Corrosion Measurement and Control

Joint Test Protocol (JTP) J-03-MAG-001-P3

for

Validation of Corrosion Protection for Magnesium Alloys

July 5, 2004

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ACRONYM LIST

US Army Armament Research Development and

AMCOM US Army Aviation and Missile Command

- **Engineering Center** ARL US Army Research Laboratory ASTM American Society for Testing and Materials A2LA American Association for Laboratory Accreditation BP **Best Performance** Chemical Agent Resistant Coating CARC CM&C **Corrosion Measurement and Control** CPC Corrosion Preventative Compounds CTC Concurrent Technologies Corporation DoD Department of Defense Rockwell B Hardness Number HRB HV Vickers Hardness Number IP Improved Performance JTP Joint Test Protocol JTR Joint Test Report MP Minimum Performance NAVAIR US Naval Air Systems Command NLT Not Less Than NVLAP National Voluntary Laboratory Accreditation Program Product Failure Laboratory PFL Saturated Calomel Electrode SCE US United States
- USAF US Air Force

ARDEC

PREFACE

This document was prepared by Concurrent Technologies Corporation (*CTC*) under Contract Number N65236-02-D-3826. This document was prepared on behalf of, and under guidance provided by, the Corrosion Measurement and Control (CM&C) Program. The structure, format, and depth of technical content of the document were determined by the CM&C technical associates, pertinent United States (US) Army personnel, government contractors, and other government technical representatives (hereafter referred to as "the stakeholders") in response to the specific needs of this project.

We wish to thank the technical stakeholders involved in the creation of this document for their invaluable contributions:

US Air Force Research Laboratory (AFRL) US Army Corrosion Office US Army Research Laboratory (ARL) US Army Aviation and Missile Command (AMCOM) US Naval Air Systems Command (NAVAIR)

We also wish to thank the contributors involved in the creation of this document for their invaluable contributions:

Sikorski Aircraft Corporation Sermatech/Teleflex Inc.

JTP Revisions History

This section will serve as a means to document revisions and discussions regarding this JTP. It is intended to help the reader identify updated versions of the JTP, and to organize periodic updates of the JTP as new materials and techniques become available.

Revision Letter	Pages Changed	Pages Added	Description	Date	Approved By (signature/division)
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1.0 INTRODUCTION

Magnesium has been a material of interest within the engineering design community for many years. Magnesium imparts good mechanical properties and specific fatigue strength, while providing low density and high strength-to-weight ratios. However, the relatively poor corrosion performance of magnesium alloys is a detriment to their consideration for many applications. Magnesium is an extremely electrochemically active metal, occupying high positions in both the electromotive force series and the galvanic series for seawater. It is anodic to all other structural metals and will corrode preferentially when coupled with virtually any other metal in the presence of an electrolyte. Untreated magnesium does form a protective oxide layer, but this oxide is not stable in acidic or even neutral pH ranges. It is therefore necessary to employ protective coatings, surface treatments, and other technologies in order to enhance the corrosion performance of magnesium in service, particularly in harsh environments.

This Joint Test Protocol (JTP) contains the critical corrosion performance requirements and tests necessary to qualify protective technologies for consideration for use on magnesium alloy components to be used on US military vehicles and weapons systems. This document is an all-inclusive protocol for testing and assessing the corrosion performance of magnesium alloys and manufacturing processes, as well as any coatings, surface treatments, sealants, assembly compounds, etc. that would be applied to these magnesium components. These potential approaches will hereafter be referred to as "candidates."

This document outlines the corrosion performance tests necessary to qualify potential candidates, either for use in place of existing components utilized on current US military assets or for use in new designs and applications for future materiel systems. A general description of the qualifying tests and the rationale behind their selection are presented. The developmental logic for the necessary criteria and the details of the selected tests are outlined.

1.1 Scope

The magnesium protection technologies to be evaluated under this document include:

- New or modified component manufacturing processes (such as sand, die, gravity, high/low pressure, or mold castings, extrusions, etc.)
- New magnesium alloys
- New pretreatment processes
- New coatings (preprimers, primers, topcoats)
- New surface treatments and sealants

This JTP establishes *only* the corrosion performance requirements that must be met for a candidate to be considered for use on military vehicles and weapons systems. In actual applications, other physical and mechanical properties of potential candidates must and will be considered (please see Feasibility Study

discussion in Section 1.2). However, the evaluation of these properties is specific to the particular application, and will be considered to be outside the scope of this Magnesium Corrosion Protection JTP.

It must be emphasized that this JTP document is not a process, material, or product specification, nor is it intended to address ongoing quality issues. The testing outlined in this document has, as its principal purpose, the evaluation of candidates for consideration for use on military vehicles and weapons systems by the relevant Program Manager. It should also be emphasized that successful completion of the procedures outlined in this JTP does not obligate the US Army or any other Department of Defense (DoD) organization to procure or use the candidate.

1.2 Execution

This section describes the utilization of this document by outlining the steps that will guide the user through the process of extracting and utilizing the corrosion data.

The corrosion performance ratings of candidates evaluated using this JTP will be determined through a series of tests. These tests have been developed from engineering, performance, and operational impact (supportability) standards formulated from research results conducted by government and industry. The tests in this document are based upon recognized commercial and military test standards that are currently in use by established test facilities. If the JTP test method conflicts with the reference standard on which it is based, the JTP test method will take precedence. The candidate must demonstrate at least Minimum Performance (MP) in a series of Performance Tests to be considered as an alternative corrosion prevention approach by this JTP. The JTP also provides guidelines for the screening of candidates (Screening Tests), in cases where initial viability must be assessed before conducting more extensive Performance Tests.

Prior to conducting the required Performance Tests, a candidate must undergo a preliminary Feasibility Study, in which the following considerations will be addressed:

• The candidate must be evaluated by appropriate tests that define the performance levels of the affected vehicle and weapons systems. These tests may include mechanical evaluations, compatibility tests, etc. The candidate **must** demonstrate compatibility with other components on existing or future materiel systems, i.e., it does not introduce permanent adverse effects on the functionality of the materiel system into which it will be incorporated. Those candidates incorporating coatings must also demonstrate the proper characteristics (e.g., adhesion, coverage, etc.) as called out in the relevant military specification. For example, a candidate to be used on magnesium helicopter gearboxes would require those tests that determine compatibility with Chemical Agent Resistant Coating

(CARC). More specifically, anodized coatings would require testing specific to that coating. Finally, the candidate must not adversely affect any other secondary materials used in related assemblies, such as gaskets, rubber seals, or critical wear surfaces.

- Occupational and environmental safety and health issues related to the application, utilization, repair, and disposal of the candidate must be considered. The candidate must conform to current military environmental regulations and concerns such as atmospheric and groundwater impact, volatile organic content, waste disposal, etc. This portion of the Feasibility Study should also identify and consider personnel health issues related to exposure during application, utilization, repair, and disposal of the candidate. Information provided in the Material Safety Data Sheet for the candidate may be sufficient for this assessment.
- Business issues for each candidate must be evaluated. Projected life cycle cost data should be made available to the relevant DoD procurement manager. Procurement of the candidate must conform with standard military business procedures. Considerations include, but are not limited to: distribution status (domestic/offshore); product cost analysis; and vendor capability, reputation, and reliability.

The Feasibility Study will be conducted prior to the execution of the test program contained in this JTP. The business issues assessment should be conducted again at the completion of the JTP testing, so that these issues can be reassessed with the test results in mind. The actual implementation of the Feasibility Study shall be conducted under the authority of the Army Corrosion Manager and/or the relevant Program Manager, and is outside the scope of this JTP.

The Performance Tests outlined in this JTP are organized into two general areas, Common and Special Testing. Common Testing involves those tests required for validating all magnesium corrosion protection candidates. The candidate must pass the Performance Tests with at least an MP rating in order to be considered for military use. Acceptance criteria for Improved Performance (IP) and Best Performance (BP) ratings are also provided, to enable quantification of greater degrees of improved corrosion performance with respect to what is used currently. Special Testing includes those tests identified by some, but not all, project stakeholders for validating candidates to be used in unique applications, such as exposure to specific hostile environments. The candidate must meet both Common and Special Testing requirements to be considered for Special applications.

A Joint Test Report (JTR) will document the testing conducted on each candidate in accordance with this JTP. The JTR will include the results of the testing and a record of experimental specifics such as sample and substrate preparation, equipment designations and calibration, and laboratory environmental conditions. If execution of the tests varies from that described in this JTP, the test procedure modifications must be documented in the JTR. The technical stakeholders, the Army Corrosion Manager, and/or the relevant Program Manager must agree upon such deviations. That JTR can then be used as a reference for other future magnesium corrosion-prevention programs, by DoD organizations, and commercial entities.

This document is organized in such a manner as to aid the user during the corrosion study planning stage, throughout the testing activity and during the reporting and interpretation phases. Section 2.0 provides a document guide and test flow diagram. Section 2.0 also includes examples of situations in which this JTP would be applied. Section 3.0 discusses application scenarios and outlines a requirements summary. Section 4.0 establishes test requirements and acceptance criteria. Section 5.0 describes test methodologies and procedures. Section 6.0 presents a short introduction to failure and failure analysis. Finally, Section 7.0 provides a list of reference documents that were utilized in the preparation of this JTP.

1.3 Document Maintenance

Annual updates of, and general maintenance for, this document will be the responsibility of a committee chaired by the Army Corrosion Manager or designee. This document should be reviewed and updated on an annual basis, with changes being noted on the JTP Revision History (page v). The entry, "No revision made for year 20XY" should be used where appropriate. This document will be considered obsolete if the latest entry on the JTP Revisions History is more than two years old. In this case, the Army Corrosion Manager or designee should be contacted for the most recent revisions before conducting testing in accordance with this JTP.

2.0 JTP DOCUMENT GUIDE

2.1 Introduction

This section of this magnesium JTP facilitates the use of this document by providing a logical implementation flow process, as well as examples of JTP validation for several candidate approaches. Use of this document for military consideration of a candidate utilizing the Common Testing section of the Performance Tests, or the preliminary screening of candidates through the Screening Tests section, is described and demonstrated.

In order to comply with this JTP, the candidate must pass the Common Testing requirements with at least an MP rating. Acceptance criteria for IP and BP ratings are provided to enable quantification of improved corrosion performance. Special Testing is comprised of additional tests identified by some project stakeholders, the Army Corrosion Manager, and/or the relevant Program Manager for validating magnesium corrosion protection candidates that will be used in unusual environments or for stringent applications.

This JTP also has provisions for the initial screening of candidates. The Screening Tests section is established to provide preliminary screening of newer, unproven candidates.

Any candidate that is to be considered technically acceptable must meet the MP criteria for each Performance Test, as established in Section 4.0, Engineering Performance and Testing Requirements. At the vendor's request, a failure analysis can be performed on any test specimen that fails either Common or Special Testing to determine the cause of failure (see Section 6.0) if said vendor feels that it can be used to show that some processing procedure or material characteristic that caused the failure can be corrected and avoid failure on retesting. Failure in any test does not necessarily disqualify a candidate (or its coating, surface treatment, etc.) for use in all possible applications. However, use of a candidate that has failed either Screening or Performance Tests is at the discretion of the Army Corrosion Manager and/or the relevant Program Manager, and is outside the scope of this document.

Figure 1 illustrates the process flow for conducting Screening and Performance Tests, as well as the analysis of candidates that have failed one of the aforementioned tests.

Note: In **Figure 1** below, there are two potential "infinite loops" that might occur due to testing failures. To avoid this, a provision has been inserted that if failure occurs for any of the Screening Tests after the third cycle, this process is to end and be documented in the JTR that is forwarded to the Program Manager to await the Program Manager's response. This provision is likewise applicable for the Performance Testing phase.

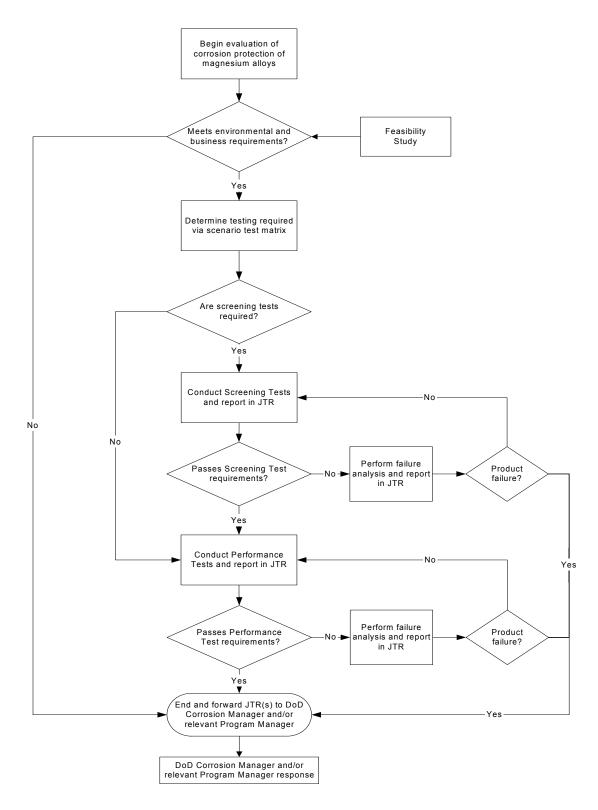


Figure 1. Test Flow Diagram

The following two examples are provided to demonstrate how the JTP can be used in Screening and Performance Tests.

Example # 1

<u>SITUATION</u>: A vendor proposes a new anodizing system to be considered for use on Army helicopter gearboxes.

EVALUATION:

- 1. The magnesium JTP directs the users to the JTP test flow diagram. The Feasibility Study is conducted to determine if initial assessments regarding compatibility and environmental concerns are met, and that the overall business risks are acceptable. The business assessment enables the users to determine if Screening Tests are warranted.
- 2. The Army Corrosion Manager and/or the relevant Program Manager determines that, since the anodizing system is new, the process will require Screening Tests prior to the initiation of Performance Tests. The JTP test flow diagram leads the users to the Applications Matrix (Table 2) to determine the degree of testing required for effective screening.
- 3. The test lab personnel begin the screening evaluation of the anodizing system.
- 4. The Screening Tests are positive. The corrosion performance represents a significant improvement over the current system and results are documented in a JTR.
- 5. Lab personnel then conduct Performance Tests. The test results are submitted to the Army Corrosion Manager and/or the relevant Program Manager in the form of the JTR.

<u>RESULT</u>: The JTP establishes the requirements for consideration, as well as guidelines for preliminary risk-reduction testing (Screening), and provides the methodology for documenting the relative corrosion prevention performance compared to that on currently produced gearboxes.

Example # 2

<u>SITUATION</u>: A vendor has developed a new magnesium sealer to be considered for gearboxes on only fifteen UH-60A Blackhawk aircraft. This is an urgent short-run application.

EVALUATION:

- 1. The magnesium JTP directs the users to the JTP test flow diagram. A feasibility study is conducted to determine if the initial assessments regarding compatibility and environmental concerns are met, and that the overall business risks are acceptable.
- 2. The Army Corrosion Manager and/or relevant Program Manager determines that only Screening Tests will be required for this system, and provided that their outcome is

positive, qualification will be granted via a special waiver (beyond the scope of this document). The JTP test flow diagram leads the users to the Applications Matrix (Table 1) to determine the degree of testing required for the Screening Tests.

- 3. The test lab personnel begin the screening evaluation of the sealer system.
- 4. Screening Test results demonstrate acceptable performance relative to currently used systems and the results are documented in the form of a JTR.
- 5. The JTR is submitted to the Army Corrosion Manager and/or the relevant Program Manager. The Army Corrosion Manager and/or the relevant Program Manager considers the results positive and issues a waiver (outside the scope of this JTP) to authorize the new system for this limited application.

<u>RESULT</u>: The JTP provides guidelines regarding testing and performance levels for preliminary risk reduction for this urgent short run requirement.

3.0 APPLICATION SCENARIOS

This section establishes the guidelines for testing a potential candidate for corrosion prevention of magnesium components, given various application scenarios.

3.1 Magnesium Alloys and Processing Variants

This document is intended to determine the corrosion-resistance characteristics of new magnesium alloys and manufacturing processes. A basic overview of selected existing magnesium alloys and their documented mechanical properties is presented in **Table 1** [Ref.1]. (NOTE: This listing is presented as an example, and is not intended to be an exhaustive list of alloys and properties)

Alloys & Casting	Elongation	Y	ield Strength, N	1Pa	Hardness,
Processes	(in 50 mm), %	Tensile	Compressive	Bearing	HRB (500 kg load, 10mm ball)
Sand & Permanent					
Mold Casting					
QE22A-T6	3	195	195		80
ZE41A-T5	3.5	140	140	350	62
AZ91C, E-T6	6	145	145	360	66
EQ21A	2	195	195		50
AZ81A-T4	15	83	83	305	55
EZ33A-T5	2	110	110	275	50
WE43A-T6	2	165			75-95
WE24					
Die Castings					
AZ91A, B and D	7	160	160		70
AS41A	15	140	140		60
AE42-F	11	145	145		60
AM50A-F	15	125	125		60
AS21-F	13	120	120		55
AM60A	13	130	130		65
Forging					
AZ61A-F	12	180	125		55
AZ31B	15	200	97	230	49
ZK61-T5					
AZ80A	11	250	195		72
Extruded Bars &					
Shapes					
AZ31B, and C	12	200	97	230	49
AZ80A-T5	7	275	240		80
AZ10A-F	10	145	69		

Table 1. Selected Existing Magnesium Alloys and Their Mechanical Properties

3.2 Magnesium Coatings Systems

A generic model of the various layers of materials that constitute a candidate magnesium corrosion prevention candidate system is shown in **Figure 2**.

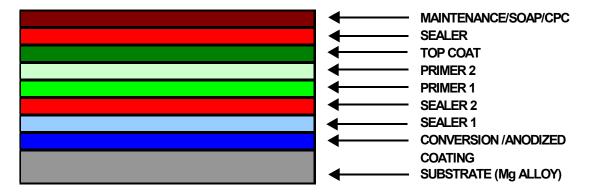


Figure 2. Generic Substrate and Corrosion Prevention Candidate System Model (not to scale)

It should be noted that the above model is based upon a typical candidate system for an actual high use magnesium component (helicopter gearbox housings). These components are typically protected by a hard coat anodizing treatment (Dow 17, HAE, or Tagnite), followed by surface sealing with multiple layers of chromate epoxy or phenolic resin (per MIL-R-3043 or the "Rockhard" resin), followed by multiple coats of chromate epoxy polyamide primer (per MIL-P-23377 Types I or II), followed by multiple coats of epoxy paint (per MIL-C-22570). Top layers could include Corrosion Preventative Compounds (CPCs), chromate pigmented sealing caulks (per MIL-S-81733 or AMS-S-8802) or other sealing compounds.

Using the above approach, guidelines for Screening and Performance Test procedures can be derived, even if the candidate contains only some of the constituents.

Table 2 establishes the test methodology to be applied to a potential candidate for both Screening and Performance Test procedures. The table lists the test methods to be employed as well as the location of that test procedure within this JTP document.

SCREENING TES (conducted on panels, total screenin month duration)		PERFORMANCE TESTS (conducted on actual or simulated parts, total performance test period about 6 months duration)		
TEST	JTP SECTION	TEST	JTP SECTION	
Adhesion, Dry Adhesion, Wet Corrosion Fatigue Hardness Neutral Salt Spray (Fog) Polarization Response	5.1.1 5.1.2 5.1.3 5.1.4 5.1.5 5.1.6	<u>COMMON TESTS</u> : Neutral Salt Spray (Fog) Galvanic Corrosion Humidity Resistance Salt Water Hydrocarbon Resistance	5.1.5 5.1.7 5.1.8 5.1.9	
	5.1.0	<u>SPECIAL TESTS</u> : Abrasion Resistance (Sandblast) Impact Resistance Modified Salt/SO ₂ Spray (Fog) Repairability Wear Resistance Stress Corrosion Cracking	5.2.1 5.2.2 5.2.3 5.2.4 5.2.5 5.2.6	

Table 2. Test Matrix for Magnesium Alloys

Based upon the summary presented in Table 2, the instructions for testing candidates under this JTP are as follows:

- 1. Choose proposed system and acquire samples from vendor.
- 2. Perform appropriate testing and document test results in a JTR.
- 3. Submit JTR to the DoD Corrosion Manager and/or the relevant Program Manager, for review.

3.3 Methodology

Screening Tests will involve the evaluation of corrosion performance of test panels fabricated from the relevant magnesium material system. Screening test panels shall be prepared utilizing all relevant coatings, surface treatments, processes, etc. The actual processes used in the preparation of the test panels will be outlined in the JTR.

Performance Tests will be conducted on manufactured parts, or samples of manufactured parts sectioned from actual fabricated magnesium components. These specimens shall be prepared utilizing all relevant coatings, surface treatments, processes, etc. and shall also incorporate relevant substrate structural features such as bends, welds, fasteners, crevices, etc. The actual processes used in the preparation of the candidate samples will be outlined in the JTR.

4.0 ENGINEERING, SCREENING, AND PERFORMANCE TESTING REQUIREMENTS

The technical requirements listed in this JTP establish the acceptable corrosion performance characteristics for corrosion prevention technologies applied to/on magnesium components. Tests to satisfy qualification requirements have been developed with detailed procedures, methodologies, and acceptance criteria to qualify the candidates can be found below.

Section 4.1 outlines Screening Testing requirements and tests. Screening Tests are performed on test panels fabricated from the candidate material, coated if applicable (see Section 5.0, Test Descriptions and Procedures).

Section 4.2 outlines Common Testing, which is conducted on manufactured parts, or pieces sectioned from manufactured parts, and coated with the relevant system as applicable. It is recommended that currently used baseline systems be tested concurrently as controls with the candidate in order to directly compare the two material systems. Questions regarding the selection of the appropriate baseline system should be directed to the Army Corrosion Manager or his designee.

Section 4.3 describes the Special Testing requirements and tests. These are programspecific requirements identified by at least one of the interested stakeholders. A design review of the functionality of the intended component, incorporating the candidate, should be conducted prior to testing, to ascertain if these or any other tests are required for the given application.

4.1 Screening Test Requirements

Table 3 lists all the Screening Test requirements identified by stakeholders for validating the corrosion performance of candidate systems.

JTP Section	Test	Acceptance Criteria, Screening Test	Test Method References
5.1.1	Adhesion, Dry	Rating \geq 4A	ASTM D3359 Method B
5.1.2	Adhesion, Wet	Rating \geq 3A	ASTM D3359 Method A
5.1.3	Corrosion Fatigue	Actual test / acceptance criteria will depend upon material, application, type of stress, and environment; note any significant improvements and / or nonconformances	ASTM E647 ASTM E8 ASTM E813
5.1.4	Hardness	\geq 400 Vickers Hardness (HV)	ASTM E384
5.1.5	Corrosion Resistance (Neutral Salt Spray (Fog))	After 240 hrs, rating beyond the scribe not less than 10. Any evidence of softening, peeling, blistering, chipping, or loss of adhesion limited to less than 1% of the unscribed areas.	ASTM B117 ASTM D714 ASTM D1654
5.1.6	Polarization Response	Similar or improved response when compared against baseline system.	ASTM G61 ASTM G5

 Table 3. Magnesium Alloys Corrosion Performance, Screening Test Requirements

Corrosion fatigue testing is especially recommended for structural components subject to cyclic stress. Corrosion fatigue failure mechanisms are relatively difficult to quantify because they are influenced by a number of factors, including material, geometry, loading spectrum, and environment. For example, some materials, such iron, steel, stainless steels, and aluminum bronzes, possess good corrosion fatigue resistance in water, but in seawater, aluminum bronzes and austenitic stainless steels retain only about 70 to 80% of their normal fatigue resistance. Likewise, high-chromium alloys retain only about 30 to 40% of their normal fatigue resistance in seawater, and quenched and drawn 0.16% carbon steel, retains only 20% of its normal fatigue strength in seawater [Ref. 2]. Furthermore, corrosion fatigue is most pronounced at low frequency tensile condition so testing in a representative environment with accurate loading rates and magnitudes is critical for characterizing the true behavior of that material system in that generic use environment.

A material / corrosion design review will be conducted by the Army Corrosion Manager to determine if corrosion fatigue and/or stress-corrosion cracking could occur in the end-use application under consideration. Testing, as it pertains to material and environment, will be determined by the Army Corrosion Manager or designee and carried out by an independent, certified lab (see Section 5.0). The Army Corrosion Manager or designee will determine the necessity of testing. The vendor will then perform mechanical stability tests through the outside engineering company as necessary.

The basic criteria for determining whether a candidate may have a high risk for corrosion fatigue failure are as follows:

1. Any material that will be exposed to cyclic stress greater than 80% yield stress in a corrosive environment (e.g., pollution, salt, chemical agents)

should be tested under experimental testing conditions simulating its generically appropriate service life environment.

2. Any material system that is historically known to be susceptible to corrosion fatigue (where susceptibility is determined by conducting a literature search or consulting with a corrosion expert) should be tested.

The basic criteria for determining a risk candidate for stress-corrosion cracking are as follows:

- 1. Any material that will be exposed to a corrosive environment known to cause stress-corrosion cracking, such as sodium hydroxide (NaOH) for carbon steel or chloride ions for stainless steels, and tensile stress due to applied load or residual stresses such as those produced by welding (e.g., any material that will experience a stress greater than 50% of the yield stress) should be tested.
- 2. Any material that is known to be subject to stress-corrosion cracking (determine susceptibility by conducting a literature search or consulting with a corrosion expert) should be tested.

It should be noted that, when testing for corrosion fatigue and/or stress-corrosion cracking, the tested specimens should be exposed to the environment (gaseous, liquid/aqueous, etc.) that simulates the exposure environment as closely as possible.

4.2 Common Testing Requirements

Table 4 lists all the Common Testing requirements identified by stakeholders for validating the corrosion performance of candidate systems.

JTP Section	Test	Acceptance Criteria, Minimum Performance (MP)	Acceptance Criteria, Improved Performance (IP)	Acceptance Criteria, Best Performance (BP)	Test Method References
5.1.5	Corrosion Resistance (Neutral Salt Spray (Fog))	After 1000 hrs, rating beyond the scribe not less than 10. Any evidence of softening, peeling, blistering, chipping, or loss of adhesion limited to less than 1% of unscribed areas.	After 1500 hrs, rating beyond the scribe not less than 10. Any evidence of softening, peeling, blistering, chipping, or loss of adhesion limited to less than 1% of unscribed areas.	After 2000 hrs, rating beyond the scribe not less than 10. Any evidence of softening, peeling, blistering, chipping, or loss of adhesion limited to less than 1% of unscribed areas.	ASTM B117 ASTM D714 ASTM D1654
5.1.7	Galvanic Corrosion	After 1000 hour exposure, D714 rating not worse than 6M(Medium), D1654 rating not worse than 6	After 1000 hour exposure, D714 rating not worse than 8M (Medium), D1654 rating not worse than 7	After 1000 hour exposure, D714 rating 10, D1654 rating not worse than 8	ASTM B117 ASTM D714 ASTM D1654
5.1.8	Humidity Resistance a) Oil Baked b) Oven Baked	After 240 hours: D1654 rating of 9, D1748 rating of "pass." Any evidence of softening, peeling, blistering, or loss of adhesion limited to less than 1% of the coating area.	After 480 hours: D1654 rating of 9, D1748 rating of "pass." Any evidence of softening, peeling, blistering, or loss of adhesion limited to less than 1% of the coating area.	After 720 hours: D1654 rating of 9, D1748 rating of "pass." Any evidence of softening, peeling, blistering, or loss of adhesion limited to less than 1% of the coating area.	MIL-PRF-3043 ASTM D1748 ASTM D1654
5.1.9	Salt Water Hydrocarbon Resistance	After 168 hours, any evidence of softening, blistering, leaching, or loss of material limited to less than 1% of the surface. No evidence of corrosion on the bare metal surfaces.	After 250 hours, any evidence of softening, blistering, leaching, or loss of material limited to less than 1% of the surface. No evidence of corrosion on the bare metal surfaces.	After 500 hours, any evidence of softening, blistering, leaching, or loss of material limited to less than 1% of the surface. No evidence of corrosion on the bare metal surfaces.	MIL-PRF-3043 ASTM D1308

Table 4. Magnesium Alloys Corrosion Performance, Common Testing Requirements

4.3 Special Testing Requirements

Table 5 lists the Special Testing requirements identified by some project stakeholders for validating the corrosion performance of candidate systems for special applications. These tests will be performed on sections of coated magnesium components unless otherwise specified in the test method (Section 5.2), or agreed upon by interested parties prior to testing and then documented in the associated JTR.

Table 5.	Magnesium	Allovs C	Corrosion	Performance.	Special	Testing Requin	rements
				,			

JTP Section	Test	Acceptance Criteria, Minimum Performance (MP)	Acceptance Criteria, Improved Performance (IP)	Acceptance Criteria, Best Performance (BP)	Test Method References	Branch/ Stakeholders/ Service Requiring Test
5.2.1	Abrasion Resistance (Sandblast)	No appearance of the substrate after 30 seconds of sandblasting.	No appearance of the substrate after 60 seconds of sandblasting.	No appearance of the substrate after 90 seconds of sandblasting.	MIL-PRF- 3043	АМСОМ
5.2.2	Impact Resistance	application, type of s Performance must be baseline/control spec	Actual test / acceptance criteria will depend upon material, application, type of stress, and projected use environment. Performance must be equivalent or improved compared to baseline/control specimen performance. Note any significant improvements and/or nonconformances.			NAVAIR
5.2.3	Modified Salt/SO2 Spray (Fog)	After 240 hrs, rating not less than 10 beyond the scribe. Any evidence of softening, peeling, blistering, chipping, or loss of adhesion limited to less than 1% of unscribed areas.	After 480 hrs, rating not less than 10 beyond the scribe. Any evidence of softening, peeling, blistering, chipping, or loss of adhesion limited to less than 1% of unscribed areas.	After 720 hrs, rating not less than 10 beyond the scribe. Any evidence of softening, peeling, blistering, chipping, or loss of adhesion limited to less than 1% of unscribed areas.	ASTM G85 ASTM B117	NAVAIR
5.2.4	Repairability	Similar or improved	Similar or improved response when compared against baseline system.			АМСОМ
5.2.5	Wear Resistance	application, type of Performance must baseline/control spe	tance criteria will dep of stress, and projected t be equivalent or impre- ecimen performance. I ments and/or nonconfo	ASTM D4060	NAVAIR	
5.2.6	Stress Corrosion Cracking	application, type of	stress, and environments (IP, BP) to be set by Manager.	nt. Any quantitative	ASTM G39 ASTM G30 ASTM G38 ASTM G47	USAF

5.0 TEST DESCRIPTIONS AND PROCEDURES

The tests identified in Tables 3, 4 and 5 are defined in more detail below to include test descriptions, scope, and methodology. Also included, as needed, are required equipment, reagents, and data reporting and analysis procedures. The test procedures list the sample preparation steps, the number of specimens for each test, the number and type of measurements for each material system being evaluated, and the acceptance criteria, as applicable. In instances where the JTP test method conflicts with the reference standard on which it is based, the JTP test method will take precedence.

All testing shall be performed by a government or independent testing laboratory, which shall be agreed upon by the stakeholders. The independent testing laboratory must either be accredited by a recognized governing body (such as the American Association for Laboratory Accreditation (A2LA) or the National Voluntary Laboratory Accreditation Program (NVLAP)) or be an ISO 9001 certified company having its own test facility to perform the testing. **Vendor-supplied testimonials shall be used for informational purposes only, and are not to be substituted for laboratory selection tests required under this JTP.** Incorporation of the results of previous studies performed on the candidate by a third-party laboratory, at the request of the vendor, will be at the discretion of the Army Corrosion Manager and/or the relevant Program Manager.

All tests shall be conducted in a manner that will eliminate duplication and maximize the data extracted from each test specimen; where possible, more than one test will be performed on each specimen. The number and types of tests that can be run on any one specimen will be dependent upon the degree of alteration imparted to the sample from previous tests. Failure in any particular test does not necessarily disqualify a candidate system for use in other possible applications. However, acceptance of a candidate that has failed either Screening or Performance Tests is at the discretion of the Army Corrosion Manager and/or the relevant Program Manager. In such cases, the use of the candidate will be justified by a special waiver, which is outside the scope of this document.

The following conditions will apply to all Screening, Common and Special Testing, unless otherwise specified:

- Test panels utilized in the Screening Tests will be comprised of panels from the candidate magnesium protective system as well those from the selected control system. 102 x 152 mm (4" x 6") panels will be employed unless otherwise specified. It is preferred that all test panels be produced during one candidate and one control system production run respectively, with both using the same magnesium alloy as the substrate.
- It is suggested that at least three (3) specimens should be used for each separate Screening Test, and at least five (5) specimens should be used for each separate Performance Test.
- Unless otherwise specified, the exposed, free surface of all the magnesium substrates should be cleaned prior to pretreatment and/or subsequence processing, to ensure that the magnesium surfaces are free of water-breaks in accordance with

the latest version of ASTM G1, "Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens." Surface cleanliness will be verified by testing in accordance with the latest version of ASTM F22, "Standard Test Method for Hydrophobic Surface Films by the Water-Break Test."

- If the candidate employs some form of protective coating, it will be applied in the appropriate, vendor specified thickness, that will be confirmed by the appropriate thickness measurement technique for that coating (e.g., dry film thickness, cross-section, etc.).
- The composition of the candidate will be determined by the analytical technique most relevant for that magnesium corrosion protection system (e.g., SEM/EDS, wet chemistry, etc.).
- Any surface preparation or pretreatment of the magnesium substrate will be specified in the JTR.

Users of this JTP should check previous magnesium corrosion protection JTRs, if available, for additional test details, or modifications that may be necessary for proper test execution. The test laboratory must document in the JTR any additional test details or minor modifications that may have been necessary to complete testing. The Army Corrosion Manager and/or relevant Program Manager must have agreed to any test procedure modifications described in the JTR.

The tests described in this JTP may involve the use of hazardous materials, operations, and/or equipment. This JTP does not address in detail most of the safety issues associated with their use. It is the responsibility of each user of this JTP to establish appropriate safety and health practices, and to determine the applicability of regulatory limitations prior to the use of such materials, operations, and/or equipment.

5.1 Screening and Common Testing

Screening and Common Testing identified in **Tables 3** and **4** are further defined in this section to include test descriptions, scope, and methodology. Also included, as needed, are any major or unique equipment and instrumentation requirements, and data analysis procedures. The test procedure includes the test specimens and substrates, definitions of the test parameters and conditions, the number of trials per specimen, any baseline (experimental control) specimens required, and acceptance criteria.

5.1.1 Adhesion (Dry)

5.1.1.1 Scope

This test method assesses the adhesion of a coating layer(s) to its magnesium substrate by applying and removing pressure-sensitive tape over cuts made into the layer. Testing is conducted on the magnesium corrosion protective system directly after application and curing steps made in accordance with the supplier's recommendation.

5.1.1.2 Equipment

<u>Cutting Tool</u>. A very sharp razor blade, scalpel, knife, or other cutting device having a cutting edge (tip) angle between 15 and 30 degrees.

<u>Cutting Guide</u>. Steel or other hard metal straightedge to ensure straight cuts. <u>Rule</u>. A steel rule graduated in 0.5 mm increments for measuring individual cuts. <u>Tape</u>. Permacel 99 (one-inch wide semitransparent pressure-sensitive tape, manufactured by Permacel, New Brunswick, NJ 08903). *NOTE*: Permacel 99 tape has a one (1) year shelf life. Utilizing the tape after this time may yield inaccurate results. It is suggested that the tape be tested for pull strength before use. The suggested pull strength for Permacel 99 tape is 50+7 oz/inch.

Roller. A 4.5-lb rubber-covered roller.

<u>Illumination</u>. A light source to determine whether the cuts have been made through the candidate system into the substrate.

Dry Film Thickness Gage. A device to measure the thickness of the applied coating.

5.1.1.3 Reagents

None.

5.1.1.4 Procedure

<u>Preparation</u>. Apply the applicable candidate system layers to the magnesium substrate for both baseline control and candidate specimens and allow them to cure in accordance with the manufacturer's recommendations. Confirm all the coating layer thicknesses are as specified with the dry film thickness gage taking five (5) readings (one (1) in each of the four corners, and one (1) on center of the panel). Make cuts in the candidate system in the grid pattern per the latest version of ASTM D3359, Test Method A. Remove two (2) laps of tape and discard. Remove an additional length of tape and cut a piece approximately 76 mm (3") long. Place the center of the tape over the grid and smooth into place by passing the roller over the area once.

<u>Test Specimens</u>. Prepare four (4) grid cut test specimens per above. For Screening Tests, $102 \times 152 \text{ mm} (4" \times 6")$ panels should be used.

<u>Test Procedure</u>. Within 90 ± 30 seconds of tape application, remove the tape by holding the free end and rapidly (without jerking) pulling back upon itself at as close to an angle of 180° as possible.

<u>Test Results</u>. Inspect the grid area for removal of any coating from the substrate or from an underlying or intermediate layer. Rate the adhesion in accordance with the latest version of ASTM D3359, Test Method A. If both ratings from either the control or candidate lots are the same or differ by only one rating point, the averaged rating should be recorded. If the difference is greater than one rating point, the results are considered suspect and two (2) additional test specimens should be prepared and the tests repeated. If applicable, use these latter two (2) ratings in the report.

<u>Report</u>. Report all information per the latest version of ASTM D3359, Test Method A. Also report the corresponding thickness readings from the dry film thickness gage measurements.

5.1.1.5 Acceptance Criteria

Substrate	Screening Test Requirements
Coated Magnesium Panels	Rating \geq 4A

5.1.2 Adhesion (Wet)

5.1.2.1 Scope

This method describes the procedure and conditions for assessing the wet adhesion of any coating layer to the magnesium substrate by applying and removing pressure-sensitive tape over cuts made in the coating after immersion in distilled water for 96 hours.

5.1.2.2 Equipment

<u>Tank and Tank Cover</u>. A tank made from corrosion-resistant materials and large enough to hold the required number of test specimens. The tank cover is required to help maintain water temperature and prevent evaporation.

<u>Test Specimen Supports</u>. Supports constructed of nonconductive and corrosion-resistant materials to hold the coated test specimens 30 mm (1.2") apart and at least 30 mm (1.2") from the bottom and sidewalls of the tank.

<u>Cutting Tool</u>. A very sharp razor blade, scalpel, knife, or other cutting device having a cutting edge (tip) angle between 15 and 30 degrees.

<u>Cutting Guide</u>. Steel or other hard metal straightedge to ensure straight cuts.

<u>Rule</u>. A steel rule graduated in 0.5 mm increments for measuring individual cuts. <u>Tape</u>. Permacel 99 (one-inch wide semitransparent pressure-sensitive tape, manufactured by Permacel, New Brunswick, NJ 08903). *NOTE*: Permacel 99 tape has a one (1) year shelf life. Utilizing the tape after this time may yield inaccurate results. It is suggested that the tape be tested for pull strength before use. The suggested pull strength for Permacel 99 tape is 50+7 oz/inch.

Roller. A 4.5 pound rubber-covered roller.

<u>Illumination</u>. A light source to determine whether the cuts have been made through the coating into the substrate.

Dry Film Thickness Gage. A device to measure the thickness of the applied coating.

5.1.2.3 Reagents

Distilled Water. Conforming to Type IV water in the latest version of ASTM D1193.

5.1.2.4 Procedure

<u>Preparation</u>. Apply the applicable candidate system layers to the magnesium substrate for both baseline and candidate specimens and allow them to cure in accordance with the manufacturer's recommendations. Confirm acceptable film thicknesses with the dry film

thickness gage taking five (5) readings (one (1) in each of the four corners, and one (1) on center of the panel).

<u>Test Specimens</u>. Four (4) replicate specimens of each coating (candidate and control) system should be prepared. For Screening Tests, $102 \times 152 \text{ mm} (4" \times 6")$ panels should be used.

<u>Test Procedure</u>. For the Screening tests, immerse the test specimens in ambient (room temperature) distilled water for 96 hours. Remove the test specimens from the water and wipe dry with a soft cloth. Within 90 ± 30 seconds after removal from the water, make cuts in the candidate system with two (2) parallel lines, 19 mm (0.75") apart, and place an "X" scribe within the parallel lines. Make the "X" lines about 38 mm (1.5") long and intersecting at 30-45 degrees in the center of the parallel lines. Remove two (2) laps of tape and discard. Remove an additional length of tape and cut a piece approximately 75 mm (3") long. Place the center of the 25 mm (1") wide tape over the center of the "X" and smooth into place by passing the roller over the area once. Remove the tape by holding the free end and rapidly (without jerking) pulling back upon itself at as close to an angle of 180° as possible.

<u>Test Results</u>. Rate the adhesion in accordance with the latest version of ASTM D3359, Method A.

<u>Report</u>. Report all information per the latest version of ASTM D3359, Method A. Also report the corresponding thickness readings from the dry film thickness gage measurements.

5.1.2.5 Acceptance Criteria

Substrate	Screening Test Requirements	
Coated Magnesium Panels	Rating \geq 3A.	

5.1.3 Corrosion Fatigue

5.1.3.1 Scope

Corrosion fatigue testing should be performed on any candidate that is considered a risk candidate as defined previously in Section 4.1. Actual test/acceptance criteria will depend upon material, application, type of stress, and environment. Note any significant improvements and/or nonconformance. The test methods listed in Table 3 for corrosion fatigue are <u>only</u> examples of a few of the tests available. The actual test used will be determined by the prospective user/stakeholder and/or an independent, certified lab (see Section 5.0) and subject to approval by the Army Corrosion Manager or designee.

5.1.3.2 Equipment

The equipment will be determined by the applicable test and the available equipment at a qualified independent lab.

5.1.3.3 Reagents

The reagents will be determined by the applicable test and the independent lab.

5.1.3.4 Procedure

The procedure will be determined by the applicable test and the independent lab. The tested specimens should be exposed to the environment that simulates the exposure environment as closely as possible.

5.1.3.5 Acceptance Criteria

The actual test will depend upon material, application, type of stress, and projected use environment. Performance must be equivalent or improved compared to baseline/control sample performance; note any significant improvements and/or nonconformance.

5.1.4 Hardness

5.1.4.1 Scope

This method is used for the determination of microindentation hardness of the candidate. The test covers microindentation tests made with a Vickers indenter.

5.1.4.2 Equipment

<u>Test machine</u>. Microhardness equipment, conforming to ASTM E384 and operated in accordance with the vendor's recommendations in the relevant equipment operators manual.

5.1.4.3 Reagents

None.

5.1.4.4 Procedure

<u>Preparation</u>. Apply the applicable candidate system layers to the magnesium substrate for both baseline and candidate specimens and allow them to cure in accordance with the manufacturer's recommendations. Set up the microhardness apparatus in accordance with the manufacturer's recommendations.

<u>Test Specimen</u>. Carefully section the test specimen in such a way that a longitudinal cross-section is exposed. Use conventional metallographic procedures to secure the test specimen in an epoxy mount with the cross-section exposed. Polish the cross-section in accordance with accepted metallurgical practices.

<u>Test Procedure</u>. Set the indenter in place. Place the test specimen on the stage or stage clamps, so that the cross sectional surface of the test specimen is perpendicular to the indenter axis. Follow operation directions in the relevant equipment operator's manual to obtain the indent diagonal lengths. Examine the indentation for its position relative to the desired location and for its symmetry. For Vickers indentation, if either diagonal dimension is not symmetric with the center of the indentation, or if the four corners of the indentation are not in sharp focus, this indicates that the test surface might not be perpendicular to the indenter axis. Check the specimen mount alignment to make sure that the cross-sectional surface is horizontal and make another indentation located at least five (5) diagonal lengths away from the original indent or other surface imperfections. If the diagonals are symmetric, measure the two diagonal lengths using Fillar objective lenses and then use the mean diagonal length value to obtain the Vickers hardness.

<u>Test Results</u>. Compute the Vickers hardness, HV by using the following equation: $HV = 1.000 \times 10^3 \times P/A_s = 1854.4(P/d^2)$, where

 $HV = Vickers hardness, gf/mm^2$

P = force, gf

 A_s = surface area of the indentation, μm^2

d = mean diagonal length of the indentation, μm

Alternatively, Table X5.2 of ASTM E384 directly provides the Vickers indentation hardness values for diagonal lengths from 1 to 200.9 μ m using 1gf. <u>Report</u>. Report test measurements and corresponding HV values, with the corresponding operational information about the number of tests, test force, magnification, mean and deviation of the test values where appropriate, and any unusual conditions encountered during the test.

5.1.4.5 Acceptance Criteria

Substrate	Screening Test Requirements	
Coated Magnesium Specimens	\geq 400 HV	

5.1.5 Corrosion Resistance (Neutral Salt Spray (Fog), per ASTM B117)

5.1.5.1 Scope

This method describes the procedure and conditions required to create and maintain the neutral salt spray (fog) test environment and the evaluation of the coated specimens with respect to corrosion, blistering associated with corrosion, loss of adhesion at a scribe mark, or other film failure.

CAVEAT: Prediction of corrosion performance in natural outdoor environments may be difficult to achieve when using neutral salt spray (fog) results as stand-alone data. Significant variability has even been observed when similar specimens are tested in different fog chambers, even though the testing conditions are nominally similar and within the ranges specified by this method.

5.1.5.2 Equipment

<u>Neutral Salt Spray (Fog) Chamber</u>. As specified in ASTM B117 and consisting of a fog chamber, a salt solution reservoir, a supply of conditioned (oil and contaminant-free) compressed air, atomizing nozzles, and specimen supports. It should also have provisions for heating the chamber and necessary means of control.

<u>Imaging System</u>. A means of visually recording corrosion on all control/baseline and candidate specimens, such as a digital camera or scanner/software system.

<u>Scribe Tool</u>. A means of scribing the coating on the specimens into the substrate. An ANSI B 94.50, style E scriber shall be used for this procedure.

<u>Straightedge</u>. Any straightedge of sufficient length to guide the scribing tool in a straight line.

<u>Air Source</u>. A source of clean, dry compressed air capable of delivering at least 10 cfm at 80 psi.

<u>Air Gun and Guard</u>. An air dusting gun and nozzle combination to meet the specification in ASTM D1654. A guard to protect the operator, such as a sandblasting cabinet. Scale. A ruler with 1 mm divisions.

Putty knife. Blunt-edged, 38 mm (1.5") wide.

5.1.5.3 Reagents

<u>Distilled Water</u>. Conforming to Type IV water in the latest version of ASTM D1193. <u>Salt</u>. Sodium chloride, substantially free of nickel and copper and containing not more than 0.1% sodium iodide and not more than 0.3% total impurities by weight.

5.1.5.4 Procedure

<u>Preparation</u>. Apply the applicable candidate system layers to the magnesium substrate and allow them to cure in accordance with the manufacturer's recommendations. Scribe a single diagonal line through the coating such that the underlying substrate is exposed for at least two (2) inches along the scratch.

Prepare the salt solution as specified in ASTM B117 such that when atomized at 35°C (95°F), the collected solution will be in the pH range from 6.5 to 7.2.

<u>Test Specimens</u>. For Screening Tests, 102 mm x 152 mm (4" x 6") panels should be used; for Performance Tests, actual or simulated components should be used. Each test specimen should contain a clear identification mark. Prepare at least three (3) test specimens each of control/baseline and candidate specimens per evaluation for Screening Tests. Prepare at least five (5) test specimens each of control/baseline and candidate specimens per evaluation for Performance Tests (hence, a minimum of fifteen (15) specimens should be considered for BP rating).

<u>Test Procedure</u>. The neutral salt spray (fog) test should be conducted in accordance with the latest version of ASTM B117, "Standard Practice for Operating Salt Spray (Fog) Apparatus." Place the scribed test specimens in the chambers, leaning at an angle between 15 and 30 degrees from the vertical. The scribed surface should be facing

upwards. The neutral salt spray (fog) chamber should be operated continuously for the specified number of hours, as shown in the Acceptance Criteria section. Test Results. At the conclusion of each exposure period (240 hrs for the screening tests and 1000 hrs, 1500 hrs, and 2000 hrs for the performance tests), remove all test specimens and clean them by gently flushing with running tap water and drying them with a stream of clean, dry compressed air. Allow the specimens to recover for twentyfour (24) hours (do not replace specimens in chamber, as they cannot be reused). Scrape the scribe on the specimens side-to-side with the putty knife at 30-degree contact angle. Evaluate the corrosion resistance and creepage of test samples (panels or components) in accordance with the latest version of ASTM D1654, "Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments." Rate the corrosion or loss of coating extending back from the scribe mark and evaluate the unscribed areas for corrosion spots, blisters, and any other types of failure that may occur. Use the rating system in ASTM D1654 for scribed areas and D714 for unscribed areas on the samples. Photographically document the surface condition of each of the test samples (panels or components) using an imaging system.

<u>Report</u>. Report all information required in ASTM B117, D714, and D1654, and include the macrographs of typical surface features recorded by the imaging system.

5.1.5.5 Acceptance Criteria

Substrate	Screening Test	Acceptance Criteria, Minimum Performance (MP)	Acceptance Criteria, Improved Performance (IP)	Acceptance Criteria, Best Performance (BP)
Coated Magnesium Specimens	After 240 hrs, rating beyond the scribe not less than 10. Any evidence of softening, peeling, blistering, chipping, or loss of adhesion limited to less than 1% of unscribed areas.	beyond the scribe not less than 10. Any evidence of softening,	After 1500 hrs, rating beyond the scribe not less than 10. Any evidence of softening, peeling, blistering, chipping, or loss of adhesion limited to less than 1% of unscribed areas.	After 2000 hrs, rating beyond the scribe not less than 10. Any evidence of softening, peeling, blistering, chipping, or loss of adhesion limited to less than 1% of unscribed areas.

5.1.6 Polarization Response

5.1.6.1 Scope

This test determines the relative susceptibility to localized corrosion of the candidates by measuring the polarization response. Polarization measurement methods involve the change in the potential of the Working Electrode that arises as a result of any current flow across the metal solution interface.

5.1.6.2 Equipment

<u>Test Cell</u>. As specified in ASTM G5 and consisting of electrodes (see below), as well as a Lugin capillary with salt bridge connection to the reference electrode, inlet and outlet for an inert gas, and a thermometer.

<u>Potentiostat</u>. Potential range from -0.6 to +1.6 V and an anodic current output range from 0.1 amps to 10^5 microamperes. The ability to measure currents as low as 0.01 amps is preferred.

<u>Electrodes</u>. The system will employ three (3) types of electrodes. The Working Electrode(s) will be the candidate material under scrutiny, and shall have the geometry specified in ASTM G5. The Auxiliary Electrode will be made of an inert material, such as platinum or graphite. The Reference Electrode, as specified in ASTM G5 Section 4.7.3, will be a Saturated Calomel Electrode (SCE).

5.1.6.3 Reagents

<u>Electrolytic Solution</u>. Electrolyte solutions that can be utilized include, but are not limited to: a) 3.15% by weight sodium chloride (NaCl) solution, b) 0.5M sodium sulfate (Na₂SO₄), c) ASTM simulated seawater, and d) filtered real seawater. The 0.5M Na₂SO₄ is intended to simulate chloride-free environments; the 3.15% NaCl, ASTM seawater, and real filtered seawater are intended to represent marine environments. Deaerated solutions are preferred.

Nitrogen or Argon Gas. Used to deaerate the electrolyte.

5.1.6.4 Procedure

<u>Preparation</u>. Set up the polarization equipment in accordance with ASTM G61. Calibrate the equipment in accordance with ASTM G59, "Standard Test Method for Conducting Potentiodynamic Polarization Resistance Measurements." For coated specimen, degrease the specimen with alcohol then proceed with the test (as received condition).

<u>Test Specimens</u>. Working Electrode(s), of the specific size and geometry specified in ASTM G5. It is suggested that at least three (3) specimens should be used for this Screening Test.

<u>Test Procedure</u>. Mount the specimen on the electrode holder and tighten the assembly. Transfer the specimen to the test cell and adjust the salt bridge tip. Measure the open circuit potential (corrosion potential) of the specimen for duration of one (1) hour. Start the potential scan at a scan rate of 10 mV/min. Proceed through +1.6 V vs. SCE in accordance with the equipment operator's manual.

<u>Test Results</u>. Plot the anodic polarization data using the measuring equipment and the relevant recording device.

<u>Report.</u> Record the polarization response. Report laboratory conditions, specimen condition, and any unusual or unexpected results

5.1.6.5 Acceptance Criteria

Substrate	Screening Test Requirements
Coated Magnesium Specimens	Similar or improved response when compared against baseline system.

5.1.7 Galvanic Corrosion

5.1.7.1 Scope

This test assesses the degree of corrosion that the candidate will resist when in contact with a more noble metal and placed in a corrosive environment. The magnesium component under scrutiny is attached to a dissimilar metal that is more noble than the magnesium (such as aluminum), usually by means of a non conducting threaded fastener, and subjected to a specific corrosive environment (such as salt spray).

NOTE: The specific configuration, the designation of a more noble metal, and the geometry of the galvanic couple test specimen should be approved prior to testing by the Army Corrosion Manager and/or the relevant Program Manager.

5.1.7.2 Equipment

<u>Neutral Salt Spray (Fog) Chamber</u>. As specified in ASTM B117 and consisting of a fog chamber, a salt solution reservoir, a supply of conditioned (oil and contaminant-free) compressed air, atomizing nozzles, and specimen supports. It should also have provisions for heating the chamber and necessary means of control.

<u>Imaging System</u>. A means of visually recording corrosion on all control/baseline and candidate galvanic couple test specimens, such as a digital camera or scanner/software system.

<u>Air Source</u>. A source of clean, dry compressed air capable of delivering at least 10 cfm at 80 psi.

<u>Air Gun and Guard</u>. An air dusting gun and nozzle combination to meet the specification in ASTM D1654. A guard to protect the operator, such as a sandblasting cabinet. <u>Scale</u>. A ruler with 1 mm divisions.

Putty knife. Blunt-edged, 38 mm (1.5") wide.

5.1.7.3 Reagents

<u>Distilled Water</u>. Conforming to Type IV water in the latest version of ASTM D1193. <u>Salt</u>. Sodium chloride, substantially free of nickel and copper and containing not more than 0.1% sodium iodide and not more than 0.3% total impurities by weight.

5.1.7.4 Procedure

<u>Preparation</u>. Apply the applicable candidate system layer(s) to the magnesium substrate and allow them to cure in accordance with the manufacturer's recommendations. [Note: It may be necessary to apply a portion (lower layers) of the candidate system, wet assemble the galvanic couple test specimen, and then apply the remaining (top) coating layers.]

Prepare the salt solution as specified in ASTM B117 such that when atomized at 35°C (95°F), the collected solution will be in the pH range from 6.5 to 7.2.

<u>Test Specimens</u>. For Performance Tests, actual or simulated components should be used. Each test specimen should contain a clear identification mark. Prepare at least five (5) test specimens each of control/baseline and candidate specimens. **The specific configuration, the designation of the more noble metal, and the geometry of the galvanic couple test specimen should be approved prior to testing by the Army Corrosion Manager and/or the relevant Program Manager.** The actual assembly process used in the manufacturing of the components in question should be replicated; for example, note that it may be necessary to apply a portion (lower layers) of the candidate system, wet assemble the galvanic couple test specimen, and then apply the remaining (top) coating layers. Inserts, gaskets, sealers, etc. should also be incorporated as required. <u>Test Procedure</u>. The neutral salt spray (fog) test should be conducted in accordance with the latest version of ASTM B117, "Standard Practice for Operating Salt Spray (Fog) Apparatus." Place the galvanic couple test specimens in the chambers, leaning at an angle between 15 and 30 degrees from the vertical. The neutral salt spray (fog) chamber should be operated continuously for 1000 hours.

<u>Test Results</u>. At the conclusion of the 1000-hour exposure period, remove all test specimens and clean them by gently flushing with running tap water and drying them with a stream of clean, dry compressed air. Allow the specimens to recover for twenty-four (24) hours (do not replace specimens in chamber, as they cannot be reused). Evaluate the corrosion resistance of test samples in all areas with specific emphasis on the points where the magnesium component contacts the dissimilar metal to which it has been connected. Use the rating system in ASTM D714 for the rating of the size of blisters on the coated areas, and note corrosion in any other areas of the galvanic couple test specimens. Use the rating system in ASTM D1654 Procedure B to evaluate the frequency and distribution of rust on the specimens. Photographically document the condition of each of the galvanic couple test specimens using the imaging system. <u>Report</u>. Report all information required in ASTM B117, D714, and D1654, and include the macrographs of typical surface features recorded by the imaging system.

5.1.7.5 Acceptance Criteria

Substrate	Acceptance Criteria, Minimum Performance (MP)		Acceptance Criteria, Best Performance (BP)
Galvanic Couple Test Specimen	After 1000 hour exposure, D714 rating not worse than 6M (Medium), D1654 rating not worse than 6	After 1000 hour exposure, D714 rating not worse than 8M (Medium), D1654 rating not worse than 7	After 1000 hour exposure, D714 rating 10, D1654 rating not worse than 8

5.1.8 Humidity Resistance (Oil Baked/Oven Baked Specimens)

5.1.8.1 Scope

This test is used for evaluating the corrosion preventive properties of a candidate (magnesium) system, given a pre-exposure treatment involving either oven baking or hot lubricating oil immersion, under conditions of 100 percent relative humidity exposure. The test consists of a) dipping coated magnesium test specimens in a hot lubricating oil or b) baking them in an oven, and then placing them in a humidity cabinet at $120^{\circ}F \pm 2^{\circ}F$ for a specified time period.

5.1.8.2 Equipment

<u>Humidity Cabinet</u>. Conforming to Appendix A.1 of ASTM D1748.
<u>Imaging System</u>. A means of visually recording corrosion on all control/baseline and candidate specimens, such as a digital camera or scanner/software system.
<u>Shaded fluorescent light</u>. A 15-watt balance illuminator type that will permit the panel to be viewed from all angles at a distance of 7.6 cm (3 in).
<u>Scribe Tool</u>. A means of scribing the coating on the test specimens into the substrate. An ANSI B 94.50, style E scriber shall be used for this procedure.
<u>Straightedge</u>. Any straightedge of sufficient length to guide the scribing tool in a straight line.
Putty knife. Blunt-edged, 38 mm (1.5") wide.

5.1.8.3 Reagents

<u>Distilled Water</u>. Conforming to Type IV water in the latest version of ASTM D1193, having a pH between 5.5 and 7.5. The water must have no evidence of oil contamination, a chloride content of less than 20 ppm, and sulfate and sulfite contents of less than 20 ppm each. <u>Hot Lubricating Oil</u>. Conforming to MIL-PRF-7808. Naphtha. TT-N-95.

Methanol. O-M-232.

Caution: Naphtha is flammable. Use only in a well-ventilated area. Methyl alcohol is both toxic and flammable. Do not allow it to be exposed to the skin or breathe its fumes. Keep all flames away from naphtha and methanol.

5.1.8.4.a Procedure for Oil Baked Humidity Resistance

<u>Preparation</u>. Prepare the humidity chamber in accordance with ASTM D1748. <u>Test Specimen</u>. Prepare at least three (3) test specimens each of control/baseline and candidate specimens per evaluation for Performance Tests (hence, a minimum of nine (9) specimens each should be considered for BP rating). Round the edges and ream out the suspension holes in accordance with Appendix A.1of ASTM D1748 before applying the candidate system. Use only samples where the candidate system layers have been applied to the magnesium substrate and have been allowed to cure in accordance with the manufacturer's recommendations. Emboss or scribe an identification number on the backside of each specimen. Oil bake nine (9) of the specimens for each batch (baseline and candidate) by immersing them to a depth of 2/3 of their length in hot lubricating oil conforming to MIL-PRF-7808 at $350^{\circ}\pm10F$ for 15 minutes, and then removing them and rinsing them clean with deionized water. Scribe one face of the oil baked specimens with a scribe so that the underlying metal is exposed at least two inches along the scratch.

Test Procedure. Place the test specimens in the humidity chamber described in Appendix A.1 of ASTM D1748 at $120^{\circ}F + 2^{\circ}F$ and 100% relative humidity for the specified exposure time (see Acceptance Criteria). Open the cabinet twice each day, except Saturday and Sunday, once for 15 minutes and again for 5 minutes (approximately 5 hours between openings). Check the chamber air temperature and water level and regulate both as needed. Maintain test room atmosphere at a temperature of $24^{\circ}C \pm 3^{\circ}C$ (75°F ± 5°F) and a maximum relative humidity of 55%. Test Results. At the end of the specified exposure period (see Acceptance Criteria), remove the panels and rinse in methanol. Follow with a rinse in naphtha and methanol and examine test surface as defined in Appendix A1.7 of ASTM D1748, using a 15watt, shaded fluorescent light. Scrape the specimens with the putty knife and evaluate the scribed area in accordance with Procedure A, Method 2 of ASTM D1654. Report. Report signs of corrosion or film failure on the specimens. Rate the scribed portion of the specimens in accordance with ASTM D1654. Rate the unscribed portions in accordance with ASTM D1748, in which a specimen receives a "pass" rating if it contains no more than three (3) spots of rust, none of which is larger than 1 mm in diameter. Visually document the condition of all specimens using the imaging system.

5.1.8.5.a Acceptance Criteria For Oil Baked Humidity Resistance

Substrate	Acceptance Criteria,	Acceptance Criteria,	Acceptance Criteria, Best
	Minimum Performance (MP)	Improved Performance (IP)	Performance (BP)
<u>Coated</u> <u>Magnesium</u> Specimen	After 240 hours: D1654 rating of 9, D1748 rating of "pass." . Any evidence of softening, peeling, blistering, or loss of adhesion limited to less than 1% of the coating area.	After 480 hours: D1654 rating of 9, D1748 rating of "pass." Any evidence of softening, peeling, blistering, or loss of adhesion limited to less than 1% of the coating area.	After 720 hours: D1654 rating of 9, D1748 rating of "pass." Any evidence of softening, peeling, blistering, or loss of adhesion limited to less than 1% of the coating area.

5.1.8.4.b Procedure for Oven Baked Humidity Resistance

<u>Preparation</u>. Prepare the humidity chamber in accordance with ASTM D1748. <u>Test Specimen</u>. Prepare at least two (2) test specimens each of control/baseline and candidate specimens per evaluation for Performance Tests (hence, a minimum of six (6) specimens each should be considered for BP rating). Round the edges and ream out the suspension holes in accordance with Appendix A.1of ASTM D1748 before applying the candidate system. Use only samples where the candidate system layers have been applied to the magnesium substrate and have been allowed to cure in accordance with the manufacturer's recommendations. Emboss or scribe an identification number on the backside of each specimen. Oven bake the six (6) specimens in accordance with MIL-PRF-3043 by placing them in an oven and baking them for 30 ± 5 minutes at 325 ± 5 °F. Scribe one face of the oven baked specimens so that the underlying metal is exposed at least two inches along the scratch.

Test Procedure. Place the test specimens in the humidity chamber described in Appendix A.1 of ASTM D1748 at $120^{\circ}F + 2^{\circ}F$ and 100% relative humidity for the specified exposure time (see Acceptance Criteria). Open the cabinet twice each day, except Saturday and Sunday, once for 15 minutes and again for 5 minutes (approximately 5 hours between openings). Check the chamber air temperature and water level and regulate both as needed. Maintain test room atmosphere at a temperature of $24^{\circ}C \pm 3^{\circ}C$ ($75^{\circ}F \pm 5^{\circ}F$) and a maximum relative humidity of 55%. Test Results. At the end of the specified exposure period (see Acceptance Criteria), remove the panels and rinse in methanol. Follow with a rinse in naphtha and methanol and examine test surface as defined in Appendix A1.7 of ASTM D1748, using a 15watt, shaded fluorescent light. Scrape the specimens with the putty knife and evaluate the scribed area in accordance with Procedure A, Method 2 of ASTM D1654. Report. Report signs of corrosion or film failure on the oven baked specimens. Rate the scribed portion of the specimens in accordance with ASTM D1654. Rate the unscribed portions in accordance with ASTM D1748, in which a specimen receives a "pass" rating if it contains no more than three (3) spots of rust, none of which is larger than 1 mm in diameter. Visually document the condition of all specimens using the imaging system.

5.1.8.5.b Acceptance Criteria for Oven Baked Humidity Resistance

Substrate	Acceptance Criteria,	Acceptance Criteria,	Acceptance Criteria, Best
	Minimum Performance (MP)	Improved Performance (IP)	Performance (BP)
<u>Coated</u> <u>Magnesium</u> Specimen	After 240 hours: D1654 rating of 9, D1748 rating of "pass." . Any evidence of softening, peeling, blistering, or loss of adhesion limited to less than 1% of the coating area.	of 9, D1748 rating of "pass." Any evidence of softening, peeling, blistering, or loss of adhesion limited to less than	After 720 hours: D1654 rating of 9, D1748 rating of "pass." Any evidence of softening, peeling, blistering, or loss of adhesion limited to less than 1% of the coating area.

5.1.9 Salt Water Hydrocarbon Resistance

5.1.9.1 Scope

This test method determines the effect of a mixed electrolyte on the candidate. The test involves the immersion of the test specimens vertically at a temperature of 95°F to 100°F in a covered glass vessel containing a double layer of liquids.

5.1.9.2 Equipment

<u>Covered Glass Vessel</u>. Placed in a temperature controlled environment and conforming to MIL-PRF-3043 Section 4.10.5.

5.1.9.3 Reagents

<u>Distilled Water</u>. Conforming to Type IV water in the latest version of ASTM D1193. <u>Sodium Chloride Solution</u>. <u>3+1</u> % salt content. <u>Hydrocarbon Fluid</u>: Conforming to ASTM D471 Reference Fuel B.

5.1.9.4 Procedure

<u>Preparation</u>. Use only samples where the candidate system layers have been applied to the magnesium substrate and been allowed to cure in accordance with the manufacturer's recommendations. Samples must be at least 6 inches long and the other dimensions small enough to fit into the glass vessel containing the double layer of liquids.

<u>Test Specimens</u>. For Performance Tests, actual or simulated components should be used. Each specimen should contain a clear identification mark. Prepare at least five (5) test specimens each of control/baseline and candidate specimens per evaluation for Performance Tests (hence, a minimum of fifteen (15) specimens should be considered for BP rating).

<u>Test Procedure</u>. Immerse the specimens in a two-layer liquid containing three percent aqueous sodium chloride solution and hydrocarbon test fluid. Suspend the specimens so that two inches of test panel is exposed to the salt mixture, two (2) inches of panel is exposed to the hydrocarbon fluid, and the balance of the panel is exposed to the air vapor mixture. Maintain solution temperature at $95\pm5^{\circ}F$. Conduct testing for the specified duration (see Acceptance Criteria).

<u>Test Results</u>. At the conclusion of the testing, remove the panels and immediately examine separately the condition of each region, as well as that of the interface regions, for any visual degradation of the surfaces of the coated specimens.

<u>Report</u>. Report any changes in the surface condition of the specimens as a function of exposure to the salt mixture, the hydrocarbon fluid, and that exposed to the air vapor mixture. Report separately the condition of each region, as well as the condition of the interface regions. Also report all other information required in ASTM D1308 and MIL-PRF-3043.

5.1.9.5 Acceptance Criteria

Substrate	Acceptance Criteria, Minimum Performance (MP)	Acceptance Criteria,Acceptance Criteria,Ainimum Performance (MP)Improved Performance (IP)			
	After 168 hours, any evidence	After 250 hours, any evidence	After 500 hours, any		
	of softening, blistering,	of softening, blistering,	evidence of softening,		
Coated	leaching, or loss of material	leaching, or loss of material	blistering, leaching, or loss		
Magnesium	limited to less than 1% of the	limited to less than 1% of the	of material limited to less		
Specimen	surface. No evidence of	surface. No evidence of	than 1% of the surface. No		
-	corrosion on the bare metal	corrosion on the bare metal	evidence of corrosion on the		
	surfaces.	surfaces.	bare metal surfaces.		

5.2 Special Testing

Special Testing is comprised of tests identified by some (but not all) project stakeholders. They are used for validating the corrosion performance of candidates for special applications. These tests will be performed on manufactured parts, or sections of larger parts, unless otherwise noted.

5.2.1 Abrasion Resistance (Sandblast)

5.2.1.1 Scope

This test determines the resistance of candidate systems and materials to surface removal/abrasion by erosive processes such as sandblasting.

5.2.1.2 Equipment

<u>Sandblasting Equipment</u>. Capable of delivering a concentrated spray of sand propelled by gases at 40±5 psig and meeting the requirements of MIL-PRF-3043. The mass flow rate of sand per unit area should be consistent with that of standard sandblast units. <u>Imaging System</u>. A means of visually recording abrasion damage on all control/baseline and candidate specimens, such as a digital camera or scanner/software system.

5.2.1.3 Reagents

<u>Silica sand</u>. White, dry, sharp. The size must meet the following sieve requirements of ASTM E323: 100% must pass through a No. 10 sieve; a minimum of 90% must pass through a No. 20 sieve; and a maximum of 10% shall be permitted to pass through a No. 50 sieve.

5.2.1.4 Procedure

<u>Preparation</u>. Use only samples where the candidate system layers have been applied to the magnesium substrate and have been allowed to cure in accordance with the manufacturer's recommendations.

<u>Test Specimens</u>. Actual or simulated components should be used. Each specimen should have a clear identification marking. At least five (5) specimens should be tested.

<u>Test Procedure</u>. In accordance with the condition as specified in MIL-PRF-3043, hold the specimens three (3) to six (6) inches from the spray nozzle and subject the specimens to a sandblast provided by any suitable means. The sandblast shall be propelled by gases at 40 ± 5 psig for the specified duration of 30 seconds for MP, 60 seconds for BP, and 90 seconds for BP. For all durations, that time when abrasion of the coating can be visually detected should be noted and recorded.

<u>Test Results</u>. At the conclusion of the testing, examine for surface degradation of the specimens. Note any chipping or peeling of the coating surface.

<u>Report</u>. Report visual condition of the specimens and all information required in MIL-PRF-3043. Record the condition of the specimens with the imaging system.

5.2.1.5 Acceptance Criteria

Substrate	Acceptance Criteria, Minimum Performance (MP)		Acceptance Criteria, Best Performance (BP)	
Coated	No appearance of substrate	No appearance of substrate	No appearance of substrate	
Magnesium	after 30 seconds of	after 60 seconds of	after 90 seconds of	
Specimen	sandblasting.	sandblasting.	sandblasting.	

5.2.2 Impact Resistance

5.2.2.1 Scope

This method evaluates the effect of rapid impact deformation on a coated substrate using an Impact Tester.

NOTE: Actual test /acceptance criteria for impact testing will depend upon material, application, type of stress, and environment. At the time of this writing this section is a placeholder that will eventually allow the actual test used to be determined by the prospective user/stakeholder and/or an independent, certified lab (see Section 5.0) and subject to approval by the Army Corrosion Manager or designee.

5.2.2.2 Equipment

The equipment will be determined by the applicable test and the available equipment at a qualified, independent lab.

5.2.2.3 Reagents

The reagents will be determined by the applicable test and the independent lab.

5.2.2.4 Procedure

The procedure will be determined by the applicable test and the independent lab.

5.2.2.5 Acceptance Criteria

Actual test / acceptance criteria will depend upon material, application, type of stress, and projected use environment. Performance must be equivalent or improved compared to baseline/control specimen performance. Note any significant improvements and/or nonconformances.

5.2.3 Corrosion Resistance (Modified Salt/SO2 Spray (Fog), per ASTM G85)

5.2.3.1 Scope

This method describes the procedure and conditions required to create and maintain the modified salt/SO₂ spray (fog) test environment, and the manner in which the coated specimens are evaluated with respect to corrosion, blistering associated with corrosion, loss of adhesion at a scribe mark, or other film/coating failure.

CAVEAT: Prediction of performance in specific acidic atmospheric environments may not correlate with modified salt/SO₂ spray (fog) results when used as stand-alone data. Significant variability may be observed when similar specimens are tested in different fog chambers, even though the testing conditions are nominally similar and within the ranges specified by this method.

5.2.3.2 Equipment

<u>Salt Spray (Fog) Chamber</u>. This equipment consists of a fog chamber, a salt solution reservoir, a supply of conditioned compressed air, atomizing nozzles, and specimen supports. The equipment should also have provisions for heating the chamber with a necessary means of control. In addition, the system should have an auxiliary means of supplying SO₂ to the chamber to include a SO₂ cylinder, regulator, flow meter, solenoid valve, timer, tubing and fittings as required per ASTM G85, Figure A4.1 (schematic of SO₂ line into salt fog cabinet).

<u>Imaging System</u>. A digital camera or scanner/software system to visually record the corrosion process on baseline and tested specimens/samples.

<u>Scribe Tool</u>. A means of scribing the specimen through the coating into the substrate on test specimens. An ANSI B 94.50, style E scriber shall be used for this procedure.

<u>Straightedge</u>. A straightedge of sufficient length to guide the scribing tool in a straight line.

<u>Air Source</u>. A source of clean, dry compressed air capable of delivering at least 10 cfm at 80 psi.

<u>Air Gun and Guard</u>. An air dusting gun and nozzle combination that meets the ASTM D1654 specifications. A guard to protect the operator such as a sandblasting cabinet. <u>Scale</u>. A ruler with 1 mm divisions.

Putty Knife. Blunt-edged, flexible and 38 mm (1.5") wide.

5.2.3.3 Reagents

<u>Distilled Water</u>. Conforming to Type IV water in the latest version of ASTM D1193. <u>Salt</u>. Sodium chloride, substantially free of nickel and copper and containing (on the dry basic) not more than 0.1% sodium iodide and not more than 0.3% total impurities by weight.

Cylinder of SO₂ Gas. 99% purity or higher.

5.2.3.4 Procedure

<u>Preparation</u>. Use only samples where the candidate system layers have been applied to the magnesium substrate and been allowed to cure in accordance with the manufacturer's recommendations. Scribe a single diagonal line through the coating, making sure that the scribed line penetrates the entire thickness of the coating down to the substrate. The modified SO₂/salt spray (fog) test should be conducted in accordance with the latest version of ASTM G85 Annex 4, "Standard Practice for Modified Salt Spray (Fog) Testing." Place the candidate and control test specimens in the chambers such that they are leaning at an angle between 15 and 30 degrees from the vertical. If there is a scribed surface it should face upwards. Configure the apparatus per ASTM G85 Annex 4. Prepare the salt solution as specified in ASTM G85 6.3 "Sodium Chloride Solution." Cycle the SO₂ gas in the chamber per the cycle parameters prescribed in ASTM G85 A4.4.1.

<u>Test Specimens</u>. Actual or sectioned parts should be used. Each test specimen should contain a clear identification mark. Prepare at least five (5) test specimens each of control/baseline and candidate specimens per evaluation for Performance Tests (hence, a minimum of fifteen (15) specimens should be considered for BP rating).

<u>Test Procedure</u>. The modified salt/SO₂ spray (fog) chamber should be operated continuously for the specified number of hours, as shown in the Acceptance Criteria section.

<u>Test Results</u>. At the conclusion of the exposure period, remove five (5) samples and clean them by gently flushing with running tap water and drying them with a stream of clean, dry compressed air. Allow the samples to recover for twenty-four (24) hours. Scrape the samples side-to-side with the putty knife at a 30-degree contact angle. Evaluate the corrosion resistance and blister creep of test samples in accordance with the latest version of ASTM D1654, "Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments." Rate the corrosion or loss of coating extending back from the scribe mark. Use the rating system in ASTM D1654 for scribed areas. Photographically document the surface condition of each sample using an imaging system. Uncoated samples should be examined for physical evidence of corrosion.

<u>Report</u>. Report all information required in ASTM G85 and D1654, and include the images from the imaging system

Substrate	Acceptance Criteria,	Acceptance Criteria,	Acceptance Criteria, Best	
Substrate	Minimum Performance (MP)	Improved Performance (IP)	Performance (BP)	
	After 240 hrs, rating not less	After 480 hrs, rating not less	After 720 hrs, rating not less	
	than 10 beyond the scribe. Any	than 10 beyond the scribe. Any	than 10 beyond the scribe. Any	
Coated Magnesium	evidence of softening, peeling,	evidence of softening, peeling,	evidence of softening, peeling,	
Specimens	blistering, or loss of adhesion	blistering, or loss of adhesion	blistering, or loss of adhesion	
	limited to less than 1% of the	limited to less than 1% of the	limited to less than 1% of the	
	unscribed area.	unscribed area	unscribed area.	

5.2.3.5 Acceptance Criteria

5.2.4 Repairability

5.2.4.1 Scope

This test assesses the candidate's performance when damaged and repaired in a controlled manner. The purpose of this test is to determine the performance impact of the candidate when repaired at the field or depot level. Test panels incorporating the candidate will be artificially damaged, placed in the salt spray chamber to initiate corrosion and simulate damage to the candidate, repaired, returned to the salt spray chamber, and then evaluated.

5.2.4.2 Equipment

<u>Neutral Salt Spray (Fog) Chamber</u>. As specified in ASTM B117 and consisting of a fog chamber, a salt solution reservoir, a supply of conditioned (oil and contaminant-free) compressed air, atomizing nozzles, and specimen supports. It should also have provisions for heating the chamber and necessary means of control.

<u>Imaging System</u>. A means of visually recording corrosion on all control/baseline and candidate specimens, such as a digital camera or scanner/software system.

<u>Scribe Tool</u>. A means of scribing the coating on the specimens into the substrate. An ANSI B 94.50, style E scriber shall be used for this procedure.

<u>Straightedge</u>. Any straightedge of sufficient length to guide the scribing tool in a straight line.

<u>Air Source</u>. A source of clean, dry compressed air capable of delivering at least 10 cfm at 80 psi.

<u>Air Gun and Guard</u>. An air dusting gun and nozzle combination to meet the specification in ASTM D1654. A guard to protect the operator, such as a sandblasting cabinet. <u>Scale</u>. A ruler with 1 mm divisions.

Putty knife. Blunt-edged, 38 mm (1.5") wide. Gravelometer.

5.2.4.3 Reagents

<u>Distilled Water</u>. Conforming to Type IV water in the latest version of ASTM D1193. <u>Salt</u>. Sodium chloride, substantially free of nickel and copper and containing not more than 0.1% sodium iodide and not more than 0.3% total impurities by weight.

5.2.4.4 Procedure

<u>Preparation</u>. Apply the applicable candidate system layers to the magnesium substrates and allow them to cure in accordance with the manufacturer's recommendations. Prepare the salt solution as specified in ASTM B117 such that when atomized at 35° C (95°F), the collected solution will be in the pH range from 6.5 to 7.2.

<u>Test Specimens</u>. 102 mm x 152 mm (4" x 6") panels should be used. Each test specimen should contain a clear identification mark. Prepare at least three (3) test specimens each of control/baseline and candidate specimens per evaluation.

Place the test panels in the gravelometer and operate in accordance with the users manual until the coated surface is uniformly damaged from the impacts (some exposed substrate is preferred). Place the "damaged" specimens in the salt spray chamber until corrosion is initiated in the damaged areas. Remove the specimens from the chamber, and clean and repair the damage in accordance with the vendor's recommendations and accepted field repair procedures. Scribe a single diagonal line through the repaired region of the coating such that the underlying substrate is exposed for at least two (2) inches along the scratch. Test Procedure. The neutral salt spray (fog) test should be conducted in accordance with the latest version of ASTM B117, "Standard Practice for Operating Salt Spray (Fog) Apparatus." Place the scribed test specimens in the chambers, leaning at an angle between 15 and 30 degrees from the vertical. The scribed surface should be facing upwards. The neutral salt spray (fog) chamber should be operated continuously for 250 hours.

<u>Test Results</u>. At the conclusion of the exposure period, remove 2 test specimens (one control and one baseline candidate) and clean them by gently flushing with running tap water and drying them with a stream of clean, dry compressed air. Allow the specimens to recover for twenty-four (24) hours. Do not put specimens back in the chamber, for they are not to be reused. Scrape the scribe on the specimens side-to-side with the putty knife at 30-degree contact angle. Evaluate the corrosion resistance and creepage of test samples (panels) in accordance with the latest version of ASTM D1654, "Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments." Rate the corrosion or loss of coating extending back from the scribe mark and evaluate the unscribed areas for corrosion spots, blisters, and any other types of failure that may occur. Use the rating system in ASTM D1654 for scribed areas and D714 for unscribed areas on the samples. Photographically document the surface condition of each of the test samples (panels) using an imaging system. Report. Report all information required in ASTM B117, D714, and D1654, and include the macrographs of typical surface features recorded by the imaging system.

5.2.4.5 Acceptance Criteria

Substrate	Acceptance Criteria, Minimum Performance (MP)	_	Acceptance Criteria, Best Performance (BP)
<u>Coated</u> <u>Magnesium</u>	After 250 hour exposure, D714 rating not worse than 6M (Medium), D1654 rating not worse than 6	After 250 hour exposure, D714 rating not worse than 8M (Medium), D1654 rating not worse than 7	After 250 hour exposure, D714 rating 10, D1654 rating not worse than 8

5.2.5 Wear Resistance

5.2.5.1 Scope

This test method determines the resistance of the candidate to abrasion applied to a plane, rigid surface.

NOTE: Actual test /acceptance criteria for wear resistance will depend upon material, application, type of stress, and environment. At the time of this writing this section is a placeholder that will eventually allow the actual test used to be determined by the prospective user/stakeholder and/or an independent, certified lab (see Section 5.0) and subject to approval by the Army Corrosion Manager or designee.

5.2.5.2 Equipment

The equipment will be determined by the applicable test and the available equipment at a qualified, independent lab.

5.2.5.3 Reagents

The reagents will be determined by the applicable test and the independent lab.

5.2.5.4 Procedure

The procedure will be determined by the applicable test and the independent lab.

5.2.5.5 Acceptance Criteria

Actual test / acceptance criteria will depend upon material, application, type of stress, and projected use environment. Performance must be equivalent or improved compared to baseline/control specimen performance. Note any significant improvements and/or nonconformances.

5.2.6 Stress-Corrosion Cracking

5.2.6.1 Scope

Stress-corrosion cracking testing should be performed on any candidate that is considered a risk candidate as defined previously in Section 4.1. Actual test /acceptance criteria will depend upon material, application, type of stress, and environment. At the time of this writing this section is a placeholder that will eventually allow the actual test to be determined by the prospective user/stakeholder and/or an independent, certified lab (see Section 5.0); the selected test will be subject to approval by the Army Corrosion Manager or designee.

5.2.6.2Equipment

The equipment will be determined by the applicable test and the available equipment at a qualified, independent lab.

5.2.6.3 Reagents

The reagents will be determined by the applicable test and the independent lab.

5.2.6.4 Procedure

The procedure will be determined by the applicable test and the independent lab.

5.2.6.5 Acceptance Criteria

Actual test / acceptance criteria will depend upon material, application, type of stress, and projected use environment. Performance must be equivalent or improved compared to baseline/control specimen performance. Any quantitative improvement factors (IP, BP) will be specified by the relevant Program Manager.

6.0 FAILURE ANALYSIS

To be considered for use as replacements for conventionally used materials, candidates must pass all tests. If the candidate fails any specific screening or performance test, at the candidate vendor's request and expense, a Failure Analysis procedure can be undertaken. Such failure analysis can be a useful vendor option if he can use it to demonstrate that some promising procedure or material characteristics that caused the observed performance failure can be corrected and then lead to acceptable performance metrics. However after failing any of the Screening Tests for the third time, further iterations of that Screening Test are not permitted. Instead, the JTP process should be ended and the results noted in that JTR. The JTR should then be forwarded to the Program Manager and await the Program Manager's response. Likewise, for failure of any specific Performance Test, if failure occurs for the third time, do not repeat the Performance Testing. Instead, the JTP process should be terminated and results noted in that JTR. The JTR should then be forwarded for the Performance Test, if failure occurs for the third time, do not repeat the Performance Testing. Instead, the JTP process should be terminated and results noted in that JTR. The JTR should then be forwarded to the Program Manager for his response.

Marginal test results must be either overcome by retesting or documented before discarding the candidate. The failure mechanism of specimens that fail one or more preliminary laboratory screening tests should be documented. In the event of a testing-related dispute between vendor and tester, such as a cause of premature failure, a third-party testing lab will be mutually agreed upon as a credible testing source by the Army Corrosion Manager and/or the relevant Program Manager. This Product Failure Laboratory (PFL) must have no pre-existing connection to either the vendor of the candidate or the original laboratory that conducted the initial testing. A failure analysis can, if requested by an interested party, be performed on any specimen that fails either Common or Special Performance Testing to determine the cause. The process flow is illustrated in **Figure 1** that appears in Section 2.0, JTP Document Guide.

Failure in any test does not necessarily disqualify a candidate coating for use in all possible applications. The initial JTR and **all** related JTRs (specifically failure analyses) must be submitted to the DoD Corrosion Manager or designee.

7.0 **REFERENCE DOCUMENTS**

The documents listed in Table 5 were referenced in the development of this JTP.

Reference Document	Title	Date	Source	Applicable Section(s) of Reference Document	JTP Test	JTP Section Cross-Reference
ASTM B117	Standard Test Method of Salt Spray (Fog) Testing	Latest edition	ASTM	All	Corrosion Resistance (Neutral Salt Spray (Fog), per ASTM B117)	5.1.5, 5.2.3, 5.2.4
ASTM D471	Standard Test Method for Rubber-Properties-Effect of Liquids	Latest edition	ASTM	All	Salt Water Hydrocarbon Resistance	5.1.9
ASTM D714	Test Method for Evaluating Degree of Blistering of Paints	Latest edition	ASTM	All	Corrosion Resistance (Neutral Salt Spray (Fog) per ASTM B117)	5.1.5 5.2.3, 5.2.4
ASTM D1193	Specification for Reagent Water	Latest edition	ASTM	All	All	5.1.2, 5.1.5, 5.2.3, 5.2.4
ASTM D1308	Standard Test Method for Effect of Household Chemicals on Clear Pigmented Organic Finishes	Latest edition	ASTM	All	Salt Water Hydrocarbon Resistance	5.1.9
ASTM D1654	Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments	Latest edition	ASTM	All	Corrosion Resistance, (Neutral Salt Spray (Fog) per ASTM B117)	5.1.5 5.2.3 5.2.4
ASTM D1748	Standard Test Method for Rust Protection by Metal Preservative in the Humidity Cabinet	Latest edition	ASTM	All	Humidity Resistance	5.1.8

Reference Document	Title	Date	Source	Applicable Section(s) of Reference Document	JTP Test	JTP Section Cross-Reference
ASTM D2794	Standard Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)	Latest edition	ASTM	All	Impact Resistance	5.2.2
ASTM D3359	Standard Test Methods for Measuring Adhesion by Tape Test	Latest edition	ASTM	All	Adhesion (Dry), Adhesion (Wet)	5.1.1, 5.1.2
ASTM E8	Standard Test Methods for Tension Testing of Metallic Materials	Latest edition	ASTM	All	Corrosion Fatigue	5.1.3
ASTM E92	Standard Test Method for Vickers Hardness of Metallic Materials	Latest edition	ASTM	All	Hardness	5.1.4
ASTM E384	Standard Test Method for Microindentation Hardness of Materials	Latest edition	ASTM	All	Hardness	5.1.4
ASTM E647	Standard Test Method for Measurement of Fatigue Crack Growth Rates	Latest edition	ASTM	All	Corrosion Fatigue	5.1.3
ASTM F22	Standard Test Method for Hydrophobic Surface Films by the Water-Break Test	Latest edition	ASTM	All	All	5.0
ASTM G1	Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens	Latest edition	ASTM	All	All	5.0
ASTM G5	Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements	Latest edition	ASTM	All	Polarization Response	5.1.6
ASTM G59	Standard Practice for Conducting Potentiodynamic Polarization Resistance Measurements	Latest edition	ASTM	All	Polarization Response	5.1.6

Reference Document	Title	Date	Source	Applicable Section(s) of Reference Document	JTP Test	JTP Section Cross-Reference
ASTM G61	Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurement for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt- based Alloys	Latest Edition	ASTM	All	Polarization Response	5.1.6
ASTM G85	Standard Practice for Modified Salt Spray (Fog) Testing	Latest edition	ASTM	All	Corrosion Resistance (Modified Salt/SO ₂ Spray (Fog))	5.2.3
MIL-C-22570	Performance Specification, Coating, Epoxy, High Solids	Latest edition	Department of Defense	All	All	3.0
MIL-P-23377	Performance Specification, Primer Coatings: Epoxy, High Solids	Latest edition	Department of Defense	All	All	3.0
MIL-PRF-3043	Performance Specification Resin Coating, Permanent, for Engine Components and Metal Parts	Latest edition	Department of Defense	All	Humidity Resistance, Salt Water Hydrocarbon Resistance Abrasion (Sandblast)	5.1.8 5.2.1 5.2.4
AMS-S-8802	Sealing Compound, Temperature-Resistant, Integral fuel Tanks and Fuel Cell Cavities, High-Adhesion	May 1999	Department of Defense	All	All	3.0
MIL-PRF-7808	Lubricating Oil, Aircraft Turbine Engine, Synthetic Base	May 1997	Department of Defense	All	Humidity Resistance	5.1.8
ASTM E-323	Standard Specification for Perforated-Plate Sieves for Testing Purposes	2001	ASTM	All	Humidity Resistance	5.1.8
ASTM E23	Standard Test Methods for Notched Bar Impact Testing of Metallic Materials	2002	ASTM	All	Impact Resistance	5.2.2
MIL-S-81733	Sealing and Coating Compounds, Corrosion Inhibitive	Latest edition	Department of Defense	All	All	3.0

Other Text References:

- 1. Magnesium and Magnesium Alloys. *ASM Specialty Handbook*, ed, M.M Avedesian and H. Baker. ASM International, Materials Park, OH, 1999.
- 2. M.G. Fontana, "Chapter Three, Eight forms of Corrosion," Corrosion Engineering, McGraw-Hill, 1986, p. 141.

APPENDIX A

List of Technical Stakeholders

List of Technical Stakeholders

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