GN-084 and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 40 Cycles of Exposure

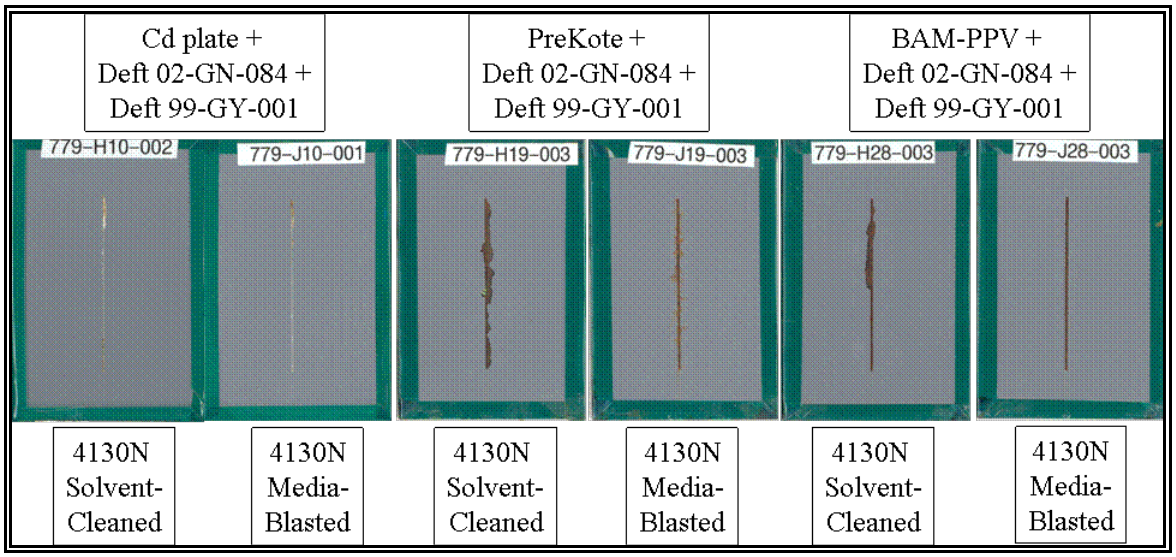


Figure 68: GM9540P Results for Pretreatments with Nonchromated Epoxy Primer Deft 02-GN-084 and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 40 Cycles of Exposure

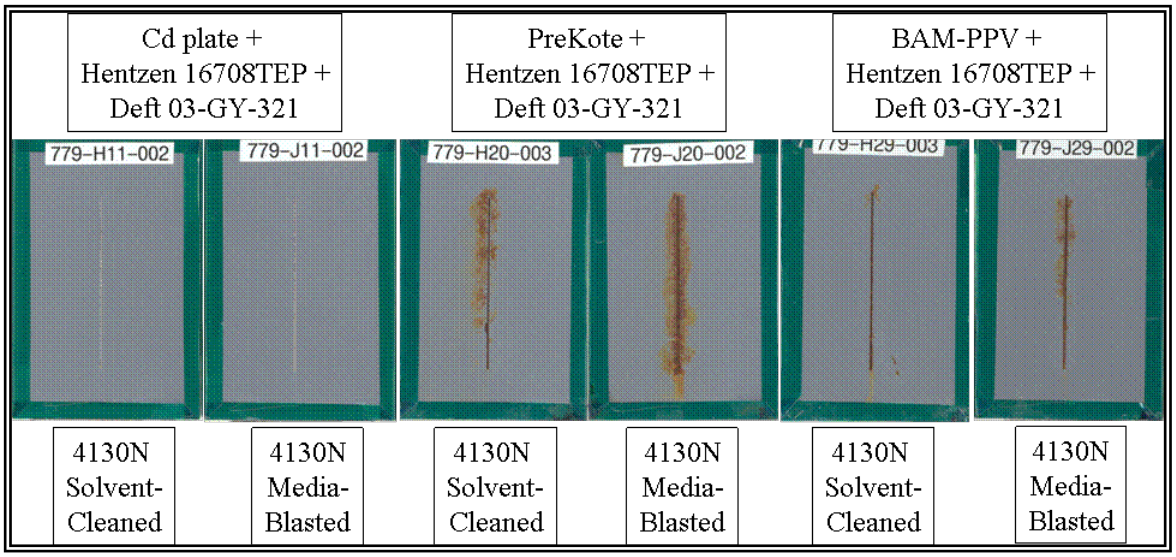


Figure 69: GM9540P Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Hentzen 16708TEP and MIL-PRF-85285 Topcoat Deft 03-GY-321 after 40 Cycles of Exposure

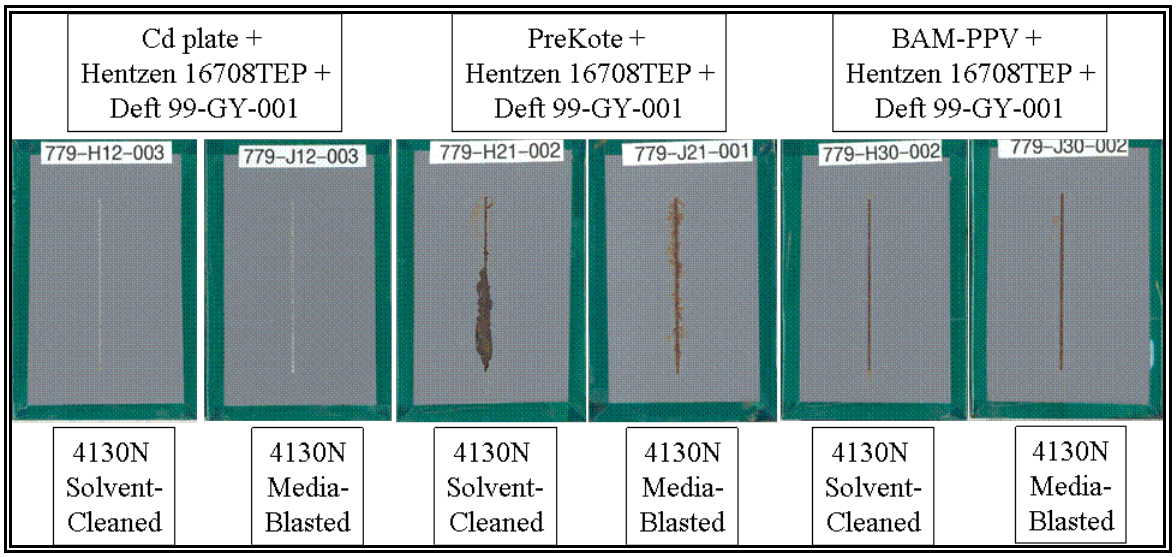


Figure 70: GM9540P Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Hentzen 16708TEP and MIL-PRF-85285 APC Topcoat Deft 99-GY-001 after 40 Cycles of Exposure

6.1.1.1.3.3 Xenon-Arc Accelerated Weathering Test

The MIL-PRF-85285 requirement states that "topcoated systems demonstrate a color change (ΔE) of no more than 1 unit after 500 hours of exposure to xenon-arc light"[78]. Color values are measured in CIE L, a, b units, and the ΔE is calculated per ASTM D 2244 from the following equation 3. All systems received a pass rating for this test. Control system is given in **green**.

Equation 3 $$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$$

Where;

$$\Delta L = L_{\text{final}} - L_{\text{initial}}$$
$$\Delta a = a_{\text{final}} - a_{\text{initial}}$$
$$\Delta b = b_{\text{final}} - b_{\text{initial}}$$

In a realistic exposure environment, coatings on aircraft will be exposed to sunlight (UV), moisture, and salt spray conditions and not simply one condition for an extended period of time. To simulate the combination of these environmental factors, alternating exposure between xenon-arc exposure and salt spray exposure was performed on topcoated systems. Exposure conditions alternated after every 500 hours until a total of 2000 hours of exposure had been reached for both exposure conditions. Color data, salt spray corrosion resistance, and crosshatch adhesion behavior are reported after 1000 hours and 2000 hours of accumulated exposure to xenon-arc and salt spray. The data are given in Tables 35-37. There are no "pass/fail" assignments. It is intended that this data will be compared to the outdoor weathering data after 24 months of exposure to determine if there are any correlations between the data sets. The control system is given in **green.**

Table 35: Xenon-Arc Exposure (MIL-PRF-85285 and APC Topcoats)			
Coating Systems	**Color Data (500 hours)**	**Coating Systems**	**Color Data (500 hours)**
Pretreatment + Primer + Topcoat	**□E**	**Pretreatment + Primer + Topcoat**	**□E**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control 0.4**	**Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)**	**control 0.6**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	0.3	Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	0.3
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	0.5	Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	0.3
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	0.8	Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	0.2
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	0.3	BAM-PPV + Deft 02-Y-40A □ chromate epoxy + Deft 99-GY-001 polyurethane (APC)	0.3
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	0.3	BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	0.4
BAM-PPV + Deft 02-GN-084 nonchromated epoxy+ Deft 03-GY-321 (85285)	0.2	BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	0.3
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	0.5	BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	0.2
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	0.5	PreKote + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	0.4
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	0.4	PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	0.3
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	0.5	PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	0.3
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	0.3	PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	0.2

Table 36: Xenon-Arc and Salt Spray Exposure (MIL-PRF-85285 Standard Topcoat) (Alternating Every 500 hours, Total of 2000 hours Each)					
Coating System	**Corrosion Resistance**	**Crosshatch Adhesion**	**Corrosion Resistance**	**Color Data**	**Crosshatch Adhesion**
Pretreatment + Primer + Topcoat	**Rating (1000 h)**	**Rating (1000 h)**	**Rating (2000 h)**	**ΔE (2000 h)**	**Rating (2000 h)**
Alodine 1200S + Deft 02-Y-40A chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control 0 0 0**	**control 5B**	**control 1 0 0**	**control 2.7**	**control 4B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	1 0 0	5B	2 0 0	2.8	5B
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	1 0 0	5B	2 1 0	2.3	4B
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	1 0 0	4B	2 0 0	2.5	3B
BAM-PPV + Deft 02-Y-40A chromate epoxy + Deft 03-GY-321 polyurethane (85285)	1 3 0	3B	2 5 0	1.6	0B
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	1 1 0	4B	2 5 0	1.9	0B
BAM-PPV + Deft 02-GN-084 nonchromated epoxy+ Deft 03-GY-321 (85285)	1 1 0	4B	2 3 0	1.9	0B
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	1 1 0	4B	2 2 0	2.9	0B
PreKote + Deft 02-Y-40A chromate epoxy + Deft 03-GY-321 polyurethane (85285)	0 0 0	4B	1 0 0	2.2	4B
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	1 1 0	5B	2 2 0	2.1	5B
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	1 1 0	4B	2 1 0	2.2	5B
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	1 1 0	3B	2 2 0	3.2	2B

Table 37: Xenon-Arc and Salt Spray Exposure (MIL-PRF-85285 APC Topcoat) (Alternating Every 500 hours, Total of 2000 hours Each)					
Coating System	**Corrosion Resistance**	**Crosshatch Adhesion**	**Corrosion Resistance**	**Color Data**	**Crosshatch Adhesion**
Pretreatment + Primer + Topcoat	**Rating (1000 h)**	**Rating (1000 h)**	**Rating (2000 h)**	**□E (2000 h)**	**Rating (2000 h)**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	**control 0 0 0**	**control 5B**	**control 1 0 0**	**control 0.7**	**control 4B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1 0 0	5B	2 0 0	0.9	5B
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1 0 0	5B	2 0 0	0.8	5B
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1 0 0	4B	2 0 0	0.7	1B
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	1 0 0	0B	2 5 0	0.6	0B
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1 3 0	1B	2 5 0	0.7	1B
BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1 4 0	0B	2 4 0	0.8	0B
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1 2 0	3B	2 5 0	0.8	0B
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	1 0 0	5B	1 0 0	0.7	4B
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1 1 0	5B	2 1 0	0.7	5B
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1 0 0	5B	2 1 0	0.7	4B
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	1 0 0	4B	2 1 0	0.6	2B

6.1.1.1.4 Additional Tests

Several additional tests were performed on BAM-PV pretreatment coatings to determine its robustness as compared to current AF pretreatment coatings. These additional tests were selected by the AF to examine the degree to which BAM-PPV pretreatment coating can perform as well as current coatings when exposed to the following tests: pencil hardness, fluid resistance, impact flexibility, repairability, storage and stability.

6.1.1.1.4.1 ASTM D 3363 Pencil Hardness (MIL-PRF-23377, MIL-PRF-85285)

This test follows ASTM D 3363 and provides a rapid determination of the hardness of organic coatings. The data are comparative in nature, meaning that the behavior of the control system is often used as a metric for the rating of the remaining systems. Often, ratings which fall within B and H are considered "acceptable", but this can vary based upon the type of the coating system. The data is presented primarily for comparative purposes, and no firm "pass/fail" ratings are given here. This test was performed on (*i*) primed and (*ii*) topcoated systems using BAM-PPV as the pretreatment coating with control samples. The data are given in Tables 38-40. The control system is highlighted in **green**.

Table 38: ASTM D 3363 Pencil Hardness (Primed Systems)

Coating System (No Topcoat)	**AA2024-T3 Data**
Pretreatment + Primer	**Rating**
Alodine 1200S Deft 02-Y-40 chromated epoxy	**control H**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	3H
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	H
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy	2H
BAM-PPV + Deft 02-Y-40A □chromate epoxy	2H
BAM-PPV + Deft 02-GN-083 nonchromated epoxy	HB
BAM-PPV + Deft 02-GN-084 nonchromated epoxy	HB
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy	HB
PreKote + Deft 02-Y-40A □chromate epoxy	F
PreKote + Deft 02-GN-083 nonchromated epoxy	2H
PreKote + Deft 02-GN-084 nonchromated epoxy	H
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy	4H

Table 39: ASTM D 3363 Pencil Hardness (MIL-PRF-85285 Topcoat)	
Coating System	**AA2024-T3 Data**
Pretreatment + Primer + Topcoat	**Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control HB**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	H
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	2H
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	HB
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	HB
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	HB
BAM-PPV + Deft 02-GN-084 nonchromated epoxy+ Deft 03-GY-321 (85285)	HB
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	2B
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	HB
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	H
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	2H
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	HB

Table 40: ASTM D 3363 Pencil Hardness (MIL-PRF-85285 APC Topcoat)	
Coating System	**AA2024-T3 Data**
Pretreatment + Primer + Topcoat	**Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	**control HB**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	F
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	2H
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	B
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	HB
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	HB
BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	HB
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	2B
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	HB
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	2H
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	H
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	HB

6.1.1.1.4.2 Hydraulic, Lubricating Oil, and JP-8 Fluid Resistance (MIL-PRF-83282, MIL-L-23699, MIL-PRF-23377, MIL-PRF-85285)

Typical operating environments for aircraft coatings will involve exposure to various fluids. The most common fluids for in-service aircraft systems are MIL-PRF-83282 hydraulic fluid, MIL-L-23699 lubricating oil, and jet fuel. Specific guidelines for how to run these tests are given in MIL-PRF-23377 and MIL-PRF-85285. These tests require the immersion of the coated panel into the fluid for the specified length of time, often at elevated temperature. This is followed by measurement of the change in coating's hardness and adhesion properties. Typically, softening of the coating below a pencil rating of HB, as defined by ASTM D 3363, is cause to reject the performance of the coating system. Adhesion ratings below a 4B, as defined by ASTM D 3359, are not acceptable. Additionally, the data are compared to the ratings of the witness panel for each data set which is exposed to the elevated temperatures but not to the fluids. This provides insight into the overall coating performance of the system but does not provide comparison for assignment of "pass/fail" ratings. Ratings that are cause to reject the performance of the coating system are given in **red**. Those systems with overall acceptable behavior are highlighted **blue**. The data are given in Tables 41-46.

Table 41: MIL-L-23699 Lubricating Fluid Exposure (MIL-PRF-85285 Standard Topcoat)				
Coating System	**AA2024-T3 Data**			
	Pencil Hardness Data			**Crosshatch Adhesion**
Pretreatment + Primer + Topcoat	**Room Temperature Rating**	**Witness Panel (121 °C) Rating**	**Post-Exposure Rating**	**Post-Exposure Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control HB**	**control F**	**control HB**	**control 5B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	H	3H	H	5B
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	2H	2H	H	5B
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	HB	2H	HB	**3B**
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	HB	F	HB	4B
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	HB	2H	HB	4B
BAM-PPV + Deft 02-GN-084 nonchromated epoxy+ Deft 03-GY-321 (85285)	HB	3H	HB	**3B**
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**2B**	2H	**2B**	4B
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	HB	HB	HB	4B
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	H	5H	2H	5B
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	2H	3H	2H	5B
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	HB	2H	HB	5B

Table 42: MIL-PRF-83282 Hydraulic Fluid Exposure (Primed Systems)				
Coating System (No Topcoat)	**AA2024-T3 Data**			
	Pencil Hardness Data			**Crosshatch Adhesion**
Pretreatment + Primer	**Room Temperature Rating**	**Witness Panel (121 °C) Rating**	**Post-Exposure Rating**	**Post-Exposure Rating**
Alodine 1200S Deft 02-Y-40 chromated epoxy	**control H**	**control 2H**	**control 2H**	**control 5B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	3H	2H	2H	5B
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	H	2H	2H	5B
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy	2H	2H	2H	4B
BAM-PPV + Deft 02-Y-40A □chromate epoxy	2H	H	F	5B
BAM-PPV + Deft 02-GN-083 nonchromated epoxy	HB	H	F	5B
BAM-PPV + Deft 02-GN-084 nonchromated epoxy	HB	F	F	5B
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy	HB	F	H	5B
PreKote + Deft 02-Y-40A □chromate epoxy	F	H	H	5B
PreKote + Deft 02-GN-083 nonchromated epoxy	2H	2H	H	5B
PreKote + Deft 02-GN-084 nonchromated epoxy	H	H	H	5B
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy	4H	2H	H	5B

Table 43: MIL-PRF-83282 Hydraulic Fluid Exposure (MIL-PRF-85285 Standard Topcoat)				
Coating System	**AA2024-T3 Data**			
	Pencil Hardness Data			**Crosshatch Adhesion**
Pretreatment + Primer + Topcoat	**Room Temperature Rating**	**Witness Panel (121 °C) Rating**	**Post-Exposure Rating**	**Post-Exposure Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control HB**	**control F**	**control F**	**control 4B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	H	2H	2H	5B
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	2H	H	2H	5B
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	HB	F	F	4B
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	HB	HB	HB	4B
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	HB	F	F	**3B**
BAM-PPV + Deft 02-GN-084 nonchromated epoxy+ Deft 03-GY-321 (85285)	HB	F	F	**3B**
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**2B**	HB	HB	4B
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	HB	HB	HB	4B
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	H	F	H	5B
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	2H	H	H	5B
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	HB	F	F	4B

Table 44: MIL-PRF-83282 Hydraulic Fluid Exposure (MIL-PRF-85285 APC Topcoat)				
Coating System	**AA2024-T3 Data**			
	Pencil Hardness Data			**Crosshatch Adhesion**
Pretreatment + Primer + Topcoat	**Room Temperature Rating**	**Witness Panel (121 °C) Rating**	**Post-Exposure Rating**	**Post-Exposure Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	**control HB**	**control F**	**control F**	**control 4B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	F	H	H	5B
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	2H	F	2H	5B
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	B	F	F	4B
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	HB	HB	HB	**3B**
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	HB	H	F	**3B**
BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	HB	H	H	**3B**
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**2B**	HB	HB	**3B**
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	HB	HB	HB	5B
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	2H	F	H	5B
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	H	H	F	5B
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	HB	HB	HB	4B

Table 45: JP-8 Jet Fuel Exposure (MIL-PRF-85285 Standard Topcoat)			
Coating System	**AA2024-T3 Data**		
	Pencil Hardness Data		**Crosshatch Adhesion**
Pretreatment + Primer + Topcoat	**Room Temperature Rating**	**Post-Exposure Rating**	**Post-Exposure Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control HB**	**control HB**	**control 5B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	H	HB	5B
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	2H	HB	5B
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	HB	**2B**	5B
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	HB	HB	5B
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	HB	HB	5B
BAM-PPV + Deft 02-GN-084 nonchromated epoxy+ Deft 03-GY-321 (85285)	HB	HB	5B
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	**2B**	**2B**	5B
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	HB	HB	5B
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	H	HB	5B
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	2H	HB	5B
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	HB	HB	5B

Table 46: JP-8 Jet Fuel Exposure (MIL-PRF-85285 APC Topcoat)			
Coating Systems	**AA2024-T3 Data**		
	Pencil Hardness Data		**Crosshatch Adhesion**
Pretreatment + Primer + Topcoat	**Room Temperature Rating**	**Post-Exposure Rating**	**Post-Exposure Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	control HB	control HB	control 4B
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	F	H	5B
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	2H	F	5B
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	B	**2B**	5B
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	HB	HB	5B
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	HB	HB	5B
BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	HB	F	5B
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	**2B**	**2B**	5B
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	HB	HB	4B
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	2H	HB	5B
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	H	HB	5B
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	HB	HB	5B

6.1.1.1.4.3 ASTM D 6905 GE Impact Flexibility Testing (MIL-PRF-23377, MIL-PRF-85285)

The impact flexibility testing, as defined by ASTM, determines "the ability of a coating film and its substrate to resist shattering, cracking, or chipping when the film and substrate are distended beyond their original form by impact." MIL-PRF-85285 defines "pass/fail" of this test in terms of the percent flexibility of the topcoat applied over anodized aluminum with no primer. Similarly, MIL-PRF-23377 defines "pass/fail" of this test in terms of the percent flexibility of the primer applied over anodized aluminum with no topcoat applied. Minimum allowable values are 40% and 10%, respectively, but because of these definitions, no "pass/fail" can be assigned to these coating systems. Rather, this data is intended for comparative purposes only, in reference to the control system highlighted in **green**. The data are given in Tables 47-49.

Table 47: ASTM D 6905 GE Impact (Primed Systems)

Coating System (No Topcoat)	**AA2024-T3 Data**
Pretreatment + Primer	**Rating**
Alodine 1200S Deft 02-Y-40 chromated epoxy	**control 40**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	20
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	20
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy	20
BAM-PPV + Deft 02-Y-40A □chromate epoxy	20
BAM-PPV + Deft 02-GN-083 nonchromated epoxy	10
BAM-PPV + Deft 02-GN-084 nonchromated epoxy	10
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy	10
PreKote + Deft 02-Y-40A □chromate epoxy	40
PreKote + Deft 02-GN-083 nonchromated epoxy	20
PreKote + Deft 02-GN-084 nonchromated epoxy	40
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy	20

Table 48: ASTM D 6905 GE Impact (MIL-PRF-85285 Topcoat)	
Coating System	**AA2024-T3 Data**
Pretreatment + Primer + Topcoat	**Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	**control 10**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	10
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	10
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	10
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	10
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	10
BAM-PPV + Deft 02-GN-084 nonchromated epoxy+ Deft 03-GY-321 (85285)	5
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	10
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 03-GY-321 polyurethane (85285)	10
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	5
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	10
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 03-GY-321 polyurethane (85285)	10

Table 49: ASTM D 6905 GE Impact (MIL-PRF-85285 APC Topcoat)	
Coating System	**AA2024-T3 Data**
Pretreatment + Primer + Topcoat	**Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	**control 10**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	5
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	5
Alodine 1200S + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	10
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	5
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	5
BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	5
BAM-PPV + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	10
PreKote + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane (APC)	10
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	5
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	5
PreKote + Hentzen 16708TEP/16709CEH nonchromated epoxy + Deft 99-GY-001 polyurethane (APC)	10

6.1.1.1.4.4 Repairability Testing (T.O. 1-1-8, Table 3-2; MIL-R-81294; MIL-PRF-25134; and TT-R-2918)

Alternative coating systems were tested for repairability and compatibility with the standard coating system of chromate conversion coating, chromate epoxy primer, and polyurethane topcoat. One alternative coating system was BAM-PPV pretreatment, replacing chromate conversion coatings in the standard system. Another alternative coating system was MEH-PPV pretreatment, replacing chromate conversion coatings in the standard system. A third alternative was BAM-PPV pretreatment and the MIL-PRF-23377 qualified nonchromated epoxy primer Deft 03-GN-083 replacing all chromates in the standard coating system. A final system was MEH-PPV pretreatment and the MIL-PRF-23377 qualified non-chromated primer Deft 03-GN-083 replacing all chromates in the standard coating system. Coating systems are given in Table 50.

Table 50: Repairability Coating System Combinations for BAM-PPV Pretreatment Coating

Location on 12 x 12 in. Panel	Primary Coating System			Repair Coating System		
	Pretreatment	**Primer**	**Topcoat**	**Pretreatment**	**Primer**	**Topcoat**
Left Side	Alodine 1200S	Deft 02-Y-40A	Deft ELT 99-GY-001	BAM-PPV	Deft 02-Y-40A	Deft ELT 99-GY-001
Right Side	BAM-PPV	Deft 02-Y-40A	Deft ELT 99-GY-001			
Left Side	Alodine 1200S	Deft 02-GN-083	Deft ELT 99-GY-001	BAM-PPV	Deft 02-GN-083	Deft ELT 99-GY-001
Right Side	BAM-PPV	Deft 02-GN-083	Deft ELT 99-GY-001			
Right Side	MEH-PPV	Deft 02-GN-083	Deft ELT 99-GY-001			

The goal was to identify coating system combinations for repairability of current systems with the alternative coating systems and of an alternative system with itself. Coating systems were tested for weather-ability and adhesion. The samples were coated with the primary coating system identified in Table 50 and exposed for 1000 hours to UVB light. Coatings were sanded down and repaired with the coating identified in Table 54. Samples were again exposed to UVB light for 1000 hours. At the end of testing, color readings were taken on the repaired coatings and adhesion was taken following ASTM B 3359, Method A. Because there are no pass or fail criteria identified for this data, comparison was made with the requirements in MIL-PRF-85285 and MIL-PRF-32239, *Performance Specification: Coating System, Advanced Performance for Aerospace Applications*, for weather-ability of a coating system. The criteria are given in Table 51. Color data is given in Table 52. When comparison is made, the results for the coating systems are satisfactory and no inadequacies were noted. Because there was no control, no “pass/fail” can be assigned. However, it is generally accepted that coating

systems that demonstrate a ΔE less than 1 are performing satisfactorily, and that data is highlighted in **blue**. Figures 71-75 show reparability of BAM-PPV coated panels. These figures show that BAM-PPV was easily removed and replaced with new BAM-PPV coating without any degradation of the coating during accelerated weathering and adhesion tests.

Table 51: Comparative Criteria for Evaluation of Repairability Color Data

Military Specification	Section/Test	Summarized Requirement
MIL-PRF-85285	3.8.2; 4.6.9 Weather Resistance	After xenon arc exposure for 500 hours, color change of less than 1 unit ($\Delta E \leq 1$)
MIL-PRF-32239	3.6.11; 4.6.17.2 Weather Resistance	After UVB exposure for 1500 hours, color change of less than 1 unit ($\Delta E \leq 1$)

Table 52: Color Data, Repairability Testing of BAM-PPV Pretreatment Coating

Primary Coating System	Repair Coating System	Δ*E* after 1000 hours UVB Exposure
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**0.8**
BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	BAM-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**0.7**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	BAM-PPV + Deft 02-GN-083 chromated epoxy + Deft ELT 99-GY-001 polyurethane	**0.7**
BAM-PPV + Deft 02-GN-083 chromated epoxy + Deft ELT 99-GY-001 polyurethane	BAM-PPV + Deft 02-GN-083 chromated epoxy + Deft ELT 99-GY-001 polyurethane	**0.8**

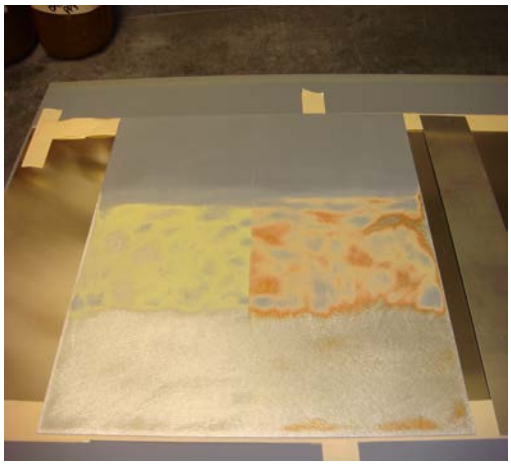


Figure 71: **(Left side)**
Standard Coating
(Alodine MIL-PRF-23377C primer, MIL-PRF-85285 APC Topcoat
Right side: Test Coating System
(BAM-PPV, MIL-PRF-23377C, MIL-PRF-85285 APC)

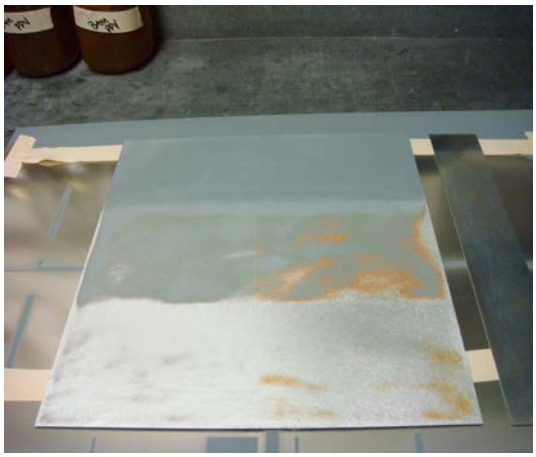


Figure 72: **(Left side)**
(Alodine, MIL-PRF-23377N, Non-chromate primer, MIL-PRF-85285 APC Topcoat
Right side: Test Coating System
(BAM-PPV, MIL-PRF-23377N, MIL-PRF-85285 APC)

Figure 73: Entire panel recoated/ Repaired with test system of BAM-PPV, MIL-PRF-23377C, MIL-PRF-85285 APC

Figure 74:CCC+MIL-PRF-23377C+ MIL-PRF-85285 APC-Repaired w/ BAM-PPV

Figure 75: CCC+ MIL-PRF-23377C+ MIL-PRF 85285APC Repaired w BAM-PPV

6.1.1.1.4.5 Storage and Stability Studies

An area of interest on EAPs has been the shelf-life stability of the material and the effect of shelf life on drying time, recoat time, adhesion, and corrosion resistance. Testing performed by WPAFB has focused on the performance of freshly mixed and 6-month-old BAM-PPV with chromate and nonchromated primers. All systems were topcoated with an advanced performance polyurethane topcoat qualified to MIL-PRF-85285. The coatings applied and the substrates are given in Table 53. All primer and topcoat materials were within date for use designated by the manufacturer. The chromate

conversion coating and PreKote pretreatments were also within date for use. The BAM-PPV was mixed in-house at WPAFB with BAM-PPV powder supplied from NAWCWD. Coatings were applied at ambient laboratory conditions of 77 °F and 50% RH, and these conditions were also maintained during coating cure of 14-days. The following tests were performed with 1000 hours neutral salt fog exposure testing on the BAM-PPV solution after standing for 6 months:

- Federal Test Method Standard 141: Method 4331 Spraying Properties
- Federal Test Method Standard 141: Method 4061 Drying Time
- Federal Test Method Standard 141: Method 6301 Wet Tape Adhesion
- American Society for Testing and Materials, ASTM B 117 Salt Spray
- American Society for Testing and Materials, ASTM D 3359, Method B Tape Test Adhesion
- American Society for Testing and Materials, ASTM D 3363 Pencil Hardness

The results from testing show that the BAM-PPV material that was applied after being held for 6 months performs similarly to the BAM-PPV that was freshly mixed. Comparative data with PreKote and chromate conversion coating data show similar results to those provided in similar testing, validating that the exposure environment and sample generation for these data are comparable to previous data in BAM-PPV studies. There is no significant difference in the adhesion or corrosion resistance performance of BAM-PPV, whether it is freshly mixed or mixed and left on the shelf for 6 months. Salt spray data suggests that the performance of the material is comparable after 1000 hours (see Table 54 and Figures 76 and 77)

Table 53: Storage Stability Laboratory Testing Performed at WPAFB

Pretreatment	**Primer**	**Topcoat**
Alodine 1200S, Chromate Conversion Coating	Deft 02-Y-40, MIL-PRF-23377 Chromated Primer	Deft 99-GY-001, MIL-PRF-85285 ELT
Alodine 1200S, Chromate Conversion Coating	Deft 02-GN-083, MIL-PRF-23377 Nonchromated Primer	Deft 99-GY-001, MIL-PRF-85285 ELT
Alodine 1200S, Chromate Conversion Coating	Deft 02-GN-084, Nonchromated Primer	Deft 99-GY-001, MIL-PRF-85285 ELT
BAM-PPV	Deft 02-Y-40, MIL-PRF-23377 Chromated Primer	Deft 99-GY-001, MIL-PRF-85285 ELT
BAM-PPV	Deft 02-GN-083, MIL-PRF-23377 Nonchromated Primer	Deft 99-GY-001, MIL-PRF-85285 ELT
BAM-PPV	Deft 02-GN-084, Nonchromated Primer	Deft 99-GY-001, MIL-PRF-85285 ELT
PreKote	Deft 02-Y-40, MIL-PRF-23377 Chromated Primer	Deft 99-GY-001, MIL-PRF-85285 ELT
PreKote	Deft 02-GN-083, MIL-PRF-23377 Nonchromated Primer	Deft 99-GY-001, MIL-PRF-85285 ELT
PreKote	Deft 02-GN-084, Nonchromated Primer	Deft 99-GY-001, MIL-PRF-85285 ELT

Table 54: Compiled Data for Storage Stability Testing, AA2024-T3

System	Salt Spray ASTM B 117 1000 h AA2024-T3	Salt Spray ASTM B 117 1000 h AA7075-T6	Adhesion ASTM D 3359	Pencil Hardness ASTM D 3363	Wet Tape
Chromate Conversion Coating MIL-PRF-23377 Chromated Primer MIL-PRF-85285 ELT Topcoat	control 1 0 0	control 0 0 0	control 4B	control B	pass 4A
Chromate Conversion Coating MIL-PRF-23377 Nonchromated Primer MIL-PRF-85285 ELT Topcoat	acceptable 2 0 0	acceptable 1 0 0	pass 5B	F	pass 4A
Chromate Conversion Coating Deft 02-GN-084 Nonchromated Primer MIL-PRF-85285 ELT Topcoat	low 2 1 0	acceptable 1 0 0	pass 5B	B	pass 5A
BAM-PPV (Fresh) MIL-PRF-23377 Chromated Primer MIL-PRF-85285 ELT Topcoat	acceptable 2 0 0	acceptable 1 0 0	fail 3B	B	pass 4A
BAM-PPV (Fresh) MIL-PRF-23377 Nonchromated Primer MIL-PRF-85285 ELT Topcoat	low 2 1 0	poor 2 4 0	fail 2B	HB	fail 2A
BAM-PPV (Fresh) Deft 02-GN-084 Nonchromated Primer MIL-PRF-85285 ELT Topcoat	low 2 1 0	low 2 2 0	fail 2B	2B	pass 5A
BAM-PPV (6 mos) MIL-PRF-23377 Chromated Primer MIL-PRF-85285 ELT Topcoat	acceptable 1 0 0	acceptable 1 0 0	pass 4B	HB	pass 4A
BAM-PPV (6 mos) MIL-PRF-23377 Nonchromated Primer MIL-PRF-85285 ELT Topcoat	acceptable 2 0 0	poor 2 4 0	fail 3B	HB	pass 4A
BAM-PPV (6 mos) Deft 02-GN-084 Nonchromated Primer MIL-PRF-85285 ELT Topcoat	low 2 1 0	poor 2 3 0	variable 2,3,4	2B	pass 4A
PreKote MIL-PRF-23377 Chromated Primer MIL-PRF-85285 ELT Topcoat	acceptable 1 0 0	acceptable 0 0 0	pass 4B	HB	pass 4A
PreKote MIL-PRF-23377 Nonchromated Primer MIL-PRF-85285 ELT Topcoat	low 2 1 0	low 2 2 0	pass 5B	F	pass 5A
PreKote Deft 02-GN-084 Nonchromated Primer MIL-PRF-85285 ELT Topcoat	low 2 1 1	poor 2 3 0	pass 5B	2B	pass 5A

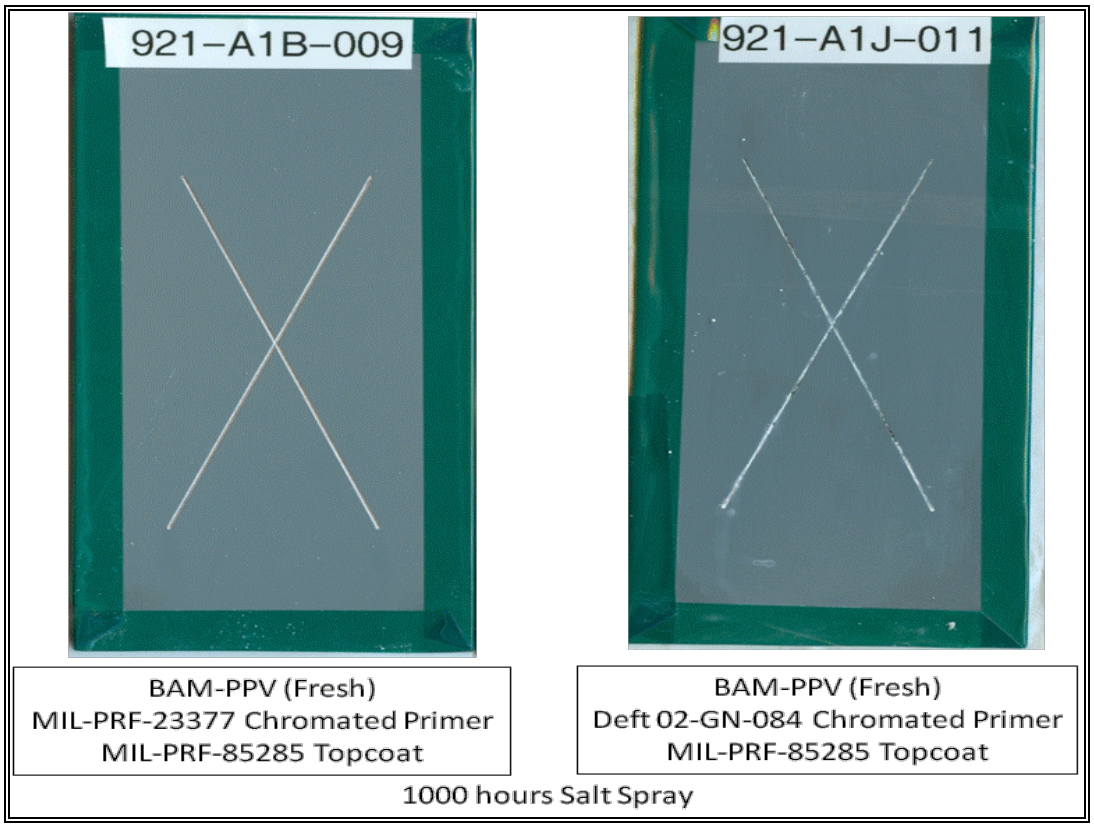


Figure 76: BAM-PPV (As Received) Performance after 1000 hours of Salt Spray

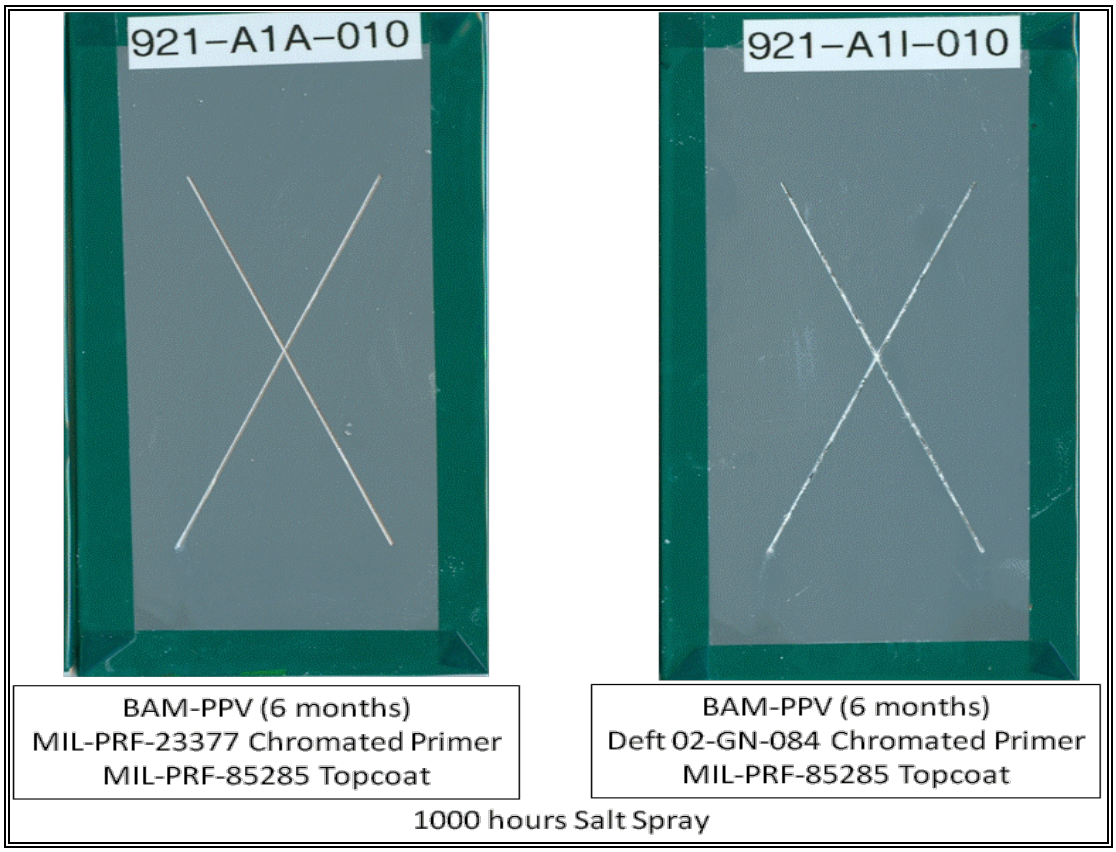


Figure 77: BAM-PPV (6 months in storage) Performance after 1000 hours of Salt Spray

6.1.1.1.5 Conclusions for BAM-PPV Laboratory Testing

The results from the extensive laboratory testing performed on BAM-PPV pretreatment coatings by WPAFB researchers are summarized below:

- ***Results from ASTM B 117 neutral salt spray (NSS) exposure testing verified that BAM-PPV and Prekote (control) pretreatment coating only on both AA 2024-T3 and AA7075-T6 coupons did not pass 336 hours NSS exposure,***

- ***Results from ASTM B 117 NSS exposure testing verified that BAM-PPV and Prekote (control) pretreatment coating only on both AA6061-T6 and AA5083-H116 passed the 336 hours NSS exposure testing,***

- ***BAM-PPV as pretreatment coating with MIL-PRF-23377C or MIL-PRF-23377N with MIL-PRF-85285 or APC topcoat passed 2000 hours NSS,***

- ***BAM-PPV has adequate adhesion characteristics in all coating systems (pretreatment + primer + topcoat),***

- ***BAM-PPV has an acceptable pencil hardness rating of HB at ambient conditions,***

- ***BAM-PPV has adequate water resistance at ambient temperature,***

- ***BAM-PPV coating systems show acceptable pull-off adhesion results as compared to Prekote coating systems,***

- ***No deficiencies in JP-8 fuel resistance was observed with BAM-PPV coating systems,***

- ***Lubricating fluid resistance results of topcoated BAM-PPV systems showed no change in pencil hardness from room temperature results,***

- ***Xenon-arc testing (500 hours) of BAM-PPV coating systems showed no change in ΔE greater than 1,***

- ***Impact flexibility showed that BAM-PPV coated systems performed as well as Alodine 1200 systems,***

- ***BAM-PPV military coating systems performed as well as the Prekote military system in GM9540P accelerated weathering tests.***

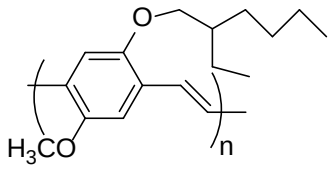
Therefore BAM-PPV has shown acceptable performance profiles in full military coatings to warrant further testing in fielded non-critical military hardware.

6.1.1.2 MEH-PPV Studies

The following sections (6.1.1.2.1-6.1.1.2.1.4) describe MEH-PPV solution processing and application onto various substrates.

6.1.1.2.1 Solubility Studies

MEH-PPV was tested for its solubility in various solvents based on the earlier testing of BAM-PPV. The suggested solvent was xylenes because of the chemical structure of MEH-PPV (Figure 78).



Where n > 1;
MEH-PPV
poly[2,(2,-(2-ethylhexyloxy)-5-meythoxy-*p*-phenylene vinylene]

Figure 78: MEH-PPV Structure

Because the goal was to generate a low-VOC compliant coating, solutions of 1 wt% MEH-PPV in a number of solutions were tested (see Table 55), again both in the heated state and at ambient temperature, followed by generation of a film using a #3

draw-down bar. Like the BAM-PPV, the films were ranked visually for the consistency of the coating produced and the evaporation rates of the solvents, both of which were criteria for the potential for application of a film using HVLP. As anticipated, the MEH-PPV produced a consistent film in either the *p*-xylene or the Oxsol-100 solvent. The MEH-PPV in the Oxsol-100 solvent produced more of a gel-like material in solution, and with the *p*-xylene solvent the material became chunky but remained fluid. Both films applied easily with a draw-down bar and by airbrush. However, the MEH-PPV material took longer to dissolve into either solvent as compared to BAM-PPV, with a total mixing time of nearly 24 hours to generate MEH-PPV solutions ready to spray. However, in comparison with BAM-PPV, the MEH-PPV had a longer "potlife" and did not continue to gel in either the Oxsol-100 or the xylene solvents once mixed, whereas the BAM-PPV demonstrated a limited potlife of less than 6 hours upon mixing.

Table 55: Solubility Data for MEH-PPV

Solvent	CAS Number	Evaporation Rate (compared to butyl acetate)	Mixing Time (hours)	Heated (Yes / No)	Draw-Down Film – Visual Description
p-xylene	106-42-3	0.60	24 (RT) 2 – 4 (heated)	Both	some particles, clean film, better solution when heat is applied
1-chloro-4 (trifluoromethyl)benzene (aka. Oxsol-100)	98-56-6	0.90	24 (RT) 2 – 4 (heated)	Both	gel-like solution, some particles, clean film, better solution when heat is applied
Solvent Blend 50/50 methyl acetate/ Oxsol-100	79-20-9 98-56-6	N/A	24	No	solvent flash-off too fast, does not dissolve
Solvent Blend 70/30 acetone/ Oxsol-100	67-64-1 98-56-6	N/A	24	No	solvent flash-off too fast, does not dissolve
Solvent Blend 30/70 acetone/ Oxsol-100	67-64-1 98-56-6	N/A	24	No	solvent flash-off too fast, does not dissolve

6.1.1.2.1.1 Panel Preparation

Using the BAM-PPV data, it was assumed from the previous studies of BAM-PPV that the MEH-PPV would have limited solubility and that corrosion resistance and adhesion were likely a function of the coating thickness and not of the concentration in solution. MEH-PPV, though available through Sigma-Aldrich, was expensive to purchase, and supply of this material for testing was dependent upon supply received from NAWCWD; therefore, it was decided to do very limited scale testing on coating

thickness (i.e., number of cross-coats) and adhesion but not to specifically test concentrations greater than 1 wt% or to test for accelerated cure options.

6.1.1.2.1.2 Solution Concentration and Coating Thickness Studies

Solutions were prepared at 1 wt% MEH-PPV in either Oxsol-100 or *p*-xylene. Solutions required additional time (more than 4 hours) to dissolve, even at elevated temperature and agitation (100–150 °C, stir plate set to 300 rpm). It is likely that this phenomena, which was seen also with BAM-PPV upon scale-up from 1 g of solvent per batch to 100 g of solvent per batch, was a function of the scale-up itself. Solutions were ready after 6 hours, but the delay required that samples be left overnight, with agitation but no heating. Samples were removed from the stir plate after 24 hours and applied to AA2024-T3 substrates using HVLP. The residual solution from the mixing process was retained for pot-life testing. Between 6 and 8 cross-coats of material were applied. The assumption was made that this would produce a comparable thickness to that generated by the BAM-PPV applications. The coating thickness, however, could not be confirmed through SEM analysis. All reasonable attempts to measure the sample in SEM (using backscatter, reduced power, and reduced spot size) did not result in successful analysis. MEH-PPV deteriorated rapidly and was highly sensitive to the electron beam. Therefore, coating thickness was not measured. Samples were tested for 1 week of exposure to salt spray to determine if the MEH-PPV material had comparable corrosion resistance. MEH-PPV demonstrated improved corrosion resistance and was blistered slightly and discolored after 1 week of exposure to salt spray, for both the Oxsol-100 and the *p*-xylene solutions (Figure 79). Adhesion testing (1-mm grid), however, demonstrated poor characteristics for the MEH-PPV material as compared to the BAM-PPV on AA2024-T3 (Figure 80).

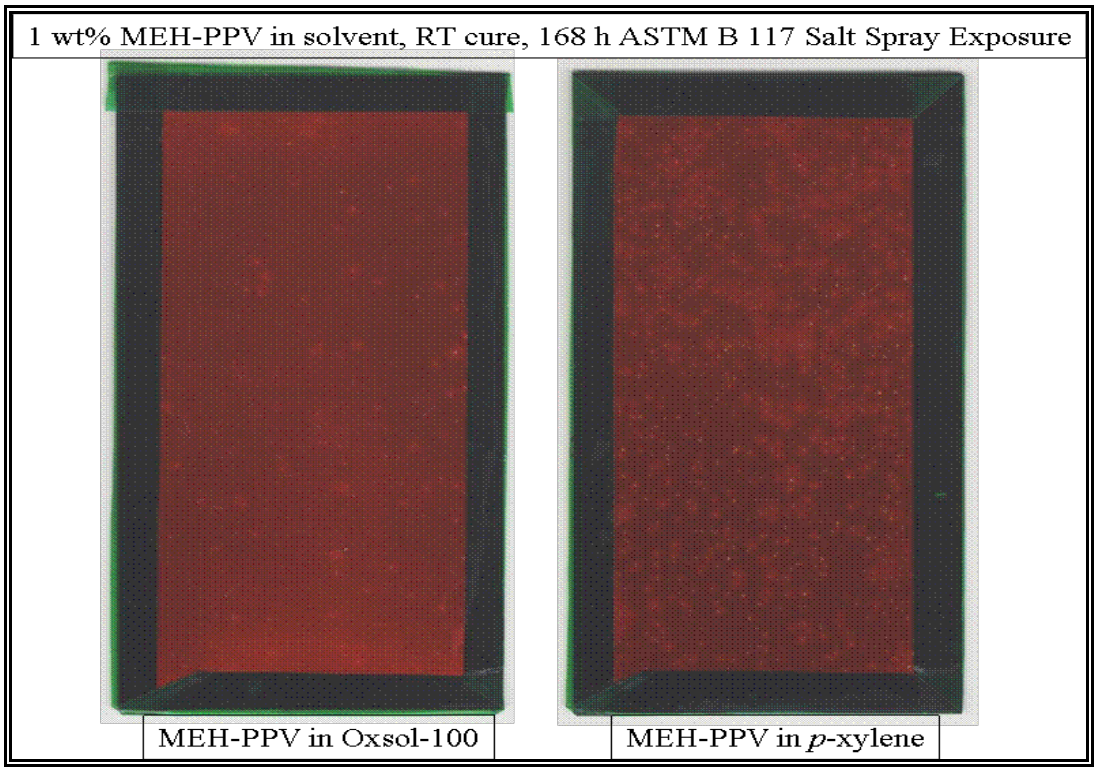


Figure 79: Neutral Salt Spray Samples, 1 wt% MEH-PPV in Oxsol-100 and p-Xylene 4-5 Days-Room Temperature Cure and Ambient Relative Humidity

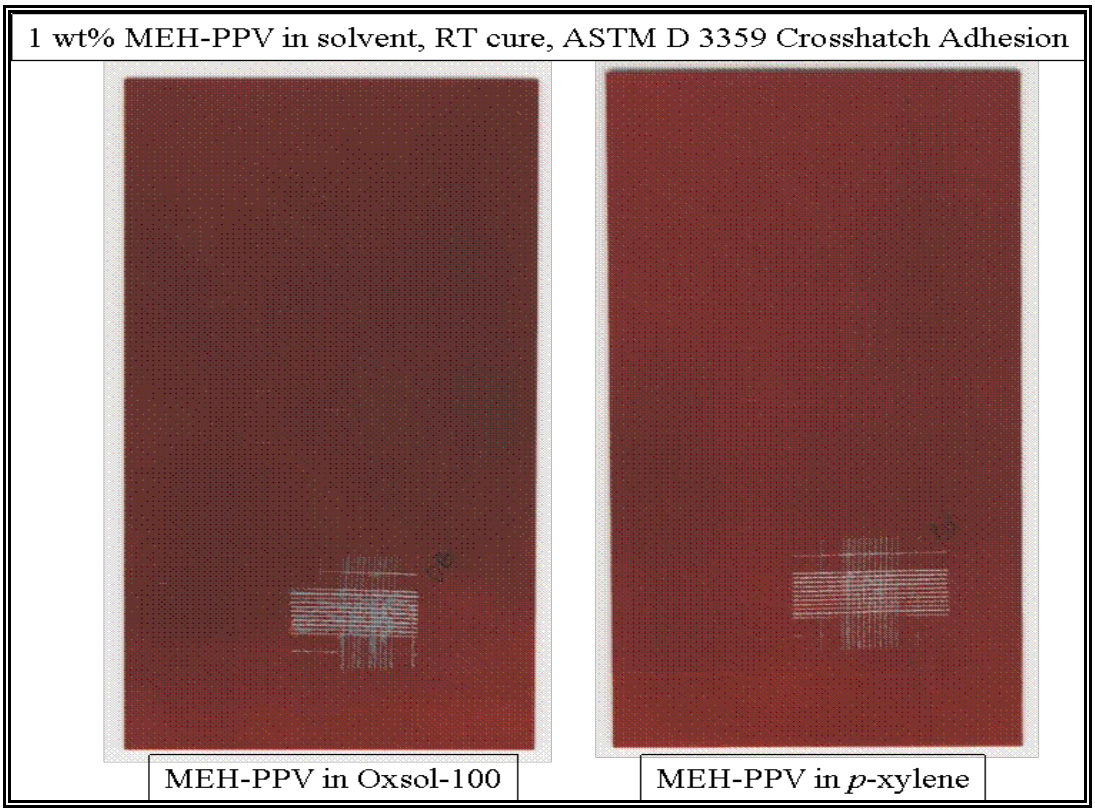


Figure 80: Crosshatch Adhesion Samples, 1 wt% MEH-PPV in Oxsol-100 and p-Xylene; 4-5 Days- Room Temperature Cure and Ambient Relative Humidity

6.1.1.2.1.3 Dwell and Cure Parameters

Because the MEH-PPV material did not demonstrate the same pot-life characteristics as BAM-PPV, there was no dwell-time required upon mixing for MEH-PPV in either Oxsol-100 or *p*-xylene solvents. MEH-PPV tends to take longer to dissolve but does not continue to agglomerate with time once it has reached a gel-like state. This is in contrast to the BAM-PPV material which does agglomerate and, within 8 hours of mixing, becomes impossible to spray-out, even upon reheating or addition of solvent to the solution. MEH-PPV was not tested for cure parameters because of limited material and the limited nature of testing.

6.1.1.2.1.4 General Application Parameters

From the experiments with BAM-PPV, it was determined that the HVLP spray application process "wastes" a minimum of 25 mL of solution with each fill and that approximately 200 mL of solution would cover approximately 36 ft^2 with single passes. It was also determined that the coating could be applied in multiple cross-coats on near-vertical surfaces with 5 minutes flash-off period after every 2–3 cross-coats. General HVLP settings for application of MEH-PPV are given in Table 56. The MEH-PPV material was more consistent in solution than the BAM-PPV material and was easier to

spray. This observation is consistent with the lab observation that MEH-PPV does not change in viscosity or become "chunky" with time, in contrast with BAM-PPV in these solvents. Limited material from NAWCWD did not permit for variation in the applied in the thickness, and the material was applied to 6–8 cross-coats with the assumption that this was comparable to the thickness achieved with BAM-PPV. Set-to-touch time for the MEH-PPV coating (dissolved in Oxsol-100) was also less than 30 minutes, a result which is comparable to that of BAM-PPV.

Table 56: HVLP Spray Application Parameters, MEH-PPV

Line PSI	30
Fluid Setting	1.5
Hose inner diameter	3/8
Hose length	30
HVLP Gun	GTI
Needle Size	413
Cap Size	100
Cap PSI	<10
Application Notes:	Material applied with a 2–3 min flash-off period between each 2–3 cross-coats

6.1.1.2.2 Adhesion Testing

The following sections (6.1.1.2.2-6.1.1.2.2.3) document the results of adhesion testing for MEH-PPV pretreatment coated coupons.

6.1.1.2.2.1 ASTM D 3359 Method B, Crosshatch Adhesion (MIL-PRF-23377)

The data for MEH-PPV coated coupons are given in Tables 57-59. The control system is highlighted in **green**. Coating systems which demonstrated acceptable performance are highlighted in **blue**. Almost all of the tested coated systems demonstrated acceptable adhesion behavior.

Table 57: ASTM D 3359 Crosshatch Adhesion (MEH-PPV Pretreated System) on Various Substrates

AA2024-T3			**AA7075-T6**	
Pretreatment Only	**Rating**		**Pretreatment Only**	**Rating**
MEH-PPV	**pass 5B**		MEH-PPV	**pass 5B**
4130 Normalized, Solvent-Cleaned			**4130 Normalized, 120-Grit-Blasted**	
MEH-PPV	**pass 5B**		MEH-PPV	**pass 5B**

Table 58: ASTM D 3359 Crosshatch Adhesion (MEH-PPV Primed Systems)

Coating System (No Topcoat)	**Rating**	
Pretreatment + Primer	**AA2024-T3 Data**	**AA7075-T6 Data**
Alodine 1200S Deft 02-Y-40A □chromate epoxy	**control 5B**	**control 5B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	**pass 5B**	**pass 5B**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	**pass 5B**	**pass 5B**
MEH-PPV + Deft 02-Y-40A □chromate epoxy	**pass 5B**	**pass 5B**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy	**pass 5B**	**pass 5B**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy	**pass 5B**	**pass 5B**
Pretreatment + Primer	**4130N Solvent-Cleaned Data**	**4130N 120-Grit-Blasted Data**
MEH-PPV + Deft 02-Y-40A □chromate epoxy	**pass 4B**	**pass 4B**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy	**pass 5B**	**pass 5B**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy	**pass 5B**	**pass 5B**

Table 59: ASTM D 3359 Crosshatch Adhesion (MEH-PPV Pretreatment with MIL-PRF-85285 Topcoated Systems)

Coating System	Rating	
Pretreatment + Primer + Topcoat	**AA2024-T3 Data**	**AA7075-T6 Data**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**control 4B**	**control 4B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**pass 4B**	**pass 5B**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**pass 5B**	**pass 5B**
MEH-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**pass 4B**	**pass 4B**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**pass 5B**	**pass 5B**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**pass 5B**	**pass 5B**
Pretreatment + Primer + Topcoat	**4130N Solvent-Cleaned Data**	**4130N 120-Grit-Blasted Data**
MEH-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**pass 4B**	**pass 4B**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**pass 5B**	**pass 5B**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**pass 5B**	**pass 5B**

6.1.1.2.2.2 FTMS 141D Method 6301, Wet-Tape Adhesion (WTA) (MIL-PRF-23377, MIL-PRF-85285)

MEH-PPV coated coupons with primers and topcoats were tested for wet-tape adhesion using the methodology of FTMS 141D, Method 6301. The data are given in Tables 60 and 61. Ratings of 4A or higher are designated considered to "pass" and are highlighted in **blue**. The control system is highlighted in **green**.

Table 60: FTMS 141, ASTM D 3359 Wet Tape Adhesion (Primed Systems)

Coating System (No Topcoat)	Rating
Pretreatment + Primer	**AA2024-T3 Data**
Alodine 1200S Deft 02-Y-40A □chromate epoxy	**control 5A**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	**pass 5A**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	**pass 5A**
MEH-PPV + Deft 02-Y-40A □chromate epoxy	**pass 5A**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy	**pass 5A**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy	**pass 5A**

Table 61: FTMS 141, ASTM D 3359 Wet Tape Adhesion
(MIL-PRF-85285 Topcoated Systems

Coating System	Rating
retreatment + Primer + Topcoat	**AA2024-T3 Data**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**control 5A**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**pass 5A**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**pass 5A**
MEH-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**pass 5A**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**pass 5A**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**pass 5A**

6.1.1.2.2.3 ASTM D 4541 PATTI, Pull-off Adhesion

This test was performed on (*i*) primed and (*ii*) topcoated systems using MEH-PPV as the pretreatment coating. Most of the systems tested demonstrated acceptable adhesion behavior in this test. No system ratings for pass/fail are assigned. The control system is highlighted in **green**. The resultant data sets for each coating system are given in Tables 62 and 63.

Table 62: ASTM D 4541 Pull-Off Adhesion (Primed Systems)

Coating System (No Topcoat)	**Rating**	
Pretreatment + Primer	**Pull-Off Value (psi)**	**Failure Mode**
Alodine 1200S Deft 02-Y-40A □chromate epoxy	**control 1336**	**P / P**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	1670	P / P
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	1571	P / P
MEH-PPV + Deft 02-Y-40A □chromate epoxy	1263	P / Pretreat
MEH-PPV + Deft 02-GN-083 nonchromated epoxy	1757	Pretreat / Pretreat
MEH-PPV + Deft 02-GN-084 nonchromated epoxy	1349	Pretreat / Pretreat

Table 63: ASTM D 4541 Pull-Off Adhesion (MIL-PRF-85285 Topcoated Systems)

Coating System	**Rating**	
Pretreatment + Primer + Topcoat	**Pull-Off Value (psi)**	**Failure Mode**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**control 792**	**P / P**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	1307	P / P
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	1752	P / P
MEH-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	1346	P / Pretreat
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	1878	Pretreat / Pretreat and P / Pretreat
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	1705	Pretreat / Pretreat

6.1.1.2.3 Accelerated Weathering Tests

The following sections (6.1.1.2.3.1-6.1.1.2.3.3) document the accelerated weathering testing of MEH-PPV pretreatment coated coupons without primer and topcoat and in full military coatings (primer + topcoat).

6.1.1.2.3.1 Neutral Salt Spray Testing (ASTM B117)

MEH-PPV as a pretreatment coating was examined using the NSS (ASTM B117) testing method without primer and topcoats and with primers and topcoats. The pretreatment data is given in Table 64 and photographs for those systems are given in Figure 81. The data for primed systems are given in Tables 65 and 66, and photographs for those systems are given in Figures 82-87.

Table 64: Neutral Salt Spray Ratings (336 hours) (MEH-PPV Pretreatment)

Coating System (No Topcoat)	Rating	
Pretreatment + Primer	**AA2024-T3 Data**	**AA7075-T6 Data**
Alodine 1200S	**control minor pitting and staining**	**control no corrosion**
MEH-PPV	minor staining	minor pitting and staining

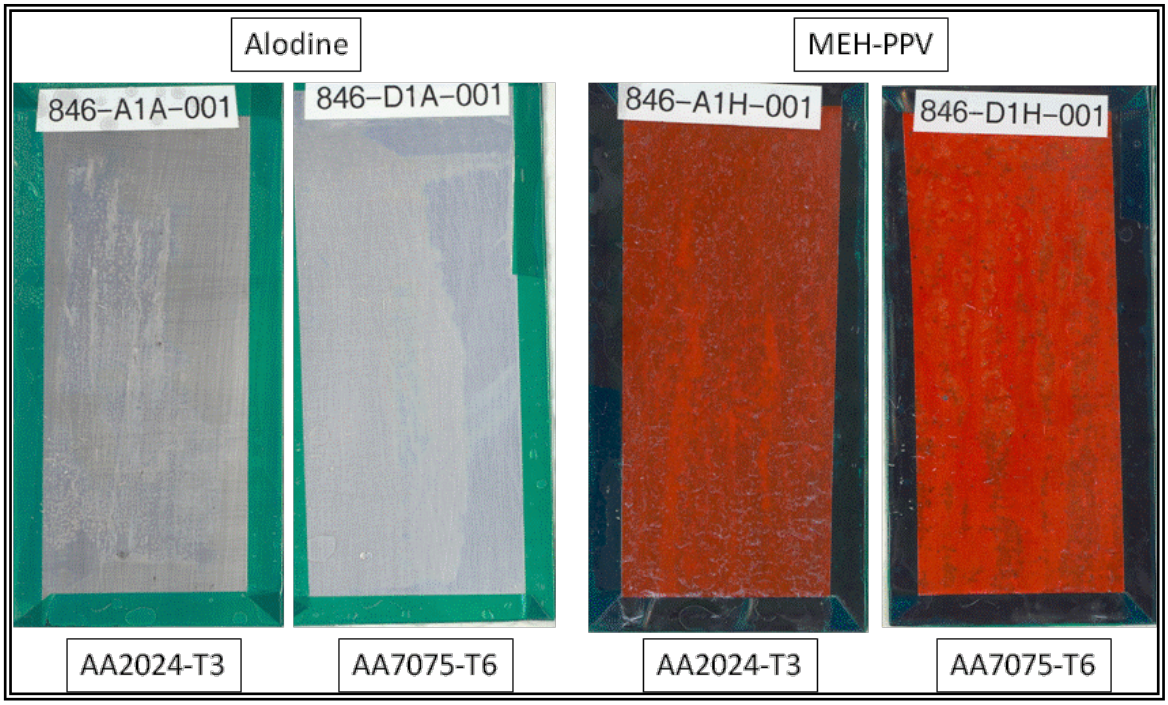


Figure 81: Neutral Salt Spray Results for MEH-PPV Pretreatment after 336 hours of Exposure

Table 65: Neutral Salt Spray Ratings (2000 hours) (MEH-PPV with Primer)

Coating System (No Topcoat)	Rating	
Pretreatment + Primer	**AA2024-T3 Data**	**AA7075-T6 Data**
Alodine 1200S Deft 02-Y-40A □chromate epoxy	**control 1 0 0**	**control 1 0 0**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	**acceptable 1 0 0**	low 2 1 0
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	**acceptable 2 0 0**	**acceptable 1 0 0**
MEH-PPV + Deft 02-Y-40A □chromate epoxy	low 1 0 1	low 2 0 1
MEH-PPV + Deft 02-GN-083 nonchromated epoxy	**poor 2 3 4**	**poor 2 4 0**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy	**poor 2 3 0**	**poor 2 3 0**

Table 66: Neutral Salt Spray Ratings (2000 hours)
(MIL-PRF-85285 Topcoated Systems)

Coating System	Rating	
Pretreatment + Primer + Topcoat	**AA2024-T3 Data**	**AA7075-T6 Data**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**control 2 0 0**	**control 2 0 0**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**acceptable 2 0 0**	**acceptable 2 0 0**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**acceptable 2 0 0**	**acceptable 2 0 0**
MEH-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**acceptable 2 0 0**	**acceptable 2 0 0**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**poor 3 4 0**	**poor 2 5 0**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**poor 3 3 0**	**poor 3 5 0**

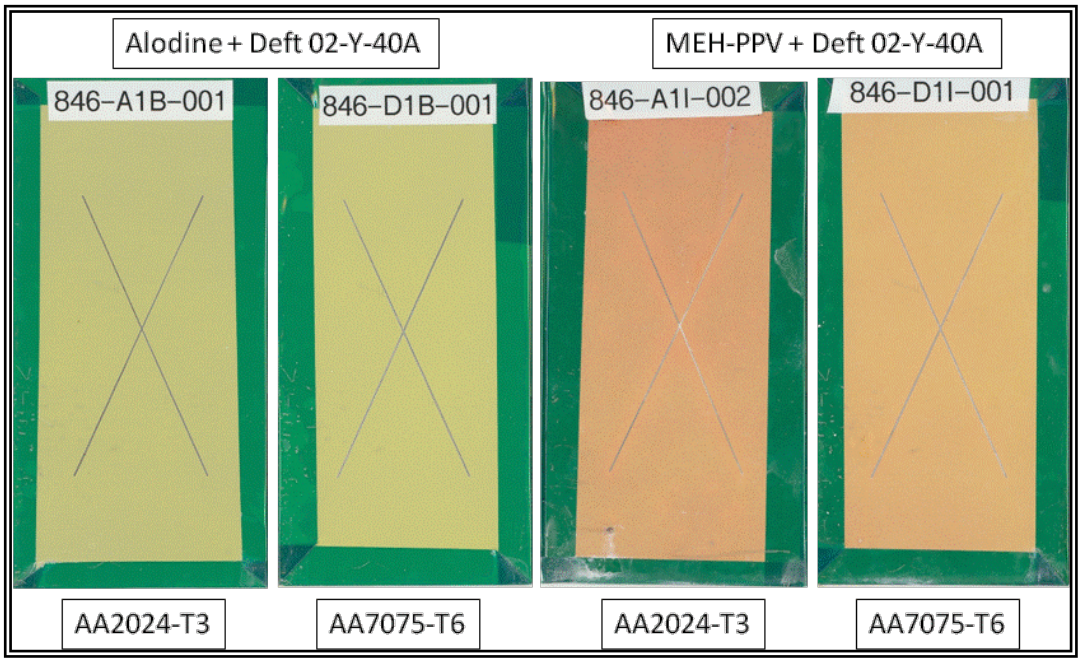


Figure 82: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A after 2000 hours of Exposure

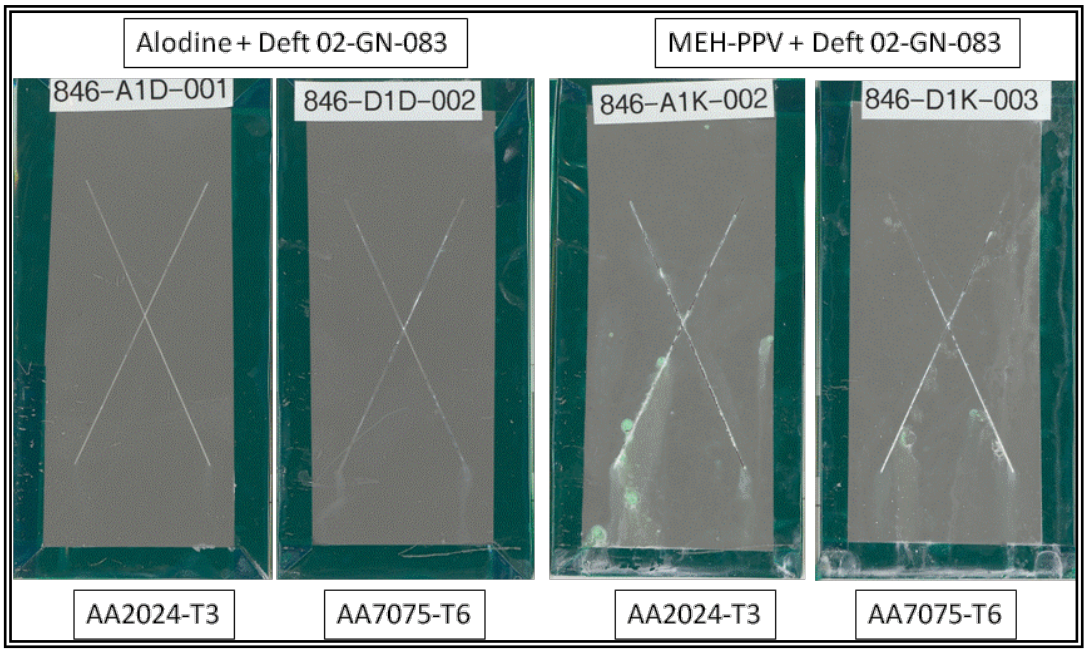


Figure 83: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 after 2000 hours of Exposure

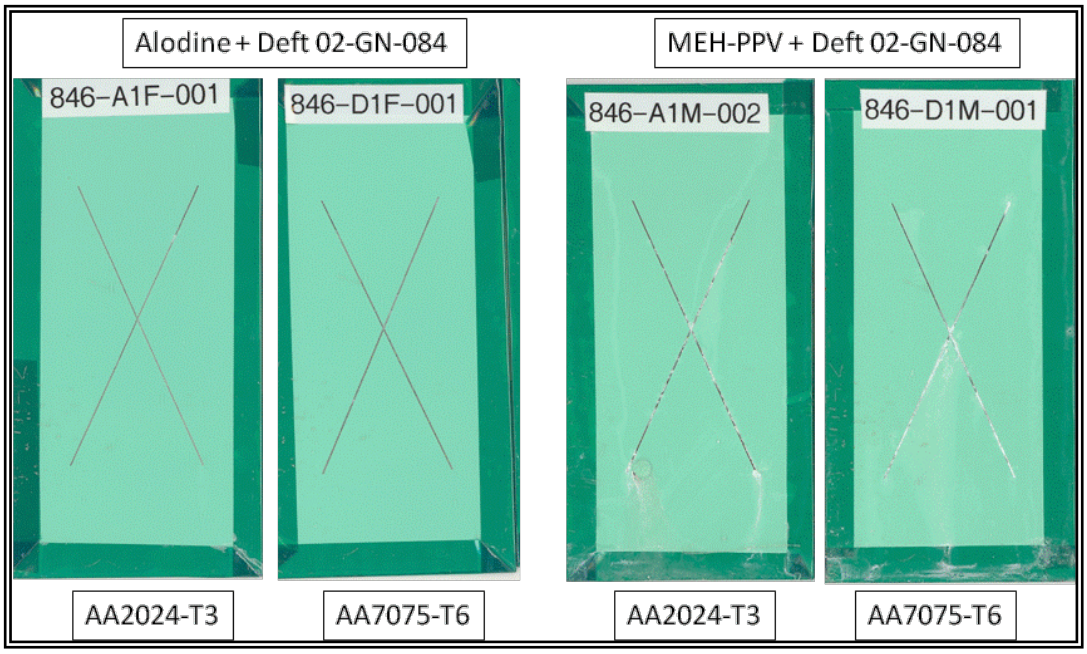


Figure 84: Neutral Salt Spray Results for Pretreatments Nonchromated Epoxy Primer Deft 02-GN-084 after 2000 hours of Exposure

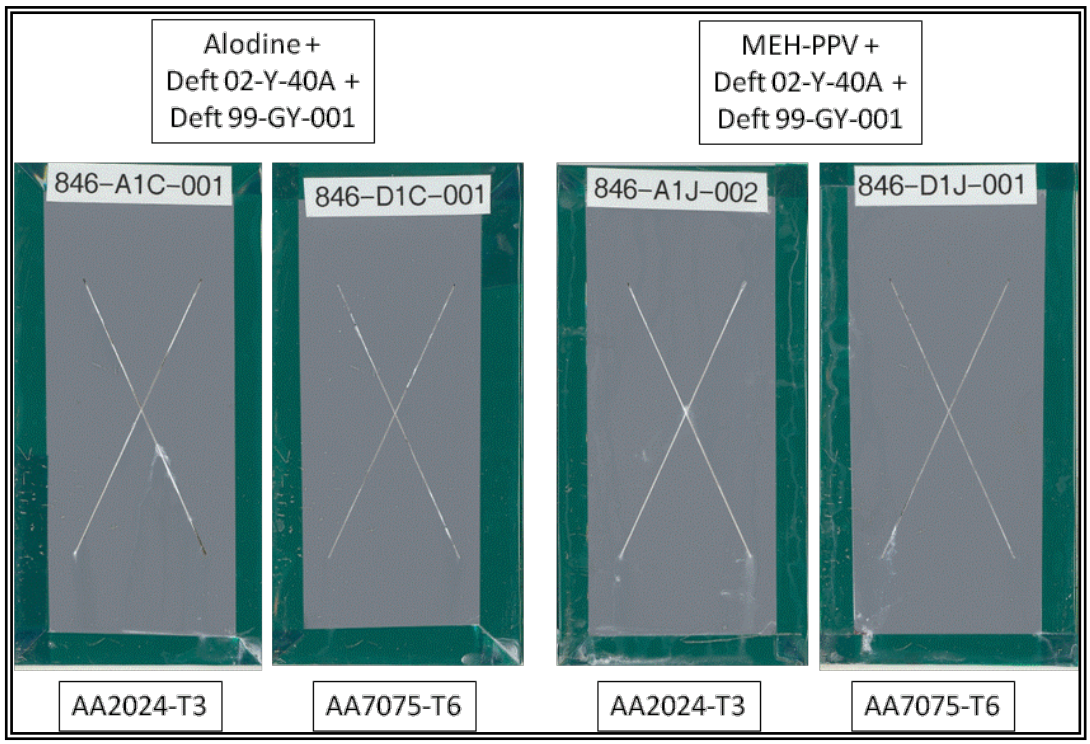


Figure 85: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer Deft 02-Y-40A and MIL-PRF-85285 Topcoat Deft ELT 99-GY-001 after 2000 hours of Exposure

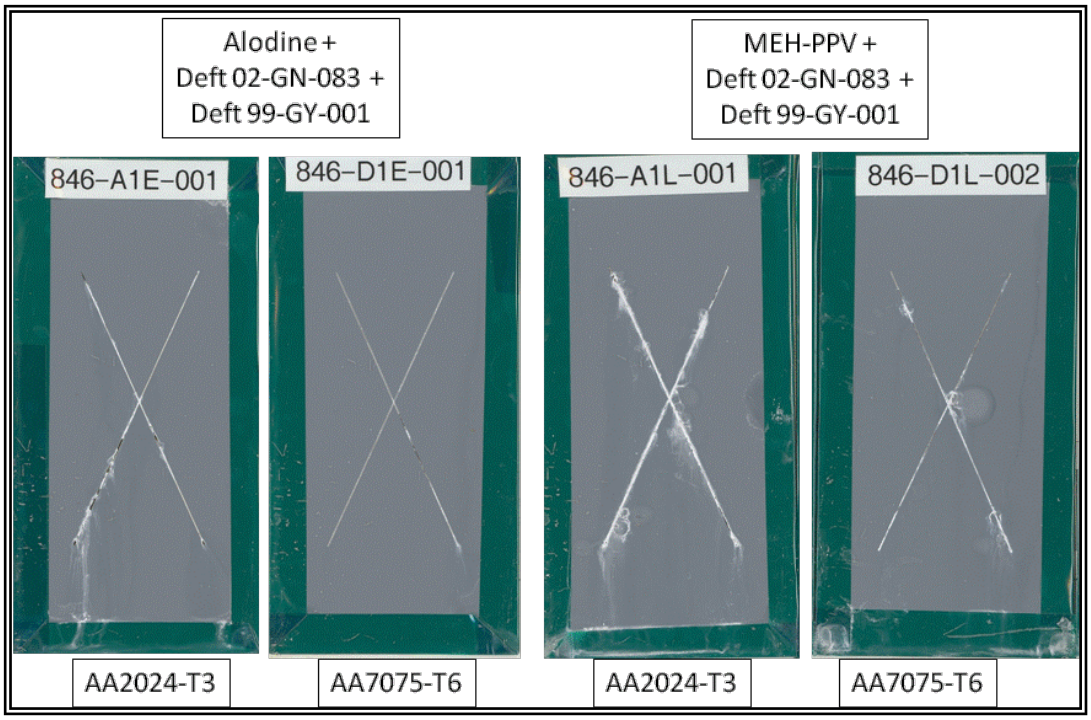


Figure 86: Neutral Salt Spray Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer Deft 02-GN-083 and MIL-PRF-85285 Topcoat Deft ELT 99-GY-001 after 2000 hours of Exposure

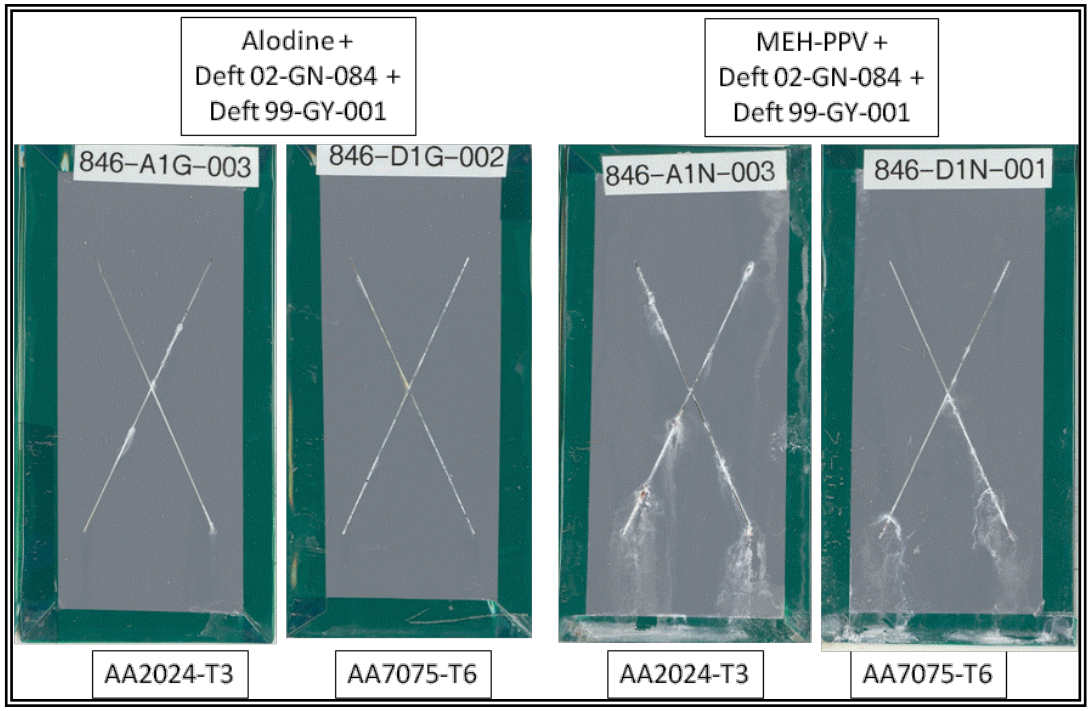


Figure 87: Neutral Salt Spray Results for Pretreatments with Nonchromated Epoxy Primer Deft 02-GN-084 and MIL-PRF-85285 Topcoat Deft ELT 99-GY-001 after 2000 hours of Exposure

6.1.1.2.3.2 GM9540P Accelerated Corrosion Test

GM9540P accelerated weathering procedure was tested on MEH-PPV coated coupons. The data are given in Tables 67 and 68. The control system is highlighted in **green**. Ratings designating acceptable performance are highlighted in **blue**. Ratings that are cause for rejection of the coating system are highlighted in **red**. Photographs of coating system behavior in GM9540P exposure are given in Figures 88-90.

Table 67: GM9540P Accelerated Corrosion on 4130 Normalized Steel, Solvent-Cleaned Samples (MIL-PRF-85285 Topcoated Systems)

Coating System	**Rating**		
Pretreatment + Primer + Topcoat	**ASTM D 1654**	**ASTM D 610**	**ASTM D 714**
Cadmium Plated + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**control 10**	**control 9S**	**control 10**
Cadmium Plated + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**10**	**9S**	**10**
Cadmium Plated + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**10**	**9S**	**10**
MEH-PPV+ Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**1**	**9S**	**10**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**1**	**9S**	**10**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**1**	**9S**	**10**

Table 68: GM9540P Accelerated Corrosion on 4130 Normalized Steel, Grit-Blasted Samples (MIL-PRF-85285 Topcoated Systems)

Coating System	Rating		
Pretreatment + Primer + Topcoat	**ASTM D 1654**	**ASTM D 610**	**ASTM D 714**
Cadmium Plated + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**control 10**	**control 9S**	**control 10**
Cadmium Plated + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**9**	**9S**	8 Few
Cadmium Plated + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**9**	**9S**	8 Few
MEH-PPV+ Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**1**	**9S**	8 Few
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**2**	**9S**	8 Few
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**2**	**9S**	8 Few

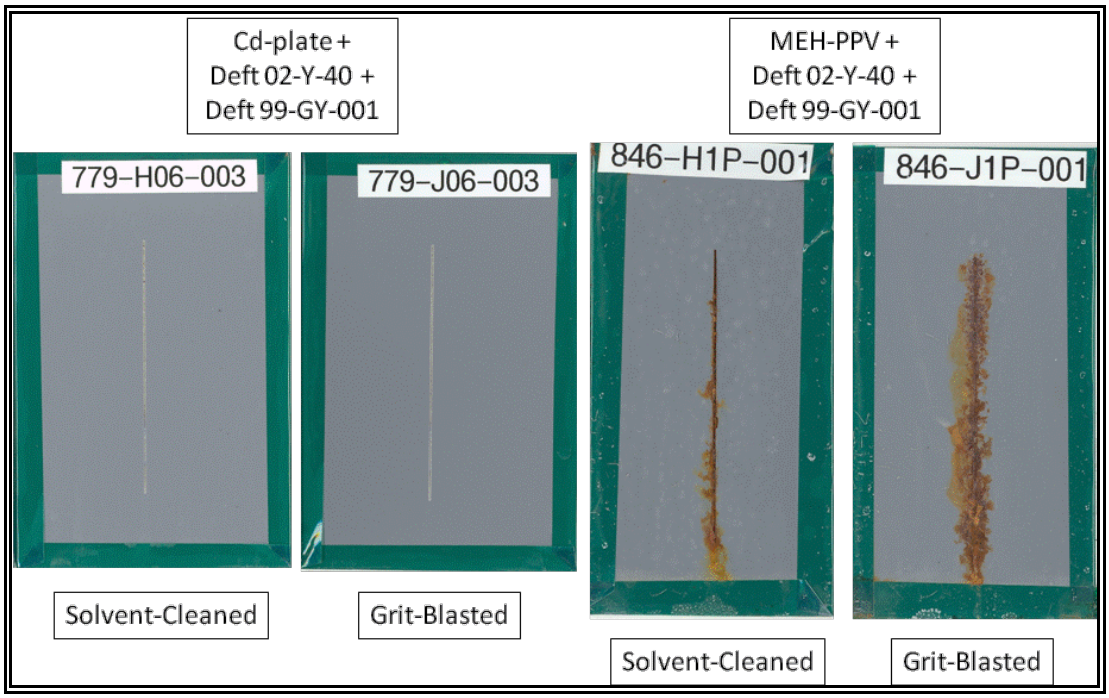


Figure 88: GM9540P Results for Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer and MIL-PRF-85285 Topcoat after 40 Cycles of Exposure

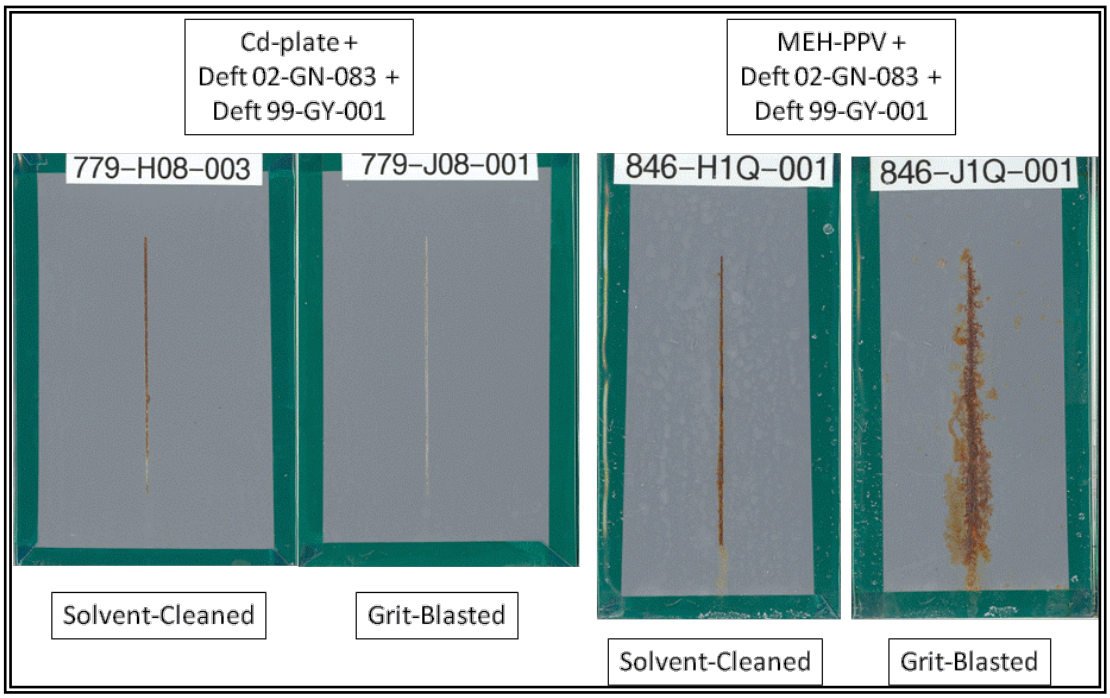


Figure 89: GM9540P Results for Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer and MIL-PRF-85285 Topcoat after 40 Cycles of Exposure

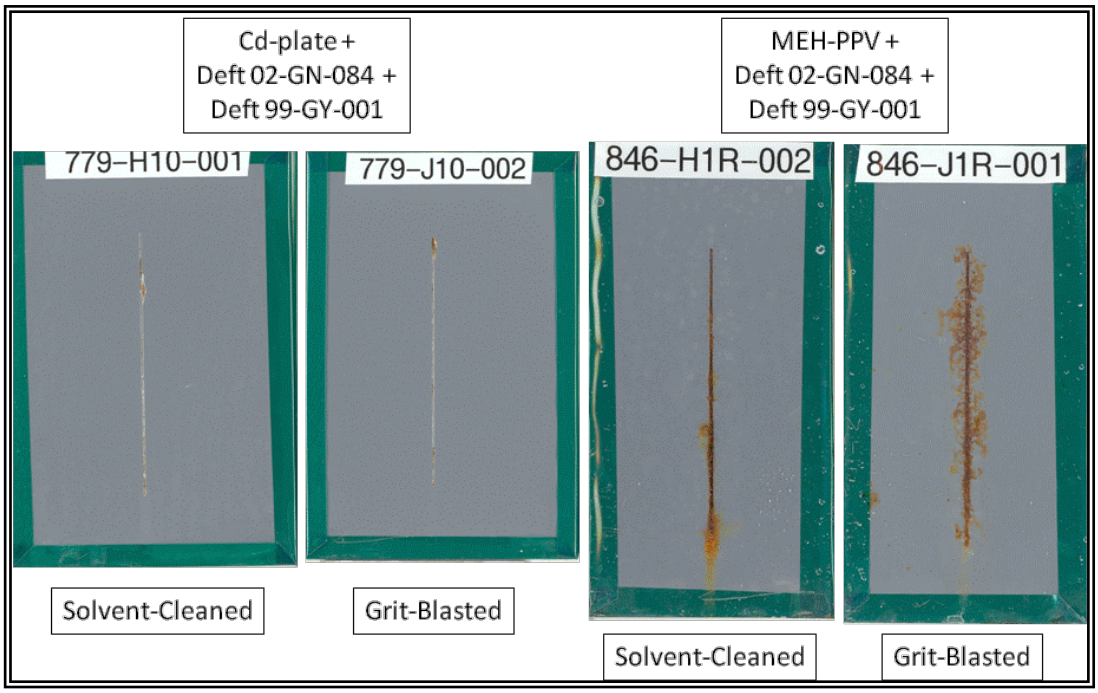


Figure 90: GM9540P Results for Pretreatments with Nonchromated Epoxy Primer and MIL-PRF-85285 Topcoat after 40 Cycles of Exposure.

6.1.1.2.3.3 Xenon Arc Accelerated Weathering Test

MEH-PPV coated coupons with primer and topcoat were tested in accelerated weathering chambers. The data is presented in Tables 69 and 70. The control system is given in **green**. Data that is comparable to the control are given in **blue**. Data that represents considerable lower ratings than the control are given in **red**.

Table 69: Xenon Arc and Salt Spray Exposure (MIL-PRF-85285 Topcoated Systems)

Coating Systems	**Color Data (Δ*E*)**	
Pretreatment + Primer + Topcoat	**Xenon Arc Exposure 500 h**	**Xenon Arc Exposure 2000 h**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**0.6**	**1.2**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**0.6**	**0.9**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**0.6**	**0.7**
MEH-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**0.6**	**0.9**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**0.6**	**0.6**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**0.6**	**0.7**

Table 70: Xenon Arc Exposure (MIL-PRF-85285 Topcoated Systems)

Coating Systems	Corrosion Resistance and Adhesion			
Pretreatment + Primer + Topcoat	**Corrosion Resistance 1000 h**	**Crosshatch Adhesion 1000 h**	**Corrosion Resistance 2000 h**	**Crosshatch Adhesion 2000 h**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**control 2 0 0**	**control 4B**	**control 2 0 0**	**control 4B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**acceptable 2 0 0**	**5B**	**poor 3 0 0**	**5B**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**acceptable 2 0 0**	**5B**	**poor 3 1 0**	**5B**
MEH-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**acceptable 2 0 0**	**4B**	**poor 2 3 0**	**2B**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**poor 2 3 0**	**5B**	**poor 3 3 0**	**3B**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	low 3 3 0	**4B**	**poor 3 3 0**	**2B**

6.1.1.2.4 Additional Tests

As was described in Section 6.1.1.1.4 for BAM-PPV, similar tests were performed on MEH-PPV pretreatment coated coupons. These additional tests were selected by the AF to examine the degree to which MEH-PPV pretreatment coating can perform as well as current AF coatings when exposed to the following tests: pencil hardness, fluid resistance and impact flexibility.

6.1.1.2.4.1 ASTM D 3363 Pencil Hardness (MIL-PRF-23377, MIL-PRF-85285)

MEH-PV coated coupons with primers and topcoats were tested for pencil hardness using the methodology of ASTM D 3363. The ratings that are considered "acceptable" are highlighted in blue. Ratings that are outside of the typical "acceptable" range are highlighted in red. The control system is highlighted in green and tables 71 and 72 summarize the results.

Table 71: ASTM D 3363 Pencil Hardness (Primed Systems)

Coating System (No Topcoat)	AA2024-T3 Data
Pretreatment + Primer	**Rating**
Alodine 1200S Deft 02-Y-40A □chromate epoxy	**control H**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	**4H**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	**H**
MEH-PPV + Deft 02-Y-40A □chromate epoxy	**H**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy	**H**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy	**F**

Table 72: ASTM D 3363 Pencil Hardness (MIL-PRF-85285 Topcoated Systems)

Coating System	Rating
Pretreatment + Primer + Topcoat	**AA2024-T3 Data**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**control HB**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**
MEH-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**HB**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**

6.1.1.2.4.2 Hydraulic, Lubricating Oil, and JP-8 Fluid Resistance (MIL-PRF-83282, MIL-PRF-23699, MIL-DTL-83133, MIL-PRF-23377, MIL-PRF-85285)

MEH-PPV coated coupons with primers and topcoats were tested for exposure to various fluids. Ratings that are cause to reject the performance of the coating system are given in **red**. Ratings depicting overall acceptable behavior are highlighted **blue**. The control system is highlighted in **green** and the data are given in Tables 73-77.

Table 73: MIL-PRF-23699 Lubricating Fluid Exposure (Primed Systems)

Coating System (No Topcoat)	**Ratings**		
	Pencil Hardness Data		**Crosshatch Adhesion**
Pretreatment + Primer	**Room Temperature Rating**	**Post-Exposure Rating**	**Post-Exposure Rating**
Alodine 1200S Deft 02-Y-40A chromate epoxy	**control H**	**control 5H**	**control 5B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	**4H**	**HB**	**5B**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	**H**	**H**	**5B**
MEH-PPV + Deft 02-Y-40A chromate epoxy	**H**	**HB**	**5B**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy	**H**	**4B**	**3B**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy	**F**	**5B**	**3B**

Table 74: MIL-PRF-23699 Lubricating Fluid Exposure
(MIL-PRF-85285 Topcoated Systems)

Coating System (No Topcoat)	**Ratings**		
	Pencil Hardness Data		**Crosshatch Adhesion**
Pretreatment + Primer + Topcoat	**Room Temperature Rating**	**Post-Exposure Rating**	**Post-Exposure Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**control HB**	**control HB**	**control 4B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	2H	**5B**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	**4H**	**5B**
MEH-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	**HB**	**5B**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	**HB**	**3B**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	**HB**	**4B**

Table 75: MIL-PRF-82382 Hydraulic Fluid Exposure (Primed Systems)

Coating System (No Topcoat)	**Ratings**		
	Pencil Hardness Data		**Crosshatch Adhesion**
Pretreatment + Primer	**Room Temperature Rating**	**Post-Exposure Rating**	**Post-Exposure Rating**
Alodine 1200S Deft 02-Y-40A □chromate epoxy	**control H**	**control 2H**	**control 5B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	**4H**	2H	**5B**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	**H**	2H	**5B**
MEH-PPV + Deft 02-Y-40A □chromate epoxy	**H**	**HB**	**5B**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy	**H**	**H**	**5B**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy	**F**	**H**	**5B**

Table 76: MIL-PRF-83282 Hydraulic Fluid Exposure
(MIL-PRF-85285 Topcoated Systems)

Coating System (No Topcoat)	**Ratings**		
	Pencil Hardness Data		**Crosshatch Adhesion**
Pretreatment + Primer + Topcoat	**Room Temperature Rating**	**Post-Exposure Rating**	**Post-Exposure Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft 99-GY-001 polyurethane	**control HB**	**control HB**	**control 5B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	2H	**5B**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	2H	**5B**
MEH-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	**HB**	**4B**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	**H**	**5B**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	**H**	**5B**

Table 77: MIL-DTL-83133 Jet Fuel Exposure (MIL-PRF-85285 Topcoated Systems)

Coating System (No Topcoat)	Ratings		
	Pencil Hardness Data		Crosshatch Adhesion
Pretreatment + Primer + Topcoat	**Room Temperature Rating**	**Post-Exposure Rating**	**Post-Exposure Rating**
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**control HB**	**control HB**	**control 4B**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	**H**	**5B**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	2H	**5B**
MEH-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	**HB**	**4B**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	2H	**5B**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**HB**	2H	**5B**

6.1.1.2.4.3 ASTM D 6905 GE Impact Flexibility Testing (MIL-PRF-23377, MIL-PRF-85285)

MEH-PPV coated coupons were tested for impact flexibility and the data are given in Tables 78 and 79 and ratings that are within the limits set by the control are given in **blue**.

Table 78: ASTM D 6905 GE Impact (Primed Systems)

Coating System (No Topcoat)	Rating
Pretreatment + Primer	
Alodine 1200S Deft 02-Y-40A □chromate epoxy	**control 40**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy	20
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy	5
MEH-PPV + Deft 02-Y-40A □chromate epoxy	10
MEH-PPV + Deft 02-GN-083 nonchromated epoxy	20
MEH-PPV + Deft 02-GN-084 nonchromated epoxy	5

Table 79: ASTM D 6905 GE Impact (MIL-PRF-85285 Topcoated Systems)

Coating System (No Topcoat)	Rating
Pretreatment + Primer	
Alodine 1200S + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**control 10**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	5
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	5
MEH-PPV + Deft 02-Y-40A □chromate epoxy + Deft ELT 99-GY-001 polyurethane	**10**
MEH-PPV + Deft 02-GN-083 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	**10**
MEH-PPV + Deft 02-GN-084 nonchromated epoxy + Deft ELT 99-GY-001 polyurethane	5

6.1.1.2.5 Conclusions for MEH-PPV Laboratory Testing

The results from the extensive laboratory testing performed on MEH-PPV pretreatment coatings by WPAFB researchers are summarized below:

- ***Results from ASTM B 117 neutral salt spray (NSS) exposure testing verified that MEH-PPV and Prekote (control) pretreatment coating only on both AA 2024-T3 and AA7075-T6 coupons <u>did not pass 336 hours</u> NSS exposure,***

- ***MEH-PPV as pretreatment coating with <u>MIL-PRF-23377C only with MIL-PRF-85285 topcoat passed 2000 hours NSS</u>,***

- ***MEH-PPV as pretreatment coating with all nonchromated primers and topcoats <u>failed the 2000 hours</u> NSS exposure testing,***

- ***MEH-PPV has adequate adhesion characteristics in all coating systems (pretreatment + primer + topcoat),***

- ***MEH-PPV has an acceptable pencil hardness rating of HB at ambient conditions,***

- ***MEH-PPV has adequate water resistance at ambient temperature,***

- ***MEH-PPV coating systems show acceptable pull-off adhesion as compared to Prekote coating systems,***

- ***<u>Serious deficiencies</u> in JP-8 fuel resistance was observed with MEH-PPV coating systems,***

- ***Lubricating fluid resistance results of topcoated MEH-PPV systems showed no change in pencil hardness from room temperature results, <u>but crosshatch adhesion results were lower</u>,***

- ***Xenon-arc testing (500 hours) of MEH-PPV coating systems showed no change in ΔE greater than 1,***

- ***Impact flexibility showed that MEH-PPV coated systems performed as well as Alodine 1200 systems,***

- ***MEH-PPV military coating systems performed as well as the Prekote military system in GM 9540P accelerated weathering tests.***

There were serious deficiencies in the MEH-PPV pretreatment coating performance as compared to BAM-PPV pretreatment coating using the same testing methods.

Therefore, BAM-PPV has shown overall acceptable performance profiles in full military coatings to warrant further testing in fielded non-critical military hardware.

6.1.2 Laboratory Testing at ARL

The following sections document the results of laboratory testing for BAM-PPV and MEH-PPV coated coupons at ARL.

6.1.2.1 BAM-PPV and MEH-PPV Laboratory Testing

The results from ARL laboratory testing of BAM-PPV are found in sections 6.1.2.1 – 6.1.2.1.2. **Bulletized summations for ARL laboratory testing are found in**

section 6.1.2.2. Their testing proves that BAM-PPV is a more processable, robust, stable compound than MEH-PPV and was therefore selected for field testing.

6.1.2.1.1 Adhesion studies

When a coating is removed from a substrate using axial tension, there are three modes of failure: adhesive debonding (Figure 91), cohesive debonding (Figure 92), and mixed mode debonding (Figure 93). Failure location within the coating system is also noted. For example, Figure 91 is adhesive failure at the pretreatment/primer interface while Figure 92 is adhesive debonding at the substrate/pretreatment interface. Both were cohesive failure within the primer layer. As can be seen in Figure 94, these results were not consistent even on a single panel. The failure location and mode reported in the following tables were those that predominate for each substrate/pretreatment/primer/topcoat combinations.

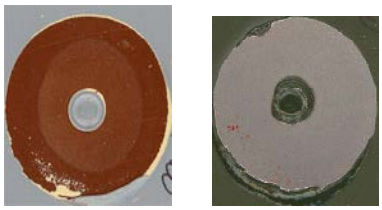


Figure 91: adhesive debonding

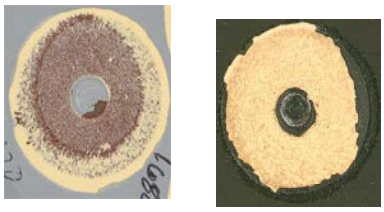


Figure 92: cohesive debonding

Figure 93: mixed mode debonding

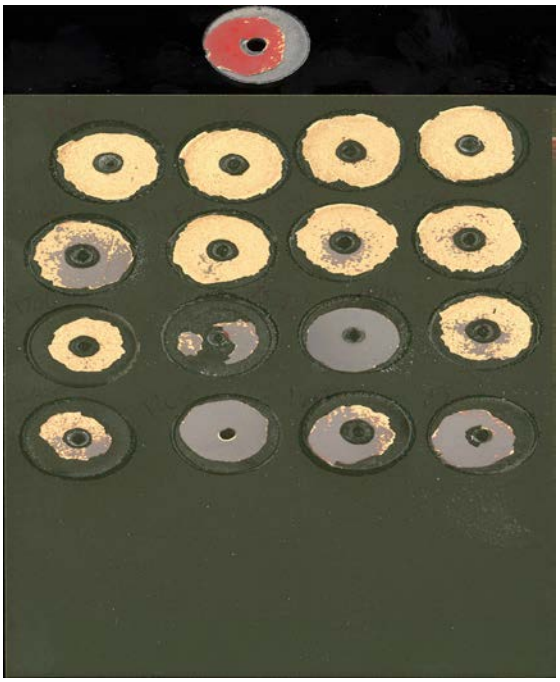


Figure 94: Example of Multiple Failure Modes and Locations

As can be seen in Table 80, when the performance was compared among all of the steel surfaces coated with MEH-PPV, there was only one instance where the clean substrate was outperformed by the grit blasted surface. That was with the Army's water reducible primer, MIL-DTL-53030, and solvent borne topcoat, MIL-DTL-53039. The BAM- PPV also had only one incidence in which the grit blasted surface finish outperformed the clean surface (see Table 81). The Army's water reducible primer, MIL-DTL-53030, with waterborne topcoat, MIL-DTL-64159, was the system in question, although the performance difference between the two is a mere 1 psi.

Table 80: Comparison of steel substrate surface preparation with MEH-PPV pretreatment*

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	MEH-PPV	MIL-P-53022	MIL-C-53039	Substrate-Pretreatment	Adhesive	1848
4130 Clean	MEH-PPV	MIL-PRF-23377N1	MIL-C-53039	Topcoat	Cohesive	1755
4130 Clean	MEH-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Primer	Cohesive	1712
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Primer	Cohesive	1439
4130 Clean	MEH-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	1403
4130 Clean	MEH-PPV	MIL-P-53022	MIL-DTL-64159	Substrate-Pretreatment **	Adhesive	1350
4130 Grit Blast	MEH-PPV	MIL-P-53022	MIL-C-53039	Substrate*-Pretreatment	Adhesive	1346
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N1	MIL-C-53039	Substrate-Pretreatment	Adhesive	1323
4130 Clean	MEH-PPV	MIL-PRF-23377N2	MIL-C-53039	Primer	Cohesive	1246
4130 Grit Blast	MEH-PPV	MIL-P-53022	MIL-DTL-64159	Substrate*-Pretreatment	Adhesive	1221
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N2	MIL-C-53039	Pretreatment-Primer & Primer	Adhesive/Cohesive	1140
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	1113
4130 Clean	MEH-PPV	MIL-P-53030	MIL-C-53039	Substrate-Pretreatment	Adhesive	757
4130 Grit Blast	MEH-PPV	MIL-P-53030	MIL-C-53039	Substrate-Pretreatment	Adhesive	752
4130 Grit Blast	MEH-PPV	MIL-P-53030	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	657
4130 Clean	MEH-PPV	MIL-P-53030	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	567

***Key: MIL-DTL-53039 =MIL-C-53039**

Table 81: Comparison of steel substrate surface preparation with BAM-PPV pretreatment*

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	BAM-PPV	MIL-PRF-23377N1	MIL-DTL-85285	Pretreatment-Substrate*	Adhesive/Cohesive	1853
4130 Clean	BAM-PPV	MIL-PRF-23377N2	MIL-DTL-85285	Primer-Pretreatment	Adhesive/Cohesive	1437
4130 Clean	BAM-PPV	MIL-P-53022	MIL-DTL-85285	Primer-Pretreatment	Adhesive	1206
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	1169
4130 Clean	BAM-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Pretreatment-Substrate*	Adhesive/Cohesive	1063
4130 Clean	BAM-PPV	MIL-PRF-23377N1	MIL-C-53039	Pretreatment-Substrate*	Adhesive	1043
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N1	MIL-C-53039	Pretreatment-Substrate*	Adhesive	991
4130 Clean	BAM-PPV	MIL-P-53030	MIL-C-53039	Pretreatment-Substrate & Primer-Pretreatment	Adhesive	936
4130 Clean	BAM-PPV	MIL-P-53022	MIL-C-53039	Pretreatment-Substrate*	Adhesive	917
4130 Clean	BAM-PPV	MIL-P-53030	MIL-DTL-85285	Primer-Pretreatment & Pretreatment-Substrate	Adhesive	879
4130 Clean	BAM-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Primer-Pretreatment	Cohesive	876
4130 Grit Blast	BAM-PPV	MIL-P-53022	MIL-C-53039	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	866
4130 Grit Blast	BAM-PPV	MIL-P-53022	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	848
4130 Clean	BAM-PPV	MIL-PRF-23377N2	MIL-C-53039	Pretreatment-Substrate	Cohesive/Adhesive	826
4130 Clean	BAM-PPV	MIL-P-53022	MIL-DTL-64159	Primer-Pretreatment	Adhesive	801
4130 Grit Blast	BAM-PPV	MIL-P-53030	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	763
4130 Clean	BAM-PPV	MIL-P-53030	MIL-DTL-64159	Pretreatment-Substrate & Primer-Pretreatment	Adhesive	762
4130 Grit Blast	BAM-PPV	MIL-P-53030	MIL-C-53039	Substrate-Pretreatment & Pretreatment	Adhesive/Cohesive	758
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	754
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N2	MIL-C-53039	Substrate*-Pretreatment & Pretreatment	Adhesive/Cohesive	744

***Key: MIL-P-53030 = MIL-DTL-53030; MIL-P-53022 = MIL-DTL-53022**

When the performance of the Army primers is compared, the adhesion of MIL-DTL-53022 is superior to MIL-DTL-53030 for both BAM-PPV and MEH-PPV pretreatments, with one exception. As can be seen in Table 82, the water reducible primer on a clean surface with MEH-PPV and MIL-DTL-53039 topcoat slightly outperforms a similarly prepared surface with the solvent borne primer. The results for MEH-PPV are provided in Table 83. When BAM-PPV is the pretreatment, the MIL-PRF-23377N1 consistently had better adhesion than the MIL-PRF-23377N2 regardless of the substrate or topcoat (Table 84). Table 85 shows that there was no consistent performance advantage when using the MEH-PPV pretreatment with the Navy primers. The performance disparity was significant ranging from 200 to 500 psi.

Table 82: Performance of BAM-PPV with Army primers

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	BAM-PPV	MIL-P-53022	MIL-DTL-85285	Primer-Pretreatment	Adhesive	1206
4130 Clean	BAM-PPV	MIL-P-53030	MIL-C-53039	Pretreatment-Substrate & Primer-Pretreatment	Adhesive	936
4130 Clean	BAM-PPV	MIL-P-53022	MIL-C-53039	Pretreatment-Substrate*	Adhesive	917
4130 Clean	BAM-PPV	MIL-P-53030	MIL-DTL-85285	Primer-Pretreatment & Pretreatment-Substrate	Adhesive	879
4130 Grit Blast	BAM-PPV	MIL-P-53022	MIL-C-53039	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	866
4130 Grit Blast	BAM-PPV	MIL-P-53022	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	848
4130 Clean	BAM-PPV	MIL-P-53022	MIL-DTL-64159	Primer-Pretreatment	Adhesive	801
4130 Grit Blast	BAM-PPV	MIL-P-53030	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	763
4130 Clean	BAM-PPV	MIL-P-53030	MIL-DTL-64159	Pretreatment-Substrate & Primer-Pretreatment	Adhesive	762
4130 Grit Blast	BAM-PPV	MIL-P-53030	MIL-C-53039	Substrate-Pretreatment & Pretreatment	Adhesive/Cohesive	758

Table 83: Performance of MEH-PPV with Army primers

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	MEH-PPV	MIL-P-53022	MIL-C-53039	Substrate-Pretreatment	Adhesive	1848
4130 Clean	MEH-PPV	MIL-P-53022	MIL-DTL-64159	Substrate-Pretreatment **	Adhesive	1350
4130 Grit Blast	MEH-PPV	MIL-P-53022	MIL-C-53039	Substrate*-Pretreatment	Adhesive	1346
4130 Grit Blast	MEH-PPV	MIL-P-53022	MIL-DTL-64159	Substrate*-Pretreatment	Adhesive	1221
4130 Clean	MEH-PPV	MIL-P-53030	MIL-C-53039	Substrate-Pretreatment	Adhesive	757
4130 Grit Blast	MEH-PPV	MIL-P-53030	MIL-C-53039	Substrate-Pretreatment	Adhesive	752
4130 Grit Blast	MEH-PPV	MIL-P-53030	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	657
4130 Clean	MEH-PPV	MIL-P-53030	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	567

Table 84: Performance of BAM-PPV with Navy primers

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	BAM-PPV	MIL-PRF-23377N1	MIL-DTL-85285	Pretreatment-Substrate*	Adhesive/Cohesive	1853
4130 Clean	BAM-PPV	MIL-PRF-23377N2	MIL-DTL-85285	Primer-Pretreatment	Adhesive/Cohesive	1437
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	1169
4130 Clean	BAM-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Pretreatment-Substrate*	Adhesive/Cohesive	1063
4130 Clean	BAM-PPV	MIL-PRF-23377N1	MIL-C-53039	Pretreatment-Substrate*	Adhesive	1043
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N1	MIL-C-53039	Pretreatment-Substrate*	Adhesive	991
4130 Clean	BAM-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Primer-Pretreatment	Cohesive	876
4130 Clean	BAM-PPV	MIL-PRF-23377N2	MIL-C-53039	Pretreatment-Substrate	Cohesive/Adhesive	826
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	754
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N2	MIL-C-53039	Substrate*-Pretreatment & Pretreatment	Adhesive/Cohesive	744

Table 85: Performance of MEH-PPV with Navy primers

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	MEH-PPV	MIL-PRF-23377N1	MIL-C-53039	Topcoat	Cohesive	1755
4130 Clean	MEH-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Primer	Cohesive	1712
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Primer	Cohesive	1439
4130 Clean	MEH-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	1403
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N1	MIL-C-53039	Substrate-Pretreatment	Adhesive	1323
4130 Clean	MEH-PPV	MIL-PRF-23377N2	MIL-C-53039	Primer	Cohesive	1246
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N2	MIL-C-53039	Pretreatment-Primer & Primer	Adhesive/Cohesive	1140
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	1113

Tables 86-89 compare the performance of the topcoats while holding the primer constant over BAM-PPV. The data in table 86 shows that the solvent borne topcoat does adhere better to the solvent borne primer than to the waterborne topcoat. Table 87 shows that the choice of Army topcoat provides no consistent performance edge. The data presented in Tables 88 and 89 shows that MIL-DTL-64159, water reducible topcoat, outperformed MIL-DTL-53039 over the Navy primers. The Navy topcoat, MIL-PRF-85285 has higher pull-off adhesion values than either Army topcoat.

Table 86: Topcoat adhesion performance of BAM-PPV and MIL-DTL-53022

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	BAM-PPV	MIL-P-53022	MIL-DTL-85285	Primer-Pretreatment	Adhesive	1206
4130 Clean	BAM-PPV	MIL-P-53022	MIL-C-53039	Pretreatment-Substrate*	Adhesive	917
4130 Grit Blast	BAM-PPV	MIL-P-53022	MIL-C-53039	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	866
4130 Grit Blast	BAM-PPV	MIL-P-53022	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	848
4130 Clean	BAM-PPV	MIL-P-53022	MIL-DTL-64159	Primer-Pretreatment	Adhesive	801

Table 87: Topcoat adhesion performance of BAM-PPV over MIL-DTL-53030

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	BAM-PPV	MIL-P-53030	MIL-C-53039	Pretreatment-Substrate & Primer-Pretreatment	Adhesive	936
4130 Clean	BAM-PPV	MIL-P-53030	MIL-DTL-85285	Primer-Pretreatment & Pretreatment-Substrate	Adhesive	879
4130 Grit Blast	BAM-PPV	MIL-P-53030	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	763
4130 Clean	BAM-PPV	MIL-P-53030	MIL-DTL-64159	Pretreatment-Substrate & Primer-Pretreatment	Adhesive	762
4130 Grit Blast	BAM-PPV	MIL-P-53030	MIL-C-53039	Substrate-Pretreatment & Pretreatment	Adhesive/Cohesive	758

Table 88: Topcoat adhesion performance of BAM-PPV over MIL-PRF-23377N1

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	BAM-PPV	MIL-PRF-23377N1	MIL-DTL-85285	Pretreatment-Substrate*	Adhesive/Cohesive	1853
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	1169
4130 Clean	BAM-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Pretreatment-Substrate*	Adhesive/Cohesive	1063
4130 Clean	BAM-PPV	MIL-PRF-23377N1	MIL-C-53039	Pretreatment-Substrate*	Adhesive	1043
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N1	MIL-C-53039	Pretreatment-Substrate*	Adhesive	991

Table 89: Topcoat adhesion performance of BAM-PPV over MIL-PRF-23377N2

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	BAM-PPV	MIL-PRF-23377N2	MIL-DTL-85285	Primer-Pretreatment	Adhesive/Cohesive	1437
4130 Clean	BAM-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Primer-Pretreatment	Cohesive	876
4130 Clean	BAM-PPV	MIL-PRF-23377N2	MIL-C-53039	Pretreatment-Substrate	Cohesive/Adhesive	826
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	754
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N2	MIL-C-53039	Substrate*-Pretreatment & Pretreatment	Adhesive/Cohesive	744

Table's 90-93 show topcoat performance when MEH-PPV is the pretreatment. The solvent borne MIL-DTL-53039 outperforms the waterborne MIL-DTL-64159 over both Army primers and over MIL-PRF-23377N1 (Tables 90-92). The waterborne topcoat performed better when in a system with MIL-RPF-23377N2 and MEH-PPV (Table 93).

Table 90: Topcoat adhesion performance of MEH-PPV over MIL-DTL-53022

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	MEH-PPV	MIL-P-53022	MIL-C-53039	Substrate-Pretreatment	Adhesive	1848
4130 Clean	MEH-PPV	MIL-P-53022	MIL-DTL-64159	Substrate-Pretreatment **	Adhesive	1350
4130 Grit Blast	MEH-PPV	MIL-P-53022	MIL-C-53039	Substrate*-Pretreatment	Adhesive	1346
4130 Grit Blast	MEH-PPV	MIL-P-53022	MIL-DTL-64159	Substrate*-Pretreatment	Adhesive	1221

Table 91: Topcoat adhesion performance of MEH-PPV over MIL-DTL-53030

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	MEH-PPV	MIL-P-53030	MIL-C-53039	Substrate-Pretreatment	Adhesive	757
4130 Grit Blast	MEH-PPV	MIL-P-53030	MIL-C-53039	Substrate-Pretreatment	Adhesive	752
4130 Grit Blast	MEH-PPV	MIL-P-53030	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	657
4130 Clean	MEH-PPV	MIL-P-53030	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	567

Table 92: Topcoat adhesion performance of MEH-PPV over MIL-PRF-23377N1

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	MEH-PPV	MIL-PRF-23377N1	MIL-C-53039	Topcoat	Cohesive	1755
4130 Clean	MEH-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	1403
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N1	MIL-C-53039	Substrate-Pretreatment	Adhesive	1323
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	1113

Table 93: Topcoat adhesion performance of MEH-PPV over MIL-PRF-23377N2

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	MEH-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Primer	Cohesive	1712
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Primer	Cohesive	1439
4130 Clean	MEH-PPV	MIL-PRF-23377N2	MIL-C-53039	Primer	Cohesive	1246
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N2	MIL-C-53039	Pretreatment-Primer & Primer	Adhesive/Cohesive	1140

When the Army primer coated BAM-PPV is compared to the MEH-PPV, there were unusual results. As seen in Table 94, when the solvent borne primer MIL-DTL-53022 was used, the MEH-PPV outperformed the BAM-PPV in every case. When the waterborne MIL-DTL-53030 was used, the BAM-PPV outperformed the MEH-PPV pretreatment in very case. When using Navy primers, the MEH-PPV outperformed the

BAM-PPV in every case (Table 95). A system that included the Navy topcoat MIL-PRF-85285 was not tested over the MEH-PPV.

Table 94: Pretreatment performance using Army Primers

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	MEH-PPV	MIL-P-53022	MIL-C-53039	Substrate-Pretreatment	Adhesive	1848
4130 Clean	MEH-PPV	MIL-P-53022	MIL-DTL-64159	Substrate-Pretreatment **	Adhesive	1350
4130 Grit Blast	MEH-PPV	MIL-P-53022	MIL-C-53039	Substrate*-Pretreatment	Adhesive	1346
4130 Grit Blast	MEH-PPV	MIL-P-53022	MIL-DTL-64159	Substrate*-Pretreatment	Adhesive	1221
4130 Clean	BAM-PPV	MIL-P-53022	MIL-DTL-85285	Primer-Pretreatment	Adhesive	1206
4130 Clean	BAM-PPV	MIL-P-53030	MIL-C-53039	Pretreatment-Substrate & Primer-Pretreatment	Adhesive	936
4130 Clean	BAM-PPV	MIL-P-53022	MIL-C-53039	Pretreatment-Substrate*	Adhesive	917
4130 Clean	BAM-PPV	MIL-P-53030	MIL-DTL-85285	Primer-Pretreatment & Pretreatment-Substrate	Adhesive	879
4130 Grit Blast	BAM-PPV	MIL-P-53022	MIL-C-53039	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	866
4130 Grit Blast	BAM-PPV	MIL-P-53022	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	848
4130 Clean	BAM-PPV	MIL-P-53022	MIL-DTL-64159	Primer-Pretreatment	Adhesive	801
4130 Grit Blast	BAM-PPV	MIL-P-53030	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	763
4130 Clean	BAM-PPV	MIL-P-53030	MIL-DTL-64159	Pretreatment-Substrate & Primer-Pretreatment	Adhesive	762
4130 Grit Blast	BAM-PPV	MIL-P-53030	MIL-C-53039	Substrate-Pretreatment & Pretreatment	Adhesive/Cohesive	758
4130 Clean	MEH-PPV	MIL-P-53030	MIL-C-53039	Substrate-Pretreatment	Adhesive	757
4130 Grit Blast	MEH-PPV	MIL-P-53030	MIL-C-53039	Substrate-Pretreatment	Adhesive	752
4130 Grit Blast	MEH-PPV	MIL-P-53030	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	657
4130 Clean	MEH-PPV	MIL-P-53030	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	567

Table 95: Pretreatment performance using Navy primers

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
4130 Clean	BAM-PPV	MIL-PRF-23377N1	MIL-DTL-85285	Pretreatment-Substrate*	Adhesive/Cohesive	1853
4130 Clean	MEH-PPV	MIL-PRF-23377N1	MIL-C-53039	Topcoat	Cohesive	1755
4130 Clean	MEH-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Primer	Cohesive	1712
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Primer	Cohesive	1439
4130 Clean	BAM-PPV	MIL-PRF-23377N2	MIL-DTL-85285	Primer-Pretreatment	Adhesive/Cohesive	1437
4130 Clean	MEH-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	1403
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N1	MIL-C-53039	Substrate-Pretreatment	Adhesive	1323
4130 Clean	MEH-PPV	MIL-PRF-23377N2	MIL-C-53039	Primer	Cohesive	1246
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	1169
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N2	MIL-C-53039	Pretreatment-Primer & Primer	Adhesive/Cohesive	1140
4130 Grit Blast	MEH-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	1113
4130 Clean	BAM-PPV	MIL-PRF-23377N1	MIL-DTL-64159	Pretreatment-Substrate*	Adhesive/Cohesive	1063
4130 Clean	BAM-PPV	MIL-PRF-23377N1	MIL-C-53039	Pretreatment-Substrate*	Adhesive	1043
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N1	MIL-C-53039	Pretreatment-Substrate*	Adhesive	991
4130 Clean	BAM-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Primer-Pretreatment	Cohesive	876
4130 Clean	BAM-PPV	MIL-PRF-23377N2	MIL-C-53039	Pretreatment-Substrate	Cohesive/Adhesive	826
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N2	MIL-DTL-64159	Substrate*-Pretreatment & Pretreatment-Primer	Adhesive	754
4130 Grit Blast	BAM-PPV	MIL-PRF-23377N2	MIL-C-53039	Substrate*-Pretreatment & Pretreatment	Adhesive/Cohesive	744

As can be seen in Table 96, the MEH-PPV outperformed the BAM-PPV when used as a pretreatment over aluminum 6061 regardless of the primer used. It also outperformed the hexavalent chromium containing MIL-C-5541 when primed with MIL-DTL-53022. There was a solvent odor immediately following the tests involving one of the electroactive polymers and the MIL-DTL-53022 indicating that there was some solvent entrapment during the coating process. When aluminum 5083 was used, the adhesion values for the MEH-PPV were significantly lower than for aluminum 6061 (see Table 97). The MIL-DTL-53022 continued to have better adhesion than the waterborne MIL-DTL-53030. BAM-PPV was not tested on this substrate.

Table 96: Pretreatment Performance on Aluminum 6061

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
6061	MEH-PPV	MIL-P-53022	MIL-DTL-85285	Substrate-Pretreatment & Topcoat	Adhesive/Cohesive	3408
6061	MIL-C-5541	MIL-P-53022	MIL-DTL-85285	Pretreatment-Primer	Adhesive	3282
6061	MIL-C-5541	MIL-P-53030	MIL-DTL-85285	Topcoat & Pretreatment-Primer	Cohesive/Adhesive	2882
6061	BAM-PPV	MIL-P-53022	MIL-DTL-85285	Substrate-Pretreatment	Adhesive	1185
6061	MEH-PPV	MIL-P-53030	MIL-DTL-85285	Substrate-Pretreatment*	Adhesive	1038
6061	BAM-PPV	MIL-P-53030	MIL-DTL-85285	Pretreatment-Primer & Substrate-Pretreatment	Adhesive	824

Table 97: Pretreatment Performance on Aluminum 5083

Substrate	Pretreatment	Primer	Topcoat	Failure Location	Mode	Average
5083	MEH-PPV	MIL-P-53022	MIL-C-53039	Topcoat & Substrate-Pretreatment	Cohesive/Adhesive	1026
5083	MEH-PPV	MIL-P-53022	MIL-DTL-64159	Substrate-Pretreatment	Adhesive	827
5083	MEH-PPV	MIL-P-53030	MIL-C-53039	Substrate-Pretreatment	Adhesive	317

6.1.2.1.2 Neutral Salt Spray Testing (ASTM B117) for BAM-PPV Pretreatment

Grit blasted and as-received steel, AA6061 and AA5083 were coated with BAM-PPV. Similarly prepared steel panels were pretreated with DoD-P-15328 for use as controls and a smaller set was pretreated with Metalast's TCP HF. Aluminum 6061 and 5083 were taken in the as-received condition, cleaned, and pretreated with BAM-PPV or Metalast TCP HF. Panels were primed with MIL-DTL-53022, MIL-DTL-53030, MIL-PRF-23377N1, MIL-PRF-23377N2, or MIL-PRF-23377C and topcoated with MIL-DTL-53039, MIL-DTL-64159 or MIL-PRF-85285 (for steel only). Coated and cured panels were exposed to 5% NaCl fog at 100% relative humidity as described in ASTM B 117 for up to 432 hours for steel and up to 2016 hours for aluminum. Panels were rated using ASTM D 1654 method A for scribed regions and method B for unscribed regions. Final images were taken upon completion of exposure. The steel panels that were coated with BAM-PPV in the as-received condition and were subsequently topcoated with MIL-DTL-64159 all failed due to coating delamination before the first rating was performed at 124 hours regardless of the primer used. Similarly prepared panels that were topcoated with MIL-DTL-53039 survived to 124 hours but were terminated because there was severe blistering of the scribed panels in the set. Steel with BAM-PPV pretreatment, MIL-DTL-53022 primer and MIL-DTL-53039 was the only as-received panel set that was exposed for 504 hours. The failure criterion was an ASTM D 1654 rating of 3 or less. Six out of eight steel panel sets whose surfaces were grit blasted prior to BAM-PPV application completed the full exposure to 432 hours. As can be seen in Tables 98 and 99, the MIL-DTL-53030 failed at 124 hours under either topcoat. It should be noted that the first three panels in each set were scribed and the remaining two were not. Also, where there are two numbers in a rating for the scribed panel, the first number indicates the ASTM D 1654 rating of the scribe and the second is the rating for those areas away from the scribe. This is usually indicative of blistering not in the immediate vicinity of the scribe. Figure 95 is fairly typical of the appearance of the remaining panels after 432 hours of ASTM B 117 exposure.

The following description provides the key to understanding the following tables regarding performance criteria in neutral salt fog chamber. BIF stands for blisters in field

and means that there was creep from scribe and blistering of the unscribed regions. The colors are a visual clue to performance and are as follows:

Green is ASTM D 1654 rating of 8-10
Yellow is ASTM D 1654 rating of 6-7
Orange is ASTM D 1654 rating of 4-5
Red is ASTM D 1654 rating of 1-3
Black is ASTM D 1654 rating of 0

A striped pattern is indicative of a rating containing BIF with the striped consisting of the colors associated with each half of the rating. If the ratings for BIF are the same, there will be a solid color field.

Table 98: ASTM D 1654 ratings for blasted steel with MIL-DTL-53039 topcoat

Substrate	Finish	Pretreatment	Primer	Topcoat	Scribe	124 hours	432 Hours
Steel	Blast	BAM	MIL-DTL-53022	MIL-C-53039	Yes	9	5 BIF 5
Steel	Blast	BAM	MIL-DTL-53022	MIL-C-53039	Yes	9 BIF 6	5 BIF 4
Steel	Blast	BAM	MIL-DTL-53022	MIL-C-53039	Yes	9	5 BIF 8
Steel	Blast	BAM	MIL-DTL-53022	MIL-C-53039	No	10	5
Steel	Blast	BAM	MIL-DTL-53022	MIL-C-53039	No	10	5
Steel	Blast	BAM	MIL-DTL-53030	MIL-C-53039	Yes	9 BIF 3	terminated
Steel	Blast	BAM	MIL-DTL-53030	MIL-C-53039	Yes	8 BIF 3	terminated
Steel	Blast	BAM	MIL-DTL-53030	MIL-C-53039	Yes	9 BIF 3	terminated
Steel	Blast	BAM	MIL-DTL-53030	MIL-C-53039	No	3	terminated
Steel	Blast	BAM	MIL-DTL-53030	MIL-C-53039	No	2	terminated
Steel	Blast	BAM	MIL-PRF-23377 N1	MIL-C-53039	Yes	9 BIF 4	5 BIF 2
Steel	Blast	BAM	MIL-PRF-23377 N1	MIL-C-53039	Yes	9 BIF 4	5 BIF 3
Steel	Blast	BAM	MIL-PRF-23377 N1	MIL-C-53039	Yes	9 BIF 4	5 BIF 2
Steel	Blast	BAM	MIL-PRF-23377 N1	MIL-C-53039	No	10	6
Steel	Blast	BAM	MIL-PRF-23377 N1	MIL-C-53039	No	10	3
Steel	Blast	BAM	MIL-PRF-23377 N2	MIL-C-53039	Yes	9 BIF 5	5 BIF 3
Steel	Blast	BAM	MIL-PRF-23377 N2	MIL-C-53039	Yes	8 BIF 6	4 BIF 3
Steel	Blast	BAM	MIL-PRF-23377 N2	MIL-C-53039	Yes	9 BIF 6	5 BIF 4
Steel	Blast	BAM	MIL-PRF-23377 N2	MIL-C-53039	No	9	4
Steel	Blast	BAM	MIL-PRF-23377 N2	MIL-C-53039	No	8	4

Table 99: ASTM D 1654 ratings for blasted steel with MIL-DTL-64159 topcoat

Substrate	Finish	Pretreatment	Primer	Topcoat	Scribe	124 hours	432 Hours
Steel	Blast	BAM	MIL-DTL-53022	MIL-DTL-64159	Yes	8 BIF 9	8 BIF 6
Steel	Blast	BAM	MIL-DTL-53022	MIL-DTL-64159	Yes	8 BIF 8	6 BIF 7
Steel	Blast	BAM	MIL-DTL-53022	MIL-DTL-64159	Yes	8 BIF 7	5 BIF 5
Steel	Blast	BAM	MIL-DTL-53022	MIL-DTL-64159	No	7	5
Steel	Blast	BAM	MIL-DTL-53022	MIL-DTL-64159	No	8	5
Steel	Blast	BAM	MIL-DTL-53030	MIL-DTL-64159	Yes	4 BIF 3	terminated
Steel	Blast	BAM	MIL-DTL-53030	MIL-DTL-64159	Yes	4 BIF 3	terminated
Steel	Blast	BAM	MIL-DTL-53030	MIL-DTL-64159	Yes	4 BIF 2	terminated
Steel	Blast	BAM	MIL-DTL-53030	MIL-DTL-64159	No	5	terminated
Steel	Blast	BAM	MIL-DTL-53030	MIL-DTL-64159	No	5	terminated
Steel	Blast	BAM	MIL-PRF-23377 N1	MIL-DTL-64159	Yes	6 BIF 4	5 BIF 4
Steel	Blast	BAM	MIL-PRF-23377 N1	MIL-DTL-64159	Yes	6 BIF 4	5 BIF 4
Steel	Blast	BAM	MIL-PRF-23377 N1	MIL-DTL-64159	Yes	5 BIF 4	4 BIF 3
Steel	Blast	BAM	MIL-PRF-23377 N1	MIL-DTL-64159	No	5	3
Steel	Blast	BAM	MIL-PRF-23377 N1	MIL-DTL-64159	No	5	4
Steel	Blast	BAM	MIL-PRF-23377 N2	MIL-DTL-64159	Yes	7 BIF 5	5 BIF 1
Steel	Blast	BAM	MIL-PRF-23377 N2	MIL-DTL-64159	Yes	7 BIF 4	4 BIF 1
Steel	Blast	BAM	MIL-PRF-23377 N2	MIL-DTL-64159	Yes	7 BIF 4	4 BIF 0
Steel	Blast	BAM	MIL-PRF-23377 N2	MIL-DTL-64159	No	5	2
Steel	Blast	BAM	MIL-PRF-23377 N2	MIL-DTL-64159	No	4	3

Figure 95: BAM-PPV on grit blasted panel with MIL-DTL-53022 and MIL-DTL-53039 after 432 hours ASTM B 117 exposure

Applying MIL-PRF-85285 topcoat in lieu of the Army CARC topcoats resulted in improved performance for all systems over the as received finishes. As can be seen in Table 100, ASTM D 1654 ratings were similar to or slightly worse that those received for comparable systems over blasted surfaces with Army topcoats. The unscribed panels with the Army primers performed better than their counterparts with the complete Army system. The grit blasted panels with MIL-PRF-23377C and MIL-PRF-85285 did surprisingly poorly considering the presence of chromate in the primer. Figure 96 shows typical performance after 432 hours for BAM-PPV topcoated with MIL-PRF-85285.

Table 100: ASTM D 1654 ratings for BAM-PPV pretreated systems with MIL-PRF-85285 topcoat

Substrate	Finish	Pretreatment	Primer	Topcoat	Scribe	124 hours	432 Hours
Steel	None	BAM	MIL-DTL-53022	MIL-PRF-85285	Yes	4 BIF 8	3 BIF 4
Steel	None	BAM	MIL-DTL-53022	MIL-PRF-85285	Yes	5 BIF 8	4 BIF 2
Steel	None	BAM	MIL-DTL-53022	MIL-PRF-85285	Yes	4 BIF 8	4 BIF 4
Steel	None	BAM	MIL-DTL-53022	MIL-PRF-85285	No	9	4
Steel	None	BAM	MIL-DTL-53022	MIL-PRF-85285	No	8	5
Steel	None	BAM	MIL-DTL-53030	MIL-PRF-85285	Yes	3 BIF 8	2 BIF 1
Steel	None	BAM	MIL-DTL-53030	MIL-PRF-85285	Yes	3 BIF 8	2 BIF 1
Steel	None	BAM	MIL-DTL-53030	MIL-PRF-85285	No	9	3
Steel	None	BAM	MIL-DTL-53030	MIL-PRF-85285	No	9	3
Steel	None	BAM	MIL-PRF-23377 N1	MIL-PRF-85285	Yes	5 BIF 7	2 BIF 3
Steel	None	BAM	MIL-PRF-23377 N1	MIL-PRF-85285	Yes	5 BIF 6	3 BIF 2
Steel	None	BAM	MIL-PRF-23377 N1	MIL-PRF-85285	Yes	4 BIF 8	4 BIF 3
Steel	None	BAM	MIL-PRF-23377 N1	MIL-PRF-85285	No	9	4
Steel	None	BAM	MIL-PRF-23377 N1	MIL-PRF-85285	No	8	4
Steel	None	BAM	MIL-PRF-23377 N2	MIL-PRF-85285	Yes	5 BIF 5	3 BIF 3
Steel	None	BAM	MIL-PRF-23377 N2	MIL-PRF-85285	Yes	5 BIF 4	2 BIF 3
Steel	None	BAM	MIL-PRF-23377 N2	MIL-PRF-85285	Yes	5 BIF 8	3 BIF 0
Steel	None	BAM	MIL-PRF-23377 N2	MIL-PRF-85285	No	5	4
Steel	None	BAM	MIL-PRF-23377 N2	MIL-PRF-85285	No	5	4
Steel	Blast	BAM	MIL-DTL-53022	MIL-PRF-85285	Yes	8	4 BIF 5
Steel	Blast	BAM	MIL-DTL-53022	MIL-PRF-85285	Yes	8	5 BIF 5
Steel	Blast	BAM	MIL-DTL-53022	MIL-PRF-85285	Yes	7	5 BIF 5
Steel	Blast	BAM	MIL-DTL-53022	MIL-PRF-85285	No	9	9
Steel	Blast	BAM	MIL-DTL-53022	MIL-PRF-85285	No	9	9
Steel	Blast	BAM	MIL-DTL-53030	MIL-PRF-85285	Yes	4 BIF 8	4 BIF 1
Steel	Blast	BAM	MIL-DTL-53030	MIL-PRF-85285	Yes	4 BIF 8	4 BIF 2
Steel	Blast	BAM	MIL-DTL-53030	MIL-PRF-85285	Yes	4 BIF 8	4 BIF 0
Steel	Blast	BAM	MIL-DTL-53030	MIL-PRF-85285	No	9	3
Steel	Blast	BAM	MIL-DTL-53030	MIL-PRF-85285	No	9	4
Steel	None	BAM	MIL-PRF-23377 C	MIL-PRF-85285	Yes	4 BIF 8	4 BIF 2
Steel	None	BAM	MIL-PRF-23377 C	MIL-PRF-85285	Yes	4 BIF 8	4 BIF 2
Steel	None	BAM	MIL-PRF-23377 C	MIL-PRF-85285	Yes	4 BIF 8	5 BIF 3
Steel	None	BAM	MIL-PRF-23377 C	MIL-PRF-85285	No	6	4
Steel	None	BAM	MIL-PRF-23377 C	MIL-PRF-85285	No	7	4

Figure 96: BAM-PPV on as received steel with MIL-DTL-53022 and MIL-PRF-85285 following 432 hours ASTM B 117 exposure

The wash primer, DoD-P-15328, improved the performance of the as-received steel to the point where everything passed at 124 hours and only the set with MIL-PRF-23377N1 primer would have had to have been removed at 432 hours (Table 100). Table 101 shows that the early performance of all comparable systems is improved by the chromate containing DoD-P-15328 over the BAM-PPV (Table 99) for grit blasted surfaces. However, that performance edge is lost with an additional 300 hours of exposure excepting the system containing MIL-DTL-53030 (see Table 102). The best performing system on either surface finish with wash primer was the one containing MIL-PRF-23377C. Figures 97 and 98 are typical examples of what the as-received and grit blasted panels looked like following 432 hours of ASTM B 117.

Table 101: ASTM D 1654 ratings for DoD-P-15328 on as received steel

Substrate	Finish	Pretreatment	Primer	Topcoat	Scribe	124 hours	432 Hours
Steel	None	DoD-P-15328	MIL-DTL-53022	MIL-DTL-64159	Yes	9 BIF 8	8 BIF 8
Steel	None	DoD-P-15328	MIL-DTL-53022	MIL-DTL-64159	Yes	9 BIF 7	6 BIF 7
Steel	None	DoD-P-15328	MIL-DTL-53022	MIL-DTL-64159	Yes	9 BIF 8	6 BIF 6
Steel	None	DoD-P-15328	MIL-DTL-53022	MIL-DTL-64159	No	8	8
Steel	None	DoD-P-15328	MIL-DTL-53022	MIL-DTL-64159	No	9	9
Steel	None	DoD-P-15328	MIL-DTL-53030	MIL-DTL-64159	Yes	8 BIF 4	4 BIF 4
Steel	None	DoD-P-15328	MIL-DTL-53030	MIL-DTL-64159	Yes	9 BIF 5	5 BIF 4
Steel	None	DoD-P-15328	MIL-DTL-53030	MIL-DTL-64159	Yes	9 BIF 5	5 BIF 5
Steel	None	DoD-P-15328	MIL-DTL-53030	MIL-DTL-64159	No	8	7
Steel	None	DoD-P-15328	MIL-DTL-53030	MIL-DTL-64159	No	8	7
Steel	None	DoD-P-15328	MIL-PRF-23377 N1	MIL-DTL-64159	Yes	8 BIF 5	5 BIF 0
Steel	None	DoD-P-15328	MIL-PRF-23377 N1	MIL-DTL-64159	Yes	7 BIF 5	7 BIF 0
Steel	None	DoD-P-15328	MIL-PRF-23377 N1	MIL-DTL-64159	Yes	6 BIF 6	6 BIF 0
Steel	None	DoD-P-15328	MIL-PRF-23377 N1	MIL-DTL-64159	No	8	0
Steel	None	DoD-P-15328	MIL-PRF-23377 N1	MIL-DTL-64159	No	9	0
Steel	None	DoD-P-15328	MIL-PRF-23377 C	MIL-DTL-64159	Yes	8 BIF 6	8 BIF 6
Steel	None	DoD-P-15328	MIL-PRF-23377 C	MIL-DTL-64159	Yes	9 BIF 8	8 BIF 6
Steel	None	DoD-P-15328	MIL-PRF-23377 C	MIL-DTL-64159	Yes	9 BIF 6	8 BIF 6
Steel	None	DoD-P-15328	MIL-PRF-23377 C	MIL-DTL-64159	No	8	7
Steel	None	DoD-P-15328	MIL-PRF-23377 C	MIL-DTL-64159	No	8	7

Table 102: ASTM D 1654 ratings for DoD-P-15328 on as received steel

Substrate	Finish	Pretreatment	Primer	Topcoat	Scribe	124 hours	432 Hours
Steel	Blast	DoD-P-15328	MIL-DTL-53022	MIL-DTL-64159	Yes	9 BIF 7	6 BIF 7
Steel	Blast	DoD-P-15328	MIL-DTL-53022	MIL-DTL-64159	Yes	7 BIF 6	5 BIF 6
Steel	Blast	DoD-P-15328	MIL-DTL-53022	MIL-DTL-64159	Yes	8 BIF 6	6 BIF 6
Steel	Blast	DoD-P-15328	MIL-DTL-53022	MIL-DTL-64159	No	5	5
Steel	Blast	DoD-P-15328	MIL-DTL-53022	MIL-DTL-64159	No	7	5
Steel	Blast	DoD-P-15328	MIL-DTL-53030	MIL-DTL-64159	Yes	8 BIF 6	5 BIF 6
Steel	Blast	DoD-P-15328	MIL-DTL-53030	MIL-DTL-64159	Yes	8 BIF 5	8 BIF 5
Steel	Blast	DoD-P-15328	MIL-DTL-53030	MIL-DTL-64159	Yes	9 BIF 5	6 BF 5
Steel	Blast	DoD-P-15328	MIL-DTL-53030	MIL-DTL-64159	No	5	5
Steel	Blast	DoD-P-15328	MIL-DTL-53030	MIL-DTL-64159	No	6	5
Steel	Blast	DoD-P-15328	MIL-PRF-23377 N1	MIL-DTL-64159	Yes	9 BIF 5	4 BIF 5
Steel	Blast	DoD-P-15328	MIL-PRF-23377 N1	MIL-DTL-64159	Yes	9 BIF 5	4 BIF 5
Steel	Blast	DoD-P-15328	MIL-PRF-23377 N1	MIL-DTL-64159	Yes	9 BIF 5	4 BIF 4
Steel	Blast	DoD-P-15328	MIL-PRF-23377 N1	MIL-DTL-64159	No	6	2
Steel	Blast	DoD-P-15328	MIL-PRF-23377 N1	MIL-DTL-64159	No	6	3
Steel	Blast	DoD-P-15328	MIL-PRF-23377 C	MIL-DTL-64159	Yes	9 BIF 7	8 BIF 7
Steel	Blast	DoD-P-15328	MIL-PRF-23377 C	MIL-DTL-64159	Yes	9 BIF 7	7 BIF 7
Steel	Blast	DoD-P-15328	MIL-PRF-23377 C	MIL-DTL-64159	Yes	9 BIF 6	7 BIF 6
Steel	Blast	DoD-P-15328	MIL-PRF-23377 C	MIL-DTL-64159	No	5	5
Steel	Blast	DoD-P-15328	MIL-PRF-23377 C	MIL-DTL-64159	No	5	5

Figure 97: DoD-P-15328 on as received steel panel with MIL-DTL-53022 and MIL-DTL-64159 after 432 hours ASTM B 117 exposure

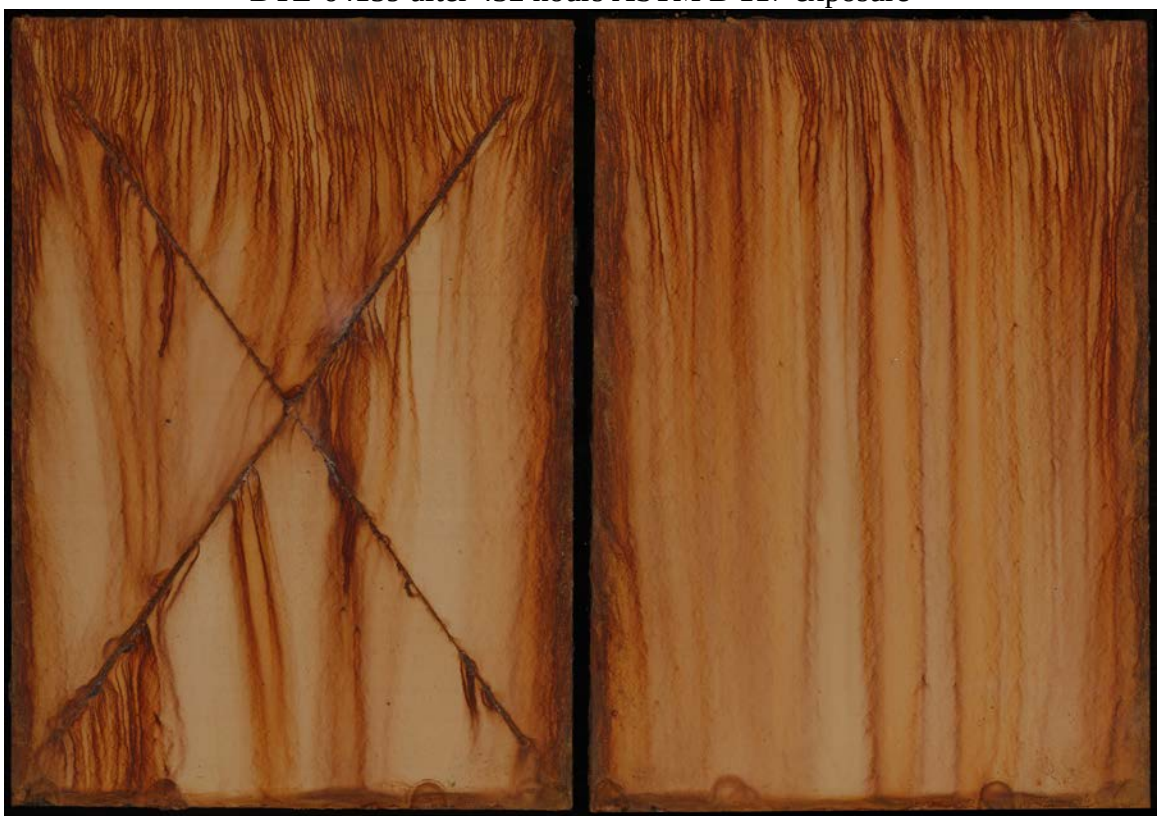


Figure 98: DoD-P-15328 on grit blasted steel panel with MIL-DTL-53022 and MIL-DTL-64159 after 432 hours ASTM B 117 exposure

There were an additional three sets of as-received steel that were coated at another facility that has more familiarity with the application of BAM-PPV. The results are presented in Table 103. When compared with like systems from Table 99, the systems applied at ARL performed as well or better than those applied at an outside facility.

Table 103: ASTM D 1654 ratings for as received steel pretreated with BAM-PPV and topcoated with MIL-PRF-85285 not applied at ARL

Substrate	Finish	Pretreatment	Primer	Topcoat	Scribe	124 hours	432 Hours
Steel	None	BAM	MIL-PRF-23377 N1	MIL-DTL-64159	Yes	4 BIF7	3 BIF 2
Steel	None	BAM	MIL-PRF-23377 N1	MIL-DTL-64159	Yes	4 BIF 8	2 BIF 2
Steel	None	BAM	MIL-PRF-23377 N1	MIL-DTL-64159	Yes	4 BIF 8	2 BIF 3
Steel	None	BAM	MIL-PRF-23377 N1	MIL-DTL-64159	No	8	4
Steel	None	BAM	MIL-PRF-23377 N1	MIL-DTL-64159	No	8	4
Steel	None	BAM	MIL-PRF-23377 N2	MIL-DTL-64159	Yes	4 BIF 8	2 BIF 2
Steel	None	BAM	MIL-PRF-23377 N2	MIL-DTL-64159	Yes	4 BIF 8	2 BIF 3
Steel	None	BAM	MIL-PRF-23377 N2	MIL-DTL-64159	Yes	4 BIF 8	2 BIF 3
Steel	None	BAM	MIL-PRF-23377 N2	MIL-DTL-64159	No	9	4
Steel	None	BAM	MIL-PRF-23377 N2	MIL-DTL-64159	No	9	3
Steel	None	BAM	MIL-PRF-23377 C	MIL-DTL-64159	Yes	5 BIF 8	5 BIF 3
Steel	None	BAM	MIL-PRF-23377 C	MIL-DTL-64159	Yes	4 BIF 8	4 BIF 3
Steel	None	BAM	MIL-PRF-23377 C	MIL-DTL-64159	Yes	4 BIF 7	3 BIF 3
Steel	None	BAM	MIL-PRF-23377 C	MIL-DTL-64159	No	9	4
Steel	None	BAM	MIL-PRF-23377 C	MIL-DTL-64159	No	9	4

All of the aluminum panels exposed had the as-received surface finish. Most of the AA 6061 panels coated with MIL-DTL-53022 and scribed through the coating survived to 1248 hours before delaminating at the scribe. Performance was consistent for either Army topcoat. As seen in Table 104, all of the unscribed panels lasted through 2016 hours. The BAM-PPV with MIL-DTL-53030 performed better than the solvent borne version. Only two of the scribed panels topcoated with MIL-DTL-53039 failed at 1248 hours of exposure. The remainder lasted to 2016 hours (Table 105). The control panels, pretreated with TCP, mirrored the performance of the BAM-PPV. As can be seen in Table 106, the TCP primed with MIL-PRF-23377C was the best performer and the TCP primed with MIL-PRF-23377N1 also did very well. There were no comparable systems for these pretreated with BAM-PPV.

Table 104: ASTM D 1654 ratings for AA6061with BAM-PPV pretreatment and MIL-DTL-53022

Substrate	Pretreatment	Primer	Topcoat	Scribe	168 hours	504 Hours	1248 Hours	2016 Hours
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-53039	Yes	8	0		
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-53039	Yes	7	6 BIF8	0	
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-53039	Yes	6	3	0	
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-53039	Yes	7	4	0	
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-53039	No	10	6	5	4
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-53039	No	10	10	10	8
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-53039	No	10	10	5	3
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-53039	No	10	10	5	4
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-64159	Yes	5	0		
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-64159	Yes	5	0		
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-64159	Yes	6	6	0	
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-64159	Yes	5	5 BIF 7	5 BIF 5	5 BIF 4
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-64159	Yes	5	3	0	
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-64159	No	10	7	6	5
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-64159	No	10	6	5	5
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-64159	No	10	10	7	7
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-64159	No	10	10	5	4
Al 6061	BAM	MIL-DTL-53022	MIL-DTL-64159	No	10	8	6	5

Table 105: ASTM D 1654 ratings for AA6061with BAM-PPV pretreatment and MIL-DTL-53030

Substrate	Pretreatment	Primer	Topcoat	Scribe	168 hours	504 Hours	1248 Hours	2016 Hours
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-53039	Yes	9	8 BIF 7	5 BIF 4	5 BIF 4
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-53039	Yes	9	7 BIF 6	0	
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-53039	Yes	9	4 BIF 5	0	
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-53039	Yes	8	7 BIF 6	7 BIF 4	5 BIF 3
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-53039	Yes	8	7 BIF 9	2 BIF9	1 BIF 3
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-53039	No	10	7	5	5
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-53039	No	10	10	8	6
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-53039	No	10	7	6	5
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-53039	No	10	10	6	4
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-53039	No	10	10	7	7
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-64159	Yes	9	6	6 BIF 7	5 BIF 5
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-64159	Yes	7	7	7 BIF 9	5 BIF 9
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-64159	Yes	9	8	7 BIF 7	5 BIF 7
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-64159	No	10	8	7	6
Al 6061	BAM	MIL-DTL-53030	MIL-DTL-64159	No	8	8	6	6

Table 106: ASTM D 1654 ratings for AA6061with TCP and MIL-DTL-64159 topcoat

Substrate	Pretreatment	Primer	Topcoat	Scribe	168 hours	504 Hours	1248 Hours	2016 Hours
Al 6061	TCP	MIL-DTL-53022	MIL-DTL-64159	Yes	7	4 BIF 7	0	
Al 6061	TCP	MIL-DTL-53022	MIL-DTL-64159	Yes	4	1 BIF 4	0	
Al 6061	TCP	MIL-DTL-53022	MIL-DTL-64159	No	9	7	4	
Al 6061	TCP	MIL-DTL-53030	MIL-DTL-64159	Yes	9	8 BIF 5	5 BIF 3	5 BIF 3
Al 6061	TCP	MIL-DTL-53030	MIL-DTL-64159	Yes	9 BIF 9	5 BIF 7	2 BIF 7	1 BIF 4
Al 6061	TCP	MIL-DTL-53030	MIL-DTL-64159	No	10	7	6	5
Al 6061	TCP	MIL-PRF-23377 C	MIL-DTL-64159	Yes	10	9 BIF 8	9 BIF 7	9 BIF 7
Al 6061	TCP	MIL-PRF-23377 C	MIL-DTL-64159	Yes	10	9 BIF 9	9 BIF 8	9 BIF 8
Al 6061	TCP	MIL-PRF-23377 C	MIL-DTL-64159	No	10	9	9	9
Al 6061	TCP	MIL-PRF-23377 NC1	MIL-DTL-64159	Yes	9 BIF 7	8 BIF 5	8 BIF 5	7 BIF 5
Al 6061	TCP	MIL-PRF-23377 NC1	MIL-DTL-64159	Yes	8 BIF 8	8 BIF 7	8 BIF 7	7 BIF 5
Al 6061	TCP	MIL-PRF-23377 NC1	MIL-DTL-64159	No	10	8	7	4
Al 6061	TCP	MIL-PRF-23377 NC2	MIL-DTL-64159	No	10	9	4	4

The scribed AA5083 panels performed relatively poorly when viewed in light of their stellar unscribed performance. As can be seen in Tables 107 and 108, nearly everything survived to 2016 hours of ASTM B 117. The exceptions were on panels of scribed BAM-PPV with MIL-DTL-53022 and MIL-DTL-53039 and three panels coated with MIL-DTL-53030 with MIL-DTL-64159 (one unscribed). The unscribed panels could easily have continued for another 1000 hours. Blistering of the unscribed panels was only a problem for those topcoated with MIL-DTL-64159. As can be seen in Table 109, the TCP pretreatment was comparable in performance to the BAM-PPV when topcoated with MIL-DTL-64159. Scribed BAM-PPV panels did slightly worse than their TCP counterparts while the unscribed ones did better. Both of the scribed sets with MIL-PRF-23377C and MIL-PRF-23377N1 performed well; however, the unscribed panels performed worse than either of the Army primers.

Table 107: ASTM D 1654 ratings for AA5083 with MIL-DTL-53022 primer

Substrate	Pretreatment	Primer	Topcoat	Scribe	168 hours	504 Hours	1248 Hours	2016 Hours
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-53039	Yes	7	6	3	2
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-53039	Yes	8	4	2	0
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-53039	Yes	8	7	2	2
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-53039	Yes	7	6	3	3
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-53039	Yes	7	4	3	3
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-53039	No	10	10	8	8
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-53039	No	10	10	10	7
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-53039	No	9	9	9	8
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-53039	No	10	10	10	10
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-53039	No	10	10	10	8
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-64159	Yes	6	6	3	2 BIF 6
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-64159	Yes	7	6	5	2 BIF 6
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-64159	Yes	6	6	3	1
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-64159	No	10	10	10	8
Al 5083	BAM	MIL-DTL-53022	MIL-DTL-64159	No	10	10	10	10

Table 108: ASTM D 1654 ratings for AA5083 with MIL-DTL-53030 primer

Substrate	Pretreatment	Primer	Topcoat	Scribe	168 hours	504 Hours	1248 Hours	2016 Hours
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-53039	Yes	9	7	5	4
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-53039	Yes	10	9	5	4
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-53039	Yes	9	9	8	5
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-53039	Yes	9	7	4	3
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-53039	Yes	10	8	5	4
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-53039	No	10	10	10	9
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-53039	No	10	10	10	8
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-53039	No	10	10	10	9
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-53039	No	10	10	10	10
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-53039	No	10	9	9	7
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-64159	Yes	9	7	6 BIF 7	4 BIF 6
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-64159	Yes	4	4	4	2 BIF 7
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-64159	Yes	5	4	4	2 BIF 7
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-64159	Yes	0			
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-64159	Yes	0			
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-64159	No	1			
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-64159	No	10	7	7	5
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-64159	No	10	8	7	5
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-64159	No	10	9	6	5
Al 5083	BAM	MIL-DTL-53030	MIL-DTL-64159	No	10	8	8	7

Table 109: ASTM D 1654 ratings for TCP with military primers and MIL-DTL-64159 topcoat

Substrate	Pretreatment	Primer	Topcoat	Scribe	168 hours	504 Hours	1248 Hours	2016 Hours
Al 5083	TCP	MIL-DTL-53022	MIL-DTL-64159	Yes	8	5	5 BIF 7	3 BIF 5
Al 5083	TCP	MIL-DTL-53022	MIL-DTL-64159	Yes	6	6	6 BIF 7	5 BIF6
Al 5083	TCP	MIL-DTL-53022	MIL-DTL-64159	No	10	8	6	6
Al 5083	TCP	MIL-DTL-53030	MIL-DTL-64159	Yes	10	8 BIF 6	8 BIF 6	8 BIF 6
Al 5083	TCP	MIL-DTL-53030	MIL-DTL-64159	Yes	10	8 BIF 6	8 BIF 6	8 BIF 6
Al 5083	TCP	MIL-DTL-53030	MIL-DTL-64159	No	10	9	8	8
Al 5083	TCP	MIL-PRF-23377 C	MIL-DTL-64159	Yes	9	9	9	9 BIF 5
Al 5083	TCP	MIL-PRF-23377 C	MIL-DTL-64159	Yes	10	9	9	9 BIF 6
Al 5083	TCP	MIL-PRF-23377 C	MIL-DTL-64159	No	10	10	8	5
Al 5083	TCP	MIL-PRF-23377 NC1	MIL-DTL-64159	Yes	10	9	9	9 BIF 6
Al 5083	TCP	MIL-PRF-23377 NC1	MIL-DTL-64159	Yes	10	8	8 BIF 7	8 BIF7
Al 5083	TCP	MIL-PRF-23377 NC1	MIL-DTL-64159	No	10	5	5	5

The poor performance of BAM-PPV as a pretreatment on as-received steel versus the improved performance on similarly prepared grit blasted steel indicates that the BAM-PPV requires a surface profile to provide adequate adhesion for steel substrates. This is significant in that the pretreatment currently in use contains Cr(VI). This material (Cr(VI) is used predominantly in the depot and rework facilities. Coatings applied in these facilities require removal of corrosion and old coating systems. This leaves behind the surface profile necessary to promote adhesion for the BAM-PPV. The currently used wash primer may not protect grit blasted surfaces as well as BAM-PPV over the long term. The best performing system overall with the wash primer (DOD-P-15328) had Cr(VI) containing MIL-PRF-23377C as the primer. This combination only slightly outperformed the best performing system with BAM-PPV as the pretreatment. The presence of an intentional defect on a BAM-PPV pretreated AA6061 panel coated with MIL-DTL-53022 caused debonding to occur radially from the scribe and failure to occur quicker than on an unscribed panel. Scribed panels with the same pretreatment and MIL-DTL-53030 primer had problems when topcoated with MIL-DTL-53039.

However, by providing a surface profile an improvement in the adhesion and corrosion performance of BAM-PPV was observed. When using the TCP, scribed panels performed slightly better, while their unscribed counterparts performed slightly worse. The AA5083 pretreated with BAM-PPV displayed a corrosion performance increase over every similar coating system combination on AA6061 save one, the MIL-DTL-53030 primer with MIL-DTL-64159 topcoat. There was significantly less blistering in the field for these panels. The adhesion near the scribe was also improved. Similarly, the performance of the TCP pretreated panels also improved for all coating systems. **Again, the corrosion performance of the BAM-PPV with MIL-DTL-53030 primer and MIL-DTL-64159, and the other systems, would be improved by increasing the surface profile.**

6.1.2.2 ARL Conclusions for BAM-PPV and MEH-PPV Laboratory Studies

The extensive laboratory testing by ARL on both BAM-PPV and MEH-PPV coated coupons have shown the following trends:

- ***MEH-PPV pretreatment showed <u>no consistent performance advantage</u> when using Navy primers,***

- ***<u>BAM-PPV pretreatment coating outperforms MEH-PPV pretreatment</u> coating when using water-borne Army primers,***

- ***<u>BAM-PPV shows improved adhesion on grit blasted steel surfaces</u>,***

- ***<u>DoD-P-15328</u> <u>does not show improved performance</u> on grit blasted steel surfaces****,*

- ***<u>BAM-PPV can provide improved corrosion and adhesion</u> performance on grit blasted steel substrates****,*

- ***BAM-PPV can provide acceptable corrosion performance and adhesion which is important from the standpoint of maintenance at Army depots and rework facilities, thereby, <u>eliminating Cr(VI) from wash primers</u> during these rework operations****.*

Therefore, BAM-PPV was selected for field demonstration based on its superior performance over MEH-PPV pretreatment. *BAM-PPV due to its adhesion profile requirement was field tested on sand blasted aluminum surface and field tested for one-year duration at the ARL ATC.*

6.1.3 Laboratory Testing at NAWCAD/NAWCWD for BAM-PPV and MEH-PPV Pretreatment Coatings

The results from NAWCWD/NAWCAD laboratory testing of BAM-PPV and MEH-PPV are found in sections 6.1.3.1 – 6.1.3.3.3 **Bulletized summations for NAWCAD/NAWCWD laboratory testing are found in section 6.1.3.4 This testing proves that BAM-PPV is a more processable, robust, stable compound than MEH-PPV and was therefore selected for field testing.**

6.1.3.1 Tape adhesion testing for BAM-PPV and MEH-PPV Pretreatments

The NAWCWD provided for NAWCAD testing purposes, BAM-PPV and MEH-PPV pretreatment coated panels. The BAM-PPV and MEH-PPV pretreatment coated panels were tested for tape adhesion. Test panel substrates were 3” x 6” AA2024-T3, AA6061-T6, AA7075-T6 and steel 4130. The test panels were painted at NAWCAD using the following primers: MIL-PRF-23377C2, MIL-PRF-23377N (02GN083 by Deft), MIL-PRF-23377N (16708TEP by Hentzen), MIL-PRF-85582N (44GN098 by Deft), TT-

P-2756, MIL-DTL-53022, and MIL-DTL-53030 (see Table 110). The BAM-PPV coated 4130 steel panels also received one of the following topcoats: MIL-DTL-53039, MIL-PRF-85285, or MIL-DTL-64159 (see Table 111). Table 112 shows the tape adhesion test matrix for aluminum alloy panels and Table 113 shows the tape adhesion test matrix for steel panels.

Table 110: BAM-PPV and MEH-PPV Coated with Various Primers

Substrate	Pretreatment(s)	Primer	Primer	Primer	Primer	Primer	Primer
AA 2024-T3	BAM-PPV/MEH-PPV	MIL-PRF-23377C2	MIL-PRF-23377N (02GN083 /Deft)	MIL-PRF-85582N (16708TEP Hentzen)	TT-P-2756	MIL-DTL-53022	MIL-DTL-53030
AA6061-T6	BAM-PPV/MEH-PPV	MIL-PRF-23377C2	MIL-PRF-23377N (02GN083 /Deft)	MIL-PRF-85582N (16708TEP Hentzen)	TT-P-2756	MIL-DTL-53022	MIL-DTL-53030
AA7075-T6	BAM-PPV/MEW-PPV	MIL-PRF-23377C2	MIL-PRF-23377N (02GN083 /Deft)	MIL-PRF-85582N (16708TEP Hentzen)	TT-P-2756	MIL-DTL-53022	MIL-DTL-53030

Table 111: BAM-PPV Pretreatment on High Strength 4130 Steel

Substrate	Pretreatment	Primer	Topcoat	Topcoat	Topcoat
4130 Steel	BAM-PPV	MIL-DTL-53022	MIL-DTL-53039	MIL-PRF-85285	MIL-DTL-64159
4130 Steel	BAM-PPV	MIL-DTL-53030	MIL-DTL-53039	MIL-PRF-85285	MIL-DTL-64159
4130 Steel	BAM-PPV	MIL-PRF-2377N (02GN083)	MIL-DTL-53039	MIL-PRF-85285	MIL-DTL-64159
4130 Steel	BAM-PPV	Mil-PRF-23377N (1680TEP Hentzen)	MIL-DTL-53039	MIL-PRF-85285	MIL-DTL-64159

Table 112: Tape Adhesion Test Matrix for Aluminum Alloy Panels

Primer	BAM-PPV AA2024	BAM-PPV AA6061	BAM-PPV AA7075	MEH-PPV AA2024	MEH-PPV AA6061	MEH-PPV AA7075
MIL-PRF-23377C2	2	-	3	2	-	3
MIL-PRF-23377N (02GN083)	3	-	3	3	-	3
MIL-PRF-23377N (16708TEP)	3	-	3	3	-	3
MIL-PRF-85582N (44GN098)	3	-	3	3	-	3
TT-P-2756	3	-	3	3	-	3
MIL-DTL-53022	-	2	-	-	1	-
MIL-DTL-53030	-	2	-	-	2	-

Table 113: Tape Adhesion Test Matrix for BAM-PPV Coated 4130 Steel Panels

Primer	MIL-DTL-53039	MIL-PRF-85285	MIL-DTL-64159
MIL-DTL-53022	1	1	1
MIL-DTL-53030	1	1	1
MIL-PRF-23377N (02GN083)	1	1	1
MIL-PRF-23377N (16708TEP)	1	1	1

After paint application, test panels were allowed to cure in accordance with primer specification prior to testing for wet-tape paint adhesion (WTA). Adhesion testing was conducted in accordance with FED-STD-141, Method 6301.3 and ASTM D 3359, Test Method A-X-Cut Tape Test. No controls were used for these WTA tests rather average ratings for the aluminum and steel test panels were recorded. Fully chromated systems (CCC + chromate primer and CCC + chromate primer and topcoat) are known to pass WTA and therefore duplication of these results was unnecessary. The investigation of alternative pretreatments with various primers and topcoats were specifically studied in order to gauge their adhesion on various substrates using different combinations of primers and topcoats.

Figures 99-129 graphically depict the tape adhesion ratings by EAP coating per test panel substrate. Each graph is followed by pictures of the test panels. The majority of adhesion failures occurred between the EAP coating and the substrate. However, as is visible in Figures 119 and 125, adhesion failure is exhibited between the TT-P-2756 and the BAM-PPV coating on AA2024 and AA7075 test panels.

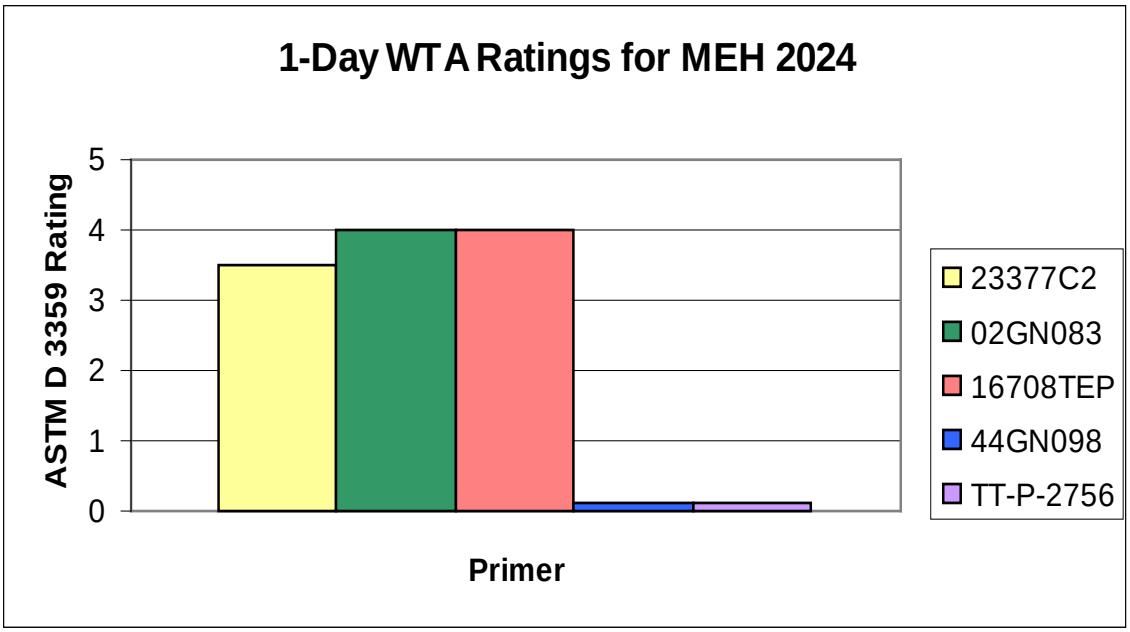


Figure 99: Average 1-Day Wet Tape Adhesion Ratings for MEH-PPV on AA2024

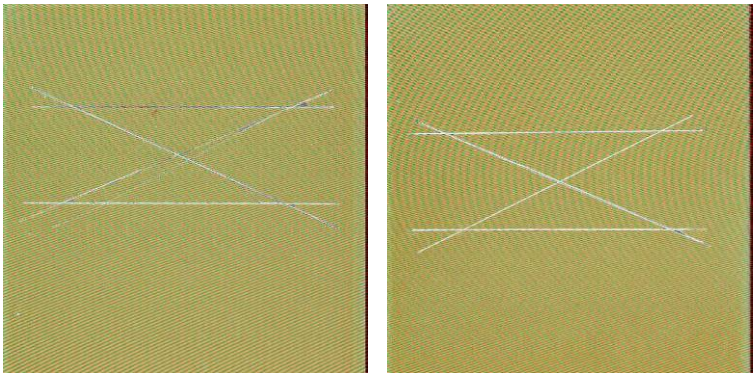


Figure 100: Test Panels after 1-Day WTA-MEH-PPV on AA2024 with MIL-PRF-23377C2

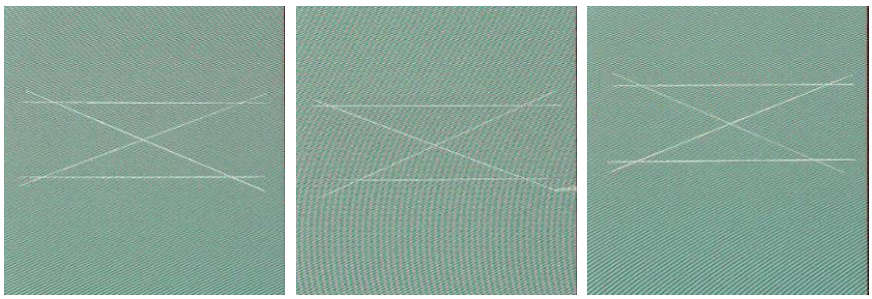


Figure 101: Test Panels after 1-Day WTA- MEH-PPV on AA2024 with 02GN083

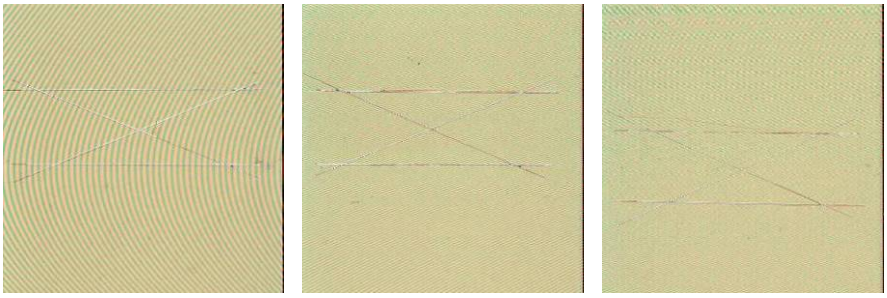


Figure 102: Test Panels after 1-Day WTA-MEH-PPV on AA2024 with 16708TEP

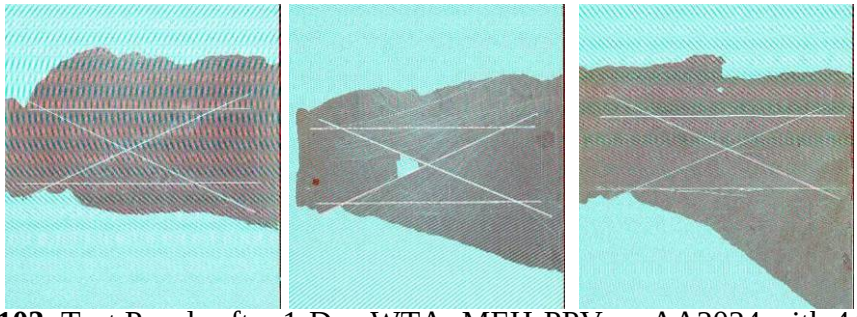


Figure 103: Test Panels after 1-Day WTA- MEH-PPV on AA2024 with 44GN098

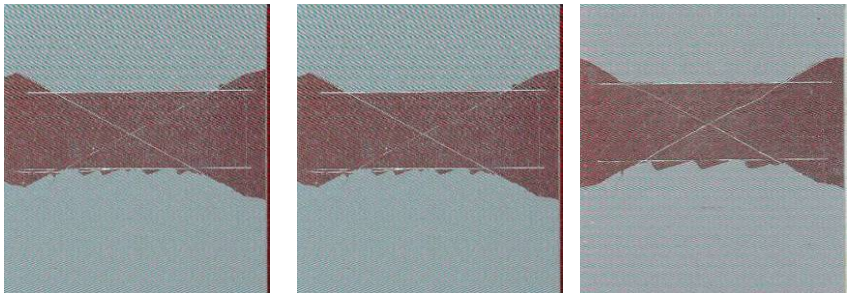


Figure 104: Test Panels after 1-Day WTA – MEH-PPV on AA2024 with TT-P-2756

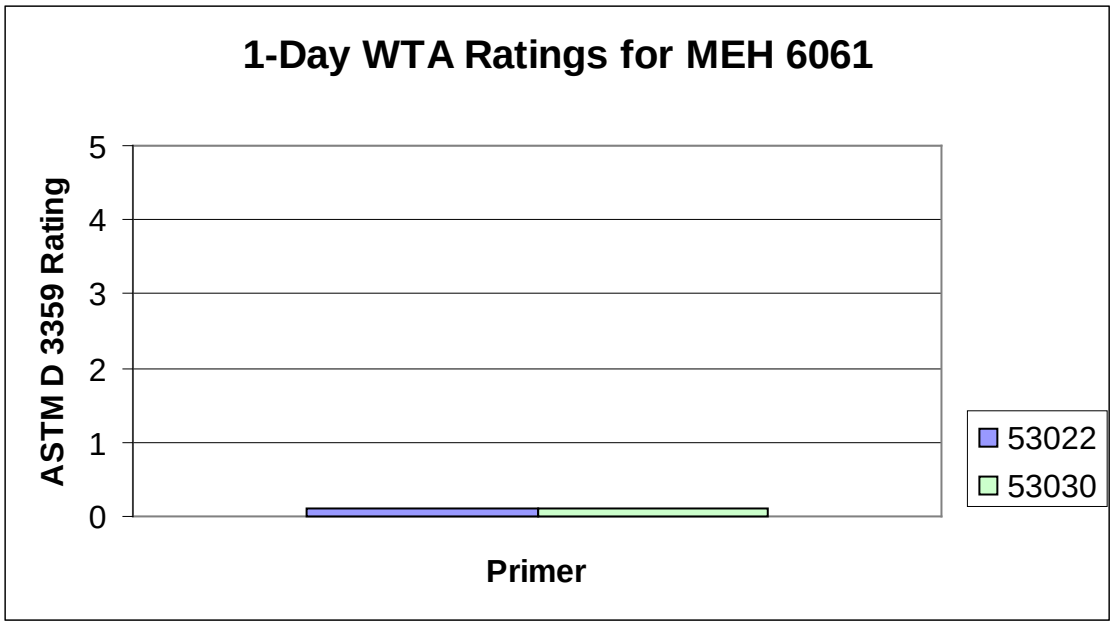


Figure 105: Average 1-Day Wet Tape Adhesion Ratings for MEH-PPV on AA6061

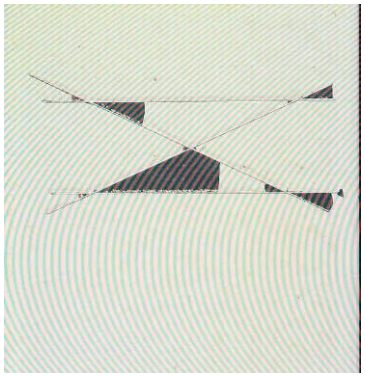


Figure 106: Test Panels after 1-Day WTA- MEH-PPV on AA6061 with MIL-DTL-53022

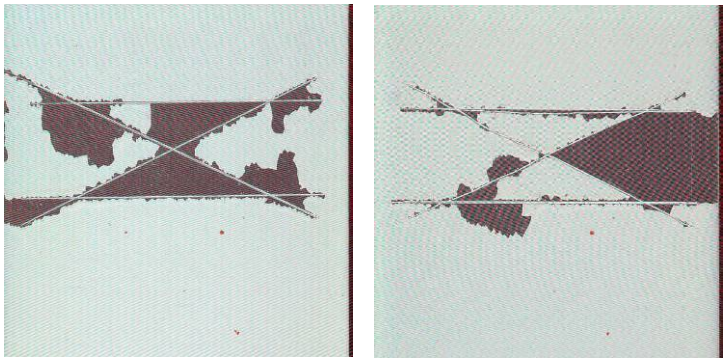


Figure 107: Test Panels after 1-Day WTA- MEH-PPV on AA6061 with MIL-DTL-53030

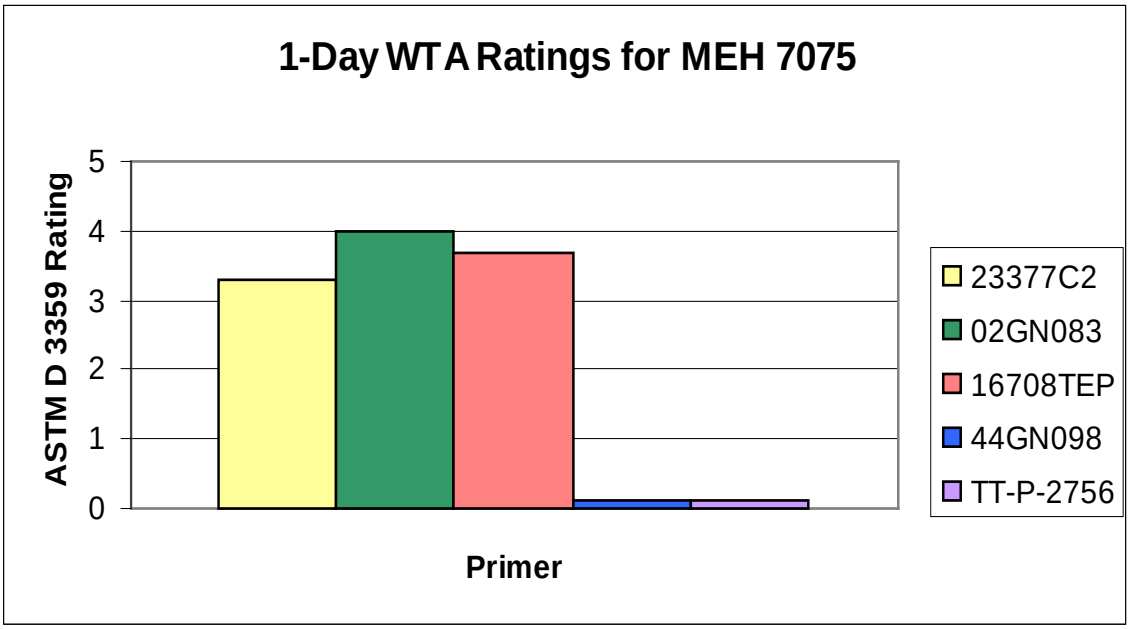


Figure 108: Average 1-Day Wet Tape Adhesion Ratings for MEH-PPV on AA7075

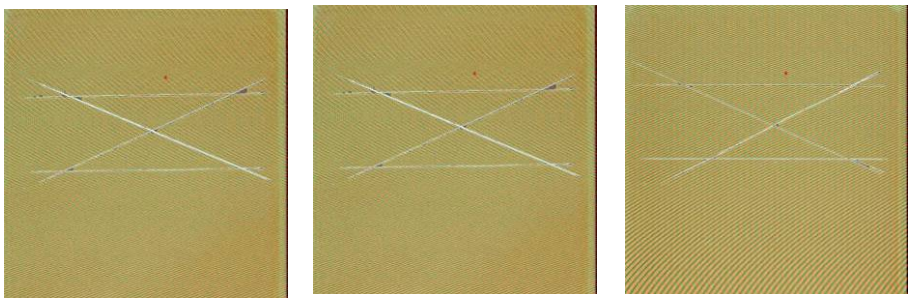


Figure 109: Test Panels after 1-Day WTA-MEH-PPV on AA7075

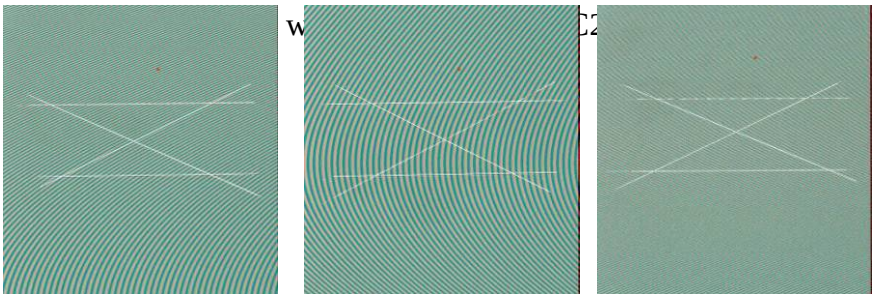


Figure 110: Test Panels after 1-Day WTA-MEH-PPV on AA7075 with 02GN083

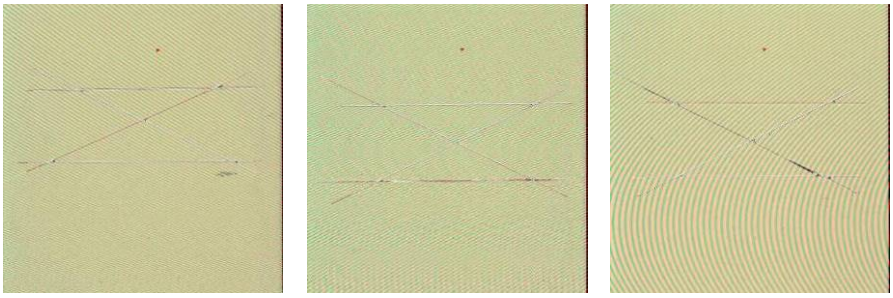


Figure 111: Test Panels after 1-Day WTA-MEH-PPV on AA7075 with 16708TEP

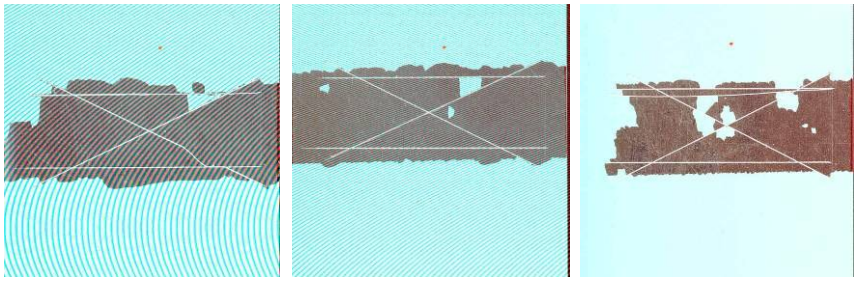


Figure 112: Test Panels after 1-Day WTA-MEH-PPV on AA7075 with 44GN098

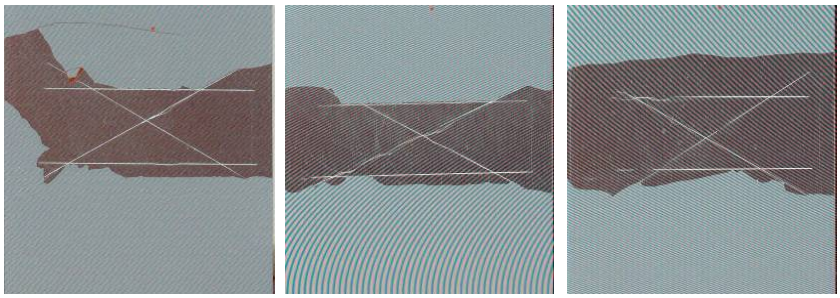


Figure 113: Test Panels after 1-Day WTA-MEH-PPV on AA7075 with TT-P-2756

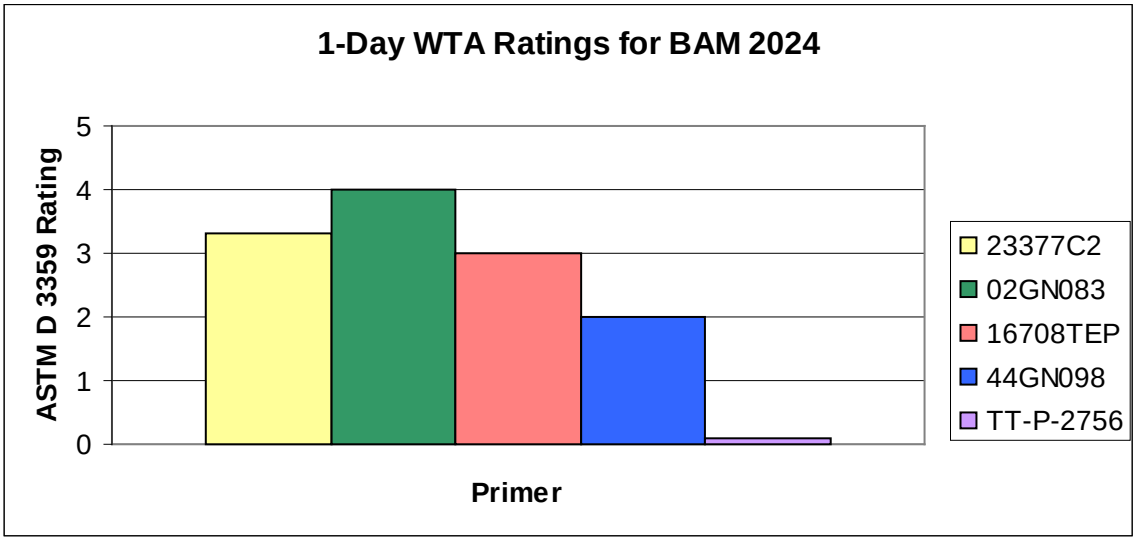


Figure 114: Average 1-Day Wet Tape Adhesion Ratings for BAM-PPV on AA2024

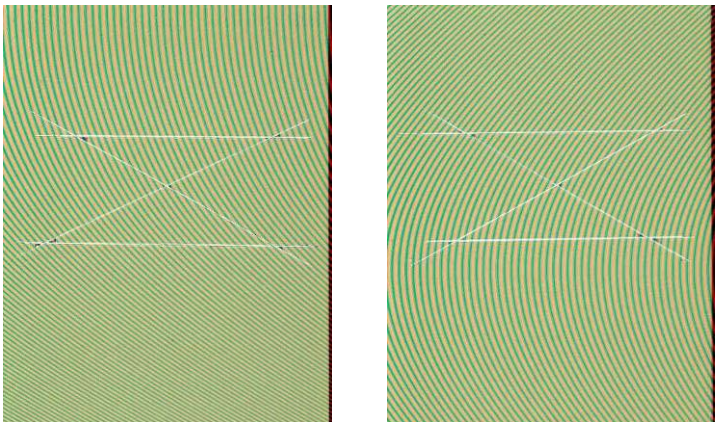


Figure 115: Test Panels after 1-Day WTA- BAM-PPV on AA2024
with MIL-PRF-23377C2

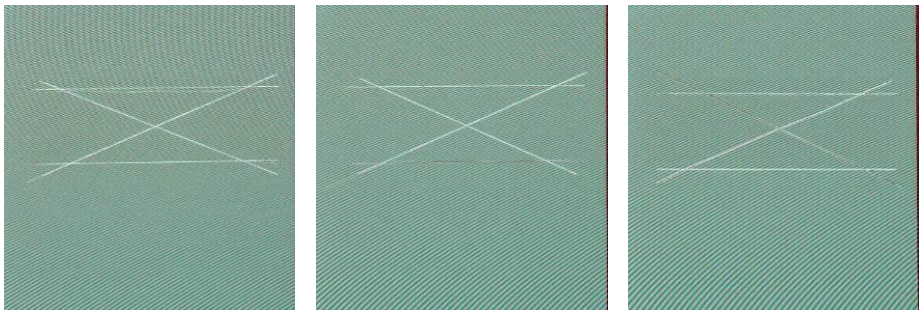


Figure 116: Test Panels after 1-Day WTA-BAM-PPV on AA2024 with 02GN083

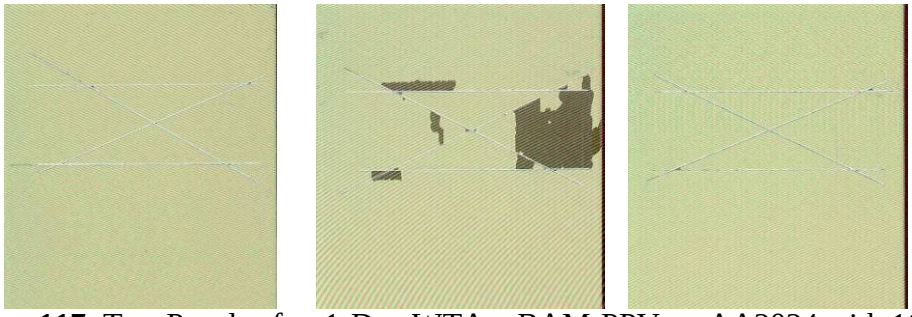


Figure 117: Test Panels after 1-Day WTA – BAM-PPV on AA2024 with 16708TEP

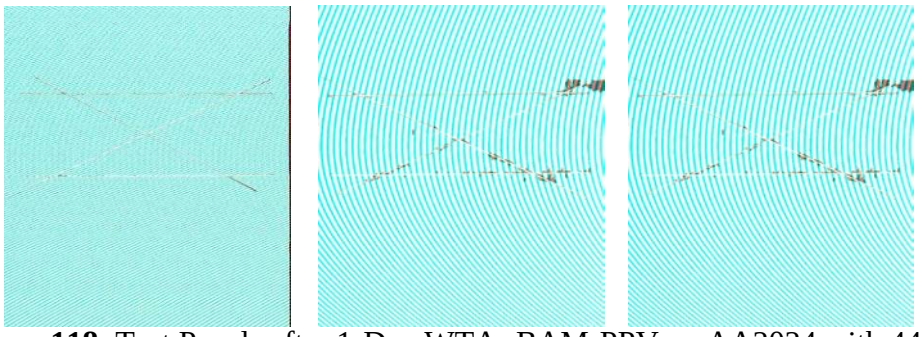


Figure 118: Test Panels after 1-Day WTA -BAM-PPV on AA2024 with 44GN098

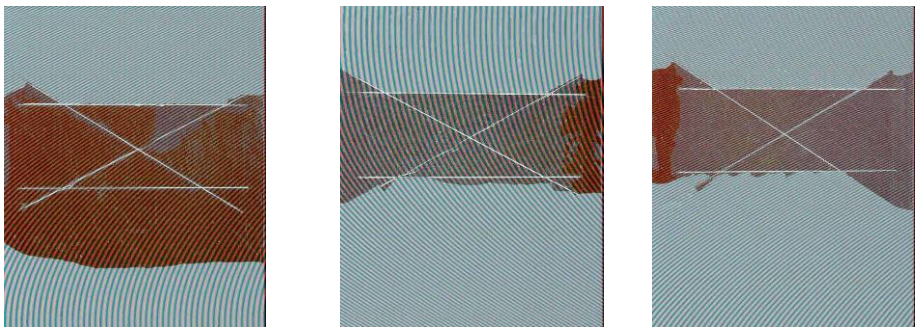


Figure 119: Test Panels after 1-Day WTA- BAM-PPV on AA2024 with TT-P-2756

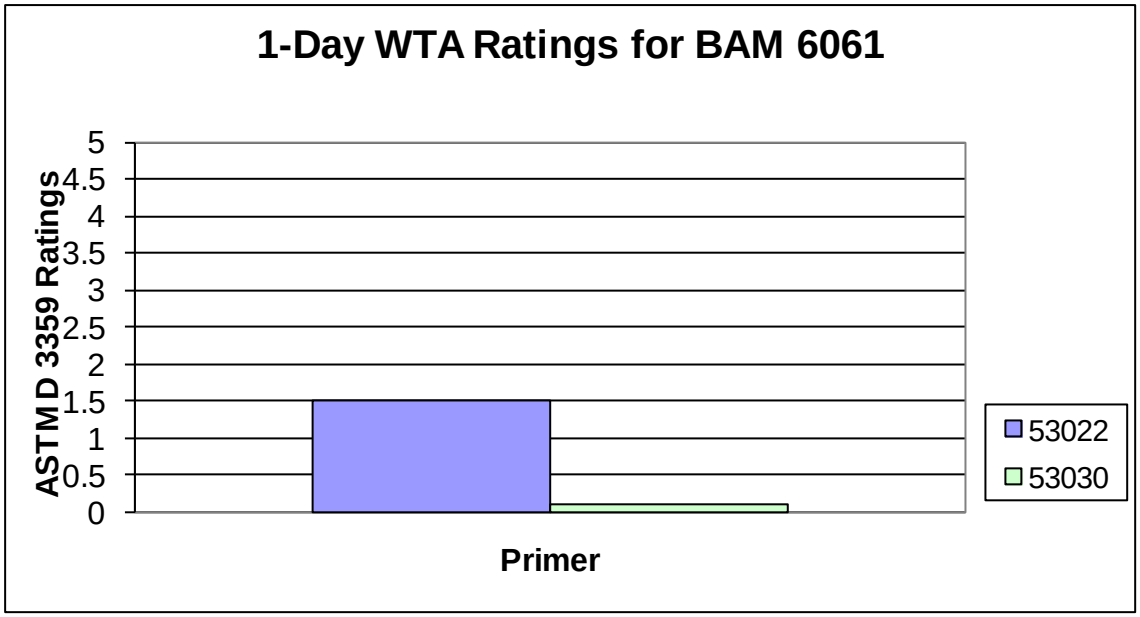


Figure 120: Average 1-Day Wet Tape Adhesion Ratings for BAM-PPV on AA6061

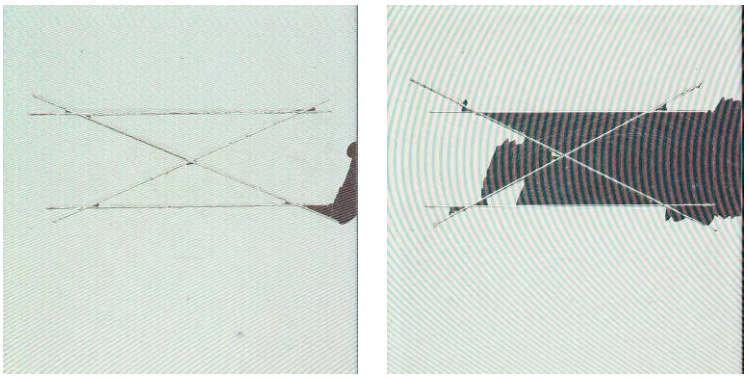


Figure 121: Test Panels after 1-Day WTA-BAM-PPV on AA6061 with MIL-DTL-53022

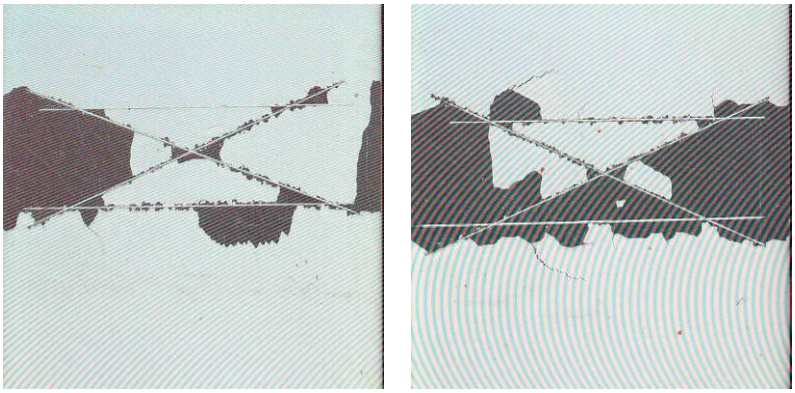


Figure 122: Test Panels after 1-Day WTA-BAM-PPV on AA6061 with MIL-DTL-53030

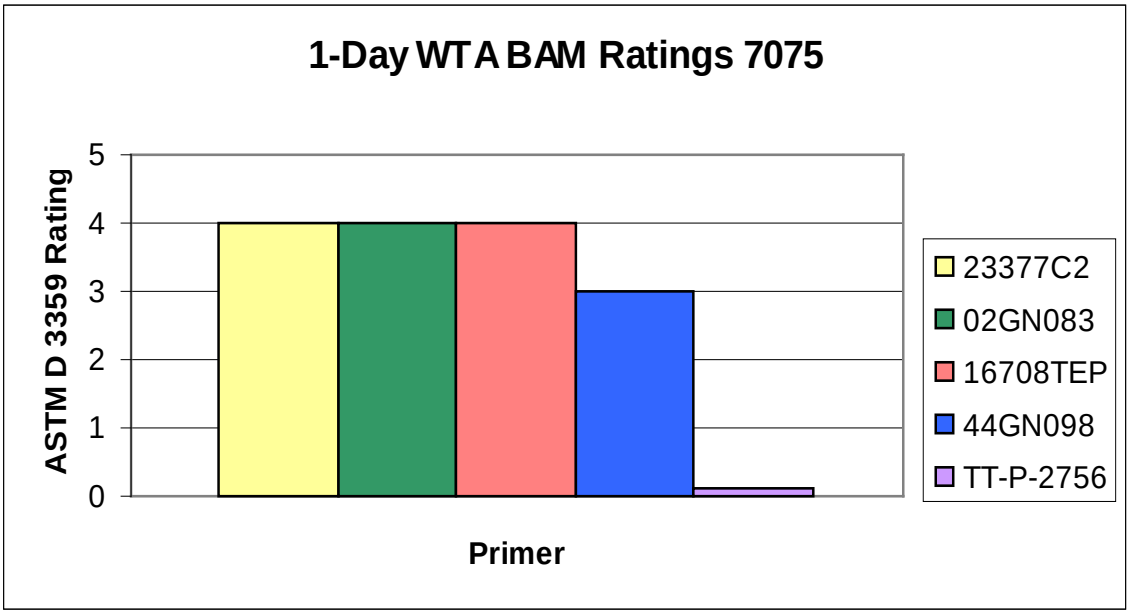


Figure 123: Average 1-Day Wet Tape Adhesion Ratings for BAM-PPV on AA7075

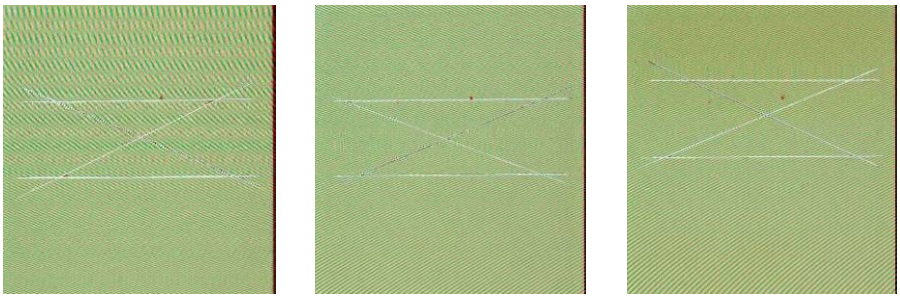


Figure 124: Test Panels after 1-Day WTA-BAM-PPV on AA7075 with MIL-PRF-23377C2

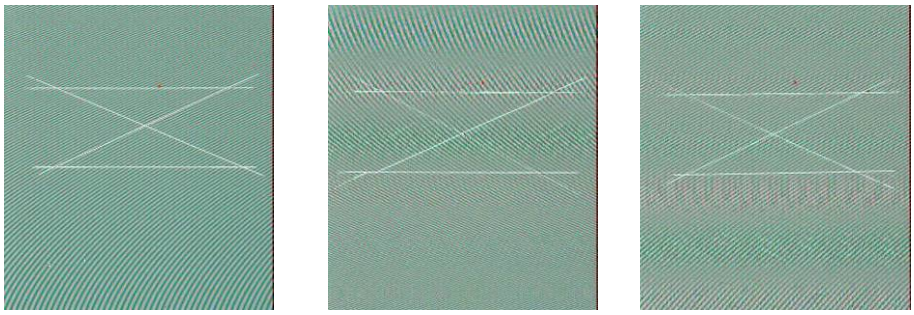


Figure 125: Test Panels after 1-Day WTA-BAM-PPV on AA7075 with 02GN083

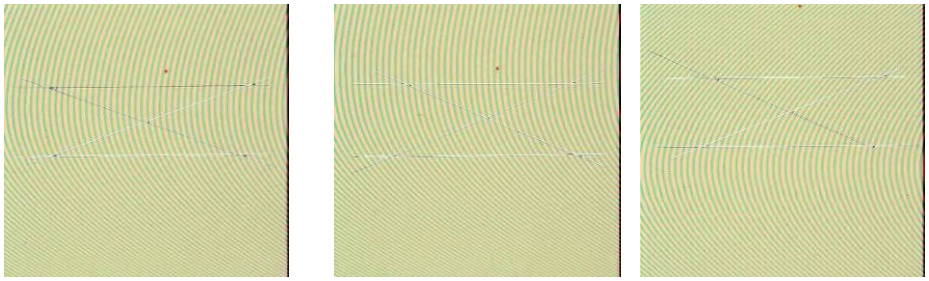


Figure 126: Test Panels after 1-Day WTA-BAM-PPV on AA7075 with 16708TEP

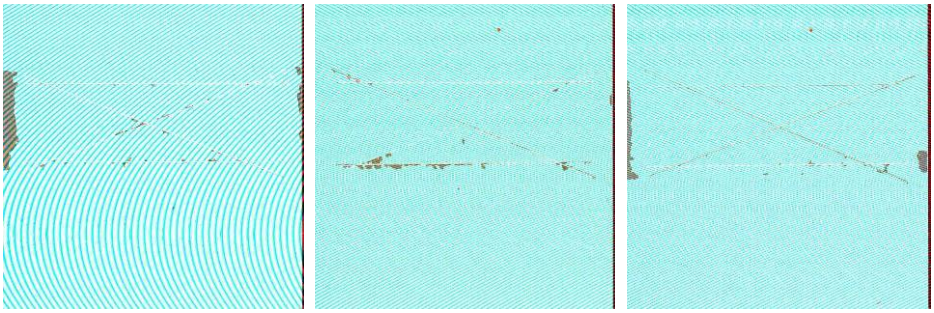


Figure 127: Test Panels after 1-Day WTA-BAM-PPV on AA7075 with 44GN098

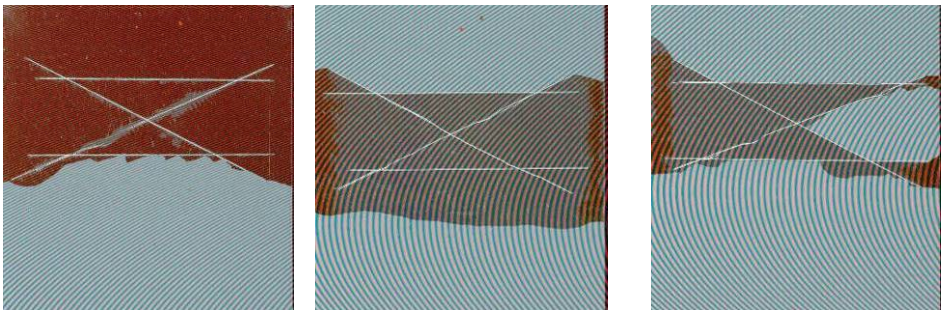


Figure 128: Test Panels after 1-Day WTA-BAM-PPV on AA7075 with TT-P-2756

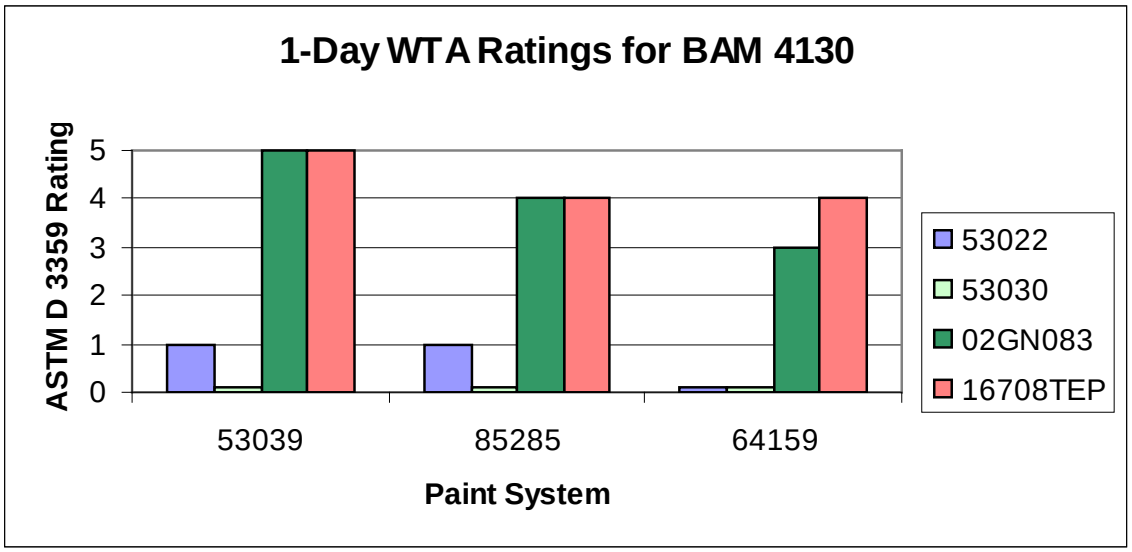
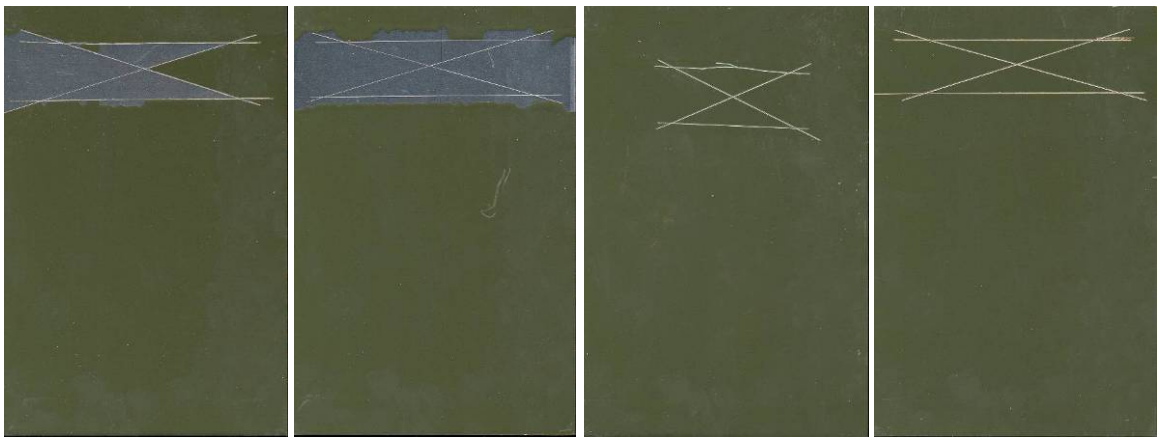


Figure 129: Average 1-Day Wet Tape Adhesion Ratings for BAM-PPV on 4130

Figures 130-137 depict the tape adhesion results by paint system. The best tape adhesion results were seen with the BAM-PPV and MEH-PPV coatings used in conjunction with the MIL-PRF-23377N primers (both 02GN083 and 16708TEP) followed by the use of the coatings with MIL-PRF-23377C2 primer. The MIL-PRF-85582N (44GN098) primer and TT-P-2756 performed the worst.



53022 53030 02GN083 16708TEP

Figure 130: Test Panels after 1-Day WTA-BAM-PPV on 4130
with MIL-PRF-53039 Topcoat

Figure 131: Test Panels after 1-Day WTA-BAM-PPV on 4130
with MIL-PRF-85285 Topcoat

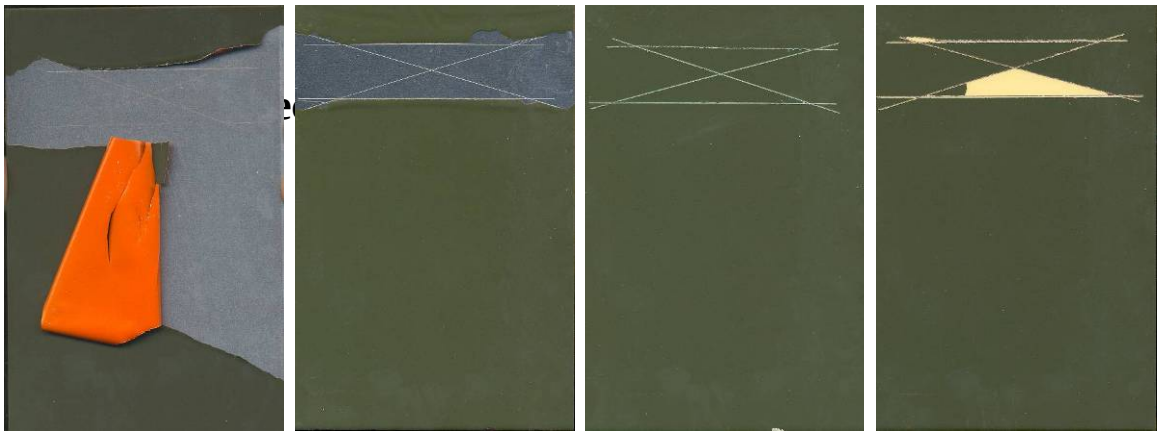


Figure 132: Test Panels after 1-Day WTA-BAM-PPV on 4130
with MIL-DTL-64159 Topcoat

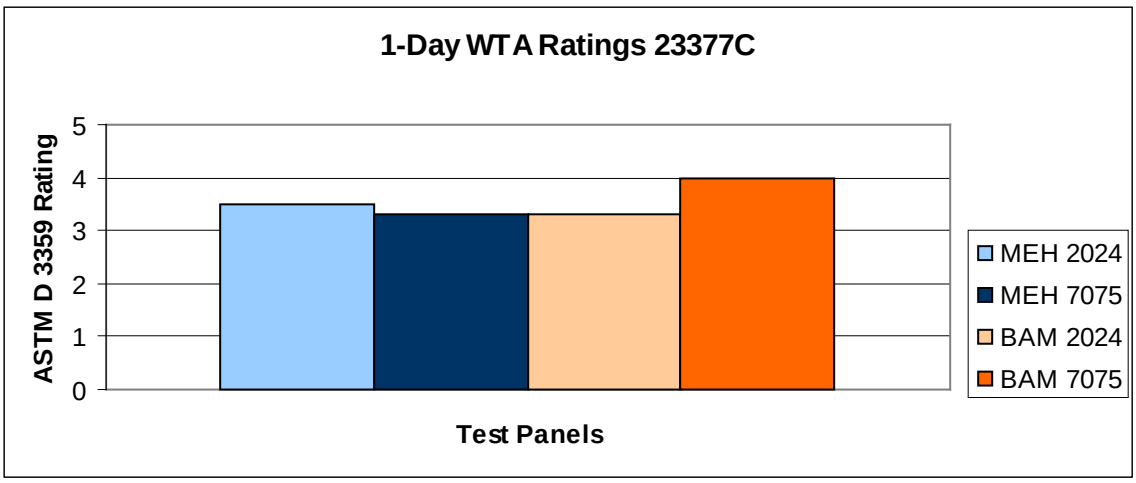


Figure 133: Average 1-Day Wet Tape Adhesion Ratings for Test Panels
with MIL-PRF-23377C2

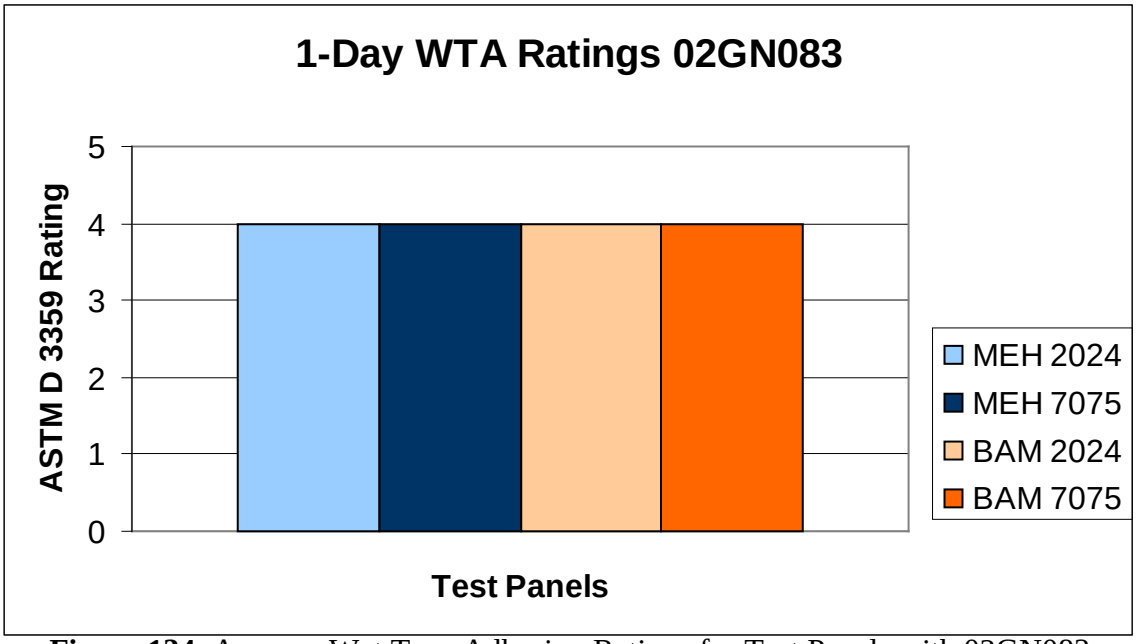


Figure 134: Average Wet Tape Adhesion Ratings for Test Panels with 02GN083

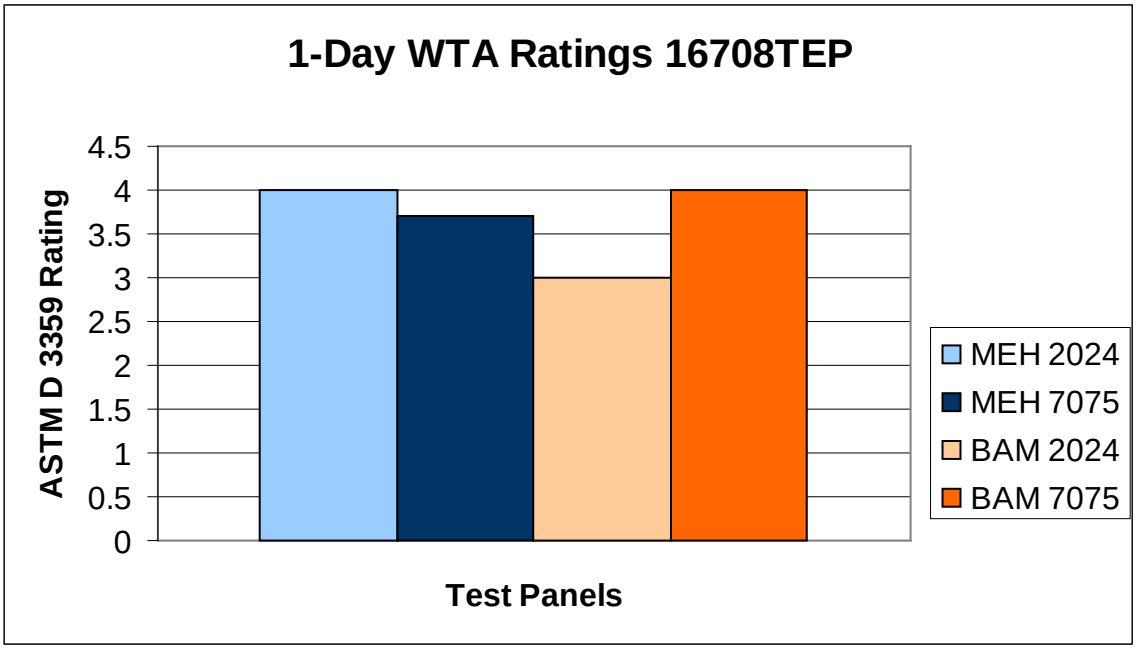


Figure 135: Average 1-Day Wet Tape Adhesion Ratings for Test Panels with 16708TEP

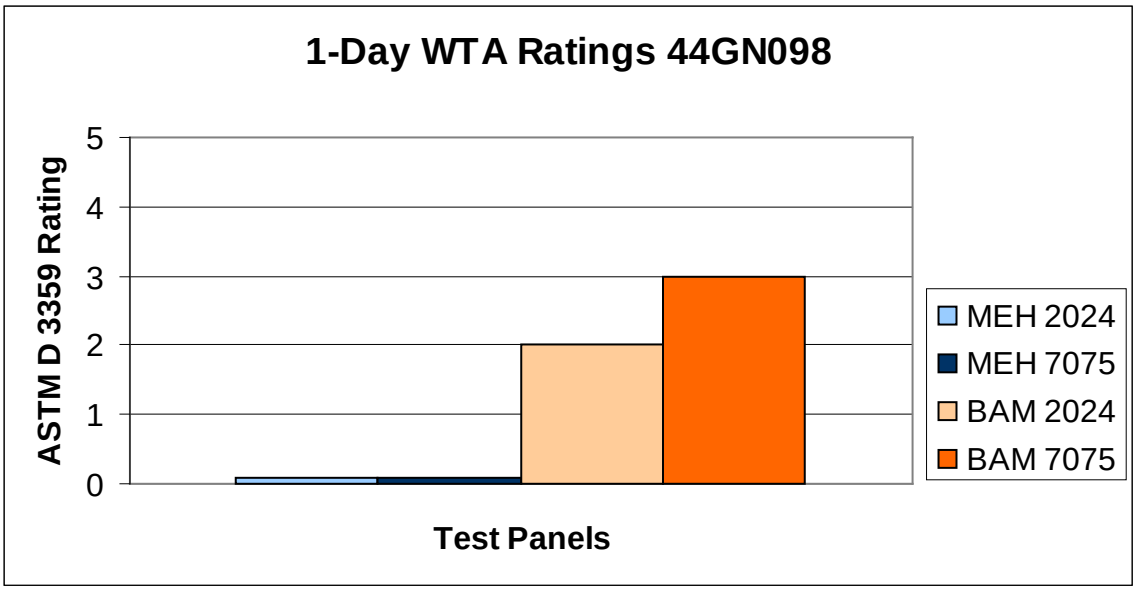


Figure 136: Average 1-Day Wet Tape Adhesion Ratings for Test Panels with 44GN098

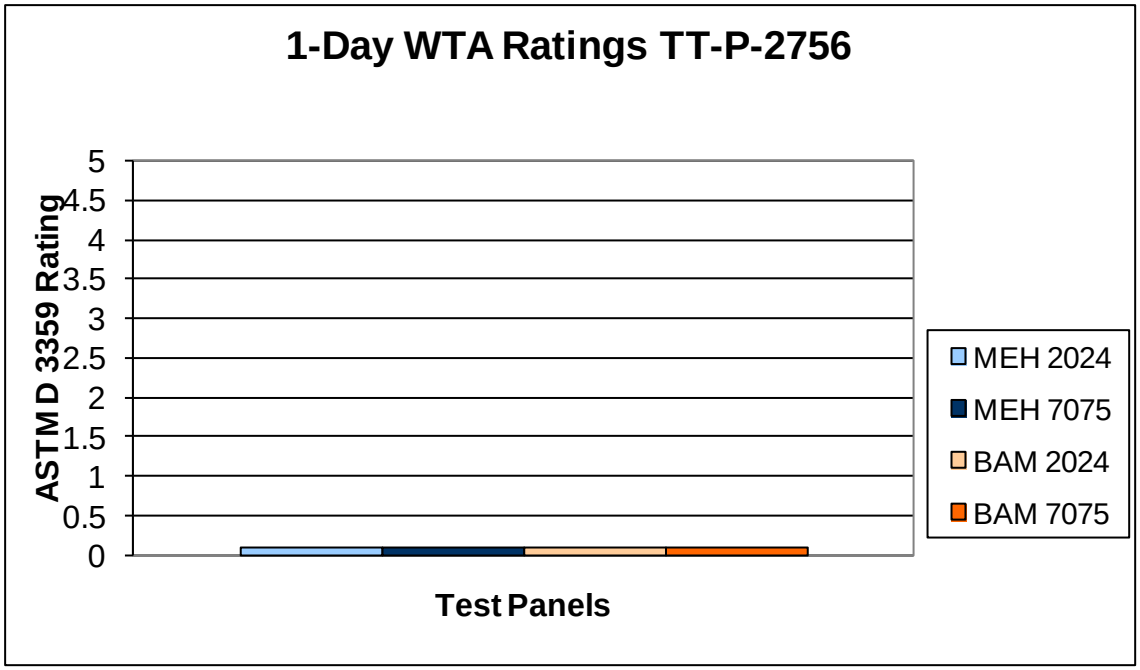


Figure 137: Average 1-Day Wet Tape Adhesion Ratings for Test Panels with TT-P-2756

The results from the WTA are summarized below in two tables. For the aluminum panels the results are summarized in Table 114 and average ratings for the steel test panels are summarized in Table 115. The tables are color coded according to pass or fail ratings (green = pass, red = fail).

Table 114: Average 1-Day Wet Tape Adhesion Results for Aluminum Test Panels with Various Primers

Primer	MEH-PPV on 2024	MEH-PPV on 7075	BAM-PPV on 2024	BAM-PPV on 7075	BAM-PPV on 6061
MIL-PRF-23377C2	3.5A	3.3A	3.3A	4A	-
MIL-PRF-23377N (02GN083)	4A	4A	4A	4A	-
MIL-PRF-23377N (16708TEP)	4A	3.7A	3A	4A	-
MIL-PRF-85582N (44GN098)	0A	0A	2A	3A	-
TT-P-2756	0A	0A	0A	0A	-
MIL-DTL-53022	-	-	-	-	1.5A
MIL-DTL-53030	-	-	-	-	0A

Table 115: 1-Day Wet Tape Adhesion Results for Steel Test Panels with Various Primers and Topcoats

Primer	53039	85285	64159
MIL-DTL-53022	1A	1A	0A
MIL-DTL-53030	0A	0A	0A
MIL-PRF-23377N (02GN083)	5A	4A	3A
MIL-PRF-23377N (16708TEP)	5A	4A	4A

6.1.3.2 Electrical Contact Resistance Testing (ECR) for BAM-PPV and MEH-PPV Pretreatment Coatings

Both BAM-PPV and MEH-PPV coated AA6061 (3”x10”) test panels were provided to NAWCAD for ECR testing. The test panels were coated on both sides. One set of BAM-PPV coated panels were subjected to a 5% salt spray test for 168 hours in accordance with ASTM B 117, except that the significant surface was inclined 6 degrees from the vertical. Test panels were rinsed with DI water and allowed to dry for 24 hours at room temperature before testing for contact electrical resistance. Ten measurements were taken on each test panel and an average panel reading was provided. When tested

in accordance with MIL-DTL-81706, the contact electrical resistance of aluminum alloy panels treated with class 3 materials under an applied electrode pressure of 200 pounds per square inch (psi) shall be not greater than 5.0 milliohms (mΩ) psi as applied and 10.0 mΩ psi after salt spray exposure. Individual readings not greater than 20 % in excess of the specified maximums shall be acceptable, provided that the average of all readings does not exceed the specified maximum resistance. ECR results for all test panels are shown in Table 116. The tables are color coded according to pass or fail ratings (green = pass, red = fail).

Table 116: ECR Results for BAM-PPV and MEH-PPV Coated Test Panels

Panel Number	MEH-PPV (mΩ)	BAM-PPV (mΩ)	BAM-PPV 168hr NSF (mΩ)
1	8.54	7.656	7.316
2	2.801	5.345	7.083
3	3.734	9.293	30.616
4	3.644	4.514	6.734
5	3.177	6.881	4.438
6	-	3.471	5.915

Figure 138 graphically depicts the ECR results for BAM-PPV and MEH-PPV coatings with no salt fog exposure (NSF). Figure 139 also depicts the ECR results for BAM-PPV coatings after salt fog exposure. Not all panels exhibited readings within the passing range as stated in MIL-DTL-81706.

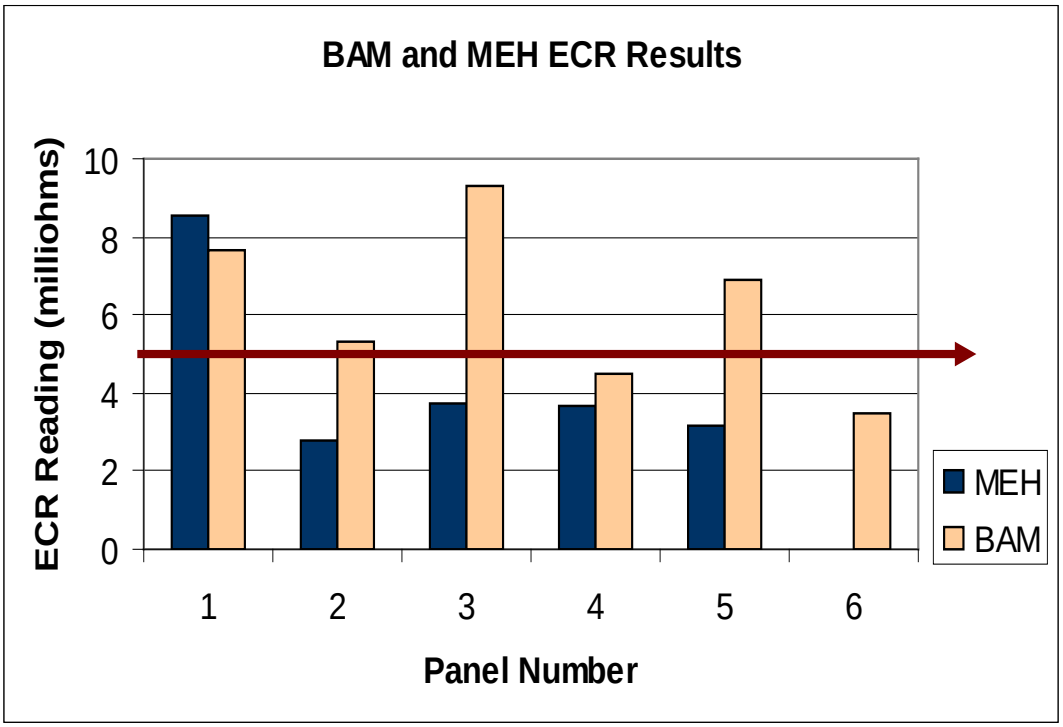


Figure 138: ECR Results for BAM-PPV and MEH-PPV Test Panels

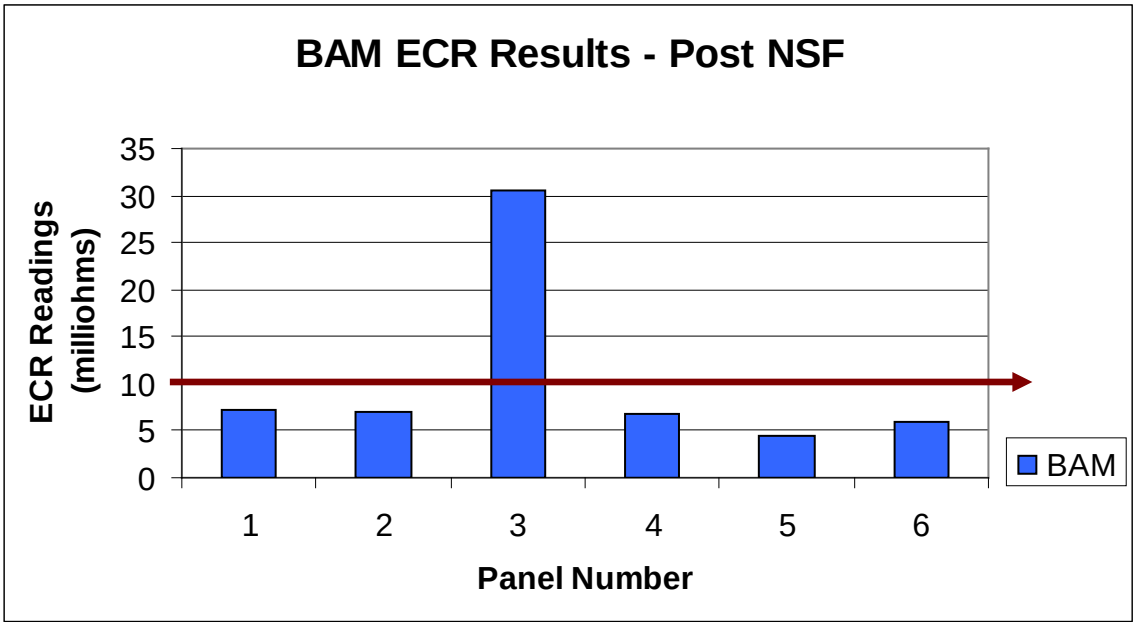


Figure 139: ECR Results for BAM-PPV Test Panels after 168hr NSF (NSS)

6.1.3.3 Accelerated Weathering Tests

The following sections (6.1.3.3.1-6.1.3.3.3) document the accelerated weathering testing of MEH-PPV pretreatment coated coupons without primer and topcoat and in full military coatings (primer + topcoat).

6.1.3.3.1 SO_2 Corrosion Testing for BAM-PPV and MEH-PPV Pretreatment Coatings

Both aluminum alloys AA2024 and AA7075 (3”x 6”) test panels were coated with BAM-PPV and MEH-PPV coatings prior to being sent to NAWCAD for SO_2 corrosion testing. **<u>All coupons</u> coated with BAM-PPV and MEH-PPV, did not meet the minimum requirement of 500 hours SO_2 exposure.**

A set of AA2024 and AA7075 (3”x5”) test panels was also sent to NAWCAD to be used as the control panels. The control panels were coated with a chromate chemical conversion coating in accordance with MIL-DTL-81706. All test panels were painted with the following primers: MIL-PRF-23377N, MIL-PRF-23377C2, MIL-PRF-85582N, MIL-PRF-85582C2, and MIL-DTL-53022. A MIL-PRF-85285 topcoat was then applied to all test panels. Table 117 shows a relative ranking of the BAM-PPV coating in conjunction with the various primers on AA2024 and AA7075. The chromate control test panels outperformed all BAM-PPV coated test panels. The BAM-PPV test panels with MIL-PRF-23377N and MIL-PRF-23377C2 as well as the AA2024 BAM-PPV test panels with MIL-PRF-85582C2 outperformed the BAM-PPV test panels with MIL-PRF-85582N and the AA7075 BAM-PPV test panels with MIL-85582C2. By far, the BAM-PPV test panels with MIL-DTL-53022 performed the worst. On these panels, the entire coating system along the scribed area lifted up and peeled away from the panel curling back on itself. In all coupons coated with BAM-PPV and MEH-PPV, they did not meet the minimum requirement of 500 hours SO_2 exposure.

Table 117: Performance Rankings for BAM-PPV AA2024 and AA7075 Panels after 500 hrs SO_2

Ranking	Alloy	Paint System
1	AA2024	MIL-PRF-23377N
2	AA2024	MIL-PRF-85582C2
3	AA7075	MIL-PRF-23377N
4	AA2024	MIL-PRF-23377C2
5	AA7075	MIL-PRF-23377C2
6	AA2024	MIL-PRF-85582N
7	AA7075	MIL-PRF-85582N
8	AA7075	MIL-PRF-85582C2
9	AA2024	MIL-DTL-53022
10	AA7075	MIL-DTL-53022

Evidence of the creepback, blistering, and “peel-away” can be seen in Figures 140-149. These figures show representative test panels from each ranking. The larger test panels shown on the left and right of each figure are coated with BAM-PPV and the smaller test panel located in the center is the Cr(VI) control test panel.

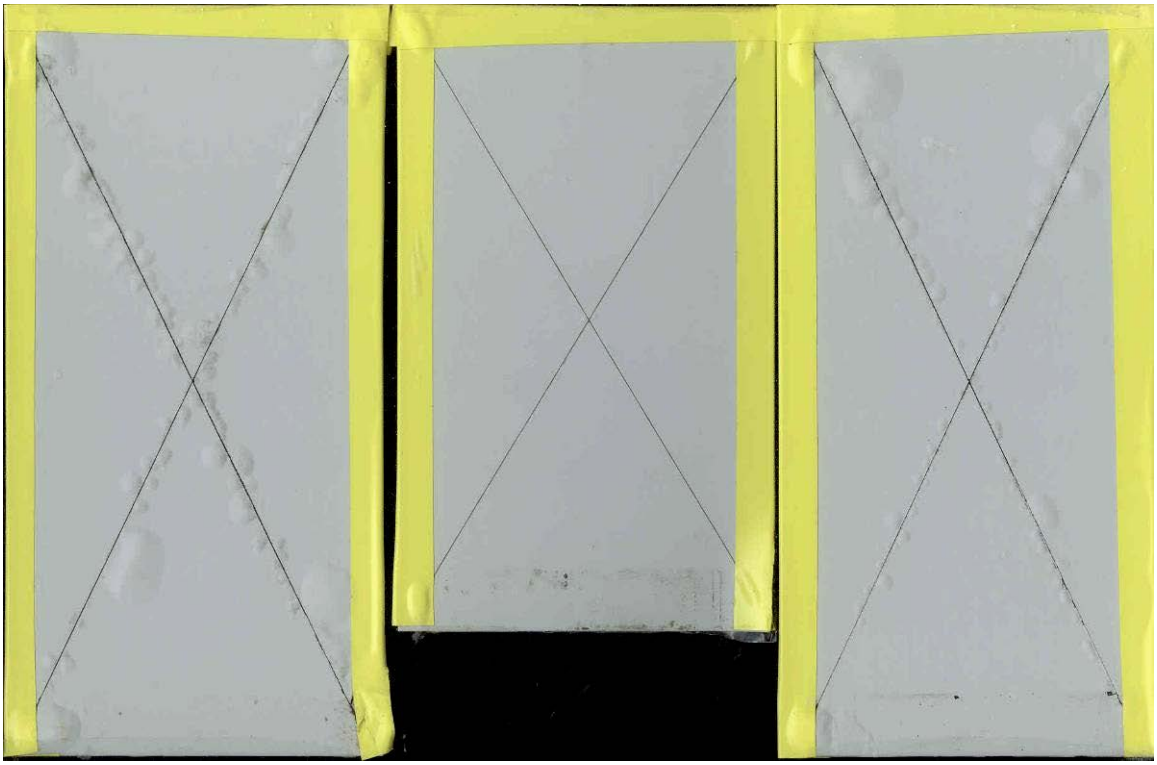


Figure 140: BAM-PPV on AA2024 with MIL-PRF-23377N after 500 hours SO_2

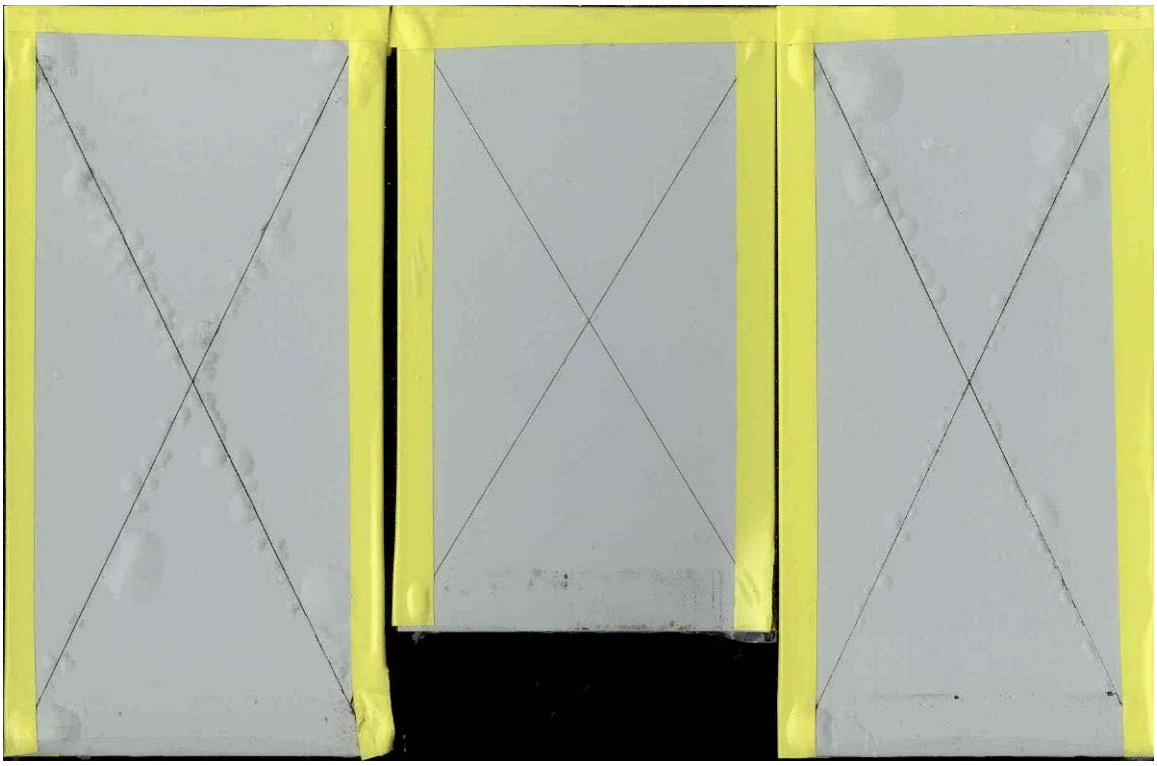


Figure 141: BAM-PPV on AA2024 with MIL-PRF-85582C2 after 500 hours SO_2

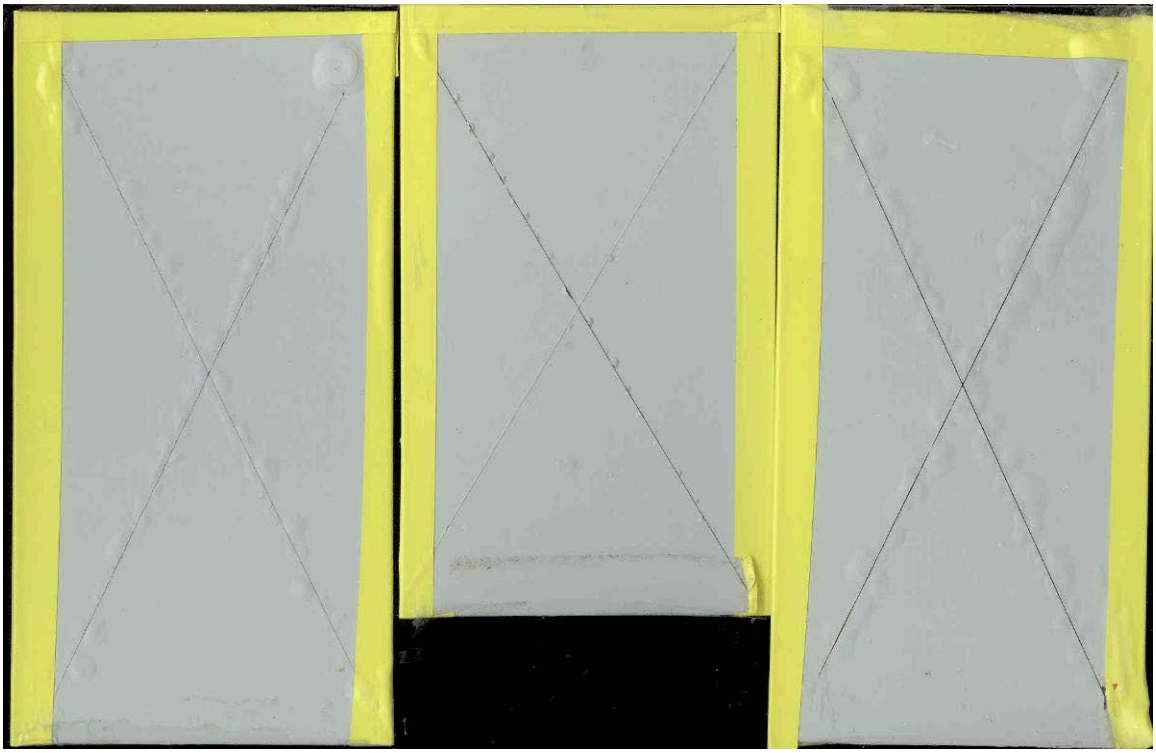


Figure 142: BAM-PPV on AA7075 with MIL-PRF-23377N after 500 hours SO_2

Figure 143: BAM-PPV on AA2024 with MIL-PRF-23377C2 after 500 hours SO_2

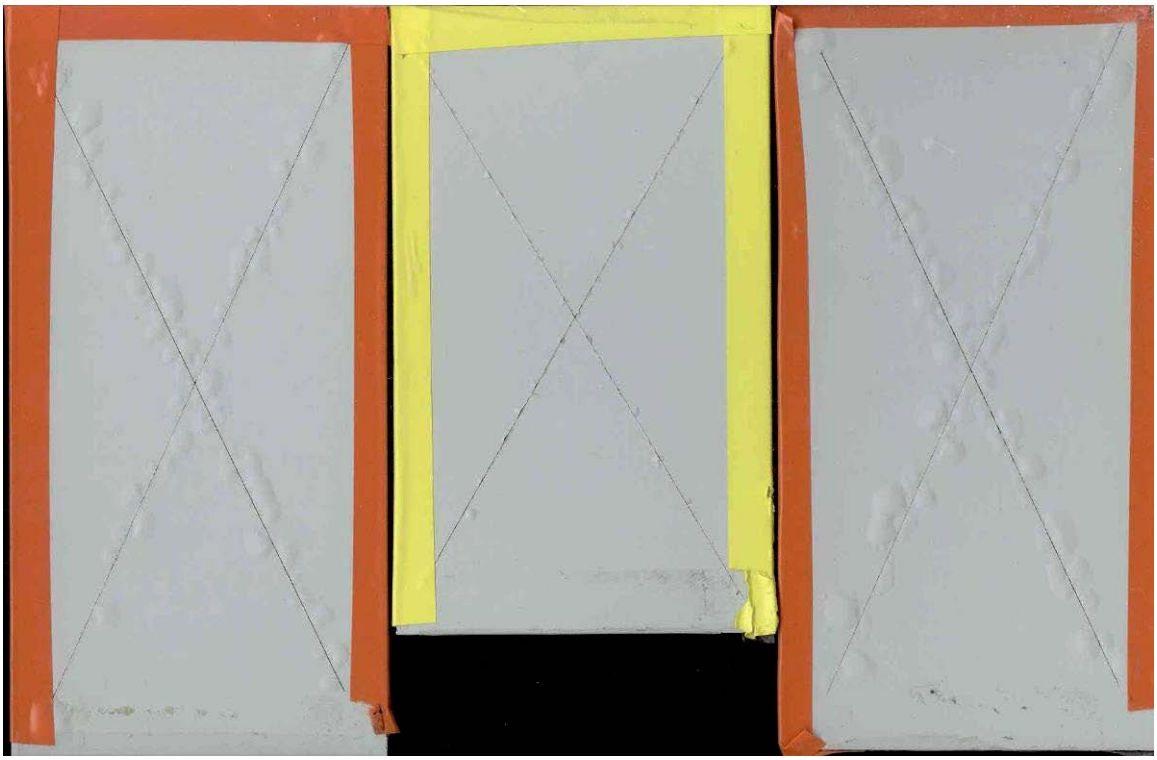


Figure 144: BAM-PPV on AA7075 with MIL-PRF-23377C2 after 500 hours SO_2

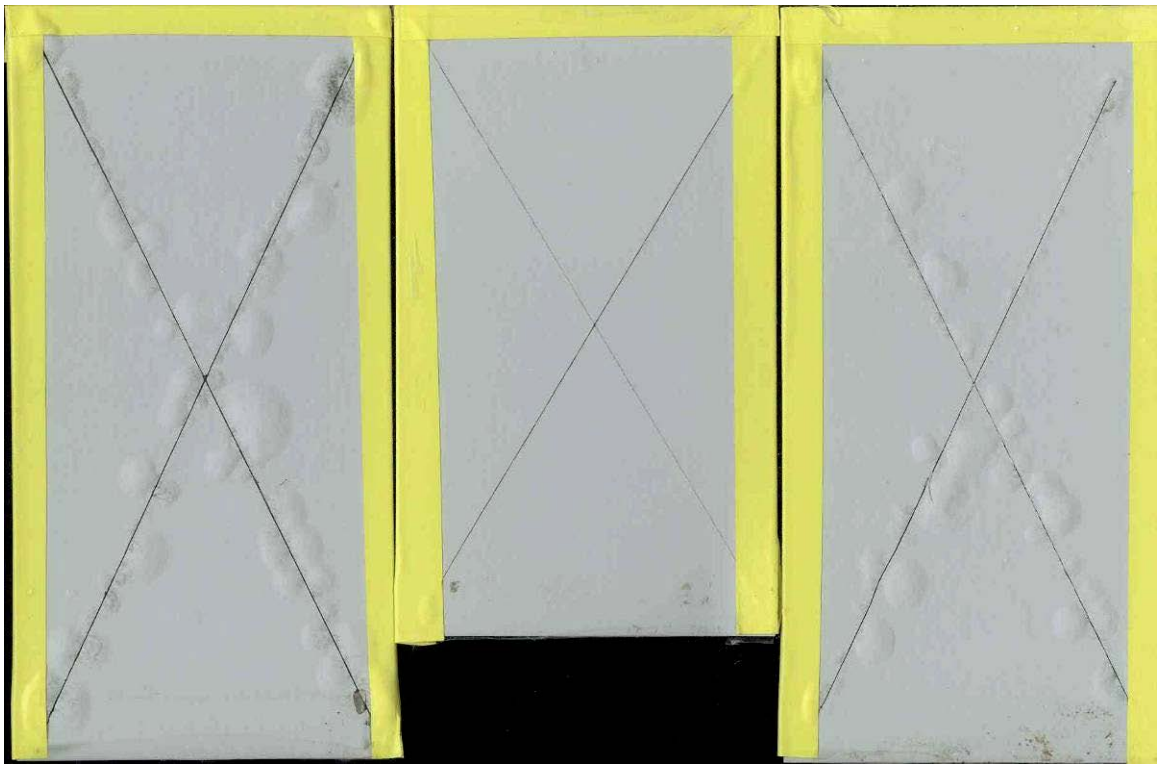


Figure 145: BAM-PPV on AA2024 with MIL-PRF-85582N after 500 hours SO_2

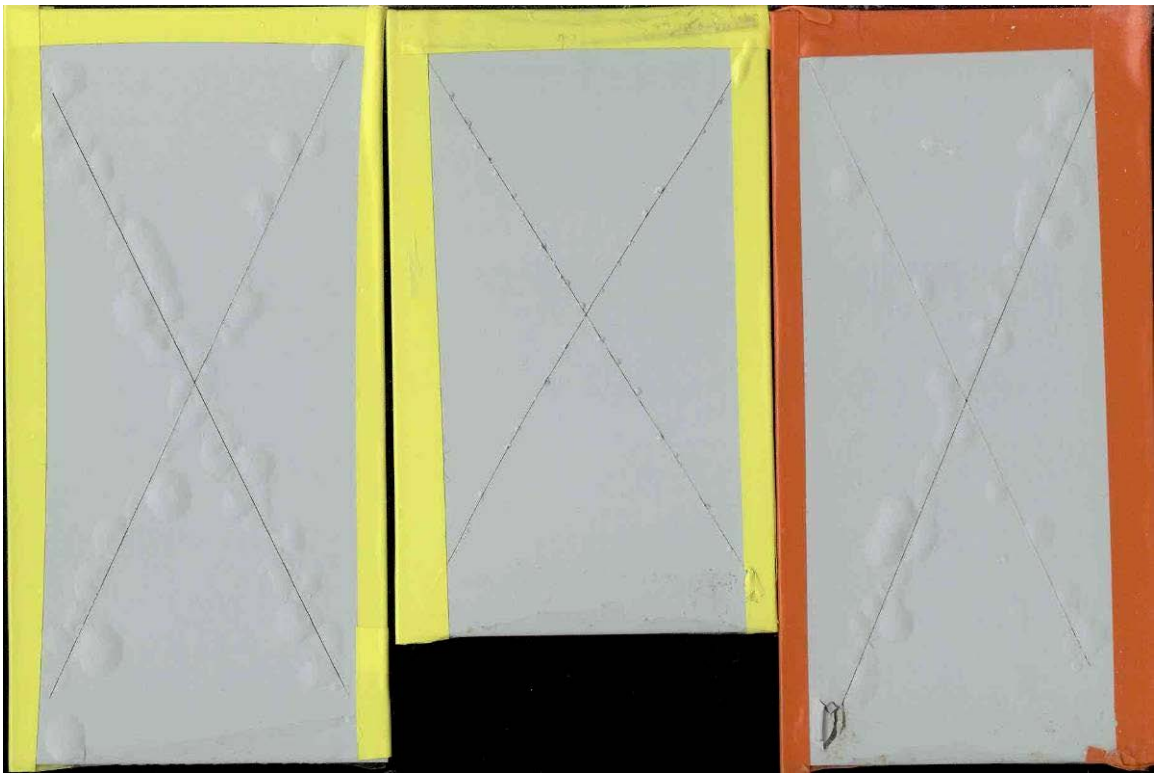


Figure 146: BAM-PPV on AA7075 with MIL-PRF-85582N after 500 hours SO_2

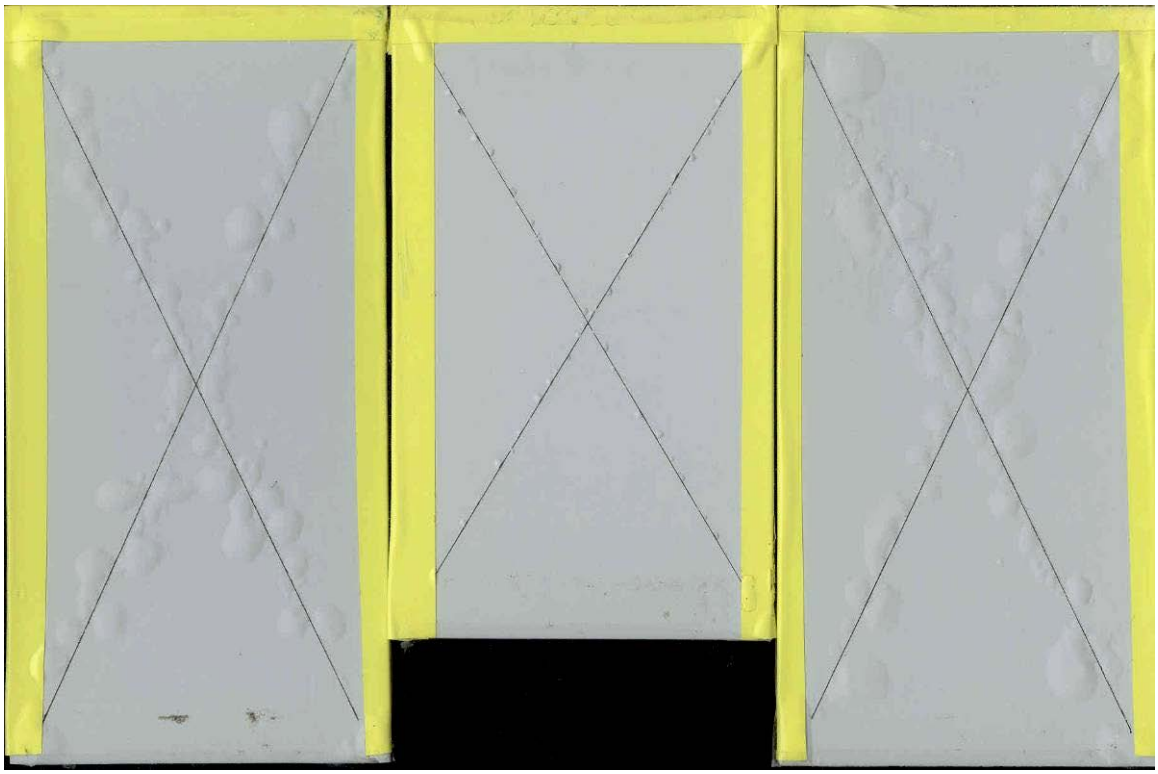


Figure 147: BAM-PPV on AA7075 with MIL-PRF-85582C2 after 500 hours SO_2

Figure 148: BAM-PPV on AA2024 with MIL-DTL-53022 after 500 hours SO_2

Figure 149: BAM-PPV on AA7075 with MIL-DTL-53022 after 500 hours SO_2

Table 118 shows a relative ranking of the MEH-PPV coating in conjunction with the various primers on AA2024 and AA7075. The Cr(VI) control test panels outperformed all MEH-PPV coated test panels. The MEH-PPV test panels with MIL-PRF-23377N and AA7075 MEH test panels with MIL-PRF-23377C2 and MIL-PRF-85582N, and MIL-PRF-85582C2 outperformed the AA2024 MEH-PPV test panels with MIL-PRF-85582N, MIL-PRF-85582C2, and MIL-PRF-23377C2 as well as the AA2024 and AA7075 test panels with MIL-DTL-53022.

Table 118: Performance Rankings for MEH-PPV AA2024 and AA7075 Panels after 500 hours SO_2

Ranking	Alloy	Paint System
1	AA7075	MIL-PRF-23377N
2	AA7075	MIL-PRF-85582N
3	AA2024	MIL-PRF-23377N
4	AA7075	MIL-PRF-85582C2
5	AA7075	MIL-PRF-23377C2
6	AA2024	MIL-PRF-23377C2
7	AA2024	MIL-PRF-85582N
8	AA2024	MIL-DTL-53022
9	AA2024	MIL-PRF-85582C2
10	AA7075	MIL-DTL-53022

The data shows evidence of the creepback, and blistering on test panels which can be seen in Figures 150-159. These figures show representative test panels from each ranking. The larger test panels shown on the left and right of each figure are coated with MEH-PPV and the smaller test panel located in the center is the Cr(VI) control test panel.

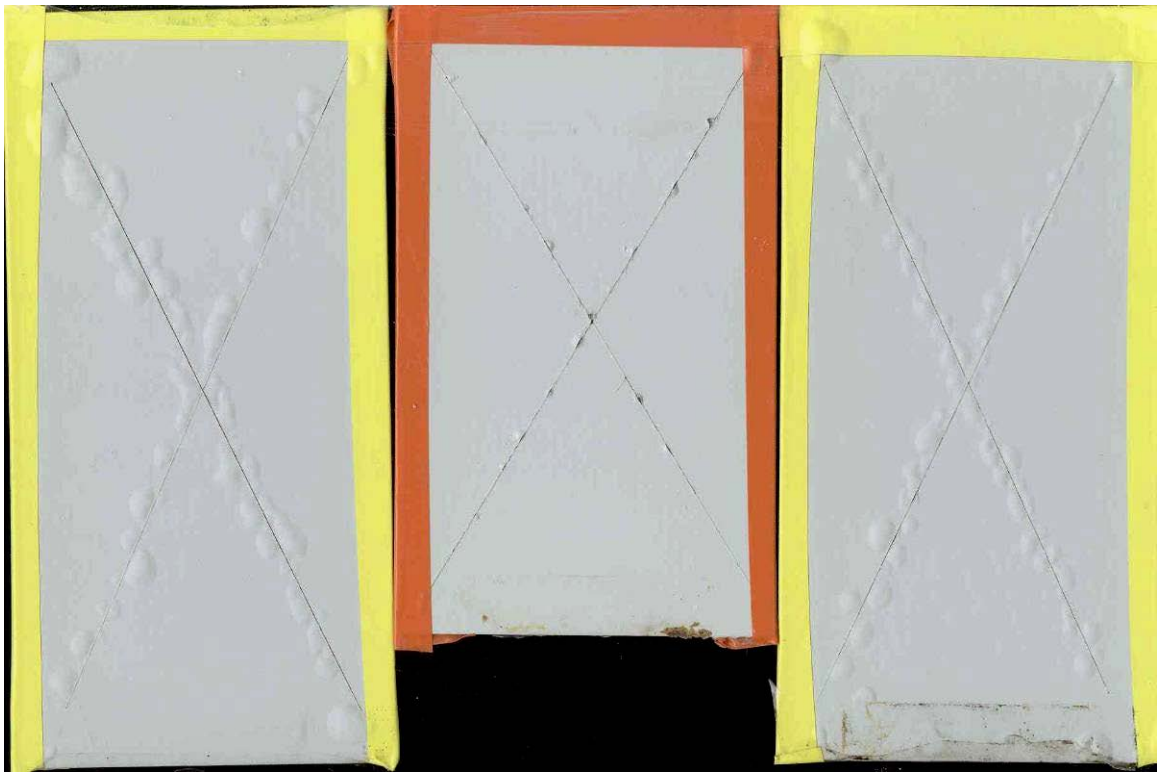


Figure 150: MEH-PPV on AA7075 with MIL-PRF-23377N after 500 hours SO_2

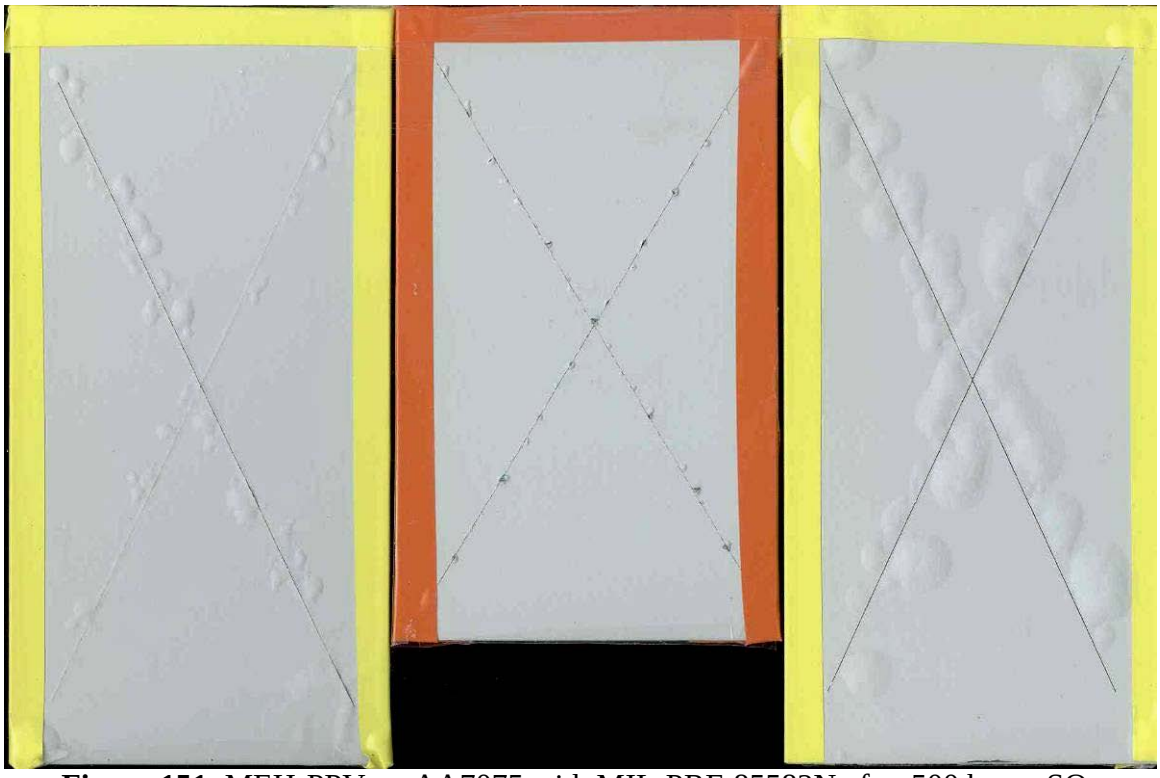


Figure 151: MEH-PPV on AA7075 with MIL-PRF-85582N after 500 hours SO_2

Figure 152: MEH-PPV on AA2024 with MIL-PRF-23377N after 500 hours SO_2

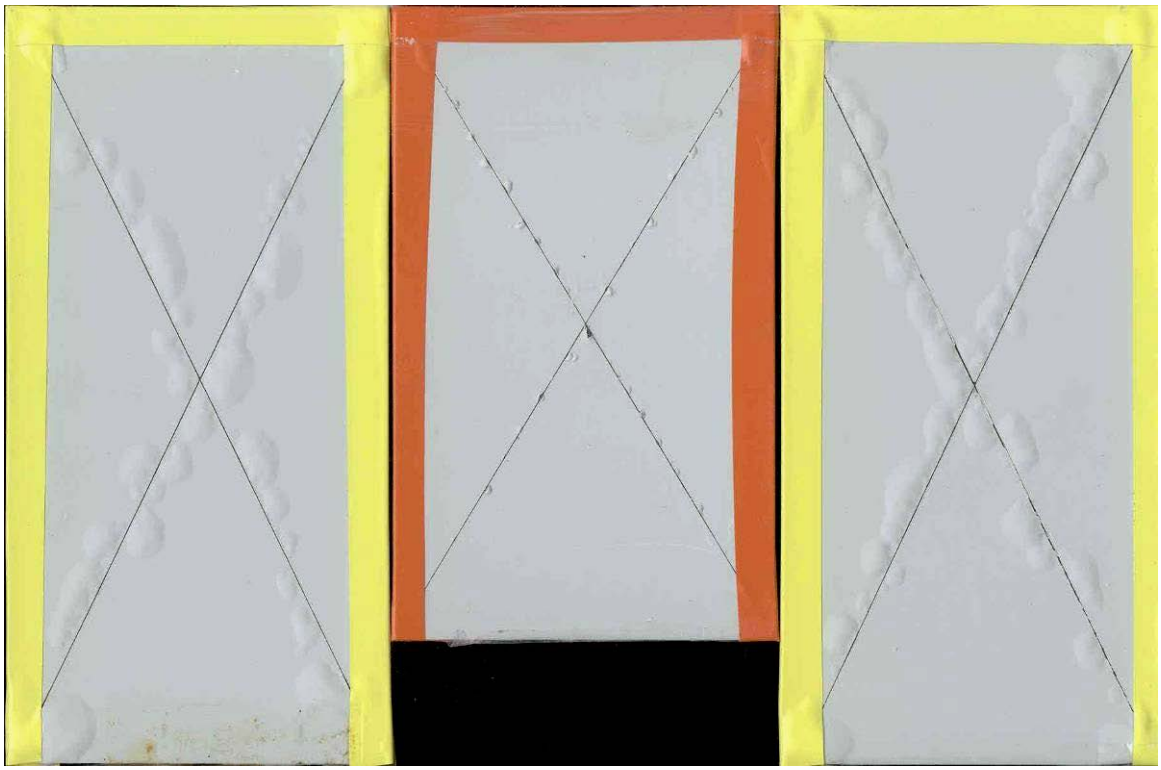


Figure 153: MEH-PPV on AA7075 with MIL-PRF-85582C2 after 500 hours SO_2

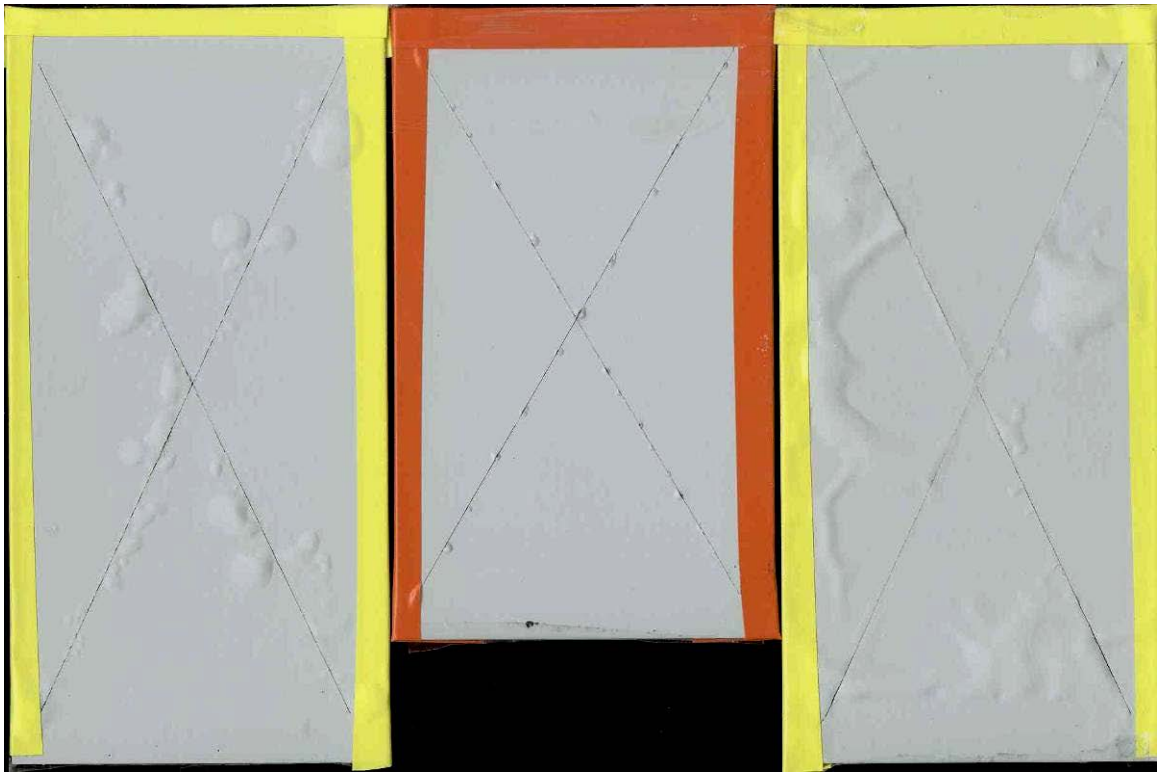


Figure 154: MEH-PPV on AA7075 with MIL-PRF-23377C2 after 500 hours SO_2

Figure 155: MEH-PPV on AA2024 with MIL-PRF-23377C2 after 500 hours SO_2

Figure 156: MEH-PPV on AA2024 with MIL-PRF-85582N after 500 hours SO_2

Figure 157: MEH-PPV on AA2024 with MIL-DTL-53022 after 500 hours SO_2

Figure 158: MEH-PPV on AA2024 with MIL-PRF-85582C2 after 500 hours SO_2

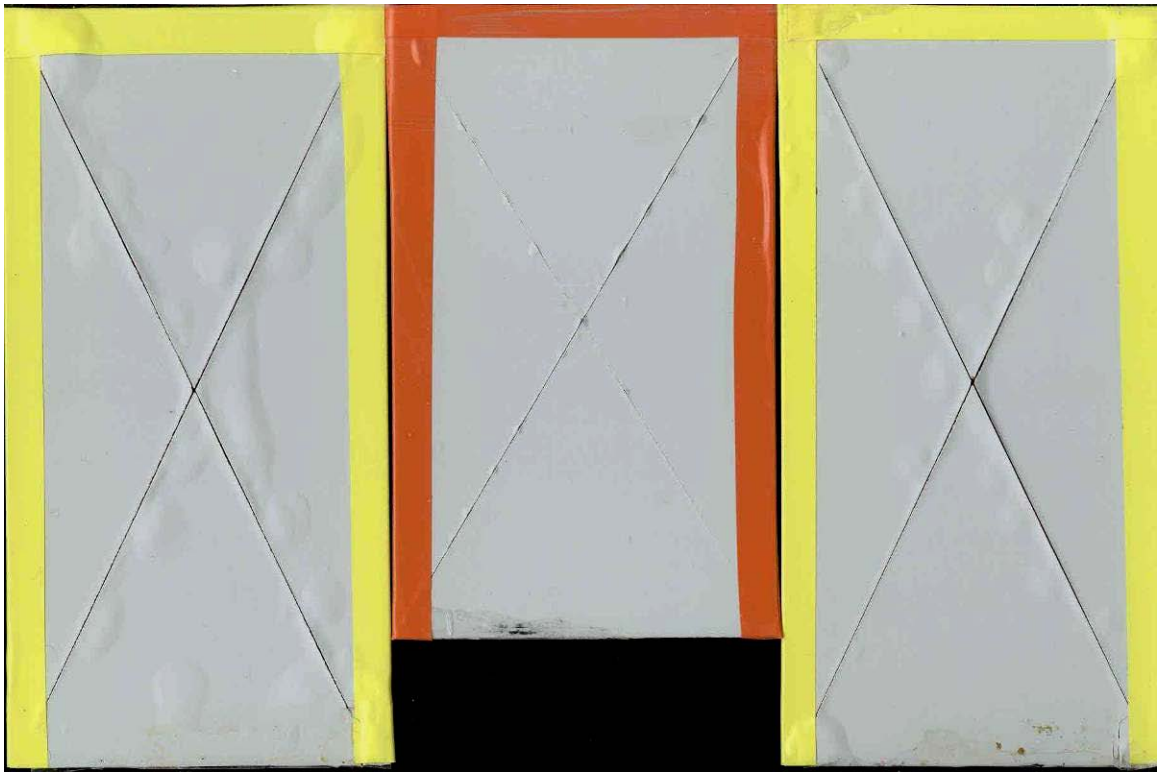


Figure 159: MEH-PPV on AA7075 with MIL-DTL-53022 after 500 hours SO_2

6.1.3.3.2 Neutral Salt Spray Exposure Testing (ASTM B 117) for BAM-PPV Pretreatment Coating

Neutral salt fog testing using ASTM B 117 was used to determine the maximum lifetime of optimized BAM-PPV. BAM-PPV was sprayed onto AA2024-T3 using *o,p*-xylenes as solvent. The BAM-PPV solution was sprayed onto the test coupons using an air brush set-up. The thickness of the BAM-PPV was 2.0μm. The BAM-PPV pretreatment coupons were placed into a neutral salt fog chamber and monitored periodically for corrosion failure and delamination. Figures 160-163 show the performance of BAM-PPV during the neutral salt fog exposure testing. BAM-PPV has shown excellent performance for the minimum military requirement of 336 hours. No blistering, delamination or corrosion was present during the 336 hours of neutral salt fog exposure testing. After 840 hours blistering and edge corrosion effects were present on these coupons.

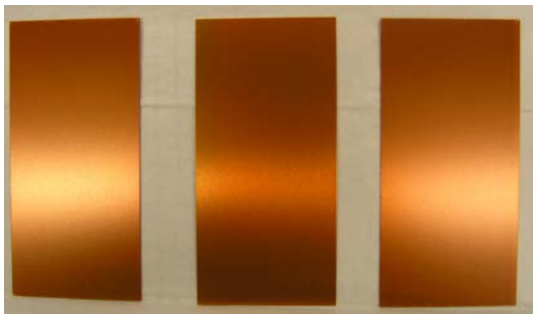


Figure 160: BAM-PPV at 0 hours

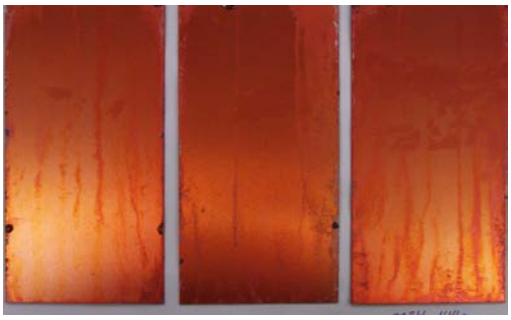


Figure 161: BAM-PPV at 336 hours

Figure 162: BAM-PPV at 840 hours

Figure 163: BAM-PPV at 1034 hours

6.1.3.3.2.1 Neutral Salt Spray Exposure Testing for BAM-PPV and MEH-PPV Pretreatments with Full Military Coatings

Neutral Salt Spray (NSS) exposure testing was performed on BAM-PPV and MEH-PPV pretreatments with chromium and non-chromium based coatings. BAM-PPV and MEH-PPV were sprayed onto AA2024-T3 and AA7075-T6 aluminum alloys from Oxsol-100 solvent. **Spraying of the BAM-PPV and MEH-PPV solutions were performed at NAWCWD and Navy Depot, Jacksonville, Florida to compare processing conditions. There was no difference in processing BAM-PPV and MEH-PPV between the solutions prepared at the NAWCWD and Navy Depot, Jacksonville, Florida. Both solutions showed similar consistency, solubility and processing features that are commonly observed at NAWCWD.** The following tables (Tables 119-121) list the results from the coatings using BAM-PPV and MEH-PPV with chromium primers. Also tested were self-priming topcoats (non-chromium) using BAM-PPV and MEH-PPV in neutral salt fog chambers. In all cases the panels were scribed and placed in a rack maintained at 15°. They were observed periodically (every 500 hours) until failure was noted (green = pass, red = failure). Both pretreatments BAM-PPV and MEH-PPV with chromated primer and topcoat passed the minimum requirement of 2000 hours neutral salt fog exposure without any evidence of corrosion or delamination. A small blister was observed for the BAM-PPV coated AA2024-T3 panels at 2448 hours. The panels were removed and testing was stopped. The pretreatments, BAM-PPV and MEH-PPV were tested with non chromium primers and topcoats. **The test results showed that both pretreatments with the new Deft non chromium primer (MIL-PRF-23377N2) did not meet performance requirements. Testing with the self-priming topcoat also showed poor performance results using both AA2024-T3 and AA7075-T6 with CCC, BAM-PPV and MEH-PPV.**

Table 119: Neutral Salt Fog Results for BAM-PPV and MEH-PPV Pretreatments with Chromium Primers and Topcoat

Alloy	Pretreatment	Primer	Topcoat	Hours	Rating
AA2024-T3	MIL-C-5541	MIL-PRF-23377C	MIL-PRF-85285	2448	Pass
AA2024-T3	BAM-PPV	MIL-PRF-23377C	MIL-PRF-85285	2448	Pass
AA2024-T3	MEH-PPV	MIL-PRF-23377C	MIL-PRF-85285	2448	Pass
AA7075-T6	MIL-C-5541	MIL-PRF-23377C	MIL-PRF-85285	1500	Fail
AA7075-T6	BAM-PPV	MIL-PRF-23377C	MIL-PRF-85285	2448	Pass
AA7075-T6	MEH-PPV	MIL-PRF-23377C	MIL-PRF-85285	2448	Pass

Table 120: Neutral Salt Fog Results for BAM-PPV and MEH-PPV Pretreatments with Non-Chromium Primer and Topcoat

Alloy	Pretreatment	Primer	Topcoat	Hours	Rating
AA2024-T3	MIL-C-5541	MIL-PRF-23377N2	MIL-PRF-85285	2448	Pass
AA2024-T3	BAM-PPV	MIL-PRF-23377N2	MIL-PRF-85285	1500	Fail
AA2024-T3	MEH-PPV	MIL-PRF-23377N2	MIL-PRF-85285	1500	Fail
AA7075-T6	MIL-C-5541	MIL-PRF-23377N2	MIL-PRF-85285	1500	Fail
AA7075-T6	BAM-PPV	MIL-PRF-23377N2	MIl-PRF-85285	1500	Fail
AA7075-T6	MEH-PPV	MIL-PRF-23377N2	MIL-PRF-85285	1500	Fail

Table 121: Neutral Salt Fog Results for BAM-PPV and MEH-PPV Pretreatments with Non-Chromium Self-Priming Topcoat

Alloy	Pretreatment	Self-Priming Topcoat	Hours	Rating
AA2024-T3	MIL-C-5541	TT-P-2756	2448	Pass
AA2024-T3	BAM-PPV	TT-P-2756	1000	Fail
AA2024-T3	MEH-PPV	TT-P-2756	1000	Fail
AA7075-T6	MIL-C-5541	TT-P-2756	1500	Fail
AA7075-T6	BAM-PPV	TT-P-2756	1000	Fail
AA7075-T6	MEH-PPV	TT-P-2756	1500	Fail

6.1.3.3.3 Filiform Testing of BAM-PPV and MEH-PPV Pretreatments

Filiform testing of BAM-PPV and MEH-PPV pretreatments on AA2024-T3 and AA6061-T6 was measured using primers MIL-DTL-53030, MIL-DTL-53022 and MIL-PRF-23377C. The topcoat used for all panels was MIL-PRF-85285. The tests were performed in accordance with ASTM D2803. The results are summarized in Table 122. **The results showed that both BAM-PV and MEH-PPV performed poorly with all non-chromium primers but acceptable performance with chromated primer.**

Table 122: Filiform Corrosion Results for BAM-PPV and MEH-PPV Pretreatments

Alloy	Pretreatment	Primer	Pass/Fail	MJ or MN*
AA2024-T3	CCC	MIL-PRF-23377C	Pass	MN
AA2024-T3	BAM-PPV	MIL-PRF-23377C	Pass	MN
AA2024-T3	BAM-PPV	MIL-DTL-53030	Fail	MJ
AA2024-T3	BAM-PPV	MIL-DTL-53022	Fail	MJ
AA2024-T3	MEH-PPV	MIL-PRF-23377C	Pass	MN
AA2024-T3	MEH-PPV	MIL-DTL-53030	Fail	MJ
AA2024-T3	MEH-PPV	MIL-DTL-53022	Fail	MJ
AA6061-T6	CCC	MIL-PRF-23377C	Pass	MN
AA6061-T6	BAM-PPV	MIL-DTL-53030	Fail	MJ
AA6061-T6	BAM-PPV	MIL-DTL-53022	Fail	MJ
AA6061-T6	MEH-PPV	MIL-PRF-23377C	Pass	MN
AA6061-T6	MEH-PPV	MIL-DTL-53030	Fail	MJ
AA6061-T6	MEH-PPV	MIL-DTL-53022	Fail	MJ

*Key (also see page 46 for full details)
MJ = majority of filiform in the scribe
MN= minority of filiform in the scribe

6.1.3.4 NAWCAD/NAWCAD Conclusions for BAM-PPV and MEH-PPV Laboratory Studies

The results from the extensive laboratory testing performed on BAM-PPV and MEH-PPV pretreatment coatings by NAWCAD/NAWCWD are summarized below:

- ***BAM-PPV pretreatment coating shows overall better adhesion performance than MEH-PPV,***
- ***ECR measurements for both BAM-PPV and MEH-PPV show similar results,***
- ***BAM-PPV passed ≥ 336 hours neutral salt spray exposure testing,***
- ***SO_2 salt spray exposure resulted in both BAM-PPV and MEH-PPV coatings failing at 500 hours which did not meet the minimum requirement for passing,***
- ***Filiform testing resulted in both BAM-PPV and MEH-PPV coatings failing,***
- ***Only BAM-PPV and MEH-PPV with chromate primer + topcoat passed filiform testing,***
- ***BAM-PPV provided marginal performance improvement over MEH-PPV and was therefore used as the pretreatment coating for field studies on non-critical military hardware.***

6.2 Outdoor Exposure Testing

The best performing system using BAM-PPV pretreatment coating with primer and topcoat was down-selected from laboratory testing. This full military coating was tested in outdoor weathering tests to determine its potential survival in field testing.

6.2.1 Outdoor Exposure Testing Performed at WPAFB

The following section (6.2.1.1) documents the results of the outdoor exposure testing on BAM-PPV pretreatment in a full military coating system with summation found in section 6.2.1.1.1.

6.2.1.1 Outdoor Exposure (ASTM D 1014) for BAM-PPV Coated Coupons

Because accelerated weathering testing can only give an estimate as to the corrosion resistance, degradation, and adhesion characteristics of a coating system, outdoor weathering was included in a battery of exposure tests to determine the effect natural weather patterns and environmental exposure did have on a coating system. These data along with accelerated corrosion and weathering data provided more detail to the anticipated performance of the electroactive polymer coating system prior to field testing. This test was performed on topcoated systems for coating systems with Alodine 1200S, PreKote, and BAM-PPV pretreatments on AA2024-T3 substrates only. MEH-

PPV was not tested since the material was not available at the start of the test 2 years previously. ASTM D 1014 was used for guidance to run the test and to evaluate the samples. Samples were scribed with an X-scribe (full size) prior to exposure. To prevent loss of the panel number during exposure, a label was applied to the backs of each sample. There were three replicates per coating system. Data is given in Table 123. The rating system was that used in salt spray exposure for scribe corrosion. Undercutting and blistering was not noted or reported. **One system demonstrated delamination at the end of the test but no surface corrosion was noted. This system was BAM-PPV with nonchromated primer Deft 02-GN-084 and polyurethane topcoat Deft ELT 99-GY-001. The absence of corrosion on the sample surface while showing insufficient adhesion during this test was unexplainable and is considered an anomaly**. All remaining systems performed satisfactorily and, those ratings are highlighted in **blue**. The control system is highlighted in **green**. Samples were photographed after 24 months of exposure, and those are given in Figures 164 and 165.

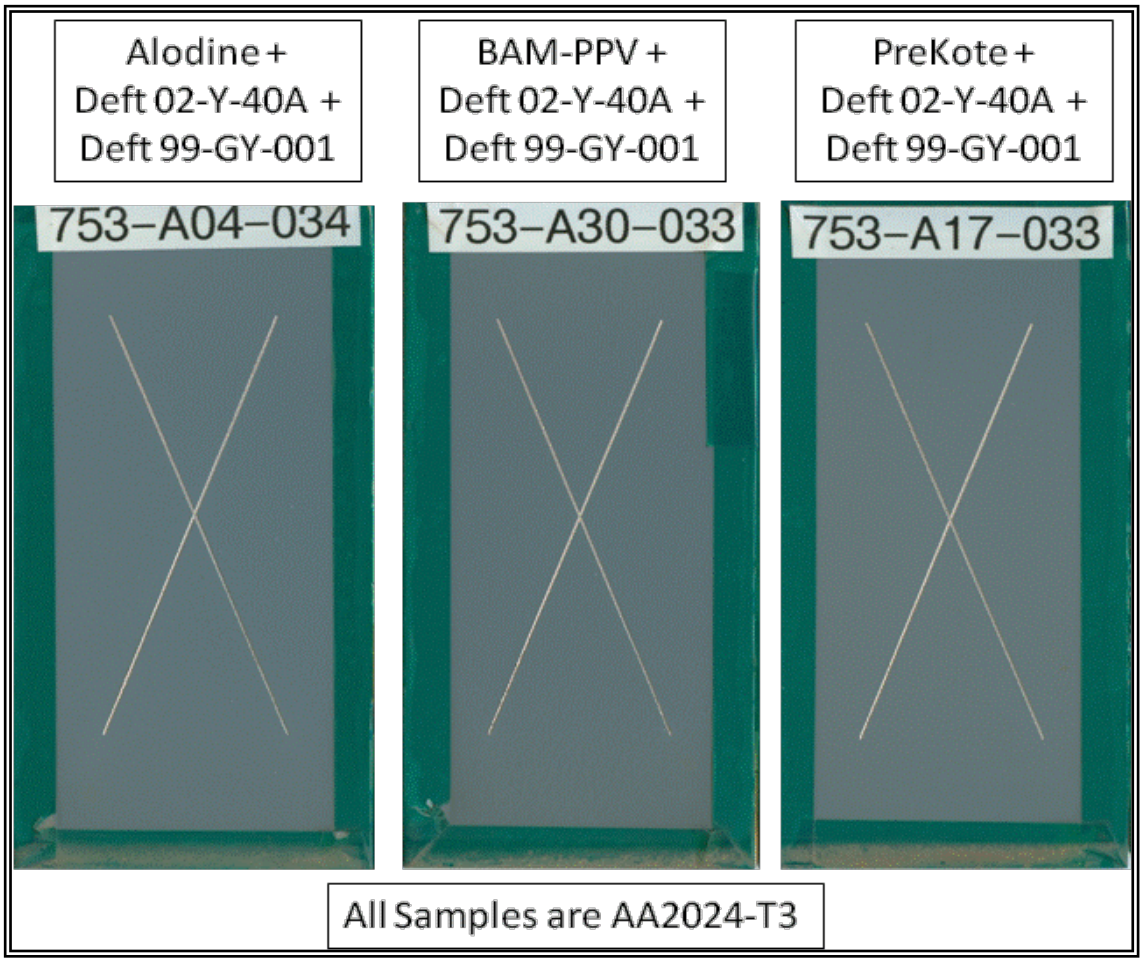


Figure 164: Outdoor Weathering after 24 months, Pretreatments with MIL-PRF-23377 Chromated Epoxy Primer and MIL-PRF-85285 Topcoat

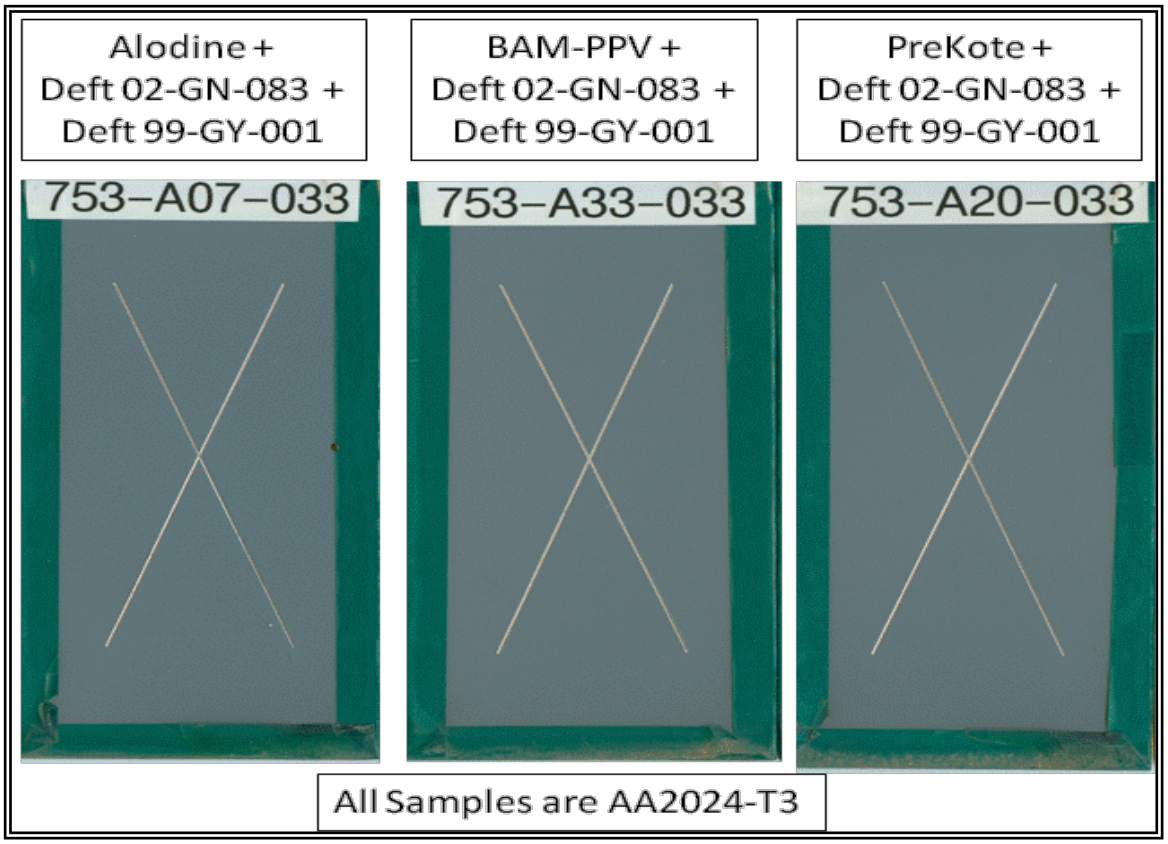


Figure 165: Outdoor Weathering after 24 months, Pretreatments with MIL-PRF-23377 Nonchromated Epoxy Primer and MIL-PRF-85285 Topcoat

Table 123: Outdoor Weathering Ratings (24 months)
(MIL-PRF-85285 Topcoated Systems) at WPAFB

Coating System	Rating
Pretreatment + Primer + Topcoat	**AA2024-T3 Data**
Alodine 1200S + Deft 02-Y-40A chromated epoxy + Deft 99-GY-001 polyurethane (ELT)	**control 0**
Alodine 1200S + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (ELT)	**0**
Alodine 1200S + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (ELT)	**0**
BAM-PPV + Deft 02-Y-40A chromated epoxy + Deft 99-GY-001 polyurethane (ELT)	**0**
BAM-PPV + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (ELT)	**0**
BAM-PPV + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (ELT)	**delamination**
PreKote + Deft 02-Y-40A chromated epoxy + Deft 99-GY-001 polyurethane (ELT)	**0**
PreKote + Deft 02-GN-083 nonchromated epoxy + Deft 99-GY-001 polyurethane (ELT)	**0**
PreKote + Deft 02-GN-084 nonchromated epoxy + Deft 99-GY-001 polyurethane (ELT)	**0**

6.2.2.1.1 Conclusions for BAM-PPV Outdoor Exposure Testing

- ***BAM-PPV pretreatment coating in both non-chromate and chromate primer with topcoat showed equal performance after 24 months outdoor exposure testing,***

- ***Therefore, BAM-PPV was selected for field testing on non-critical military hardware due to its outstanding outdoor exposure performance.***

6.2.2 Marine Outdoor Exposure Testing

The following sections document the marine outdoor exposure testing at the KSC. These tests were performed in order to examine the robustness of BAM-PPV and MEH-PPV survival to harsh marine environments.

6.2.2.1 Testing Conducted at the NASA-KSC

University of Dayton Research Institute (UDRI) in cooperation with WPAFB supplied one hundred and forty test substrates to the Kennedy Space Center (KSC) for marine outdoor atmospheric exposure and evaluation. All panels were sorted, scribed, photo-documented and placed at the NASA Beach Corrosion Test Site for a 1-year duration. All panels were coated according to the methods described in Sections 5.2.0 and 5.2.1.

The following evaluations were performed on the panels:

A) Corrosion from the Scribed Surface (ASTM 1654)
B) Degree of Rusting on Painted Steel Surface (ASTM D 610)
C) Degree of Blistering of Paints (ASTM D714)
D) Color in CIE Lab format. (ASTM D 2244)
E) Gloss (ASTM D 523)
F) Overview photo-documentation of the samples.

The test matrix and labeling of the 140 samples provided by UDRI is shown in Table 124. The substrates consisted of 2024 and 7075 aluminum, as well as 4130 and 1020 steel. These samples were coated with the system shown Table 124. The numbering sequence provided in the three tables (Tables 125-127) was used throughout the coastal environmental exposure section of this report.

Table 124: Test Matrix for samples received from UDRI

Project # 906		Electroactive Polymers Technology Transfer			Account # 3701020104
EAPTT					Mgr : Diane Buhrmaster
Encon @ 77 F / 50% RH					
Panel Prep & Paint					
It is unknown if the supplied substrates are Bare or Clad, nor is the temper known. Therefore unassigned substrate identifiers were used I.A.W. Lab procedure: CLG-LP-001 Test Panel numbering system.					
System	Substrate / Code	Conversion Coat	Primer	Topcoat	Panel ID #s
A	2024	Chromate Conversion Coating (customer supplied)	Deft 02-Y-40 MIL-PRF-23377	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1A-001...005
	7075				906-G1A-001...005
B	2024	TCP (customer supplied)			906-F1B-001...005
C	2024	BAM-PPV (customer supplied)			906-F1C-001...005
	7075				906-G1C-001...005
D	2024	MEH-PPV (customer supplied)			906-F1D-001...005
E	2024	Chromate Conversion Coating (customer supplied)	Deft 02-GN-083 MIL-PRF-23377	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1E-001...005
	7075				906-G1E-001...005
F	2024	TCP (customer supplied)			906-F1F-001...005
G	2024	BAM-PPV (customer supplied)			906-F1G-001...005
	7075				906-G1G-001...005
H	2024	MEH-PPV (customer supplied)			906-F1H-001...005
I	2024	Chromate Conversion Coating (customer supplied)	Deft 02-GN-084	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1I-001...005
	7075				906-G1I-001...005
J	2024	TCP (customer supplied)			906-F1J-001...005
K	2024	BAM-PPV (customer supplied)			906-F1K-001...005
	7075				906-G1K-001...005
L	2024	MEH-PPV (customer supplied)			906-F1L-001...005
M	2024	Chromate Conversion Coating (customer supplied)	Akzo Nobel Mg-rich 2100P001	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1M-001...005
	7075				906-G1M-001...005
N	2024	TCP (customer supplied)			906-F1N-001...005
O	2024	BAM-PPV (customer supplied)			906-F1O-001...005
	7075				906-G1O-001...005
P	2024	MEH-PPV (customer supplied)			906-F1P-001...005
Q	4130 Steel	Cd plated	Deft 02-Y-40 MIL-PRF-23377	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-H1Q-001...005
R	4130 Steel	BAM-PPV (customer supplied)			906-H1R-001...005
S	1020 Steel	No Pretreatment	Deft 02-W-36 MIL-PRF-53022	Deft 99-W-009 MIL-PRF-85285 color 17925	906-I1S-001...005
T	1020 Steel	BAM-PPV (customer supplied)			906-I1T-001...005

All 140 samples were mounted on racks at the NASA Beach Corrosion Test Site, and remained in place for 12 month duration. Each rack was oriented so the face of each

coupon was in direct exposure with the coastal marine atmospheric environment from the Atlantic Ocean (Figure 166).

Figure 166: NAWCWD Panels at the NASA Beach Corrosion Test Site

6.2.2.2 Corrosion Resistance Performance

The coating performance parameters, with respect to corrosion control, were ascertained using the following methodologies:

- ASTM D 1654 - Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments
- ASTM D 610 - Standard Test Method for Evaluating Degree of Rusting on Painted Surfaces
- ASTM D 714 - Standard Test Method for Evaluating Degree of Blistering of Paints

6.2.2.2.1 ASTM D 1654 - Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments

The gloss and color measurements were performed to determine aesthetic and technical parameters related to the coating systems. NASA Corrosion Technology Laboratory personnel evaluated and measured the maximum, minimum and mean creepage from the scribe. All evaluations were performed non-destructively by visually examining the surface of the coating for signs of corrosion or lifting of the coating. Throughout the marine coating section of this final report, ASTM 1654 and ASTM D 610 ratings were color coded according to the severity of corrosion. A rating of 10 was color

coded in green. This rating indicates that no visible sign of corrosion transcended from the scribed area, and no lifting of the coating was evident. A rating number from 7 to 10 (non inclusive of 10) was color coded in yellow. Finally, a rating below 7 was color coded in red.

A summary of the maximum creep as attributable to corrosion from the scribe is shown in Table 125. In some instances, ratings were unobtainable because of significant delamination to the coating, or an inability to ascertain whether the corrosion was the result of edge versus scribe-induced effects. In these circumstances, no rating (N/R) was assigned. After 12 months of exposure at the NASA Beach Corrosion Test Site, the samples utilizing BAM-PPV and MEH-PPV pretreatments on the 2024 aluminum substrates showed significant signs of corrosion from both the scribe and edges of the panels. Several of the coatings exhibited significant signs of delamination. Corrosion from the scribe (while less pronounced than for the other BAM-PPV and MEH-PPV pretreatments) was also significant for the samples labeled 906-F1C and 906-F1D. These series utilized the MIL-PRF-23377C primer and MIL-PRF-85285 color 36173 topcoat.

The 7075 aluminum samples utilizing BAM-PPV or MEH-PPV conversion coatings showed significant signs of corrosion from the scribe, though to a lesser extent than that displayed on the AA2024 substrates. Coating performance (for the 7075 alloy) fared the best for the samples labeled 906-G1C. This series utilized the MIL-PRF-23377C primer and MIL-PRF-85285 color 36173 topcoat. All samples were rated as a 10 after 12 months of environmental exposure. The performance of the coatings that utilized either the chromate or tri-chrome conversion (TCP) coating showed excellent overall performance characteristics on both the 2024 and 7075 aluminum samples throughout the 12 month exposure.

Ten steel coupons (4130 alloy) were used to compare the performance of the BAM-PPV conversion coating, to a similar set that was cadmium plated. As the summary in Table 125 indicates, the corrosion control performance of the cadmium plating was superior to the BAM-PPV pretreatment. Both coating systems utilized a MIL-PRF-23377C primer and MIL-PRF-85285 color 36173 topcoat. Ten steel coupons (1020 alloy) were used to compare the performance of the BAM-PPV conversion coating, to a similar set that used no pretreatment of any type. This comparison utilized a MIL-DTL-53022 primer (non-Cr(VI)) and MIL-PRF-85285 color 17925 topcoat. As the summary in Table 125 indicates, both samples showed significant signs of corrosion emanating from the scribe. The BAM-PPV pretreated samples, in contrast to the non-pretreated samples, exhibited significant signs of delamination.

Table 125: Summary of ASTM D 1654 Ratings – Corrosion from Scribe

System	Substrate / Code	Conversion Coat	Primer	Topcoat	Panel ID #s 4 Month Exposure	4 Month Performance	6 Month Performance	9 Month Performance	12 Month Performance
A	2024	Chromate Conversion Coating (customer supplied)	Deft 02-Y-40 MIL-PRF-23377	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1A-001…005				
	7075				906-G1A-001…005				
B	2024	TCP (customer supplied)			906-F1B-001…005				
C	2024	BAM-PPV (customer supplied)			906-F1C-001…005				
	7075				906-G1C-001…005				
D	2024	MEH-PPV (customer supplied)			906-F1D-001…005				
E	2024	Chromate Conversion Coating (customer supplied)	Deft 02-GN-083 MIL-PRF-23377	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1E-001…005				
	7075				906-G1E-001…005				
F	2024	TCP (customer supplied)			906-F1F-001…005				
G	2024	BAM-PPV (customer supplied)			906-F1G-001…005				
	7075				906-G1G-001…005				
H	2024	MEH-PPV (customer supplied)			906-F1H-001…005				
I	2024	Chromate Conversion Coating (customer supplied)	Deft 02-GN-084	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1I-001…005				
	7075				906-G1I-001…005				
J	2024	TCP (customer supplied)			906-F1J-001…005				
K	2024	BAM-PPV (customer supplied)			906-F1K-001…005				
	7075				906-G1K-001…005				
L	2024	MEH-PPV (customer supplied)			906-F1L-001…005				
M	2024	Chromate Conversion Coating (customer supplied)	Akzo Nobel Mg-rich 2100P001	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1M-001…005				
	7075				906-G1M-001…005				
N	2024	TCP (customer supplied)			906-F1N-001…005				
O	2024	BAM-PPV (customer supplied)			906-F1O-001…005				
	7075				906-G1O-001…005				
P	2024	MEH-PPV (customer supplied)			906-F1P-001…005				
Q	4130 Steel	Cd plated	Deft 02-Y-40 MIL-PRF-23377	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-H1Q-001…005				
R	4130 Steel	BAM-PPV (customer supplied)			906-H1R-001…005				
S	1020 Steel	No Pretreatment	Deft 02-W-36 MIL-PRF-53022	Deft 99-W-009 MIL-PRF-85285 color 17925	906-I1S-001…005				
T	1020 Steel	BAM-PPV (customer supplied)			906-I1T-001…005				

6.2.2.2.2 ASTM D 610 - Standard Test Method for Evaluating Degree of Rusting on Painted Surfaces

All coupons were rated according to ASTM D 610 "*Standard Test Method for Evaluating Degree of Rusting on Painted Surface.*" ASTM D 610 rates the degree of rusting on painted steel surfaces on a scale in which each rating number correlates to the area of a coupon that is corroded. To determine the degree of corrosion emanating from the unscribed metal surface, a series of pictorial examples (from ASTM D 610) were used as a reference. The coating performance was color coded according to the extent of corrosion away from the scribe or edges of the samples. A perfect rating of 10 was color coded in green. A rating number from 7 to 10 (non inclusive of 10) was color coded in yellow. Finally, a rating below 7 was color coded in red. The results of this study are summarized in Table 126. Most of the samples did not show any rusting; however, there were several coupons that showed significant rusting that they could not be rated (N/R). This was due to the large amount of corrosion that either emanated from the scribe or edges.

Table 126: Summary of ASTM D 610 Ratings-Degree of Rusting on Painted Surface

System	Substrate / Code	Conversion Coat	Primer	Topcoat	Panel ID #s	4 Month	6 Month	9 Month	12 Month
A	2024	Chromate Conversion Coating (customer supplied)	Deft 02-Y-40 MIL-PRF-23377	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1A-001…005				
	7075				906-G1A-001…005				
B	2024	TCP (customer supplied)			906-F1B-001…005				
C	2024	BAM-PPV (customer supplied)			906-F1C-001…005				
	7075				906-G1C-001…005				
D	2024	MEH-PPV (customer supplied)			906-F1D-001…005				
E	2024	Chromate Conversion Coating (customer supplied)	Deft 02-GN-083 MIL-PRF-23377	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1E-001…005				
	7075				906-G1E-001…005				
F	2024	TCP (customer supplied)			906-F1F-001…005				
G	2024	BAM-PPV (customer supplied)			906-F1G-001…005				
	7075				906-G1G-001…005				
H	2024	MEH-PPV (customer supplied)			906-F1H-001…005				N/R
I	2024	Chromate Conversion Coating (customer supplied)	Deft 02-GN-084	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1I-001…005				
	7075				906-G1I-001…005				
J	2024	TCP (customer supplied)			906-F1J-001…005				
K	2024	BAM-PPV (customer supplied)			906-F1K-001…005			N/R	N/R
	7075				906-G1K-001…005				
L	2024	MEH-PPV (customer supplied)			906-F1L-001…005				
M	2024	Chromate Conversion Coating (customer supplied)	Akzo Nobel Mg-rich 2100P001	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1M-001…005				
	7075				906-G1M-001…005				
N	2024	TCP (customer supplied)			906-F1N-001…005				
O	2024	BAM-PPV (customer supplied)			906-F1O-001…005			N/R	N/R
	7075				906-G1O-001…005				
P	2024	MEH-PPV (customer supplied)			906-F1P-001…005			N/R	N/R
Q	4130 Steel	Cd plated	Deft 02-Y-40 MIL-PRF-23377	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-H1Q-001…005				
R	4130 Steel	BAM-PPV (customer supplied)			906-H1R-001…005				N/R
S	1020 Steel	No Pretreatment	Deft 02-W-36 MIL-PRF-53022	Deft 99-W-009 MIL-PRF-85285 color 17925	906-I1S-001…005				
T	1020 Steel	BAM-PPV (customer supplied)			906-I1T-001…005				N/R

6.2.2.2.3 ASTM D 714 - Standard Test Method for Evaluating Degree of Blistering of Paints

All samples were rated according to degree of blistering that emanated between the coatings and metal surface of the sample. The extent of blistering was color coded as a function of blistering. A summary of the ASTM D 714 blister ratings (by coating type) is shown in Table 127. A perfect rating value of 10 (indicating no blisters) was color coded in green. A system that exhibited blisters was color coded in red. Yellow indicates that the coating had previously blistered.

As shown in Table 127, the non-cadmium plated 4130 alloy steel samples exhibited blisters early in the duration of exposure. The blisters formed near the scribe and edges of the coupons. As the exposure period lengthened, the blisters subsided, and subsequently, initiated corrosion under the surface of the coating. All 1020 steel alloy samples exhibited blistering after 4 months of exposure. Blistering was only visible on steel coupons. No blistering was evident on the aluminum substrates.

Table 127: Summary of ASTM D 714 Ratings-Degree of Blistering

System	Substrate / Code	Conversion Coat	Primer	Topcoat	Panel ID #s	4 Month	6 Month	9 Month	12 Month
A	2024	Chromate Conversion Coating (customer supplied)	Deft 02-Y-40 MIL-PRF-23377	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1A-001…005				
	7075				906-G1A-001…005				
B	2024	TCP (customer supplied)			906-F1B-001…005				
C	2024	BAM-PPV (customer supplied)			906-F1C-001…005				
	7075				906-G1C-001…005				
D	2024	MEH-PPV (customer supplied)			906-F1D-001…005				
E	2024	Chromate Conversion Coating (customer supplied)	Deft 02-GN-083 MIL-PRF-23377	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1E-001…005				
	7075				906-G1E-001…005				
F	2024	TCP (customer supplied)			906-F1F-001…005				
G	2024	BAM-PPV (customer supplied)			906-F1G-001…005				
	7075				906-G1G-001…005				
H	2024	MEH-PPV (customer supplied)			906-F1H-001…005				
I	2024	Chromate Conversion Coating (customer supplied)	Deft 02-GN-084	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1I-001…005				
	7075				906-G1I-001…005				
J	2024	TCP (customer supplied)			906-F1J-001…005				
K	2024	BAM-PPV (customer supplied)			906-F1K-001…005				
	7075				906-G1K-001…005				
L	2024	MEH-PPV (customer supplied)			906-F1L-001…005				
M	2024	Chromate Conversion Coating (customer supplied)	Akzo Nobel Mg-rich 2100P001	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-F1M-001…005				
	7075				906-G1M-001…005				
N	2024	TCP (customer supplied)			906-F1N-001…005				
O	2024	BAM-PPV (customer supplied)			906-F1O-001…005				
	7075				906-G1O-001…005				
P	2024	MEH-PPV (customer supplied)			906-F1P-001…005				
Q	4130 Steel	Cd plated	Deft 02-Y-40 MIL-PRF-23377	Deft 99-GY-001 MIL-PRF-85285 color 36173	906-H1Q-001…005				
R	4130 Steel	BAM-PPV (customer supplied)			906-H1R-001…005				
S	1020 Steel	No Pretreatment	Deft 02-W-36 MIL-PRF-53022	Deft 99-W-009 MIL-PRF-85285 color 17925	906-I1S-001…005				
T	1020 Steel	BAM-PPV (customer supplied)			906-I1T-001…005				

6.2.2.2.4 Aesthetic and Other Technical Parameters

The gloss and color measurements were performed to determine aesthetic and technical parameters related to the coating system. The ASTM tests performed to determine the metrics are as follows:

- ASTM D 2244- Standard Practice for Calculation of Color Tolerances and Color Differences from Instrumentally Measured Color Coordinates
- ASTM D 523-Standard Test Method for Specular Gloss

6.2.2.2.5 ASTM D 2244- Standard Practice for Calculation of Color Tolerances and Color Differences from Instrumentally Measured Color Coordinates

Color measurements were recorded with a handheld portable color meter using the CIE *a*b* format, D-65 illuminant and a 10° observer. Delta E (ΔE) (change in color) values were calculated from the initial and coastal environmental exposure dependent measurements. Generally, a ΔE value of one is discernible by the human eye in a side-by-side comparison. However, in less than ideal lighting, a ΔE value of two or three can still be considered the same color.

Color measurements were collected near the center of each coupon to avoid deviations in color that might coincide with degradation of the coating from the edges of the sample. Color values for individual coupons were based upon the average of three measurements for each sample.

The average ΔE change in color for each coating type and duration of exposure is shown in Table 128. The sample column in Table 128 shows the sample ID number which is also found in the previous 4 Tables (Tables 124-127). The panel ID numbers are correlated to the substrate/pretreatment coating/primer/topcoat found in Tables 124-127.

Color values for individual coupons were based upon the average of three measurements for each sample. A rating of N/M indicates that a sufficient sample surface was unavailable for measurement because of a significant degree of coating delamination. For the coated aluminum samples, calculated delta E (ΔE) color values varied between 2 and 4. The steel samples did exhibit somewhat larger ΔE values, but this phenomenon was most probably related to the proliferation of corrosion products from the edges and scribe. Color measurements were recorded in areas as far removed from the corroded locations as possible.

As might be expected, significant changes in color were exhibited from the white corrosion products that formed on the uncoated AA2024-T3 aluminum substrates. This provides a measure that helps delineate the degree of protection in the field. For the coated aluminum samples (906-F1A through 906-F1P) calculated ΔE color values are between 1.65 and 3.21. The steel samples (906-H1Q through 906-I1T) exhibited ΔE values which were slightly larger and varied between 1.92 and 3.56. The slightly increased values were most probably related to the proliferation of corrosion products from the edges and scribes.

Table 128: Average Delta E (ΔE) Color Calculations by Coating Type

	Average Delta E Calculations by Group			
Sample	4 Month Delta E	6 Month Delta E	9 Month Delta E	12 Month Delta E
906-F1A	2.69	2.48	1.70	1.89
906-F1B	2.88	2.32	2.50	2.46
906-F1C	2.65	2.47	1.97	2.52
906-F1D	2.60	2.91	2.57	2.49
906-F1E	2.30	2.33	3.26	2.21
906-F1F	2.15	2.47	3.74	2.05
906-F1G	2.12	2.38	2.59	2.34
906-F1H	2.16	2.25	2.40	2.24
906-F1I	2.48	2.27	2.16	2.34
906-F1J	2.46	2.10	1.94	2.10
906-F1K	2.26	2.21	2.03	2.24
906-F1L	2.27	2.11	1.87	2.22
906-F1M	2.53	2.44	2.08	1.80
906-F1N	2.54	2.32	1.94	2.62
906-F1O	2.38	2.29	2.11	3.21
906-F1P	2.31	2.08	2.29	2.62
906-G1A	2.68	2.75	2.06	2.48
906-G1C	2.57	2.82	2.23	2.06
906-G1E	2.28	2.28	4.97	1.99
906-G1G	2.09	2.22	2.01	1.81
906-G1I	2.72	2.24	2.02	2.00
906-G1K	2.41	2.45	1.99	2.06
906-G1M	2.50	2.35	2.17	1.65
906-G1O	2.33	1.96	1.74	1.73
906-H1Q	2.35	2.49	2.38	1.92
906-H1R	2.69	3.12	3.15	2.84
906-I1S	5.13	3.66	4.63	3.56
906-I1T	3.56	2.27	2.26	3.07
2024 T3	24.26	23.89	31.09	22.36

6.2.2.2.6 ASTM D523-Standard Test Method for Specular Gloss

Gloss measurements were evaluated for each coating system. The gloss meter records the amount of reflective illuminated light at specified angles of 20°, 60°, or 85°; and gives a value in gloss units (GU). The 60° geometry is used for most specimens, and is the initial angle used to determine whether the 20° or 85° angles may be more applicable. The 20° angle is used when the 60° angle gloss values are higher than 70 GU's, while the 85° angle is used when the 60° angle gloss values are less than 10 GU's. Because of the dull finish of the samples, the 85° angle was reported for all unscribed coupons in this study.

The measurement scale, Gloss Units (GU), of a gloss meter is a scaling based on a highly polished reference black glass standard with a defined refractive index having a specular reflectance of 100GU at the specified angle. This standard is used to establish an upper point calibration of 100 with a lower end-point established at 0 on a perfectly matt surface.

Gloss measurements were recorded using a calibrated BYK Gardner Tri-Gloss portable gloss meter. Measurements were recorded in three locations near the center of the face of each non-scribed coupon and the results were averaged. The measurements were recorded prior to, and after every three months of exposure. The change in gloss in relation to the initial gloss readings was calculated and reported.

The coated steel coupons (906-I1S and 906-I1T) exhibited a large deviation in gloss. Much of the variation was attributable to the liberation of corrosion products that deposited on the face of the white coupons that originally exhibited a high level of gloss, as well a surface that was heavily disturbed by corrosion underneath the coating. The gloss measurements for the bare aluminum substrates were heavily influenced by the corrosion products that formed on the face of the unprotected samples (AA2024-T3). Gloss measurements for all coated samples showed negligible changes in gloss.

6.2.2.2.7 Sample Photodocumentation

The photodocumentation of the coated samples are shown in Figures 167-170. These general figures are shown in order to give the reader an impression of corrosion failure and survival in marine outdoor exposure testing. The initial set of photos (Figures 167-170) show scribed panels prior to marine outdoor exposure testing. As shown from these photos, no corrosion, delamination is evident at the start of this test. The second set of photographs (Figures 171-174) showed the condition of the panels after twelve months of coastal atmospheric exposure. As can be seen from these photos no corrosion, delamination is evident at the start of this test. Figures 171-174 document the 12 month marine outdoor exposure testing at the KSC facility and after 12 months, numerous failures were evident. This includes significant corrosion, delamination and blistering. BAM-PPV and MEH-PPV with various primers (non-chromate) and topcoat failed early in the marine outdoor testing (~4 months) except BAM-PPV + chromate primer + topcoat lasted 12 months. The best performing systems were fully chromated military coating (CCC + chromate primer + topcoat) and TCP pretreatment with both chromate and non-chromate primer and topcoat. Figures 175-179 give specific examples of several coupons that failed this test vs. several that passed (chromate controls).

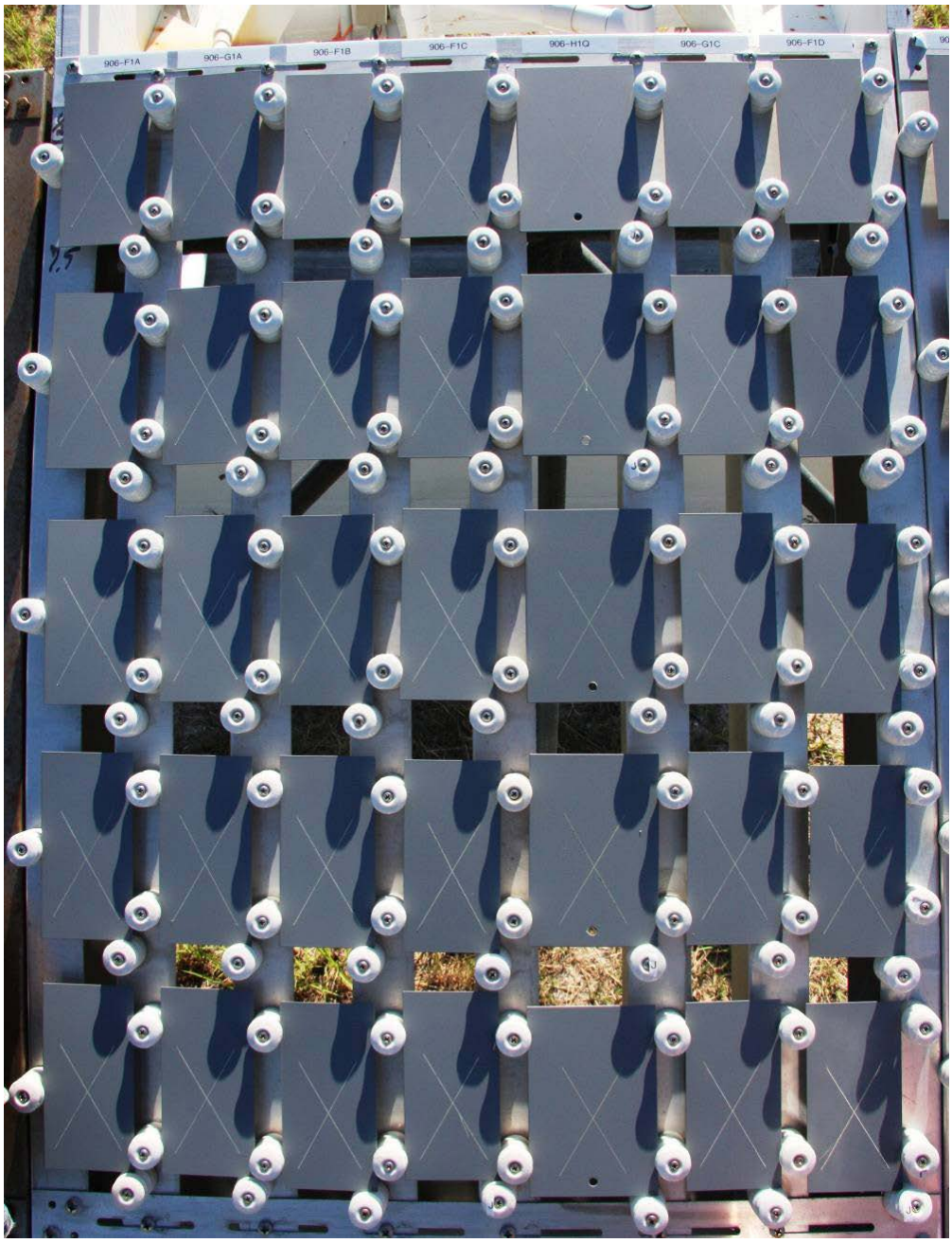


Figure 167: Initial-Rack 1

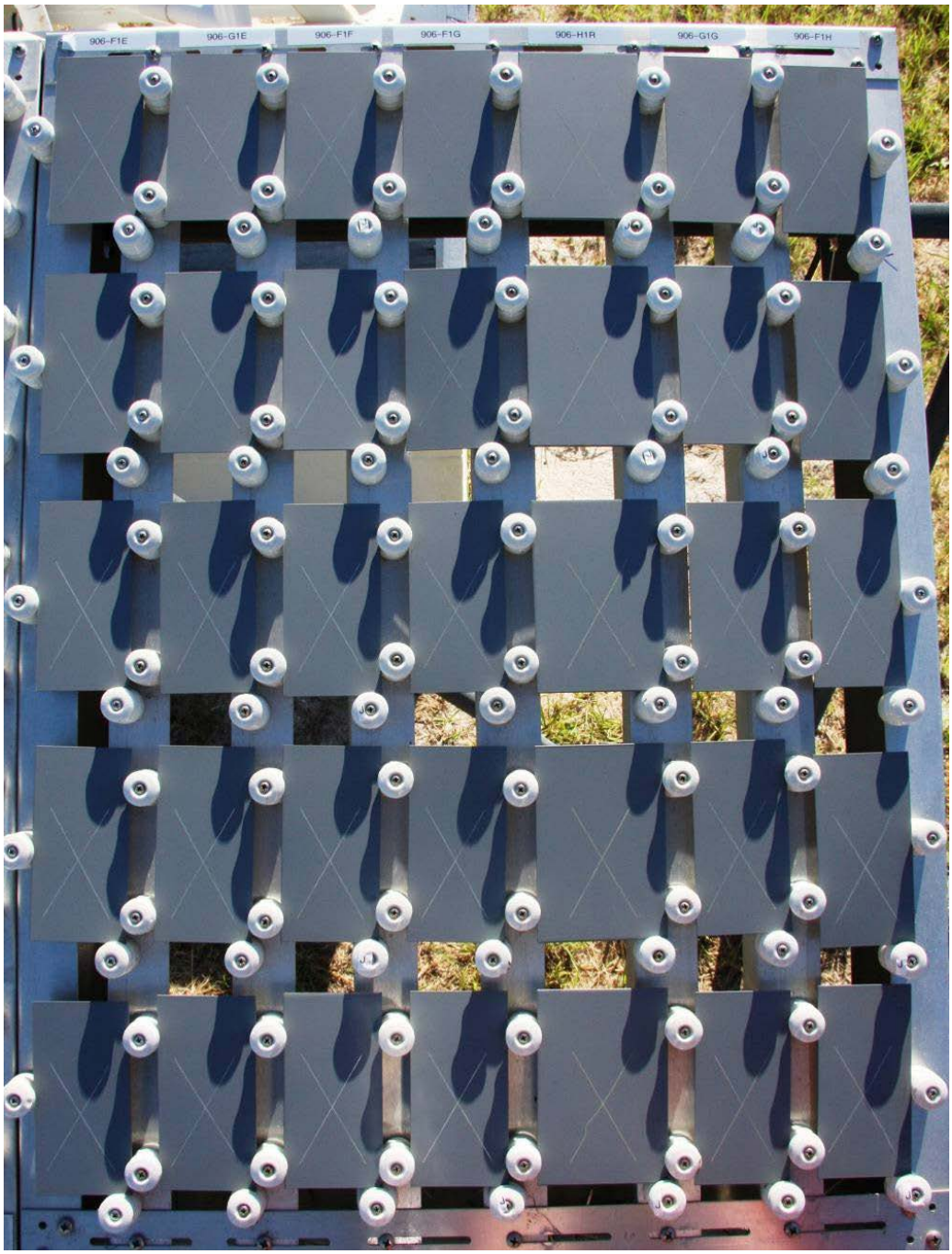


Figure 168: Initial-Rack 2

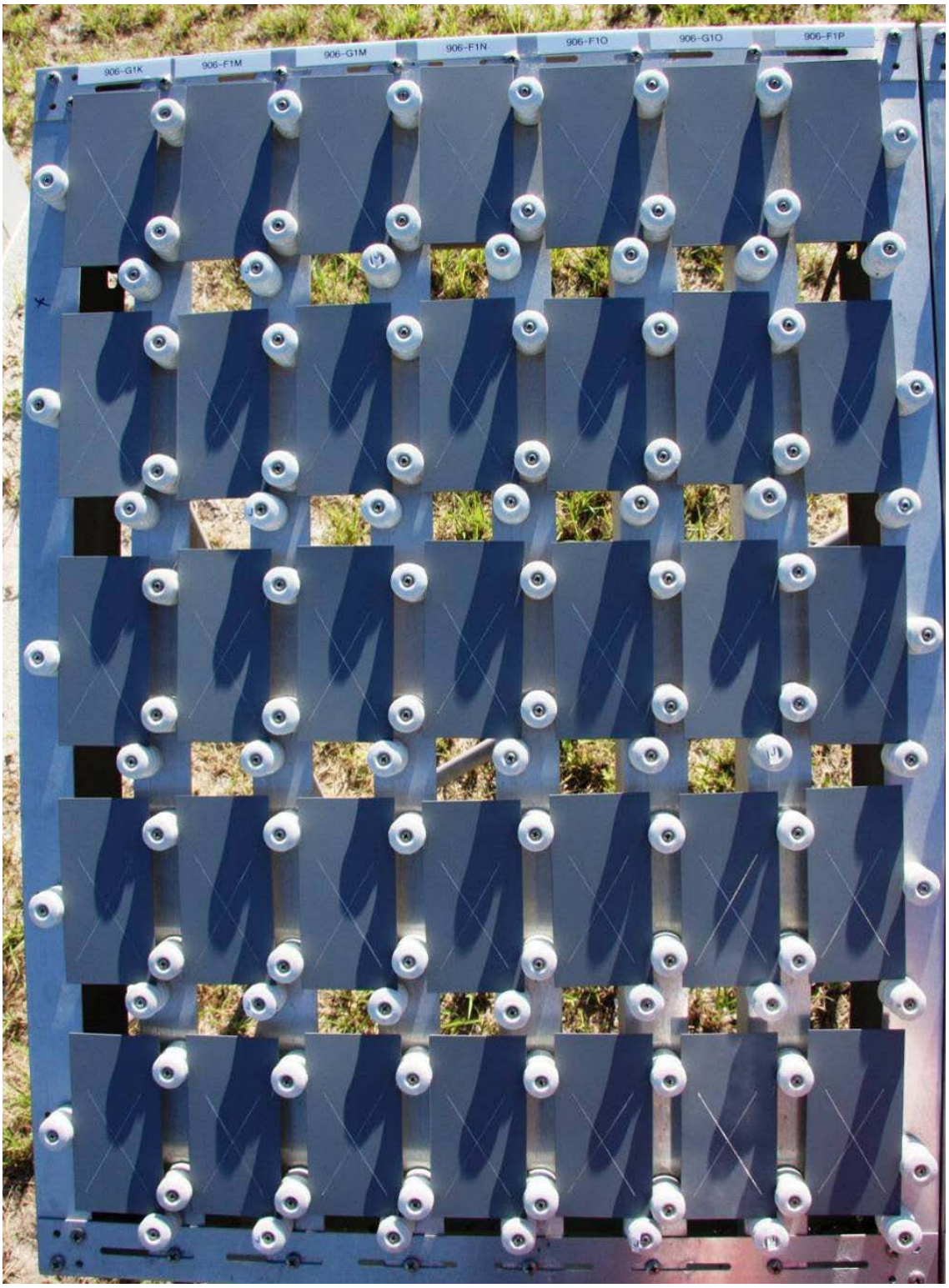


Figure 169: Initial-Rack 3

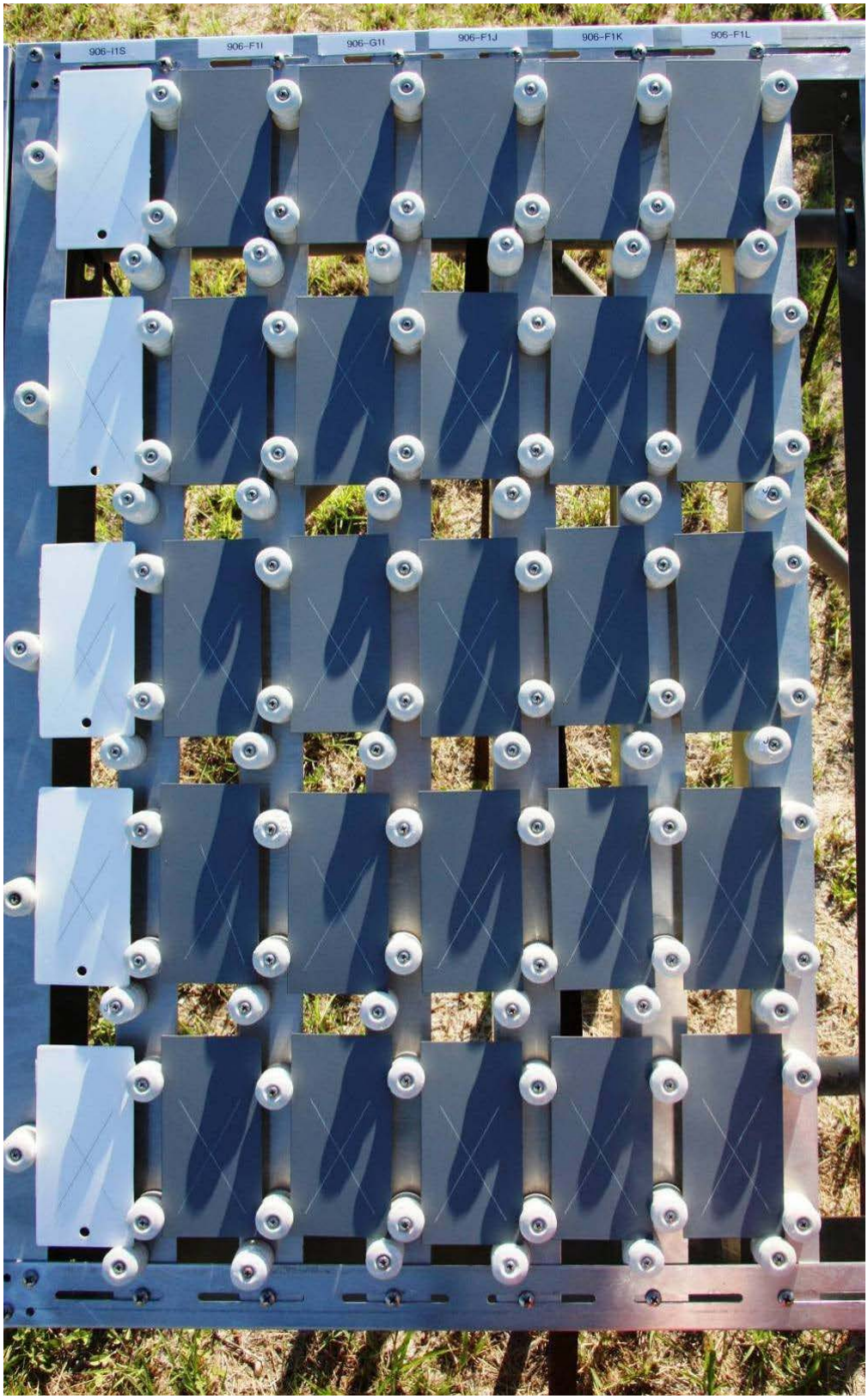


Figure 170: Initial-Rack 4

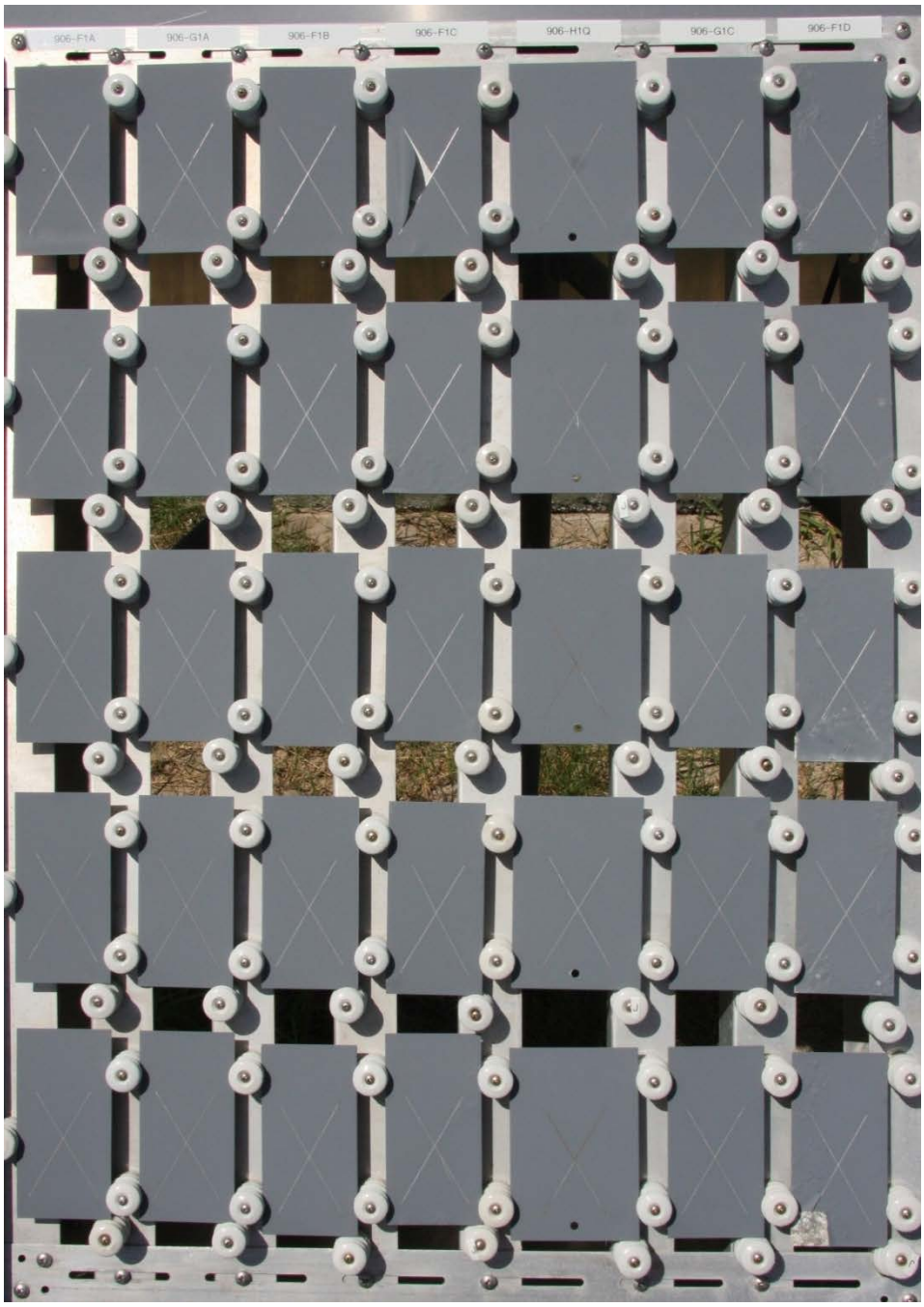


Figure 171: 12 Month Exposure-Rack 1

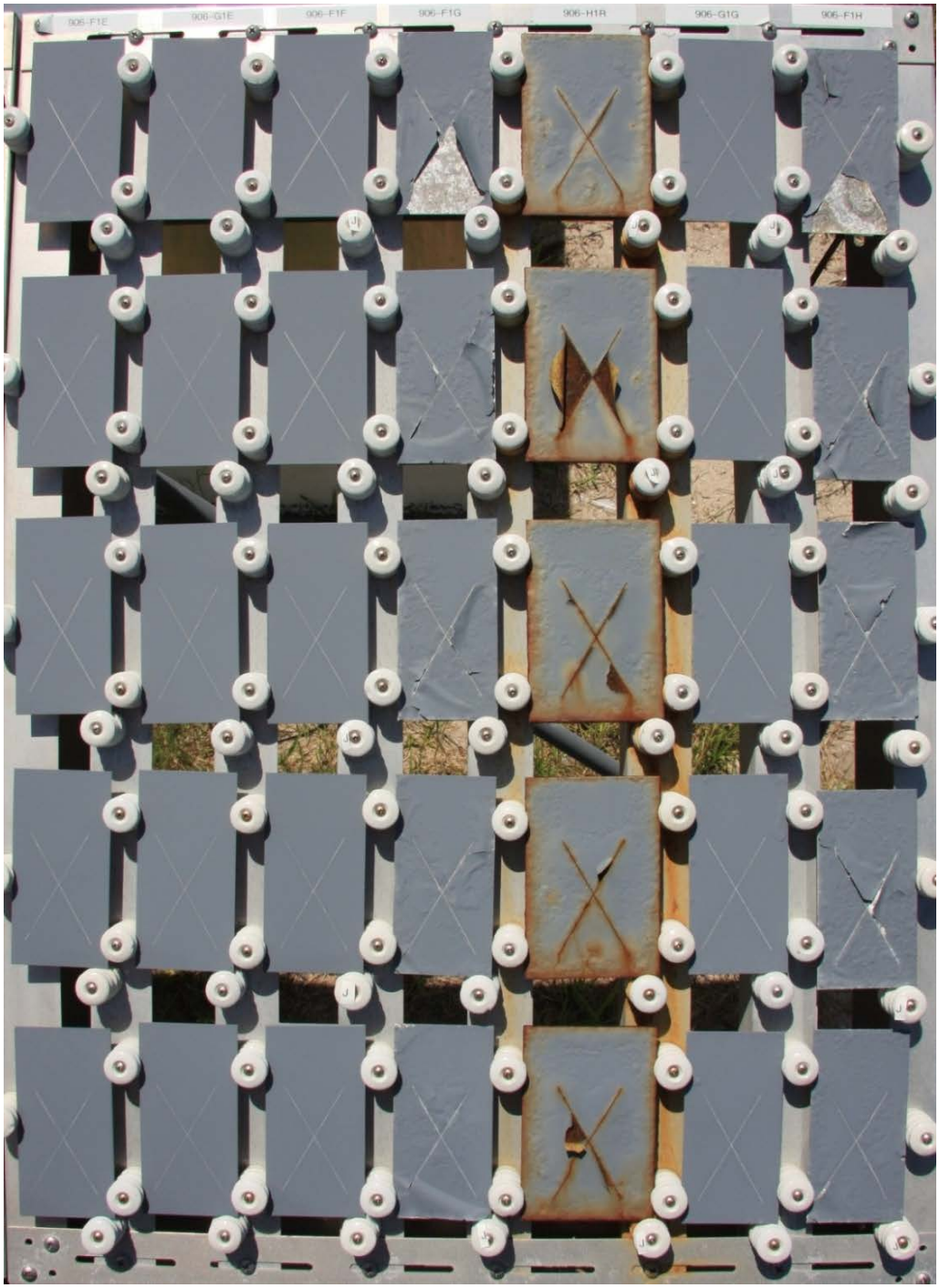


Figure 172: 12 Month Exposure-Rack 2

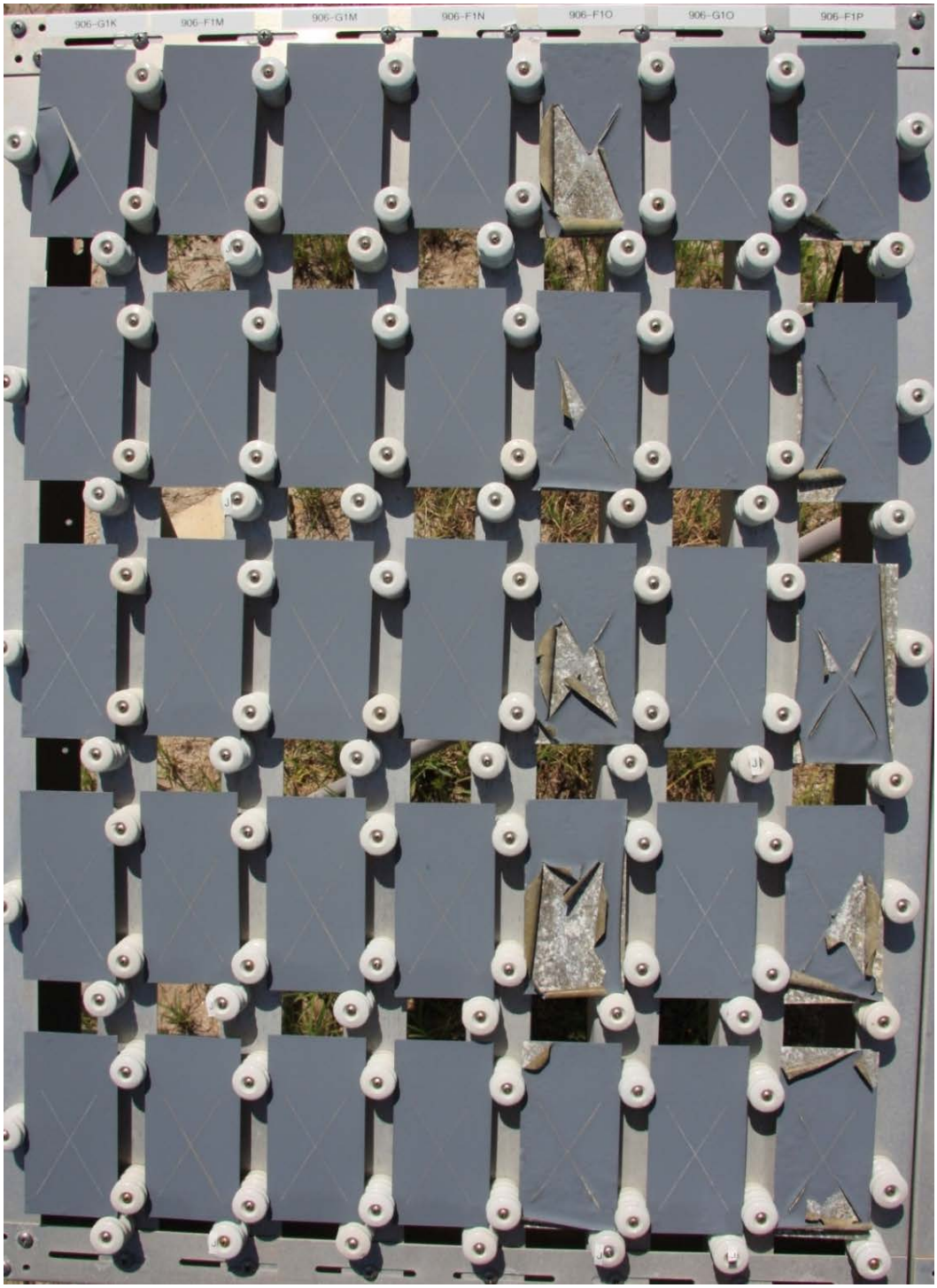


Figure 173: 12 Month Exposure-Rack 3

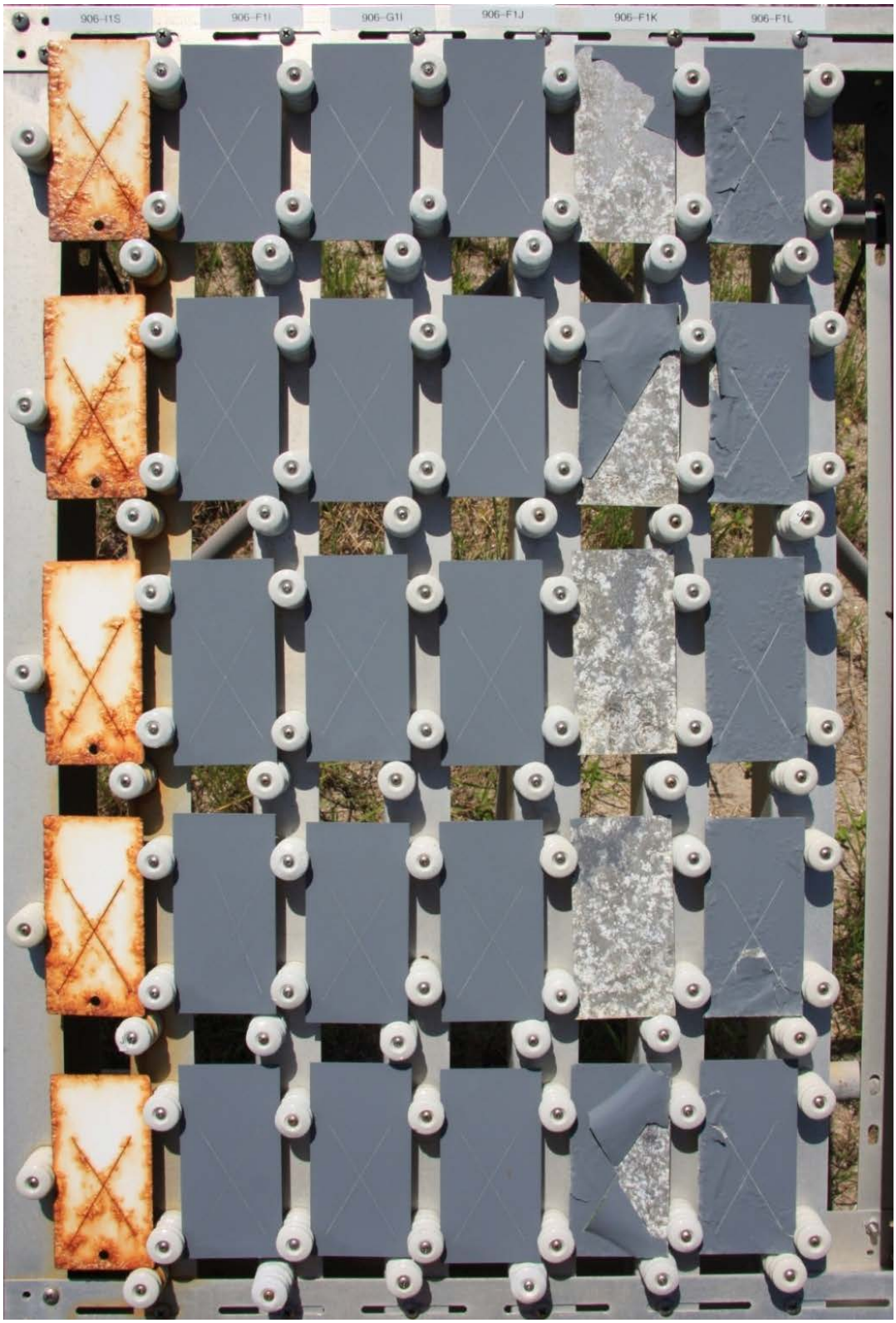
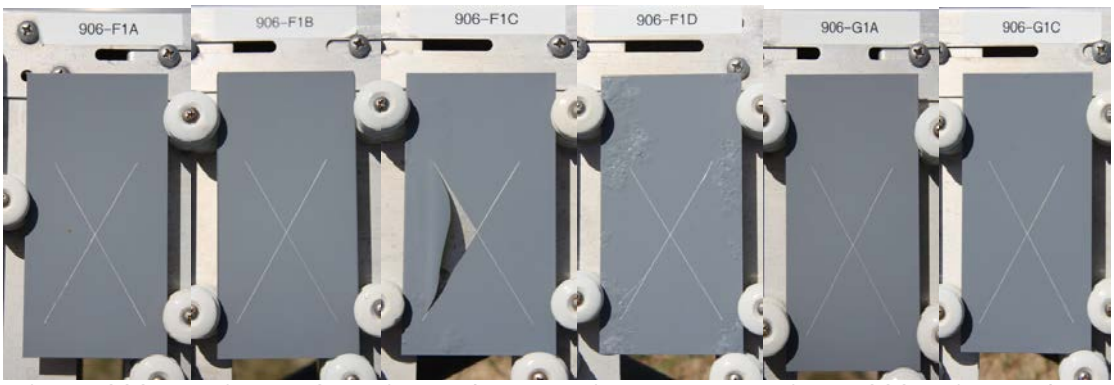


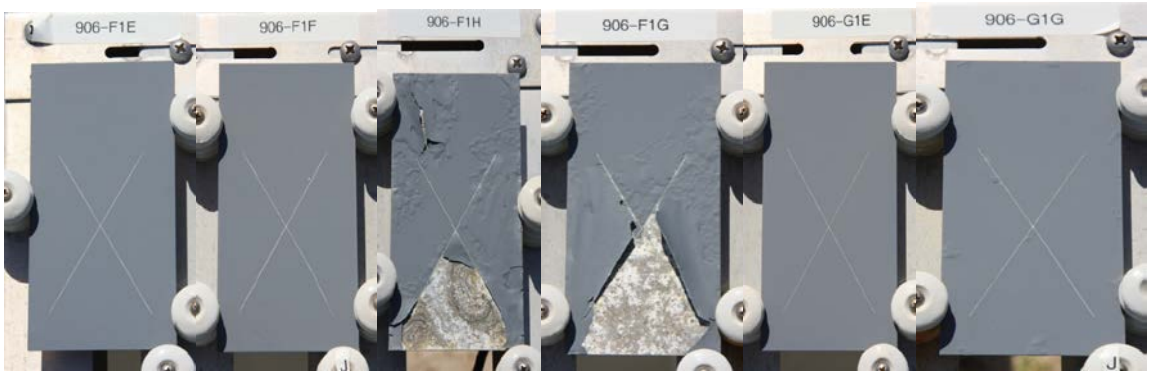
Figure 174: 12 Month Exposure-Rack 4

Further photodocumentation of panels at the KSC marine outdoor exposure test facility are shown in Figures 175-179 after 12 months exposure time. The BAM-PPV and MEH-PPV pretreatments on AA2024-T3 showed after 6 months delamination and corrosion in the scribe. At 12 months exposure time the BAM-PPV coated coupons on AA2024-T3 showed the worst performance as compared to both CCC, TCP and MEH-PPV pretreatment coupons using a chromate primer and topcoat. BAM-PPV and MEH-PPV coated coupons with both non-chromated epoxy primers systems and the same topcoat showed failure at about 6 months of exposure time. The BAM-PPV coated AA7075-T6 coated coupons using chromate epoxy primer and topcoat showed comparable performance to the CCC control after 12 months of exposure. BAM-PPV coated 4130 high strength steel and 1020 steel showed failure at about 6 months exposure.



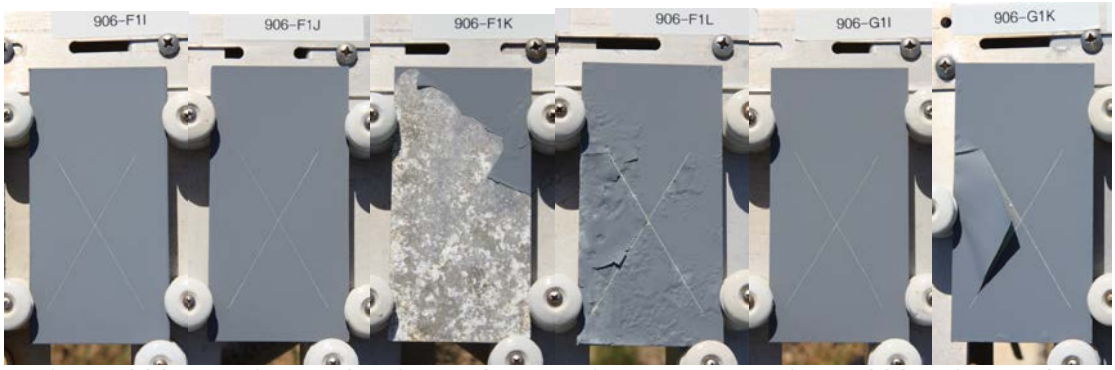
2024-T3 CCC *2024-T3 TCP* *2024-T3BAM-PPV* *2024-T3MEH-PPV* *7075-T6 CCC* *7075-T6 BAM-PPV*

Figure 175: Deft 02-Y-40 Primer (Chromated epoxy primer MIL-PRF-23377C): Deft 99-GY-001 Topcoat (Polyurethane APC topcoat MIL-PRF-85285



2024-T3 CCC *2024-T3 TCP* *2024-T3BAM-PPV* *2024-T3MEH-PPV* *7075-T6 CCC* *7075-T6 BAM-PPV*

Figure 176: Deft 02-GN-083 Primer (Non chromate epoxy primer MIL-PRF-23377N): Deft 99-GY-001 Topcoat



2024-T3 CCC *2024-T3 TCP* *2024-T3BAM-PPV* *2024-T3MEH-PPV* *7075-T6 CCC* *7075-T6 BAM-PPV*

Figure 177: Deft 02-GN-084 Primer (Non chromated epoxy primer MIL-PRF-23377N): Deft 99-GY-001 Topcoat

4130 Cadmium Plated *4130 HHS BAM-PPV*
High Strength Steel (HSS)

Figure 178: Deft 02-Y-40 Primer: Deft 99-GY-001 Topcoat

1020 – No pretreatment *1020 – BAM-PPV*

Figure 179: Deft 02-W-36 Primer: Deft 99-W-009 Topcoat

6.2.2.3 NASA-KSC Conclusions for Marine Outdoor Exposure Testing

The results from the marine outdoor exposure testing are summarized below:

- ***<u>BAM-PPV and MEH-PPV failed the marine outdoor exposure testing</u>***,
- ***BAM-PPV and MEH-PPV showed signs of corrosion after several months of marine outdoor exposure***,
- ***<u>BAM-PPV showed improved performance (survived 9 months)</u> with chromated primer vs. MEH-PPV with chromated primer***,
- ***Best performing systems were CCC + chromated or non-chromated primer + topcoat and TCP + chromated or non-chromated primer + topcoat***,
- ***<u>Therefore, BAM-PPV with moderately improved performance</u> over MEH-PPV was selected for field studies***.

6.3 Toxicology Results

The results shown below summarize the toxicology testing that is required for new materials prior to field testing. The tests performed on BAM-PPV were done in accordance with the December 2000 JTP [78].

6.3.1 Acute Oral Toxicity - Up and Down Procedure (UDP)

Initially, a single female Wistar albino rat was dosed orally with BAM-PPV, at a dose level of 2000 mg/kg. Based on the survival of the initial animal, four additional females were dosed at 2000 mg/kg. Since the dose volume exceeded the maximum amount to be delivered in a single administration at 2000 mg/kg, the animals were administered two separate doses over a period of approximately one hour. The rats were observed at 15 minutes, 1, 2 and 4 hours following the second dose and once daily for 14 days for toxicity and pharmacological effects. All animals were observed twice daily for mortality. Body weights were recorded immediately pretest, weekly and at termination. All animals were examined for gross pathology. The potential for toxicity was based on the mortality response noted. All animals survived the single 2000 mg/kg oral dose. There were no abnormal physical signs noted during the observation period. Body weight changes were normal. Necropsy results were normal. Therefore, the LD_{50} of BAM-PPV is greater than 2000 mg/kg.

6.3.2 Acute Dermal Toxicity/LD_{50} in Rabbits

Five healthy male and five healthy female New Zealand White rabbits were dosed dermally with BAM-PPV at 2000mg/kg of body weight. The test article (BAM-PPV) was kept in contact with the skin for 24 hours. Dermal responses were recorded at 24 hours post dose and on days 7 and 14. Animals were observed for toxicity and pharmacological effects at 1, 2, and 4 hours post dose and once daily for 14 days. All animals were observed twice a day for mortality and body weights were recorded pretest, weekly and at termination. All animals were examined for gross pathology. All animals survived the 200mg/kg dermal application and instances of soiling of the anogenital area were the only abnormal physical signs noted during the observation period. Dermal responses were very slight at 24 hours after the BAM-PPV was removed and absent on days 7 and 14. However, instances of yellow staining and flaking skin were noted at 24 hours and on day 7. Body weight changes were normal as well as necropsy. Therefore, the LD_{50} of BAM-PPV is greater than 2000 mg/kg of body weight.

6.3.3 Acute Dermal Irritation in Rabbits

Since the test article was not expected to produce severe irritation or corrosion, three healthy New Zealand White rabbits (males) were dosed dermally with BAM-PPV. The test article (0.5 g) was applied dermally to one intact site per rabbit. The test article was kept in contact with the skin for 4 hours at which time the wrappings were removed. Dermal reactions were scored at 60 minutes after removal of wrappings. Reactions were scored again at 24, 48 and 72 hours. The skin was also evaluated for ulceration and

necrosis or any evidence of tissue destruction at these time periods. Body weights were recorded pretest and at termination. There was no erythema or edema noted at any observation period. One instance of wetness of the anogenital area, noted in one animal, was the only abnormal physical sign noted during the observation period. All body weight changes were normal and, therefore, BAM-PPV is not a dermal irritant.

6.3.4 Acute Eye Irritation in Rabbits

Three healthy New Zealand White rabbits (1 male - 2 females), free from evidence of ocular irritation and corneal abnormalities, were dosed with BAM-PPV. The test article (0.1 ml equivalent (54 mg)) was placed into the conjunctival sac of one eye of each rabbit. The contralateral eye served as a control. The eyes were examined and scored by the Draize technique at 1, 24, 48 and 72 hours. Sodium fluorescein dye procedures were used at the 24-hour observation interval. Body weights were recorded pretest. There was no corneal opacity or iritis noted at any observation period. Conjunctival irritation, noted in 3 of the 3 eyes, cleared within 24 hours. There were no abnormal physical signs noted during the observation period. Ocular administration of BAM-PPV produced irritation which cleared within 24 hours.

6.3.5 Delayed Contact Dermal Sensitization Test-Buehler Method

Fifteen healthy male and fifteen healthy female Hartley Albino guinea pigs were assigned to the study. Group 1 (10 males & 10 females) was induced with BAM-PPV at a concentration of 100%. Group 2 (5 males & 5 females) was not induced and served as a naive control. Group 1 received three topical induction applications, one per week for three weeks. Skin reactions of the animals in Group 1 were recorded 24 and 48 hours following patch removal. Based on the results of the induction application, 100% was chosen as the highest non-irritating concentration for the challenge and was administered to both groups two weeks after the third induction. The skin reactions of all animals were recorded at 24 and 48 hours following patch removal. Body weights were recorded pretest and at termination. Animals in Groups 1 and 2 were observed once a day for mortality and toxicity.

Induction 1 - Erythema was absent to very faint with orange staining on the dose sites.

Induction 2 - Erythema was absent with orange staining on the dose sites.

Induction 3 - Erythema was absent with orange staining on the dose sites.

Challenge - Erythema was absent in both the induced and naive control group with orange staining on the dose sites (see Table 129).

Table 129: Buehler Method Test Results for BAM-PPV

	Incidence Index		Severity Index	
	24 hours	48 hours	24 hours	48 hours
poly (2,5-bis(-N-methyl-N-hexylamino) phenylene vinylene), (BAM-PPV), PZ1615-30, Lot/Batch# PZ1615-30	0	0	0	0
Naive Control	0	0	0	0

One male in Group 1 was noted with soiling of the anogenital area. All other animals appeared normal throughout the observation period. Body weight changes were normal. The BAM-PPV is a non-sensitizer.

6.3.6 Acute Inhalation Toxicity/LC_{50} in Rats

Acute inhalation studies in rats were not performed on samples of BAM-PPV due to the inherent difficulty of preparing BAM-PPV particle sizes in the range of 1-4 micrometers. This small particle range is needed to obtain meaningful data regarding the lethality of BAM-PPV when test specimens are exposed to inhalation of an unknown material. Several attempts at grinding BAM-PPV powder into the appropriate size proved impossible. The BAM-PPV when repeatedly ground in a mixer under high shear forces produced particles that clumped together. The BAM-PPV particles sizes ranged from 25-250 micrometers in diameter. Figure 180 shows SEM images of BAM-PPV particles aggregating in which there are numerous individual particles and clumps of particles present.

In consultation with MB Research the decision to cancel any studies of BAM-PPV was made due to the lack of any meaningful data that can provide information regarding the LC_{50} toxicity in rats using acute inhalation testing conditions.

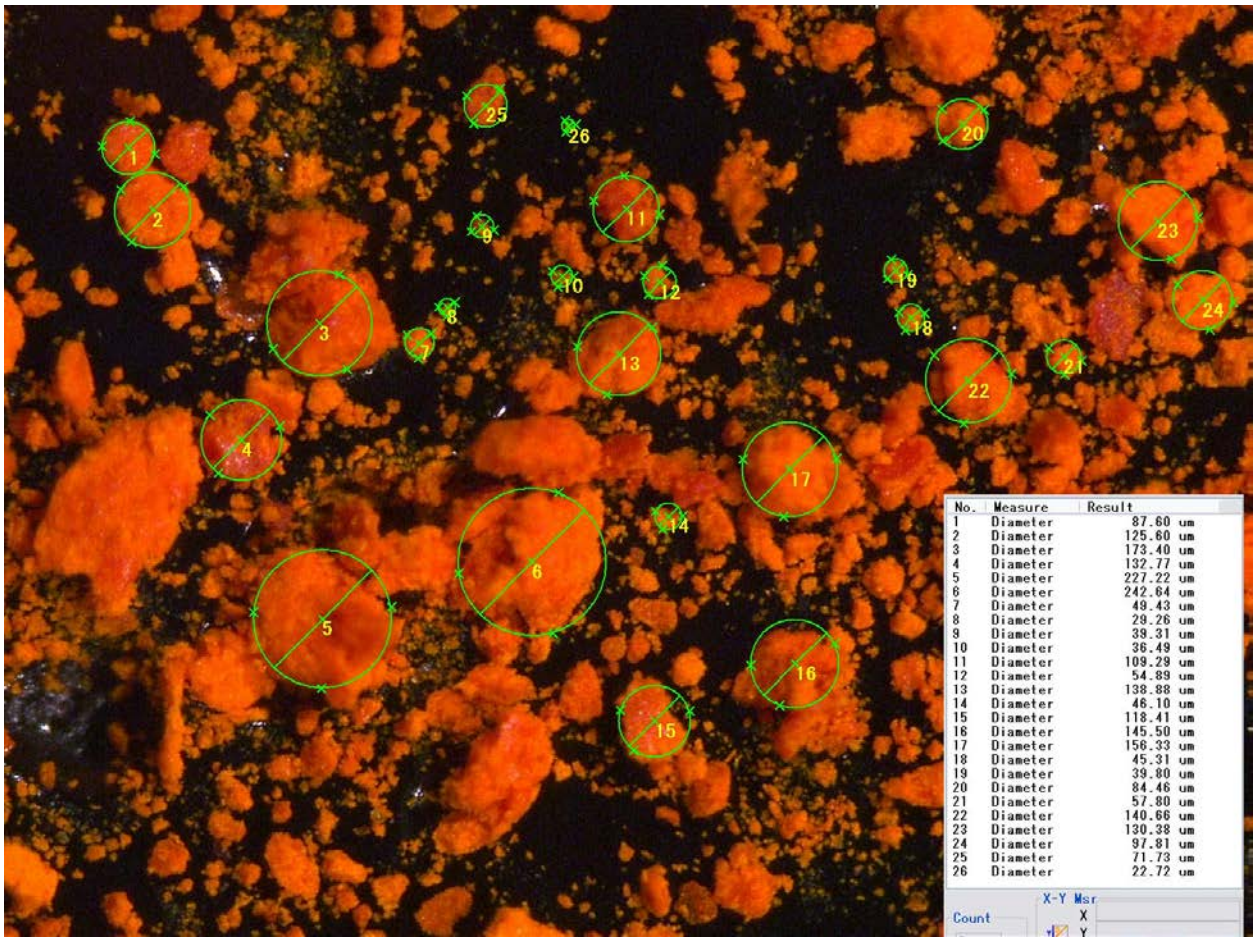


Figure 180: SEM Image of Ground BAM-PPV Powder

6.3.7 Conclusions for BAM-PPV Toxicity Testing

The following bullets summarize the results found for BAM-PPV powder toxicology testing:

- ***BAM-PPV is a non-toxic, non-hazardous material,***
- ***BAM-PPV has an LD_{50} ≥2000 mg/kg for both acute dermal and acute oral toxicity,***
- ***BAM-PPV is not a dermal irritant,***
- ***BAM-PPV is not a sensitizer,***
- ***BAM-PPV does not appear to be an acute inhalation material due to its difficulty in producing small particles that can enter the lungs,***
- ***BAM-PPV conforms to EPA/OSHA standards.***

6.4 Field Tests for BAM-PPV Pretreatment Coating Systems

The criteria were determined from all laboratory and outdoor exposure testing (Phase I) for BAM-PPV and MEH-PPV to determine which if any of these pretreatment coating would be a viable candidate for field testing. **BAM-PPV after a thorough review by each performing organization determined that BAM-PPV provided acceptable data to warrant a field test**. Field tests were conducted on BAM-PPV pretreatment coatings after a down-selection during Phase II.

6.4.1 Field Tests for BAM-PPV Pretreatment Coating Conducted at WPAFB

Of the systems tested, BAM-PPV and MIL-PRF-23377 non-chromated epoxy primer Deft 02-GN series with MIL-PRF-85285 APC polyurethane topcoat Deft 99-GY-001 demonstrated the best overall performance. This system was identified as the prime candidate for field testing alongside the current Cr(VI) coating system typically found on aircraft. For BAM-PPV coating systems, ASTM B 117 salt spray data verified that these coating systems performed as well or better than PreKote coating systems, and as well or slightly worse than Alodine 1200S coating systems. Additional tests such as crosshatch adhesion, pencil hardness, and wet-tape adhesion results were similar to Alodine 1200S and PreKote coating systems with two exceptions: (*i*) BAM-PPV systems with the Hentzen 16708TEP primer demonstrated softer pencil hardness values and lower wet-tape adhesion results; and (*ii*) PATTI results were comparable to PreKote and showed less variability. However, Alodine 1200S systems had slightly higher pull-off values. Fluid resistance results identified a weakness of the BAM-PPV systems when exposed to hydraulic fluids, but no weaknesses were noted for jet fuel or lubricating oils exposure. Xenon-arc exposure did not identify loss of color greater than ΔE of 1 for BAM-PPV systems, and no coating deficiencies were noted after 500 hours of exposure. Impact flexibility testing did not identify excessive vulnerabilities in BAM-PPV beyond what is seen with PreKote and Alodine 1200S coating systems. GM9540P accelerated corrosion results for BAM-PPV coating systems were similar to PreKote systems and slightly lower than cadmium-plated systems. As a result of the acceptable laboratory performance of BAM-PPV, field testing was done at WPAFB using BAM-PPV as the pretreatment coating.

A 1 wt% solution of BAM-PPV dissolved in Oxsol-100 was used to coat the C-5 aircraft door. HVLP was used as the delivery system to apply the BAM-PPV solution onto the C-5 cargo door using multiple passes to get an approximate thickness of 2 μm. This coating thickness was selected due to the good corrosion performance seen in the neutral salt fog studies of BAM-PPV. The BAM-PPV pretreatment coating was applied onto the C-5 cargo plane's rear hatch door with a non-Cr(VI) primer and topcoat. A Cr(VI) full military coating [CCC + MIL-PRF-23377C + MIL-PRF-85285] was used as the control for this field demonstration. The rear hatch door was divided into four quadrants and coated with the controls and BAM-PPV (see Table 130 and Figures 181-183).

Table 130: BAM-PPV and Cr(VI) controls on C-5 Cargo Plane Rear Hatch Door

Test Coating System	Pretreatment	Primer	Topcoat
Quadrant 1	CCC	MIL-PRF-23377N1 (non-Cr(VI) epoxy primer)	MIL-PRF-85285 IV (Polyurethane topcoat)
Quadrant 2	BAM-PPV	MIL-PRF-23377N1 (non-Cr(VI) epoxy primer)	MIL-PRF-85285 IV (Polyurethane topcoat)
Quadrant 3	BAM-PPV	MIL-PRF-23377C2 Cr(VI) epoxy primer	MIL-PRF-85285 IV (Polyurethane topcoat)
Quadrant 4	CCC	MIL-PRF-23377C2 Cr(VI) epoxy primer	MIL-PRF-85285 IV (Polyurethane topcoat)

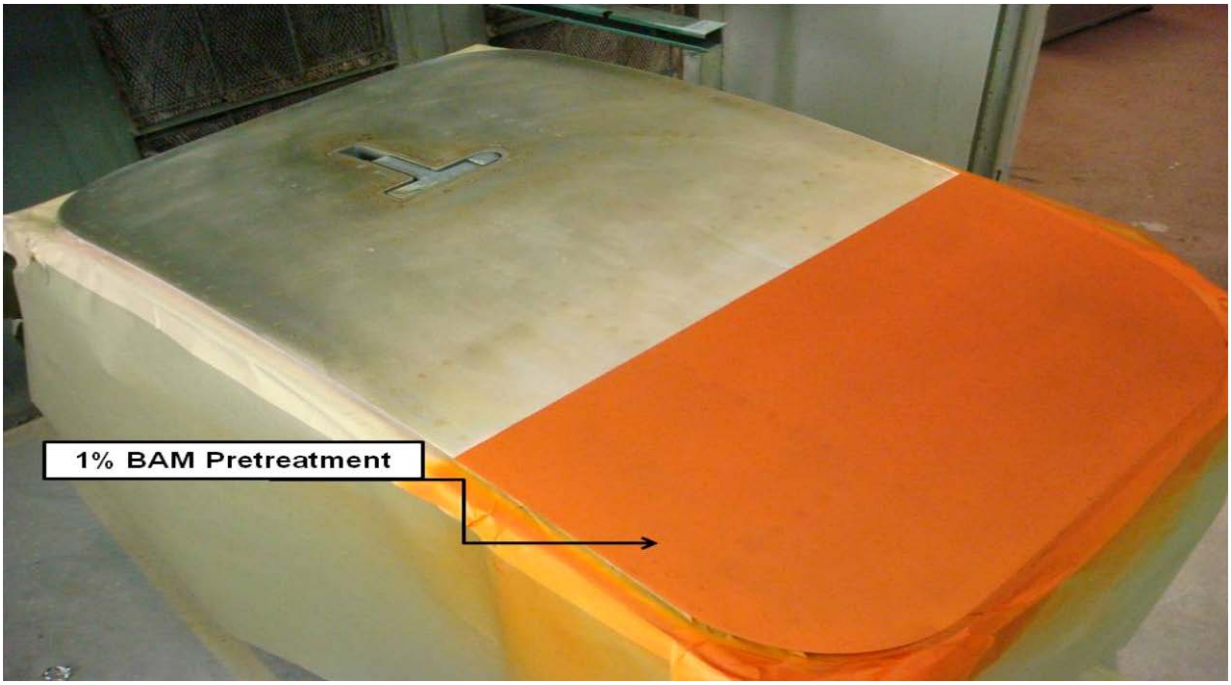
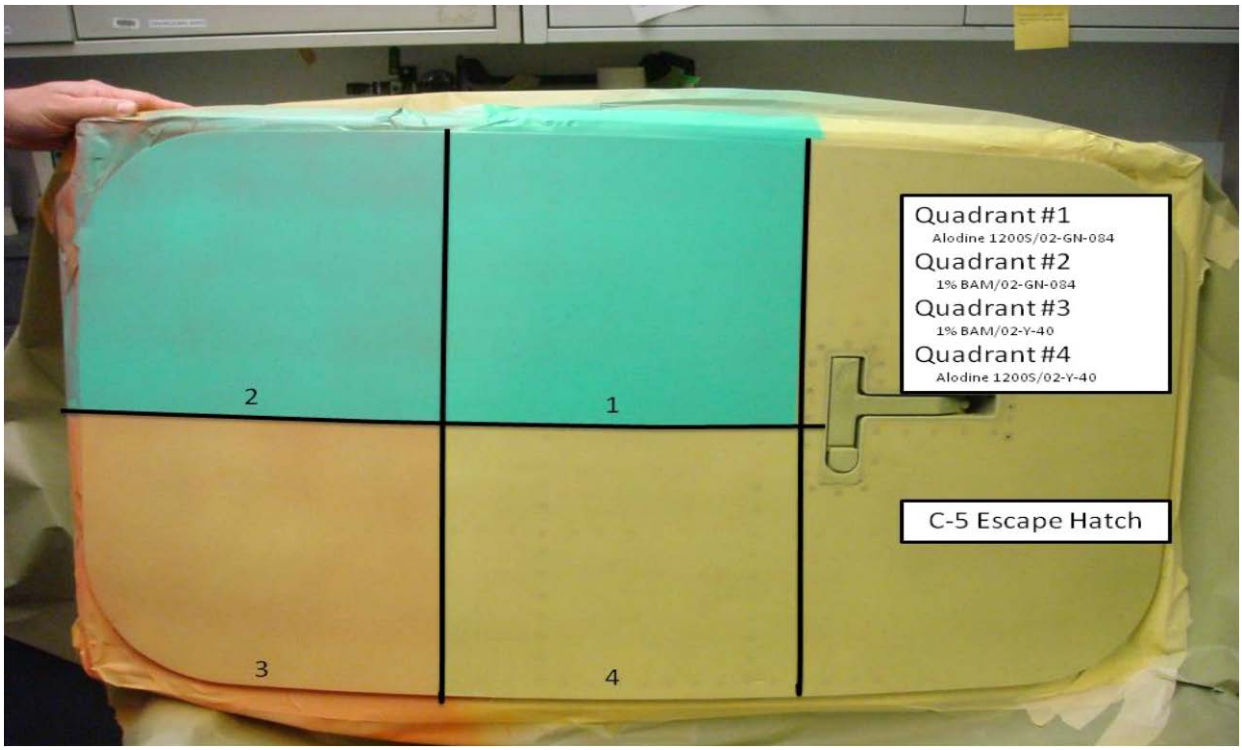


Figure 181: C-5 Rear Hatch Door Coated with 1wt. % BAM-PPV Pretreatment solution



Figures 182: C-5 Rear Hatch Door with Primer Coatings

Figure 183: C-5 Escape Hatch Door with Full Coating System

The coated door was flown for 12 months and examined periodically (visual inspection) every three months. The aircraft was flown under ambient conditions in the Midwest and Northeast regions of the United States with one overseas flight to Europe, giving a total of 296.7 flight hours. The conditions included down-time at military depots for routine maintenance and inspection. The C-5 cargo plane was exposed to normal weather conditions found in these regions of the United States which included rain, sleet, snow, northern coastal moisture, and sun. The door with the BAM-PPV pretreatments and controls survived the field demonstration intact without loss of adhesion or corrosion. No significant change in dry film thickness was observed during the 296.7 flight hours. The small changes that were observed were due to dirt build-up on the coatings. **The overall assessment of this BAM-PPV coating via visual inspection over a 12-month period showed no corrosion damage to the coating or delamination (see Table 131 and Figure 184).**

Figure 184: C-5 Test Door at 12 months (299.7 hours)

Table 131: Assessment of BAM-PPV Field Test on C-5 Cargo Aircraft Rear Hatch Door

Visual Inspection Test	**Performance at 12 months**
ASTM D 660 Degree of Checking of Exterior Paints	***No Damage***
ASTM D 661 Degree of Cracking of Exterior Paints	***No Damage***
ASTM D 662 Degree of Erosion of Exterior Paints	***No Damage***
ASTM D 714 Degree of Blistering of Paints	***No Damage***
ASTM D 772 Degree of Flaking of Exterior Paints	***No Damage***
ASTM D 1654 Evaluation of Painted Specimens Subjected to Corrosive Environments	***No Damage***
ASTM D 4214 Degree of Chalking of Exterior Paints	***No Damage***

6.4.1.1 Conclusion for Field Studies Conducted at WPAFB

The following conclusion can be made from field studies of BAM-PPV:

- ***Results from WPAFB field testing confirms that BAM-PPV military coating performs as well as a chromated military coating for a one-year field test without any visual appearance of failure of the BAM-PPV coating.***

6.4.2 Field Tests for BAM-PPV Pretreatment Coating Conducted at ARL

For the ARL field tests, AA 2024-T3 headlight cover (see Figure 185) was selected due to BAM-PPV showing acceptable laboratory performance results. The laboratory tests showed that for Army field applications a rough surface profile must be present in order for the BAM-PPV pretreatment to adhere to the AA 2024-T3 substrate. Also the BAM-PPV coating must have a film thickness > 2 μm using Oxsol-100 as solvent in order to show adequate performance. The Bradley vehicle headlight cover was sand blasted prior to coating to achieve the appropriate surface roughness. BAM-PPV was sprayed onto the substrate using HVLP spray equipment (Figure 186), coated with MIL-DTL-53022 non-Cr(VI) primer and top coated with MIL-DTL-64159 (waterborne chemical agent resistant coating,(CARC) polyurethane topcoat) (Figure 187). The vehicle was field tested on the ARL outdoor weathering test tract (ATC) in Maryland and exposed to rain, snow, sleet, sun, wind, coastal moisture and humidity. Visual inspections for corrosion, adhesion, color change, gloss, chalking, cracking, peeling and alligatoring were performed every three months. **After one year of field testing, no significant corrosion, no blistering near edges and no undercutting of the coating adjacent to chip sites was observed. This is considered acceptable performance for ARL field**

testing new pretreatment coatings with primers and topcoats. However, there was slight corrosion present in chip sites where all coatings were removed including BAM-PPV (Figures 188-189 and Table 132).

Figure 185: Headlight Cover Sand Blasted Prior to Coating with BAM-PPV

Figure 186: BAM-PPV Coated Headlight Cover

Figure 187: Headlight Cover Coated with MIL-DTL-53022 Primer and CARC Topcoat

Figure 188: Abrasion/Wear Damage after 1 Year Field Testing Headlight Cover

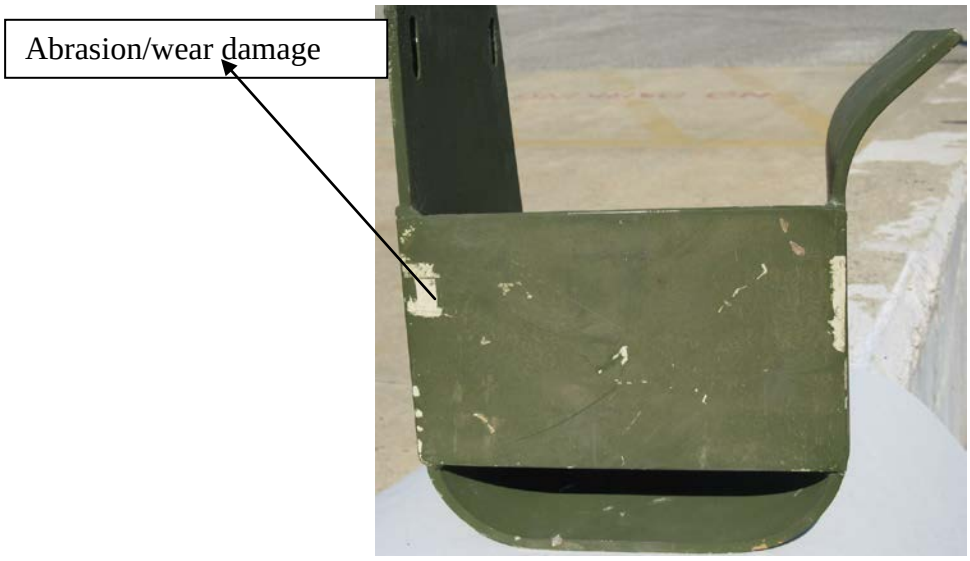


Figure 189: Abrasion/Wear Damage after 1 Year Field Testing (Backside View)

Table 132: Assessment of BAM-PPV Field Test on Headlight Cover of Bradley Vehicle

Visual Inspection Test	**Performance at 12 months**
ASTM D 523 Gloss Measurements	***No Damage***
ASTM E 308 Color Measurements	***No Damage***
ASTM D 1654 Evaluation of Painted Specimens Subjected to Corrosive Environments	***Limited corrosion damage to areas where all coatings were removed including BAM-PPV***
ASTM D 4214 Degree of Chalking of Exterior Paints	***No Damage***

6.4.2.1 Conclusion from Field Studies Conducted at ARL

- ***Results from ARL field testing confirm that BAM-PPV military coating provides acceptable field performance, after proper surface roughness is achieved to enhance the BAM-PPV pretreatment coating adhesion. Also BAM-PPV can be used as the depot level where rework of coatings is most critical and the surface profiles after grit blasting provides for BAM-PPV adhesion onto various substrates.***

6.4.3 Field Tests for BAM-PPV Pretreatment Coating Conducted at FRC-NW

Bomb hoists and towbars were used as non-critical military hardware for testing BAM-PPV pretreatment due to its acceptable performance in laboratory studies. The bomb hoists and towbars were pretreated via HVLP spray gun with BAM-PPV (coating thickness ~2 μm, dissolved in Oxsol-100), primed (MIL-PRF-23377N) and

topcoated with MIL-PRF-85285. The parts were put into service and monitored monthly via visual inspection for corrosion, chipping, abrasion and delamination of coating during normal operations. After 6 months of service, the BAM-PPV coated bomb hoists showed significant chipping and scratches (Figures 190-191). **There was rapid deterioration of the coating system and corrosion was evident in the chipped areas. This deterioration of the BAM-PPV pretreatment coating was unacceptable at 6 months and showed no improvement after 12 months. The performance of BAM-PPV was considered a failure during the field testing. This was due to unacceptable performance of the BAM-PPV coated bomb hoists and towbars (see Figures 192-193 and Table 133).**

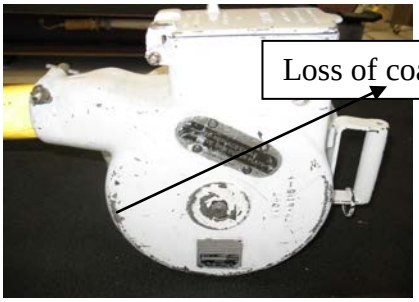


Figure 190: BAM-PPV coated bomb hoist prior to field testing (left)
BAM-PPV coated with MIL-PRF-23377N and topcoated with MIL-PRF-85285 after 6 months of field testing (right)

Figure 191: BAM-PPV coated towbars prior to field testing (left)
BAM-PPV coated with MIL-PRF-23377N and topcoated with MIL-PRF-85285 after 6 months of field testing (right)

Figure 192: BAM-PPV bomb hoist coated with MIL-PRF-23377N and topcoated with MIL-PRF-85285 after 1 year of field testing

Figure 193: BAM-PPV coated towbars with MIL-PRF-23377N and topcoated with MIL-PRF-85285 after 1 year of field testing

Table 133: Assessment of BAM-PPV Field Test on Bomb Hoists and Towbars

Visual Inspection Test	**Performance at 12 months**
ASTM D 660 Degree of Checking of Exterior Paints	***Failure ≤6 months***
ASTM D 1654 Evaluation of Painted Specimens Subjected to Corrosive Environments	***Failure ≤6 months***
ASTM D 4214 Degree of Chalking of Exterior Paints	***Failure ≤6 months***
ASTM 661 Degree of Cracking of Exterior Paints	***Failure ≤6 months***
ASTM 772 Degree of Flaking of Exterior Paints	***Failure ≤6 months***

6.4.3.1 Conclusions from Field Studies Conducted at FRC-NW

- ***Results from FRC confirm that BAM-PPV coated onto bomb hoists and towbars <u>does not</u> provide acceptable adhesion or corrosion inhibition after 6 months of field studies.***

- ***This may be the result of improper surface preparation resulting in the BAM-PPV pretreatment failing to adhere onto the metal surface or to shelf-life issues with BAM-PPV.***

7.0 COST ASSESSMENT

7.1 Cost Model

If EAPs are approved for use by military organizations, the cost to implement within the AF and Army would be minimal (see Table 134). This is because the EAP material can be applied via HVLP, the primary system utilized by AF, Army and Navy maintenance operations. The need for extensive PPE, hazardous waste removal, employee monitoring, and other expenses associated with CCC do not exist with the EAP-based coating systems. There are no storage stability issues with EAPs and this does not deter the implementation of this system in place of CCC. Packaging and availability of the final product will need to be determined by the vendor who will receive this technology.

As there are no costs associated with equipment change for application of the EAPs any cost increase would be due to increased preparation time (mixing the EAP), change in application time. There would be significant cost saving from elimination of hazardous waste disposal costs and savings in depainting costs because EAPs are Cr(VI)-free.

Table 134: Determination of Cost Drivers for EAP Field Testing

Baseline Matrix	Cost Factors
Equipment Purchases	**No cost increase** associated with using BAM-PPV solutions (HVLP)
Personnel Protective Equipment	**No cost increase** associated with using BAM-PPV solutions
Permit Requirements	**No additional permits** are required for this material
Training	**Training costs increases would be minimal** using BAM-PPV solutions with exiting HVLP spray equipment
Waste Stream	**No hazardous materials generated** with BAM-PPV solutions or removal processes

7.2 Cost Analysis and Comparison

The cost drivers for the manufacture of BAM-PPV will be dependent on obtaining a manufacturer of this material. BAM-PPV has only been synthesized at the kilogram level by scientists at the NAWCWD and through an SBIR contract (ONR sponsored) with Spectra Group Ltd, Toledo, OH. Multiple kilogram quantities have been prepared during the previous SERDP program and completed ESTCP program. The costs associated with preparing multiple kilogram quantities based on the synthetic process developed at NAWCWD is ~ $10.00/gram. The improvement in the synthesis of BAM-PPV by Spectra Group Ltd will reduce the cost of manufacturing BAM-PPV. Spectra Group Ltd has provided NAWCWD with estimated costs on preparing the monomer for BAM-PPV. The immediate precursor (monomer) - the ammonium salt dichloride costs about $20/g to make 100 grams ($2,000). Since the price drops the more you make, 500 grams would cost approximately $13.40/gram and for 10 kg it would be approximately $6.02/gram (see Table 135 for more details).

Table 135: Estimated Cost of Making BAM-PPV

Amount of Scale grams	Estimated Price/gram
100	$20/gram
500	$13.40/gram
2,500	$8.98/gram
10,000	$6.02/gram

Both NAWCWD and Spectra Group Ltd used recovered solvents. Almost all the reactions can be done using the recovered solvent and reuse for the same reaction. Reactions at NAWCWD and Spectra Group Ltd. were done on a 10 and 22 liter scale, respectively, distilling the used solvent, and then using the recycled solvent for repeating the reaction. There were no ill effects from using recycled solvent during any of the

synthetic steps used to make BAM-PPV. This process could be easily scaled-up to a manufacturing level and several manufacturers of specialty chemicals could use reactors that hold up to greater than 10,000 liters. We believe that there would be no problems with doing any of the reactions to produce the BAM-PPV on a larger scale.

8.0 IMPLEMENTATION ISSUES

8.1 Environmental Checklist

Removal of chromates from the pretreatment process would allow for improved compliance with 29 CFR 1910.1026 which calls for a permissible exposure limit (PEL) on Cr(VI) of 5 micrograms per cubic meter, TWA with an action level of 2.5 micrograms per cubic meter TWA. This should not be an issue since the substitution involves trading one high VOC (albeit with exempt solvent) pretreatment for another high VOC pretreatment containing a known human carcinogen that has prohibitive PELs and restrictive disposal options. The EAP coatings for the each facility will be applied by the appropriate coatings group and each depot has the appropriate safety permits and EPA reporting requirements to implement this technology.

The only implementation issue that each co-performer identified was the number of passes that BAM-PPV requires for appropriate thickness.

Currently CCC, TCP and Prekote require only:

- **two passes to produce adequate thickness for pretreatment coatings,**
- **BAM-PPV requires between 7-9 passes for adequate thickness.**

This will be a problem for implementation as it will increase time and costs for applying BAM-PPV onto various metal substrates.

8.2 Future Plans

Currently a potential joint venture with Dr. Fredrik von Kieseritzky, CEO, Arubedo AB, Sigtunagatan 10, 113 22 Stockholm, Sweden (fvk@arubedo.com, 46 70 751 5353) and NAWCWD has been initiated during December 2012. The research group under Dr. von Kieseritzky, is trying to develop a solvent system that will improve the solubility of BAM-PPV during coating processes. No Cooperative Research and Development Agreement (CRADA) have been signed between our two groups but we are pursuing potential funding from European sources.

8.3 Regulatory Issues

It is not anticipated that there will additional regulatory issues involved since BAM-PPV is non-hazardous, the solvent is VOC-exempt, and the coatings de-paint and application activities will be performed in a permitted area where proper approvals from base Bioenvironmental already exist.

8.4 End-user/Original Equipment Manufacturer (OEM) Issues

Because BAM-PPV can be applied using HVLP, no special equipment would be necessary; therefore, this technology can be used as a drop-in replacement for CCCs with no change in equipment required.

Potential end users include all facilities that apply the polyurethane topcoats, CARC system that is not an OEM. With a successful outcome of this demonstration, the end user concerns should be minimal. The product is a drop-in replacement that does not require special equipment for application and needs only an adjustment in application procedure (number of passes increase). The concern that may negatively affect stakeholder buy-in is the number of passes required to apply the BAM-PPV solution.

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2. P. Zarras, *Organic-Polymeric Coatings As Replacements for Hexavalent Chromium Based Coatings: A Tutorial*, ACS Polymer Preprints, August 28-September 1, 2011, Denver, CO.
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APPENDIX A: POINTS OF CONTACT

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Amy Fowler	NAWCAD, Code 4.3.4.2 48066 Shaw Road Bldg 2188 Patuxent River, MD 20670-1906	(P) 301-342-0986 (F) 301-342-7566 Amy.Fowler@navy.mil	Coating management
Christopher S. Mahendra	Naval Air Warfare Center Aircraft Division Lakehurst Rte 547 South 486JB562-2 Lakehurst, NJ 08733-5060	(P) 732-323-7131 (F) 732-323-4025/4917 christopher.mahendra @navy.mil	Coating management
Christopher E. Miller	Army Research Laboratory Coatings and Corrosion Building 4600 ARSRD-ARL-WM-SG Aberdeen Proving Ground, M21005-5069	(P) 410-306-0849 (F) 410-306-0613 cemiller@arl.army.mil	Coating management
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Mark R. Kolody	ASRC Applied Technology NASA Corrosion Technology Laboratory MS ASRC-24 Kennedy Space Center, FL 32899	(P) 321-867-6659 Mark.r.kolody@nasa.gov	Corrosion Monitoring
Dan Cerven	MB Research Laboratories 1765 Wentz Road Spinnerstown, PA 18968	215-536-4110	Toxicology Testing

APPENDIX B: MATERIALS AND METHODS

Appendix B1.0 Materials and General Analytical Methods

The monomer, (2,5-bis(chloromethyl)-4-(hexamethylamino)-phenyl)hexamethylamine dihydrochloride) was prepared according to published work [57 -65, 72]. BAM-PPV polymer was ground to a fine powder using a commercial coffee grinder. BAM-PPV solutions were prepared and thin films of BAM-PPV were coated onto aluminum substrates (Al 2024-T3) using an airbrush technique (film thickness approximately ~1.5-2.0 μm). *o, p*-Xylenes and 4-chlorobenzotrifluoride (Oxsol-100) (Volatile organic compounds , VOC-exempt) were used as the solvents to dissolve BAM-PPV. The solvents were heated with 1-3 wt% of the BAM-PPV powder added to the solvents with gentle heating for 3 days. After 3 days the solution was homogenous and the solution was filtered through course filter paper and the filtrate kept for future use. Acetonitrile, methanol, toluene, tetrahydrofuran (THF), potassium *t*-butoxide (K-*t*-OBu) were obtained from Aldrich Chemical Co. and used without further purification. N, N-dimethylformamide (DMF), 99% purchased from Acros Organics and used as received.

Differential scanning calorimetry (DSC) was performed using a TA Instruments 2910 Differential Scanning Calorimeter at a heating rate of 10°C/min under nitrogen. Thermogravimetric analysis (TGA) was performed using a TA Instruments 2950 TGA scanning at 5°C/min (under nitrogen and air).

The melting points (mp) of synthesized compounds were collected on an electrothermal capillary melting point apparatus (Melt-Temp) and are not corrected.

^{1}H and ^{13}C NMR data was acquired using a Bruker Advance 400MHz NMR spectrometer at 300K. The FTIR spectrum was collected using a Nicolet Nexus 870 FTIR spectrometer with a liquid nitrogen cooled MCT detector. The spectrum is an average of 100 scans with 4 cm^{-1} resolution. The polymer film was placed in contact with a Germanium crystal on a "Thunderdome" attenuated total reflectance (ATR) accessory. Gel-Permeation Chromatography (GPC) was done at 1.5mL/min using a Jordy mixed-bed column at 20°C in THF using polystyrene standards. Elemental analysis (EA) was performed by Atlantic Microlab, Inc. (Norcross, GA).

Appendix B1.1 Polymerization of Monomer and Characterization of BAM-PPV Polymer

The polymerization of the monomer was performed in a 5L reactor with a mechanical stirrer (Figure 194). The reactor was placed inside a large stainless steel secondary container to allow cooling. Toluene (2L) and THF (2L) was added to the reactor. Dry ice was added to the secondary container with acetonitrile to cool the reactor to -45°C. While the solvent was cooling, 100-150g of monomer was added. When the temperature reached -45°C, 8 molar equivalents of potassium *t*-butoxide (K-*t*-OBu) was added (1 mol monomer/8 mol K-*t*-OBu). The temperature was kept between -45°C and -55°C for 2 hours. The solution was then allowed to warm to room temperature overnight. After 18 hours, the reaction mixture was found to be a very viscous orange, semi-gelatinous material. The polymer was precipitated by pouring this material into

methanol, filtered through a fine glass frit, and dried under vacuum at 100°C overnight. After drying, the polymer was placed in a soxhlet thimble and extracted with methanol to remove oligomers and some remaining potassium chloride salts, yielding an orange-yellow powder in 93% yield. The extraction was followed by rinsing with methanol, filtering and drying to constant weight. The crude polymer was mechanically agitated with vigorous stirring using De-ionized water (DI water): methanol (90:10, v/v) as extractant for one week to remove any remaining potassium chloride (KCl) salts. The suspension was filtered and the powder dried for one day under vacuum (25°C, 0.5 mmHg). The ^{1}H NMR (400 MHz, d_8-toluene) and the ^{13}C NMR (400 MHz, d_8-toluene) are shown in Tables 136 and 137 respectively. The FTIR spectrum of BAM-PPV is provided in Figure 195 and the peak positions and assignments are shown in Table 138. The FTIR spectrum is consistent with the proposed polymer structure. A polymer molecular weight (M_w) of ~85,000 and (M_n) of ~67,000 was observed using gel permeation chromatography (GPC) against polystyrene standards. The EA calculated for BAM-PPV are C: 80.43, H: 11.05, N: 8.53, Cl: 0.00. The EA values obtained by Altlantic Microlab are as folows: C: 79.77, H: 11.05, N: 8.53, Cl: trace, <0.25%. The EA was measured for total Cl on all BAM-PPV samples to insure complete removal of the KCl salts formed during polymerization. The purity of BAM-PPV was determined by EA to be > 99%. The glass transition temperature (T_g) for BAM-PPV was measured at 9.0^{o}C. Thermogravometric analysis, TGA showed that initial decomposition begins at 300°Cfor BAM-PPV. The 5% weight loss temperature is about 387°C in nitrogen and 320°C in air (heating rate 10°C/minute).

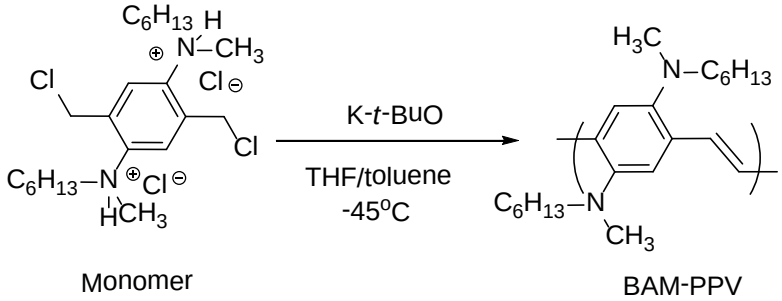

Figure 194: BAM-PPV Polymerization

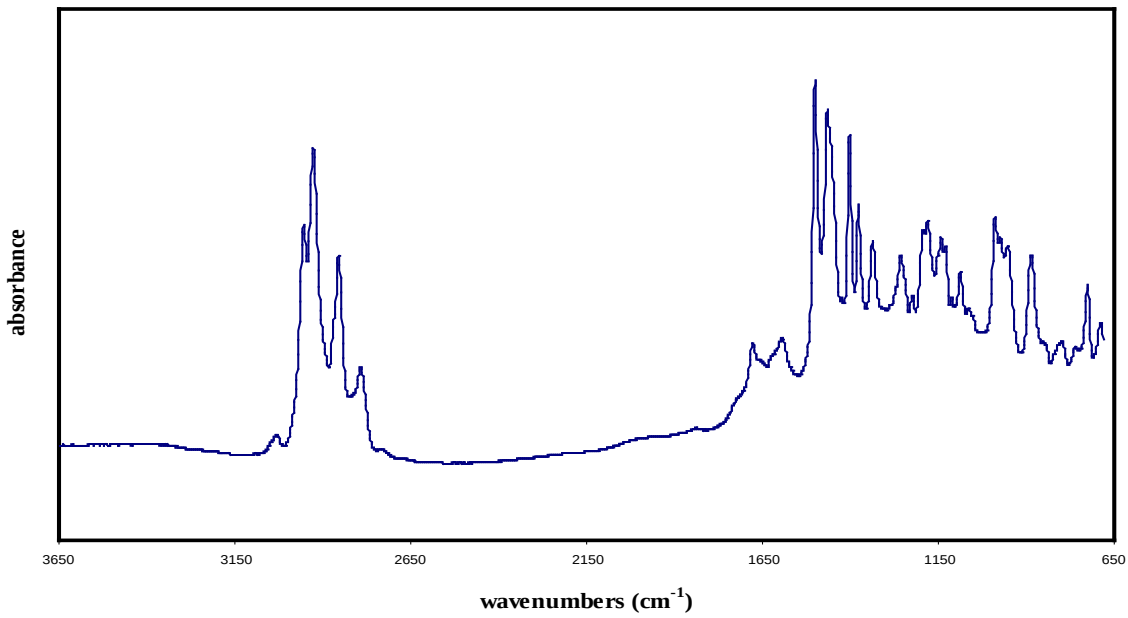

Figure 195: FTIR spectrum of BAM-PPV film

Table 136: ^{1}H NMR (400 MHz, δ ppm, d_8-toluene) of BAM-PPV

Peak Shift (ppm)	Assignment
0.88	$N(CH_2)_5C\underline{H}_3$
1.27	$N(CH_2)_3C\underline{H}_2$ and $N(CH_2)_4C\underline{H}_2$
1.37	$N(CH_2)_2C\underline{H}_2$
1.70	$NCH_2C\underline{H}_2$
2.80	$NC\underline{H}_3$
3.04	$NC\underline{H}_2$
7.78	Ar
8.01	alkenyl

Table 137: ^{13}C NMR (400 MHz, δ ppm, d_8-toluene) of BAM-PPV

Peak Shift	**Assignment**
14.3	$N(CH_2)_5\underline{C}H_3$
23.2	$N(CH_2)_4\underline{C}H_2$
27.5	$N(CH_2)_2\underline{C}H_2$
28.3	$NCH_2\underline{C}H_2$
32.3	$N(CH_2)_3\underline{C}H_2$
42.2	$N\underline{C}H_3$
57.9	$N\underline{C}H_2$
118.2	$\underline{C}_{Ar}$-H
126.0	$\underline{C}_{alkenyl}$
133.9	$\underline{C}_{Ar}$-$C_{alkenyl}$
148.4	$\underline{C}_{Ar}$-N

Table 138: FTIR Peaks and Assignments for BAM-PPV Polymer Film

Vibrational peaks and proposed peak assignments of BAM-PPV polymer film

peak position (cm^{-1})		assignment	peak position (cm^{-1})		assignment
3032	w	aromatic C-H stretch	1257	m	aromatic C-N stretch
2954	s	asym. methyl C-H stretch	1224	w	
2927	vs	asym. methylene C-H stretch	1192/1179	doublet	C-N aliphatic stretch
2869	sh	sym. methyl C-H stretch	1141	m	
2855	s	sym. methylene C-H stretch	1127	m	
2792	m	C-H stretch (C bonded to N)	1111	w	
1678	w	vinyl C=C stretch	1087	m	
1593	w,b	aromatic C=C stretch	989	s	C-H out of plane on vinyl group
1500	vs	aromatic skeletal vib.	973	sh	
1465	s	asym. methyl def.	953	sh,s	
1455	sh	CH_2 scissor	886	s	C-H out of plane on 1,2,4,5 sub. phenyl
1424	w	N-CH_3 sym. methyl bend	797	w	
1403	s		725	m,s	CH_2 rock
1377	s	sym. CH_3 bend	687	m	
1337	m				

w=weak, m=medium, s=sharp, vs=very sharp, sh=shoulder, b=broad

Appendix B.1.2 Improvement in the Synthesis of the Monomer 1,4-Bis(chloromethyl)-2-(2-ethylhexyloxy)-5-methoxybenzene to obtain Poly[2-(2-(2-ethylhexyloxy)-5-methoxy-*p*-phenylene vinylene] (MEH-PPV)

The typical conditions for the alkylation of 4-methoxyphenol with 2-ethylhexyl bromide are refluxing in ethanol with potassium hydroxide as base [95]. By using these conditions, the reaction took days to complete. A much faster alkylation was found to take place by using a polar, aprotic solvent and a strong, non-nucleophilic base such as *N*,*N*-dimethylformamide and potassium *tert*-butoxide [73]. To make the reaction go to completion, a 1.75 fold excess of potassium *tert*-butoxide and 2-ethylhexyl bromide were added. In this way, the reaction went to completion in three hours at two mole scale. It is believed that a portion of the 2-ethylhexyl bromide reacts with the base, undergoing dehydrohalogenation and producing an unknown amount of 2-ethylhexene by-product. The Structure 1 (see Figure 196) is recovered in 97 % yield from the aqueous work-up

and analytically pure by reduced pressure distillation. The standard conditions for chloromethylation include saturation of the reaction mixture with gaseous hydrogen chloride and/or multiple additions of excess paraformaldehyde and aqueous hydrochloric acid [96]. A serious hazard of chloromethylation under these conditions is the formation of toxic bis(chloromethyl)ether (BCME) [97]. It was found that running the chloromethylation of Structure 1 (Figure 196) in a mixture of concentrated hydrochloric acid and acetic acid with only four equivalents of paraformaldehyde gave the bis-chloromethylated, Structure 2 (Figure 196) in just six hours at 80 °C. Although we did not analyze the reaction for BCME, it is most likely present. However, since large excesses of hydrochloric acid and paraformaldehyde were not used, the amount of BCME generated is minimized. The chloromethylation reaction is accelerated due to the acetic acid co-solvent. The reaction is a two-phase system with the upper phase consisting of the reactant/product and acetic acid. The reaction progress was measured by proton nuclear magnetic resonance taking an aliquot of the upper phase. The crude product precipitates upon cooling the reaction to ambient temperature but was difficult to filter in this state, so an extractive work-up was necessary. Two recrystallizations from ethanol gave Structure 2 in analytically pure form in 63 % yield with a mp = 45-48°C.

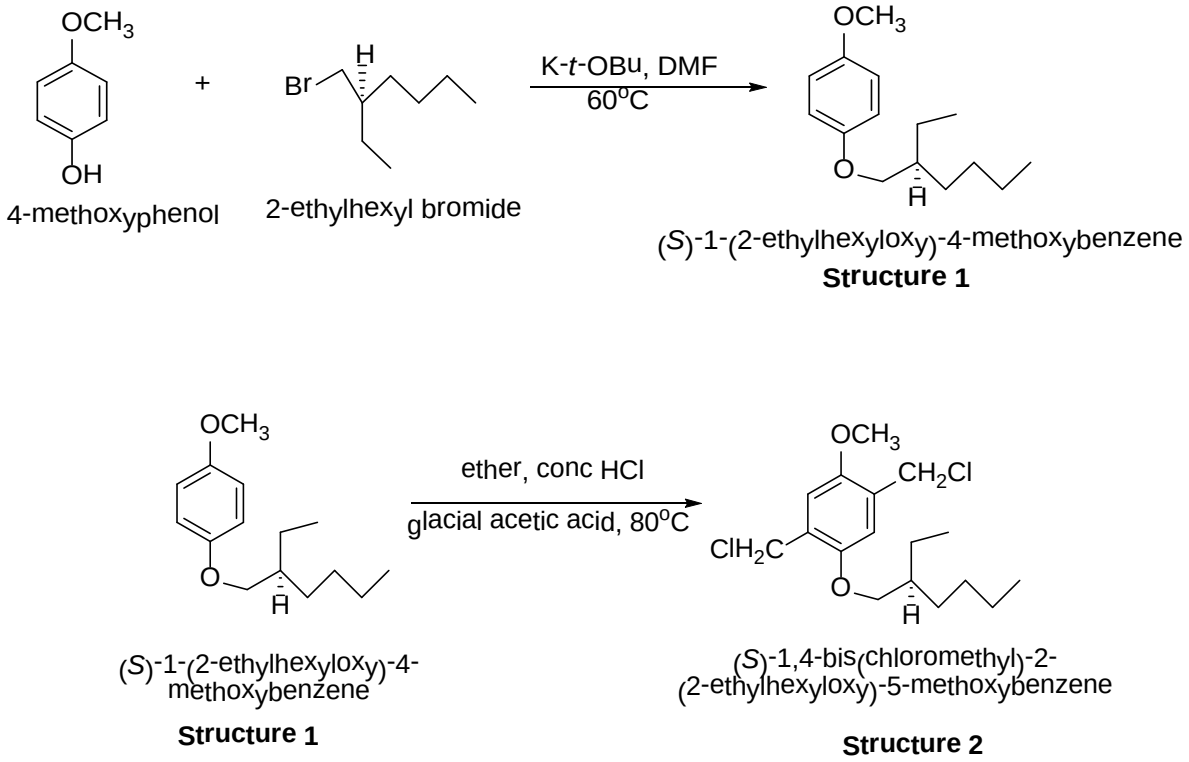

Figure 196: Synthesis of (S)-1,4-Bis(chloromethyl)-2-(2-ethylhexyloxy)-5-methoxybenzene (Structure 2)

Appendix B 1.3 Synthesis of racemic 1-(2-Ethylhexyloxy)-4-methoxybenzene (Structure 1)

A 3L 3-necked, round-bottomed flask equipped with mechanical stirrer and thermometer was charged with 248 g 4-methoxyphenol (2 mol) and 1L DMF. The mixture was stirred until all the solids dissolved. In one portion, 224 K- *t*-OBu (2 mol, 1 equiv) was added. After 30 minutes, all the solids had dissolved and the mixture was a greenish colored solution and the internal temperature was 50 °C. An addition funnel was equipped and charged with 386 g 2-ethylhexyl bromide (2 mol, 1 equiv) which was run into the reaction over 45 minutes. Solids began to precipitate shortly after the start of the addition and the temperature peaked at 60 °C. After 1h, the temperature had cooled to 50 °C and 112 g K-*t*-OBu (1 mol, 0.5 equiv) was added to the reaction. The temperature again rose to 60 °C. The addition funnel was again charged with 193 g 2-ethylhexyl bromide (1 mol, 0.5 equiv) which was run into the reaction over 30 minutes. The internal temperature reached 65 °C during the addition. After 1 hour, a final addition was made of 56 g K-*t*-OBu (0.5 mol, 0.25 equiv) followed by 96.5 g 2-ethylhexyl bromide (0.5 mol, 0.25 equiv) in similar fashion. After stirring for 3 hours, the internal temperature was near room temperature and thin layer chromatography (TLC) showed all the phenol was consumed. Water (1L) was added to the mixture and all the solids were allowed to dissolve. The mixture was extracted three times with 1 L portions of ether. The organic extracts were collected and washed once with 500 mL water, followed by 500 mL brine. The organic phase was dried over 20 g anhydrous magnesium sulfate, filtered and rotary evaporated leaving a slightly brown liquid. The crude product was distilled at 0.1 torr, discarding all volatiles < 90 °C, which contained residual 2-ethylhexyl bromide, to give the title compound (bp 115 °C, 0.1 torr) as a colorless liquid (461.5 g, 97%). The ^{1}H NMR (400 MHz, $CDCl_3$) and the ^{13}C NMR (400 MHz, $CDCl_3$) analysis are shown in Tables 139 and 140 respectively. The EA was calculated for $C_{15}H_{24}O_2$ to be the following: C: 76.23; H: 10.24. The EA analysis performed by Atlantic Microlabs found the following: C: 76.21; H: 10.42.

Table 139: ^{1}H NMR Analysis (400 MHz, δ ppm, $CDCl_3$) of Structure 1

Peak Shift (ppm)	Mutiplicity	Integration	Assignment
0.92-0.82	m	6H	$CH_2CH_2C\underline{H}_3$ and $CH_2C\underline{H}_3$
1.53-1.2	m	8H	$C\underline{H}_2CH_3$ and $C\underline{H}_2C\underline{H}_2C\underline{H}_2CH_3$
1.65	m	1H	$CH_2C\underline{H}CH_2$
3.72	t	3H	O-$C\underline{H}_3$
3.74	d	2H	Ar-O-$C\underline{H}_2$-
6.79	s	4H	Ar-$\underline{H}$

S = singlet, m= mutiplet, d = doublet and t = triplet

Table 140: ^{13}C NMR Analysis (400 MHz, δ ppm, $CDCl_3$) of Structure 1

Peak Shift	Assignment
11.32	$CH_2\underline{C}H_3$
14.29	$CH_2CH_2\underline{C}H_3$
23.29	$CH_2\underline{C}H_2CH_3$
24.10	$\underline{C}H_2CH_3$
29.33	$CHCH_2\underline{C}H_2CH_2CH_2$
30.77	$CH\underline{C}H_2CH_2CH_2CH_3$
39.71	$CH_2\underline{C}HCH_2$
55.96	$O\text{-}\underline{C}H_3$
71.47	$O\text{-}\underline{C}H_2CHCH_2CH_2CH_2$
114.83	$\underline{C}_{Ar}O\text{-}CH_3$
115.67	$\underline{C}_{Ar}O\text{-}CH_2CHCH_2$
153.86	$\underline{C}_{Ar\text{-}quarternary}$

Appendix B.1.4 Synthesis of racemic 1,4-Bis(chloromethyl)-2-(2-ethylhexyloxy)-5-methoxybenzene (Structure 2)

Warning! The following reaction must be conducted in a well-ventilated fume hood.

A 3L round-bottomed flask equipped with mechanical stirrer was charged with 461.5 g **Structure 1** (1.96 mol), 891 mL glacial acetic acid and 235.2 g paraformaldehyde (7.84 mol, 4 equiv). The mixture was stirred while 1.1 L conc. HCl was added. An endotherm of 3 °C was observed. A heating mantle was equipped and the mixture was heated and stirred at 80°C. Shortly after heating, all the solids dissolved. After 6 h, virtually all the starting material had been consumed by ^1H NMR and the heating mantle was removed. During cooling to ambient temperature, copious white solids precipitated from solution. The mixture was extracted three times with 1.25 L portions of hexanes. The combined extracts were washed twice with 1 L portions of water followed by two washings with 500 mL portions of brine. The organic phase was dried over 20 g anhydrous magnesium sulfate, filtered and rotary-evaporated, leaving 630 g of a pale yellow liquid that would slowly solidify at ambient temperature into a waxy, white solid. The crude product was kept warm and liquid, and transferred to a 5L round-bottomed flask equipped with mechanical stirrer containing 2 L ethanol. The mixture was stirred and heated until complete dissolution and then allowed to stir until the mixture reached room temperature. The white precipitate was filtered on a coarse porosity (145-175 μm) glass frit. Recrystallization again from 2 L ethanol and drying (30 °C, 10 torr) overnight gave 403 g (1.2 mol, 63 %) of the desired compound as a soft, snow-white powder. mp 45-48 °C. The ^1H NMR (400 MHz, $CDCl_3$) and the ^{13}C NMR (400 MHz, $CDCl_3$) analysis are shown in Tables 141 and 142 respectively. The EA was calculated for $C_{17}H_{26}Cl_2O_2$ as follows: C: 61.26; H: 7.86. The EA analysis performed by Atlantic Microlabs found the following: C: 61.39; H: 7.86.

Table 141: ^{1}H NMR Analysis (400 MHz, δ ppm, $CDCl_3$) of Structure 2

Peak Shift (ppm)	**Mutiplicity**	**Integration**	**Assignment**
0.86	t	3H	$CH_2CH_2C\underline{H}_3$
0.89	t	3H	$CH_2C\underline{H}_3$
1.21-1.32	m	4H	$C\underline{H}_2C\underline{H}_2CH_3$
1.33-1.56	m	4H	$CHC\underline{H}_2CH_2CH_3$ and $C\underline{H}_2CH_3$
1.69	m	1H	$CH_2C\underline{H}CH_2$
3.79	s	3H	$O\text{-}C\underline{H}_3$
3.82	d	2H	$OC\underline{H}_2CH$
4.51	s	2H	$C\underline{H}_2Cl$
6.86	s	1H	Ar-<u>H</u>
6.87	s	1H	Ar-<u>H</u>

Table 142: ^{13}C NMR Analysis (400 MHz, δ ppm, $CDCl_3$) of Structure 2

Peak Shift	Assignment
11.45	$CH_2\underline{C}H_3$
14.29	$CH_2CH_2\underline{C}H_3$
23.27	$CH_2\underline{C}H_2CH_3$
24.27	$\underline{C}H_2CH_3$
29.35	$CH_2\underline{C}H_2CH_2CH_3$
30.87	$CH\underline{C}H_2CH_2CH_2CH_3$
39.86	$CH_2\underline{C}HCH_2$
41.54	$O\underline{C}H_2CHCH_2$
41.55	$\underline{C}H_2Cl$
56.56	$O\underline{C}H_3$
113.65	$\underline{C}$-$_{Ar}$
114.31	$\underline{C}$-$_{Ar}$
127.10	$\underline{C}$-$_{Ar\ quarternary}$
127.29	$\underline{C}$-$_{Ar\ quarternary}$
151.14	O-$\underline{C}$-$_{Ar\ quaternary}$
151.21	O-$\underline{C}$-$_{Ar\ quarternary}$

Appendix B 1.5 Polymerization of 1,4-Bis(chloromethyl)-2-(2-ethylhexyloxy)-5-methoxybenzene to obtain MEH-PPV

A 5L reaction vessel was charged with 2.0 L THF and 62.9 grams monomer (Structure 2). After monomer added, reaction mixture stirred via mechanical stirrer under positive nitrogen blanket for 30 minutes. The reaction flask was placed in a dry ice bath maintained at -40°C for 1 hour. 128 gram of K-*t*-OBu was added at this point and the contents stirred for 1 hour under a positive nitrogen blanket at -40°C. After 1 hour, 300 mL THF was added to the solution. At this point, the solution is brick red in color. The polymer suspension is precipitated into an excess of methanol. The gel-like pieces are broken-up using a coffee grinder. After grinding the crude MEH-PPV, the material is rewashed using an excess of methanol and filtered through a medium frit. The crude polymer is extracted in a soxhlet apparatus using methanol as the extractant for 5 days, after which time, it is dried and finally extracted using DI water for several days. The polymer is dried under vacuum (25°C, 0.5 Torr) to constant weight to give yields between 60-70%. (see Figure 197). The EA calculated for MEH-PPV is C: 78.21, H: 10.21, O: 11.58 and Cl: 0.00, found: C: 78.12, H: 9.39, O: not determined and Cl: trace <0.25%. The polymer is dissolved in *o,p*-xylenes and/or Oxsol-100 as described for BAM-PPV and applied the same way.

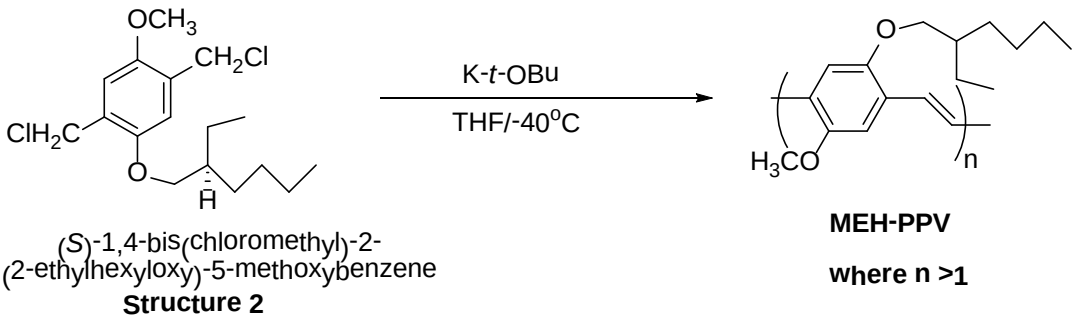

Figure 197: MEH-PPV polymerization