ARMY RESEARCH LABORATORY

Water-Dispersible Polyurethane Coating Test Protocol

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Abstract

The Coatings Technology Team of the Polymers Research Branch has developed a water-reducible 180-g/L volatile organic compound (VOC) formulation that is also a chemical agent resistant coating (CARC). This report describes the substrates, pretreatments, and primers to be used in comparative testing of the new formulation vs. current CARC. The tests and evaluation criteria will also be introduced. This report will be used as a reference for the data presentation reports that follow.

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1. Background

The Coatings Technology Team of the Polymers Research Branch, U.S. Army Research Laboratory (USARL), has developed a water-reducible 180-g/L volatile organic compound (VOC) formulation that is also a chemical agent resistant coating (CARC).[1] This coating material is designed to meet or exceed all the current applicable CARC requirements within MIL-C-46168.[2,3] The primary reason for this effort was the continual demands for environmentally compliant materials due to various regulatory requirements. Federal and local regulations resulting from the Clean Air Act restrict the amount of VOCs emitted during the application of protective coatings, while Occupational Safety and Health Administration (OSHA) regulations restrict human exposure to many materials used in coating manufacture. The development of this material has also required serious consideration of its durability and compatibility with the Army's existing pretreatments, primers, and topcoats.

2. Approach

A joint effort between the Polymers Research Branch and the Metals and Ceramics Research Branch was initiated to develop a comprehensive test panel matrix for the newly developed water-reducible polyurethane CARC topcoat project. This effort has three primary objectives. First, since this is intended to be an alternative replacement for the current CARC topcoat, the new product had to be tested for compatibility with existing pretreatments and primers used by the Army and other services requiring CARC. Second, the water-reducible topcoat had to meet or exceed the performance requirements detailed in the MIL-C-46168 specification. Finally, this data will be used to establish a baseline for compatibility and performance to successfully predict the life-cycle of the coating systems and the associated application and disposal costs to the Army. Using these guidelines, a matrix was developed that consists of 7 alloy substrates, 3 pretreatments, 2 primers, and 2 topcoats, in 14 tests with multiple samples and 1 control set for a total of 2,868 test panels.

Substrates were chosen that represent a large portion of the Army's painted inventory. Low-alloy carbon steel panels were pretreated using either zinc phosphate per TT-C-490[4] to represent original equipment manufacturer (OEM) coatings or 0.3–0.5 mil of DOD-P-15328[5] wash primer to represent field-repaired or depot-applied coatings. Panels made from three commercial grades of aluminum (2024, 6061, and 7075) and three armor grades of aluminum (2519, 5083, and 7039) were also pretreated with wash primer to represent field or depot coatings. Alodine 1200 was also used as a pretreatment on the commercial aluminum alloys and the 2519 armor alloy to represent OEM systems. All alloy and pretreatment combinations were primed with one of the two Army primers used in the CARC system, MIL-P-53022 or MIL-P-53030.[6,7] Finally, all alloy, pretreatment, and primer combinations were topcoated with either the Army's two-component CARC polyurethane, MIL-C-46168, or with the new water-reducible polyurethane that should become an environmentally friendly alternative to MIL-C-46168.

Many of the tests that were chosen are performance requirements cited in the current primer (MIL-P-53022 and MIL-P-53030) and topcoat (MIL-C-46168 and MIL-C-53039[8]) specifications. Any topcoat that is to be used where CARC is specified would have to meet these requirements in addition to agent resistance. Also included were some accelerated tests that may assist in predicting long-term performance of these coating systems better than those included in the current specifications. Whether these are included in the future specification for water-reducible polyurethane or not, all tests will provide baseline data that can be used to predict the coating's performance in a variety of service environments. The following pages provide a brief description of the methods and parameters that will be used in this matrix.

Dry adhesion will be conducted following procedures outlined in ASTM D3359 method B.[9] The test will be performed by cutting two sets of six parallel lines through the coating to the substrate in a cross-hatch pattern. Standard adhesive tape will be firmly applied and removed. Evaluations will be made on a scale of 0B–5B, where 5B represents no coating removal by the tape. The purpose of this test is to determine if there are incompatibilities between the constituents of the system and the substrate. Failure will also occur when the coating is brittle.

Wet adhesion is a more severe test than dry adhesion. It is referenced in the primer specifications (MIL-P-53022 and MIL-P-53030). The test that will be used is a modified version of that found in FED-STD-141 method 6301,[10] with evaluations made using ASTM D3359 method A. In this test, a panel is immersed in deionized water for 24 hr, removed, and dried. The panel is then scribed with two intersecting sets of two parallel lines, 1 in apart. Standard tape is firmly applied and removed to determine the adhesion properties. Evaluations are made on a scale of 0A–5A, where 5A represents no coating removal by the tape. In addition to determining incompatibilities between the coating constituents, this test also demonstrates the system's sensitivity to moisture.

Salt spray resistance is also referenced in the primer specifications (MIL-P-53022 and MIL-P-53030) and is based on procedures defined in ASTM B117.[11] This test includes two panels with scribes through the coatings to the substrate and three panels without scribes; both exposed initially for 336 hr. If no apparent or severe degradation of the coating is observed, the test is continued to 1,000 hr of exposure and beyond. Salt fog testing can show a coating system's resistance to water permeability, general corrosion resistance, and resistance to corrosion undercutting of the film on the scribed panels. This test is widely used by the paint industry even though it is considered to be more of a quality control test and not necessarily indicative of long-term performance of the coating.

GM 9540P[12] is an accelerated cyclic corrosion test that was developed by the automotive industry to better replicate long-term outdoor performance of coatings. Scribed panels are exposed to multiple stages consisting of several cycles of salt mist (0.9% NaCl, 0.1% $CaCl_2$, and 0.25% $NaHCO_3$), high-temperature drying, and ambient rest followed by a cycle of humidity exposure, high-temperature drying, and ambient soak. Standardized calibration coupons are run concurrently to ensure compliance with specified tolerances. The previously described process repeated 80 times is claimed by industry to be equivalent to 10 yr of field exposure in South Florida.

The Prohesion test is another cyclic corrosion test developed by the paint industry to improve correlation between actual and accelerated tests. The test is performed according to ASTM G85 annex A5.[13] Specimens are alternatively exposed to 1-hr intervals of salt fog followed by dry off. The solution for the salt fog is 0.05% NaCl and 0.35% ammonium sulfate. The test must be run for a minimum of 336 hr before a decision is made about continuing the test. The data generated by this test will be compared to the exposure test data.

Because the failure modes in accelerated corrosion are varied, the panels from the previous tests are evaluated using a combination of three specifications. ASTM D610[14] is used when corrosion products bleed through the coating at active corrosion sites. ASTM D714[15] is used when corrosion products and water form a blister between the coating and the substrate. ASTM D1654[16] is used to assess performance of the coating in the vicinity of a scribe through the coating system. All 3 standards use a 10-point scale for their rating system, and a score below 5 constitutes failure.

Accelerated weathering using a xenon arc bulb for ultraviolet (UV) exposure is currently called for in the topcoat specifications (MIL-C-46168 and MIL-C-53039). The test is run according to ASTM G26 method A[17] for 300 hr. The method is programmed for continuous cycles of 102 min of UV exposure followed by 18 min of UV with water spray to the front of the specimen. Exposure for 300 hr at an irradiance level of 0.35 W/m^2 corresponds to an eighth of an average year in Florida. These panels will be run to failure, where failure is defined as a color change of 2.5 National Bureau of Standards (N.B.S.) units, an increase in 60° gloss to above 1.0, or an increase in 85° gloss to above 3.5. Spectrocolorimetry and specular gloss readings will be taken every 300 hr.

Another accelerated weathering method, conforming to ASTM G53,[18] that will be implemented is cyclic UV and condensation. This method simulates the effect of sunlight by exposing specimens to fluorescent UV lamps while it simulates rain and dew with condensing

humidity. Exposure times for each step in the cycle is 4 hr. These panels will also be run to failure. Spectrocolorimetry and specular gloss readings will be taken every 500 hr.

Emmaqua is an accelerated test offered by the Arizona division of the South Florida Test Service. In this test, natural sunlight is concentrated on test specimens using mirrors. The rack assembly tracks the sun. Approximately 7 weeks of Emmaqua exposure is equivalent to a year of exposure in Florida. This test will evaluate a 3-yr equivalent exposure with color and gloss readings taken at half-year equivalent intervals. If results are comparable, this may provide tremendous insight into the long-term durability of these coatings with a relatively short exposure time.

Electrochemical impedance spectroscopy (EIS) and electrochemical noise analysis are two methods for assessing the porosity and permeability of a coating to ionic species. In both procedures, a portion of the panel is immersed in a 0.5N NaCl solution and measurements are taken at prescribed intervals. In EIS, an electrical signal is impressed on the sample and the frequency response is measured. This method can also be used to periodically check the condition of coatings during other testing. Electrochemical noise takes readings of signals that are generated by local electrochemical processes caused by the environment at the coating/substrate interface. The advantage of this method is that it can be used to noninvasively monitor the performance of the coating system as it is being tested without perturbing the system. EIS will be conducted for 650 hr or until failure of the system. Readings will be taken with more frequency during the early part of the exposure until a steady state is achieved. Electrochemical noise will utilize the same test methodology with a different experimental setup.

Long-term outdoor exposure is the test by which all accelerated testing is compared. Nothing is as predictive as placing coated samples in the environment in which they must perform and allowing sun, humidity, natural chemicals, and time to degrade the coating. However, no one is willing to wait 18 months to 10 years to determine whether a coating can or cannot satisfactorily do the job that it was formulated to accomplish. Currently, aspects of coating performance are

tested in accelerated tests and outdoor exposure is used to validate the results from the accelerated tests. Most of the time this process works well. This is the compromise that must be accepted for timely implementation of new coating technology. For this experiment, a set of 52 groups of 5 panels each will be exposed for 3 yr in Florida or Arizona. Two panels from each group will be scribed. The Florida test site offers a semi-tropical marine environment, which is one of the most severely corrosive to which Army materiel would be exposed. The Arizona test site offers a desert environment with high solar loading and cyclic humidity.

3. Conclusion

The overall intent and objectives set forth by the various tests and evaluations will establish a comprehensive database of existing and newly developed coating systems. It will also demonstrate the compatibility and performance of the new water-reducible chemical agent resistant topcoat. Published reports will be generated for the various tests outlined previously, and the final objective will incorporate updates to the database as new materials are formulated and existing formulations are modified. Ultimately, this database will be available to assist all scientists and engineers in selecting or evaluating the best coating and/or substrate for the end user, the soldier.

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