CORROSION TESTS OF HIGH-STRENGTH ALUMINUM ALLOYS: A REVIEW

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Abstract

Accelerated laboratory corrosion tests are essential for efficiently evaluating the performance of high-strength aluminum alloys, particularly in the context of rapidly developing new materials. By adhering to established standards like ASTM, these tests not only provide valuable insights into the materials' corrosion resistance but also ensure that the results are reliable and comparable to those obtained from long-term exposure studies. This duality of testing methodologies emphasizes the importance of thorough corrosion assessment in the development and application of aluminum alloys in various environments.

Keywords*: Corrosion Testing, High-Strength Aluminum Alloys, Long-term tests, Laboratory Tests, Salt Spray Tests, Electrochemical Tests, Exfoliation Corrosion, ASTM Standard*

1. INTRODUCTION

Corrosion testing of aluminum alloys is divided into two categories: long-term exposure tests and laboratory tests. In long-term tests, material specimens are exposed to specific natural environments such as marine, rural, urban, or industrial environments [1-3]. In particular, the exposure of specimens to the atmosphere for conducting outdoor atmospheric corrosion tests is described in ASTM G50 [4]. A drawback of these tests is the very long time required to evaluate corrosion resistance due to the relatively mild corrosive environment. This duration can vary from a few years to decades, depending on the material and the exposure environment. On the other hand, because they involve real service conditions, as opposed to laboratory simulations, these tests provide a reliable assessment of the corrosion resistance of materials [5].

Laboratory tests were developed to provide a rapid assessment of the corrosion resistance of materials. Often, the rapid development of new alloys and treatments leaves no time for lengthy evaluations via long-term testing. Accelerated laboratory tests fill this gap, simulating the real environment, and obtaining reliable and comparable results to long-term tests remains a major challenge for laboratory tests [6,7].

In laboratory tests, aggressive corrosive environments are used to accelerate the corrosion phenomenon. Generally, we can classify these tests into the following categories: (a) immersion tests, (b) salt spray tests, and (c) electrochemical tests.

Laboratory tests are usually carried out according to specific standards such as those of ASTM and ISO. These standards detail the specifications of test specimens, the composition of the corrosive environment, the procedure and conditions of the test, as well as the methodology for evaluating the results. It is clear that following standard test methods ensures greater reliability and comparability of results. Standard laboratory tests are presented in detail below.

2. IMMERSION TESTS OF ALUMINUM ALLOYS

In immersion tests, standardized specimens of the materials under examination are submerged in aggressive chemical solutions for a specified period of time. The evaluation of material resistance is carried out in various ways depending on the form of corrosion and the type of test. Specifically, in the case of an exfoliation corrosion test, the surfaces of the specimens are compared with reference photographs. In the intergranular corrosion test, specimens are prepared metallographically, and the

phenomenon is examined via optical microscopy. In the case of pitting corrosion, alternative methods can be used, such as counting pit density per area or measuring the depth and size of pits. Table 1 summarizes the types of standard immersion tests depending on the material, the form of corrosion they examine, and the evaluation criterion used.

Standard	Material	Corrosion form	Evaluation criterion
ASTM G34 (EXCO test)	2xxx and 7xxx series	Exfoliation	Comparison with reference photographs
ASTM G66 (ASSET test)	5xxx series	Exfoliation	Comparison with reference photographs
ASTM G110	2xxx and 7xxx series (and other alloys, including cast)	Intergranular	Metallographic examination
ASTM G67 (NAMLT test)	5xxx series	Intergranular	Mass loss measurement
ASTM G44 (alternate immersion)	2xxx and 7xxx series	Pitting, intergranular, galvanic	The occurrence of failure or a specified duration of exposure
ASTM G47	2xxx and 7xxx series	Stress-corrosion cracking (SCC)	Visual inspection for cracks, followed by metallographic examination to determine if the fracture is intergranular (in which case it is considered an SCC failure)

Table 1. Immersion tests of aluminum alloys

Immersion tests are a useful tool for the rapid assessment of the corrosion resistance of aluminum alloys. The effectiveness of each test depends on its sensitivity in distinguishing the corrosion resistance of alloys of different compositions and heat treatment. Finally, it is very important that the test results show satisfactory correlation with those of long-term exposure tests.

Below, the most important standard immersion tests for high-strength aluminum alloys are presented.

2.1 Exfoliation Corrosion Tests (EXCO test)

The exfoliation corrosion test (EXCO test), according to ASTM G34, is widely used to evaluate the exfoliation corrosion behavior of 2xxx and 7xxx series aluminum alloys. According to this standard, specimens of aluminum alloy from the above series are immersed in a standard EXCO solution consisting of 4.0 M NaCl, 0.5 M KNO_3, and 0.1 M HNO_3. The maximum immersion time is 48 hours for 7xxx series alloys and 96 hours for 2xxx series alloys. During the test, the solution pH increases from 0.4 to 3. Immediately after removal from the solution, the specimen surfaces are evaluated by comparing them with reference photographs and are classified into the following categories: (a) N if there is no attack on the material, (b) P if pitting corrosion is observed, and (c) EA through ED if exfoliation corrosion is observed (the letters A–D indicating increasing severity) [8]. Figure 1 shows the experimental setup of the EXCO test, specimens after the test, and the standard photographs used for specimen evaluation. The images in Figure 1 are from an exfoliation corrosion test conducted according to the above standard at the Corrosion Center of Aristotle University of Thessaloniki (AUTH).

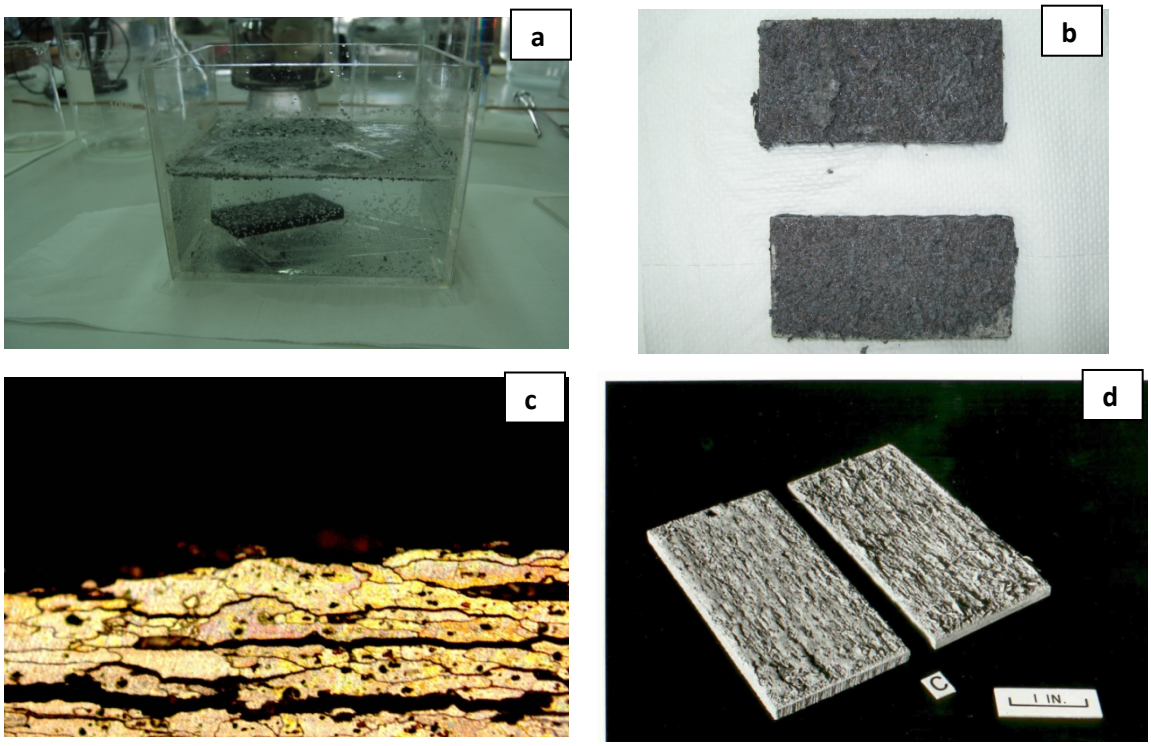

Fig. 1. Photographs from an exfoliation corrosion test (EXCO test) conducted at the AUTH Corrosion Center. (a) EXCO test experimental setup, (b) AA7075-T6 alloy specimens after the test, (c) Microstructure of AA7075-T6 after exposure (cross-section, x200), (d) Standard reference photographs for specimen evaluation.

The exfoliation corrosion test successfully differentiates the resistance of the T6 and T73 tempers of AA7075 aluminum alloy. It also shows satisfactory correlation with long-term tests in marine and industrial environments for AA7075 and AA7178 alloys [9]. However, this test has problems in predicting the resistance of AA2024 and AA7050 alloys in the T7 temper, as the EXCO solution appears to be too aggressive [10]. Furthermore, for predicting the exfoliation corrosion behavior of Al-Li alloys, the EXCO test shows poor correlation with atmospheric exposure data [10].

To address the above issues, Lee and Lifka proposed a modification of the EXCO solution in which the solution pH is adjusted to 3.2 by adding HCl and Aluminum ions [11]. The modified EXCO test shows better correlation with natural environment tests for alloys such as AA2024 and AA2090. However, for Al-Li alloys, it causes more severe exfoliation than the standard EXCO solution.

For evaluating the exfoliation corrosion of 5xxx series wrought alloys, ASTM G66 (ASSET test) is applied. According to this standard, specimens of the material are immersed in a solution containing 1.0 M NH_4Cl, 0.25 M NH_4NO_3, 0.01 M $(NH_4)_2C_4H_4O_6$, and 0.09 M H_2O_2 for 24 hours at 65 ±1 °C. The susceptibility to exfoliation corrosion is determined by visual assessment through comparison with reference photographs. Specimens are classified by degree of attack into category N when there is no attack on the material, P when pitting corrosion is present, and E when exfoliation corrosion is present [12]. Figure 2 shows the ASSET test setup, photographs of specimens after exposure, and reference photographs for evaluation. The images in Figure 2 are from an exfoliation corrosion test conducted according to the above standard at the AUTH Corrosion Center. This test shows good correlation with natural exfoliation behavior [13].

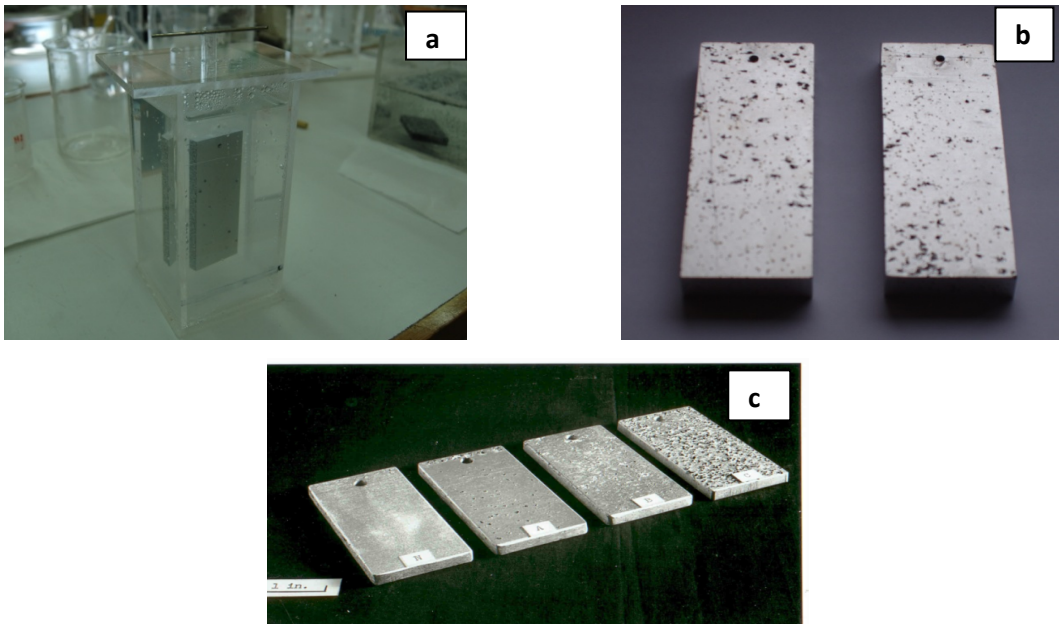

Fig. 2. Photographs from an exfoliation corrosion test (ASSET test) conducted at the AUTH Corrosion Center. (a) ASSET test experimental setup, (b) AA5083-H111 alloy specimens after the test, (c) Standard reference photographs for specimen evaluation.

2.2 Intergranular Corrosion Tests

The intergranular corrosion test according to ASTM G110 primarily applies to wrought alloys from the 2xxx and 7xxx series. It can also be used on other aluminum alloys, including cast alloys. In this test, chemically pre-etched specimens are immersed in a NaCl + H_2O_2 solution for at least 6 hours. After immersion, metallographic specimens are examined under an optical microscope to determine the extent of intergranular corrosion [14]. Figure 3 shows the microstructure of an AA2024 aluminum alloy following an intergranular corrosion test, performed in accordance with the above standard at the AUTH Corrosion Center.

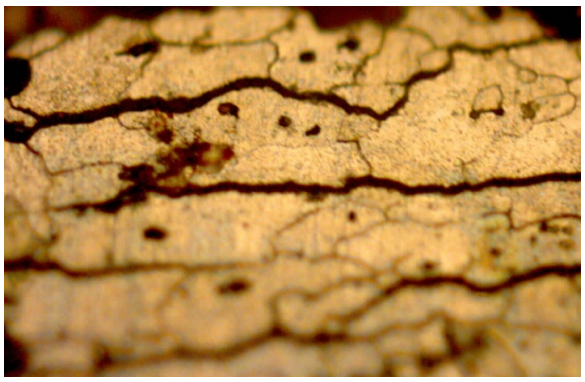

Fig. 3. Microstructure of AA2024 aluminum alloy after an intergranular corrosion test, according to ASTM G110, conducted at the AUTH Corrosion Center (x200).

To evaluate the resistance to intergranular corrosion of 5xxx series aluminum alloys, ASTM G67 (NAMLT test) is utilized. This standard assesses the susceptibility of these alloys to intergranular

corrosion by measuring their mass loss after immersing specimens for 24 hours in a nitric acid solution [15].

2.3 Alternate Immersion Test

This test involves exposing metals and alloys to alternating immersion in a neutral 3.5% NaCl solution. Notably, either stressed or unstressed specimens can be used. Historically, the method was used to test the Stress Corrosion Cracking (SCC) resistance of stressed specimens of 2xxx and 7xxx series alloys, but it can also be used on unstressed specimens that exhibit pitting, intergranular, or galvanic corrosion.

In the alternate immersion test according to ASTM G44, specimens of the material under examination are subjected to a repeated exposure cycle of one hour. Specifically, they are immersed in an aqueous solution of 3.5% NaCl for 10 minutes and then allowed to dry for 50 minutes out of the solution. This cycle is repeated continuously, 24 hours a day, for a duration that depends on the alloy's corrosion resistance in seawater. The test can be concluded after a specified period or when failure of the material occurs [16]

2.4 Stress Corrosion Cracking Test (SCC)

Stress corrosion cracking (SCC) is defined as the failure or degradation of a material's properties due to the combined effect of a corrosive environment and the application of a constant tensile stress, which may be in the elastic or elastoplastic range [17]. The corrosive environment for SCC can be created using the alternate immersion test described above. The design and preparation of the specimens used in the test are conducted according to ASTM G49. This standard covers the use of standardized axially loaded specimens (as per ASTM specifications) to evaluate resistance to SCC. Additionally, the standard addresses the selection of appropriate specimen types and the design of the loading fixture [18]. The standard specimens used in SCC testing are cylindrical and have standardized dimensions. Alternatively, depending on the form of the component to be tested, C-ring type specimens can be used. The dimensions and characteristics of C-ring specimens are specified by ASTM G38 [19]. Figure 4 shows cylindrical and C-ring SCC test specimens before and after an SCC test conducted at the AUTH Corrosion Center.

Fig. 4. a) Cylindrical and C-ring SCC specimens before and b)after an SCC test, conducted at the AUTH Corrosion Center.

The determination of susceptibility to SCC in 2xxx and 7xxx series aluminum alloys is conducted according to ASTM G47. This standard outlines procedures for sampling various product forms, examining the exposed specimens, and interpreting the results. Specifically, the surfaces of the specimens are inspected for cracks, followed by a metallographic examination to determine whether the fracture is intergranular, if so, it is classified as an SCC failure [20].

3. SALT SPRAY TESTS

Salt spray (fog) testing has been used as a method for studying corrosion since the 1930s. It was initially employed to evaluate the quality of organic and inorganic protective coatings on metal substrates, primarily in industries producing painted metal objects, such as the automotive industry. Specifically, salt spray testing allows for quick and easy determination if a batch of industrial materials is problematic, as such a batch will corrode faster than the rest of the production, which will be visibly evident.

Today, the method is used to examine the corrosion behavior of both coated and uncoated metallic materials.

When a metal is exposed to the atmosphere, it becomes the anode of a galvanic cell, forming ions that leave its surface, which results in a reduction of its mass. The cathode of the galvanic cell is an area of the metal with a different potential due to varying composition, stress accumulation, or local changes in the electrolyte composition. In the case of atmospheric exposure, the electrolyte is the atmospheric moisture that gathers on the metal surface.

Atmospheric corrosion is accelerated by the dissolution of various ions in the moisture condensed on the metal surface, such as Cl^- ions, which are found in high concentrations in coastal areas. Other factors that enhance this phenomenon include atmospheric pollutants like SO_2 and NO_x, which, when dissolved in atmospheric moisture, lead to a drop in pH.

The usefulness of the salt spray test lies in reproducing the corrosive atmospheric conditions observed in coastal areas and accelerating the atmospheric corrosion process by imposing more intense conditions. The desired conditions are achieved using a salt spray chamber, where specimens of the material under test are placed and sprayed with a suitable corrosive medium, typically an aqueous NaCl solution.

A salt spray chamber consists of the test chamber, the systems for storing, supplying, and atomizing the corrosive solution, and the system for controlling and recording the conditions within the chamber. Figure 5 illustrates the salt spray cabinet at the AUTH Corrosion Center, the arrangement of specimens inside the chamber, and the specimens following the test.

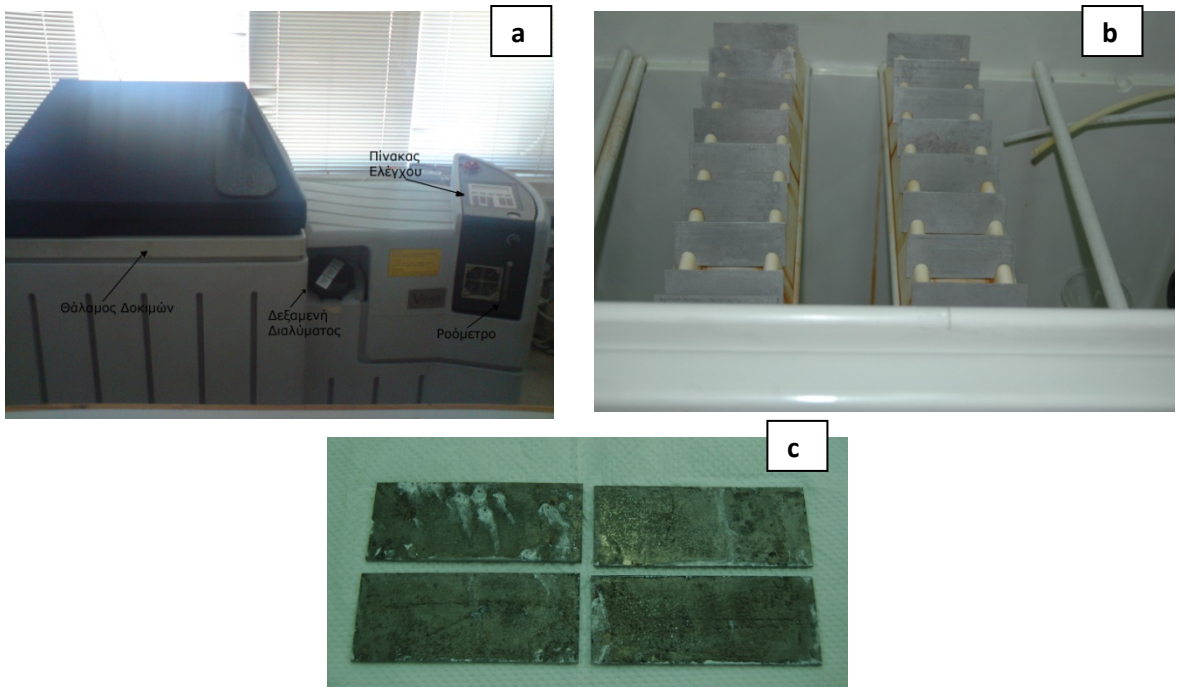

Fig. 5. Conducting a salt spray test according to ASTM B117 at the AUTH Corrosion Center. (a) Salt spray chamber at AUTH, (b) Placement of specimens inside the chamber, (c) Specimens after the test.

To ensure the comparability of salt spray test results, specific standards are adhered to, including ISO 9227, ASTM B117, and ASTM G85. These standards detail the equipment, conditions, procedures, and evaluation of test results, which are discussed below.

3.1 ASTM B117 Standard Salt Spray (Fog) Test

The ASTM B117 standard outlines the equipment, procedure, and conditions necessary to establish and maintain the proper environment during a salt spray test. This standard is applicable to metals and alloys.

The performance of the salt spray chamber is checked using cold-rolled carbon steel specimens as per SAE 1008. The number of test specimens is determined by the experiment at hand and is typically an even number (e.g., 2, 4, 6). The specimens measure 127 mm × 76 mm and have a thickness of 0.6 mm. Their surface must be free of pores, marks, and scratches; therefore, before each experiment, the specimens are cleaned with deionized water and scrubbed with a special brush, followed by drying, cleaning with ethanol, and then drying again. The mass of the specimens is measured before the experiment with an accuracy of ±1 mg. The specimens are placed in separate quadrants inside the salt spray chamber so that they are not close to one another, with the exposure surface facing upward at an angle of 15°–30° from vertical [21].

The solution used is 5% NaCl by mass in deionized water, with a density of 1.025–1.04 g/cm³ at 25 °C. The experimental conditions include the temperature of the saturator (humidifier), the temperature of the salt spray chamber, the air pressure for atomization, the pH of the solution, and the duration of the experiment. The saturator temperature is set according to the air pressure of the salt spray apparatus, ranging from 46–49 °C for an air pressure of 0.83–1.24 bar. The chamber temperature is maintained at 35 °C (with an allowable deviation of +1.1/–1.7 °C). The pH of the solution collected by the chamber's collectors must range from 6.5 to 7.2. Special methods are used to adjust the pH accurately. Finally, the duration of the experiment ranges from 48 to 168 hours, depending on the material and the agreement between buyer and seller [21].

3.2 ASTM G85 Modified Salt Spray Tests

This standard applies to ferrous and non-ferrous alloys, along with organic and inorganic coatings. It includes modified tests that are useful when a different or more aggressive environment is desired compared to the simple salt spray test of ASTM B117 described above.

Specifically, the standard includes five modified tests presented in the order of their development:

a) Continuous salt spray testing with the addition of acetic acid. In this test, the solution sprayed is 5% NaCl in deionized water. The pH of the solution is adjusted to a range of 3.1 to 3.3 by adding acetic acid, while the chamber temperature is maintained at 35 °C (tolerance +1.1/–1.7 °C) [22].

b) Cyclic salt spray test: In this test, the spray solution consists of 5% NaCl in deionized water, with the pH adjusted to between 2.8 and 3.0 by adding acetic acid. The chamber temperature is maintained at 49 °C (tolerance +1.1/–1.7 °C). The cycle lasts for 6 hours and includes 45 minutes of spraying, followed by 2 hours of exposure to dry air, and 3 hours and 15 minutes of high humidity exposure (with the solution retained in the chamber). The specimens are positioned at a 45° angle. The test duration varies based on the sensitivity of the alloy, and after completion, the resulting surfaces are compared with reference photographs to assess the extent of exfoliation corrosion [22]. This test demonstrates a good correlation with long-term outdoor exposure results for Al-Li alloys. Additionally, it aligns more closely with long-term outdoor exposure results than the EXCO exfoliation test. However, it is complex and requires specialized equipment and a relatively extended exposure period.

c) Cyclic seawater immersion-spray test. In this test, the solution consists of synthetic seawater at a 5% concentration, made acidic by adding 10 mL of acetic acid per liter of solution, resulting in a pH of approximately 2.8–3.0. The saturator temperature is set to 47 ±1 °C for a chamber temperature of 35 °C and 57 ±1 °C for a chamber temperature of 49 °C. The test follows a cycle of 30 minutes of spraying, followed by 90 minutes of exposure at over 98% relative humidity. This test is suitable for production control of heat treatments that are resistant to exfoliation corrosion in 2xxx, 5xxx, and 7xxx series alloys [23]. For this purpose, a chamber temperature of 49 °C is required; whereas for organic coatings on

various metal substrates, the chamber temperature is maintained between 24 °C and 35 °C because temperatures above 35 °C may lead to blister formation in the coating.

d) Cyclic salt spray test with SO_2. This test consists of spraying a salt solution along with the periodic introduction of SO_2 gas into the chamber. The solution to be sprayed is, as previously mentioned, synthetic seawater, and the chamber temperature is kept at 35 °C (with a tolerance of +1.1/–1.7 °C). The pH of the solution collected in the chamber should range between 2.5 and 3.2. The test cycle is defined by the specifications of the material being tested and by agreement between the buyer and seller. Example test cycles include continuous spraying with SO_2 gas introduction for one hour, four times a day (i.e., every 6 hours), or half an hour of spraying, followed by half an hour of SO_2 introduction, and 2 hours of exposure to high relative humidity [22].

Standard	Material	Corrosive medium (pH)	Evaluation criterion
ASTM B117, continuous spray	Metals and alloys	None (6.5–7.2)	Depends on the type of corrosion
ASTM G85, Annex A1, continuous spray (AASS)	Ferrous and non-ferrous alloys, organic and inorganic coatings	Acetic acid (pH 3.1–3.3)	Depends on the type of corrosion
ASTM G85, Annex A2, cyclic spray/dry	Ferrous and non-ferrous alloys, organic and inorganic coatings	Acetic acid (pH 2.8–3.0)	Depends on the type of corrosion
ASTM G85, Annex A3, cyclic spray with synthetic seawater (SWAAT)	Ferrous and non-ferrous alloys, organic and inorganic coatings	Acetic acid (pH 2.8–3.0)	Depends on the type of corrosion
ASTM G85, Annex A4, cyclic spray with SO_2	Ferrous and non-ferrous alloys, organic and inorganic coatings	SO_2 (pH 2.5–3.2)	Depends on the type of corrosion

Table 2. Salt spray tests (ASTM B117 and G85)

Damborenea et al. [24] conducted accelerated and long-term atmospheric exposure tests on two Al-Cu alloys (AA2024-T4 and AA7075-T7351) and two Al-Li alloys (AA2091-T84 and AA8090-T7351). These alloys were exposed for two years in an aggressive marine environment, and their corrosion behavior was compared with that observed in accelerated exfoliation corrosion and intergranular corrosion tests. The results of the EXCO exfoliation tests did not correlate well with those from the outdoor exposure tests, even with the use of the modified EXCO solution. The EXCO test produced a significantly greater degree of exfoliation than the outdoor exposure for the AA2091-T84 and AA8090-T7351 alloys. Furthermore, the laboratory tests did not replicate the pitting and intergranular corrosion seen in the outdoor exposure.

Braun also conducted laboratory tests on the alloys mentioned above [10]. The results of his experiments indicated that the cyclic salt spray test replicated the outdoor exposure data. Furthermore, the standard EXCO solution reproduced the exfoliation corrosion behavior of all the alloys, except for AA8090-T6 and AA7075-T7351.

4. ELECTROCHEMICAL TESTS

Several standard electrochemical tests are available for assessing the corrosion behavior of metallic materials, as well as for research purposes. The measurement of the open-circuit potential (OCP) of aluminum alloys is carried out according to ASTM G69. This standard outlines the procedure for

measuring the corrosion potential (open-circuit potential) of aluminum alloys. This measurement is an effective tool for characterizing the metallurgical state of 2xxx and 7xxx series aluminum alloys, as it is highly sensitive to changes in the composition of the solid solution matrix [25].

Potentiodynamic polarization curves facilitate the evaluation of susceptibility to localized corrosion in intermetallic compounds (by comparing the breakdown potential) as well as intergranular and exfoliation corrosion (by comparing the current density).

The ASTM G5 method is a standard reference method for potentiostatic and potentiodynamic anodic polarization measurements. This test method includes a procedure to verify the experimental technique and equipment. The test ensures repeatability and reproducibility of potentiostatic and potentiodynamic anodic polarization measurements using specimens from the same lot of 430 stainless steel [26].

The required equipment for performing electrochemical tests includes a potentiostat connected to an electrochemical cell composed of three electrodes. The working electrode is the location where the test specimen is mounted. The reference electrode is a saturated calomel electrode (SCE), and the auxiliary (counter) electrode is made of platinum (Pt). OCP measurements and the plotting of potentiodynamic curves can be conducted using specialized software.

5. CONCLUSIONS

Accelerating corrosion tests are widely used in researching corrosion behavior and developing metallic materials. However, the inconsistency of results between accelerated laboratory tests and long-term outdoor exposure tests highlights the limitations of laboratory evaluations. The appropriate synthetic environment for accelerated tests that adequately simulate natural exposure conditions remains an open question, and the qualitative assessment of test results does not provide sufficient quantitative information on the depth, extent, and kinetics of the phenomenon. Therefore, to obtain additional information for evaluating this phenomenon, metallographic cross-sections and post-analysis of the resulting surfaces are usually necessary [27].

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