Torsional shear strength of adhesive joined steel in saline environments

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This study investigates the impact of exposure to aggressive environments, specifically saline conditions per ASTM B117 standard, on the shear strength of adhesive-bonded joints, crucial for ensuring structural integrity in various industrial applications like automotive, aerospace, and construction. Employing an epoxy-based adhesive to bond AISI304 steel, specimens undergo 14- and 28-day salt spray treatments to simulate harsh working conditions. Torsional shear strength assessment of steel-to-steel adhesive joints is conducted using a torsion test methodology, with an analytical approach used to extrapolate adhesive joint shear strength. This research contributes to understanding adhesively bonded joint performance in challenging environmental conditions, and how the shear strength of the bonded joints changes in relatively small amount of time, offering valuable insights for robust structural designs across industrial sectors.

1. Introduction

Over the past few decades, structural adhesives have experienced a significant increase in their application and use in various industries. [1] Structural adhesives are high-strength bonding agents that can join two or more materials together, often without the need for additional mechanical fasteners such as screws, bolts, or rivets. These adhesives provide an effective and efficient solution for joining materials that are difficult or impossible to bond using traditional joining methods.[2]

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The use of structural adhesives has increased in many industries, including automotive, aerospace, construction, and electronics. In the automotive industry, for example, structural adhesives are used to bond various parts of a vehicle, such as the roof, body panels, and chassis. This results in weight reduction and improved fuel efficiency. [3] In the aerospace industry, adhesives are used to join aircraft structures and components, allowing for greater strength and improved aerodynamics. [4] In the construction industry instead, they could be used to join materials that should not be joined with traditional joining techniques, such as glass, to avoid the introduction of cracks or localized stresses. [5, 6] Generally speaking, the use of adhesives guarantees more uniform stress along the joint compared with the other traditional joining techniques. [7]

One of the major reasons for the growth in the use of structural adhesives is their ability to offer improved performance and durability compared to traditional joining methods. They can also be more cost-effective and provide a cleaner, more aesthetically pleasing finish.[8] As a result, the use of structural adhesives is expected to continue to grow in the coming years as new, innovative formulations are developed and as more industries recognize the benefits of this technology. [9]

Salt spray treatment is a type of corrosion testing that is commonly used in various industries, such as automotive, to evaluate the corrosion resistance of materials and coatings. [10] The test involves exposing a material or coating to a salt spray solution, which creates a harsh, corrosive environment that accelerates the corrosion process, which can help manufacturers identify potential weaknesses in materials or coating. [11]

ASTM B117 is a widely recognized standard test method for salt spray corrosion testing. It is used to evaluate the corrosion resistance of metallic materials and coatings, as well as the effectiveness of corrosion inhibitors and surface treatments. [12]

There are several available tests to evaluate the mechanical properties of adhesive joints, such as the double cantilever beam test or single lap test. [13] One of the more interesting parameters from an industrial point of view is the joint shear strength: indeed, and before any other one, the shear strength is the first design property of practical use in a design process. [14]

To measure the shear strength of bonded joints some testing methods, such as the asymmetrical four-point bending or Arcan test [15][16] both characterized by some drawbacks, in terms of the specimen manufacturing process or difficulties in performing the test: as an alternative to obtaining the shear properties of an adhesive joint, the authors proposed a torsion test on hourglass shaped samples, as discussed in reference. [17]

In the scientific literature, several studies have addressed the impact of a saline environment on the shear resistance of adhesive joints using the single lap joint test method, as documented in [18, 19].

The impact of humidity on torsion strength has been discussed in[20], where stiffness measurement was the main focus and no change in the maximum shear force was detected.

In contrast, this research uniquely investigates the influence of an aggressive saline environment through the utilization of a torsion test, aimed at characterizing the adhesive joints under pure shear stress conditions. This approach allows for a comprehensive assessment of the effects of a harsh environment on both the maximum and elastic shear stress properties of the adhesive joints, providing a more thorough understanding of the behavior of adhesive joints exposed to adverse conditions. In real-world applications, there are some examples of adhesive joints used in environment close to the seaside, such as the one reported in Figure 1 in [6], which can be affected by the aggressive saline environment.

2. Materials and methods

As joining material an epoxy-based adhesive EPX DP490 (3M™Scotch-Weld™) was used to join the hourglass shaped AISI304 steel samples as reported in Figure 1. More details on the choice of this torsion test and on the samples' shape and size can be found in [6, 17, 21].

Figure 1. Hourglass-shaped AISI304 steel substrates for torsion test (size in mm).

Steel surfaces were polished with sandpaper (P2000) and then ultrasonic cleaned with acetone before joining. The joining process was done by using a fixture designed to guarantee the alignment and a constant joint thickness. A constant amount of adhesive has been put on the two half hourglass substrates avoiding gaps and spew fillets, and the same weight (5 grams) has been used to keep each sample in place by applying the same pressure on each sample. The specimens were cured according to the adhesive datasheet at room temperature for 7 days.

The thickness of each sample has been measured by a digital caliper before and after joining to obtain the joint thickness by difference. The joint thickness of each specimen was measured in the range of 30 µm – 80 µm. Nine specimens were produced, 3 tested as prepared, while the other 6 were submitted to salt spray treatment, for 14 days and 28 days respectively, according to ASTM B117. The test requires keeping specimens in a continuous salt spray mist in a controlled environment for a specified time (Weiss Technik SC/KVT1000, Grand Rapids, MI (USA)). The mixture used to age the specimens is a water solution with 5% weight of sodium chloride. The mixture is kept in a reservoir inside the machine: during the treatment an oil and grease free compressed air is sprayed through an atomizing nozzle to obtain the aging environment in the fog chamber where the specimens are placed. The torsion machine (Test Resources, Shakopee, MN, USA) used to perform the tests is shown in Figure 2. The machine is equipped with a torque cell of 10Nm and the angular speed used for the tests was 2 degrees per minute. The torsion results were then analyzed through an analytical elastoplastic model to extrapolate the maximum elastic shear stress resistance. In particular, the total torsional moment (M) was split into two different contributions, elastic (M_{el}) and plastic (M_{pl}). The analytical model has been fully described in reference [21]

Finally, a comparison between the results of the as cured specimen and the ones subjected to a salt spray treatment was done and the fracture surfaces were inspected through a Field Emission Scanning Electron Microscopy (TESCAN MIRA3 XMH, Brno, CZ, J-Tech@PoliTO laboratories).

Figure 2.Torsion machine at J-Tech@Polito laboratories.

3. Results and discussion

3.1. Mechanical tests

The purpose of this paper is to establish how the shear strength resistance of this joint is influenced by a saline environment. Three conditions have been analyzed: the first one is the as-cured joint for comparison purposes, and the other two typologies are related to joints submitted to a salt spray treatment for 14 and 28 days, respectively. For each specimen typology, tests have been done in triplicate. Firstly, the maximum shear strength was analyzed, but more interesting, is the elastic shear strength evaluation which defines the pure shear stress, i.e. the maximum working stress to remain in the elastic regime. The values of the maximum shear strength and elastic shear strength are reported in Table 1.

Table 1. Elastic shear strength and maximum shear strength obtained from the experimental torsion tests on as cured and after salt spray treatment, 14 and 28 days.

Specimen	τ_{el} [MPa]	τ_{max} [MPa]
1 [As Cured]	42	53
2 [As Cured]	49	62.8
3 [As Cured]	48	59.5
Mean value As Cured	46.3	58.4
1 [14 Days]	25	31
2 [14 Days]	29	36.7
3 [14 Days]	32	41.2
Mean value 14 days	28.7	36.3
1 [28 Days]	21.5	26.9
2 [28 Days]	29	37.5
3 [28 Days]	28.5	36.7
Mean value 28 Days	26.3	33.7

The maximum shear strength is computed by the equation (1):

$$\tau_{max} = G\gamma_m = \frac{M_{max} l}{J\theta_m}\gamma_m = \frac{M_{max} r}{J} \quad (1)$$

Where G is the shear modulus (1550 MPa from the adhesive datasheet), γ_m is the shear strain at the maximum moment, M_{max} the maximum torsional moment, l the adhesive thickness, J the polar moment of inertia, θ_m the angle of rotation at the peak of the torque and r is the radius of the circular joint. The elastic shear resistance, τ_{el}, instead is estimated through an analytical elastoplastic model (2):

$$M_{tot} = M_{el} + M_{pl} = \tau_{el}\frac{\pi r^4}{2r} + \tau_{el}\frac{2\pi}{3}(r^3) \quad (2)$$

Where M_{el} is the elastic contribution to the total moment while M_{pl} is the plastic contribution. By using the mean values of each configuration the mean curves have been obtained. The mean experimental curves are reported with the corresponding analytical model in Figure 3.

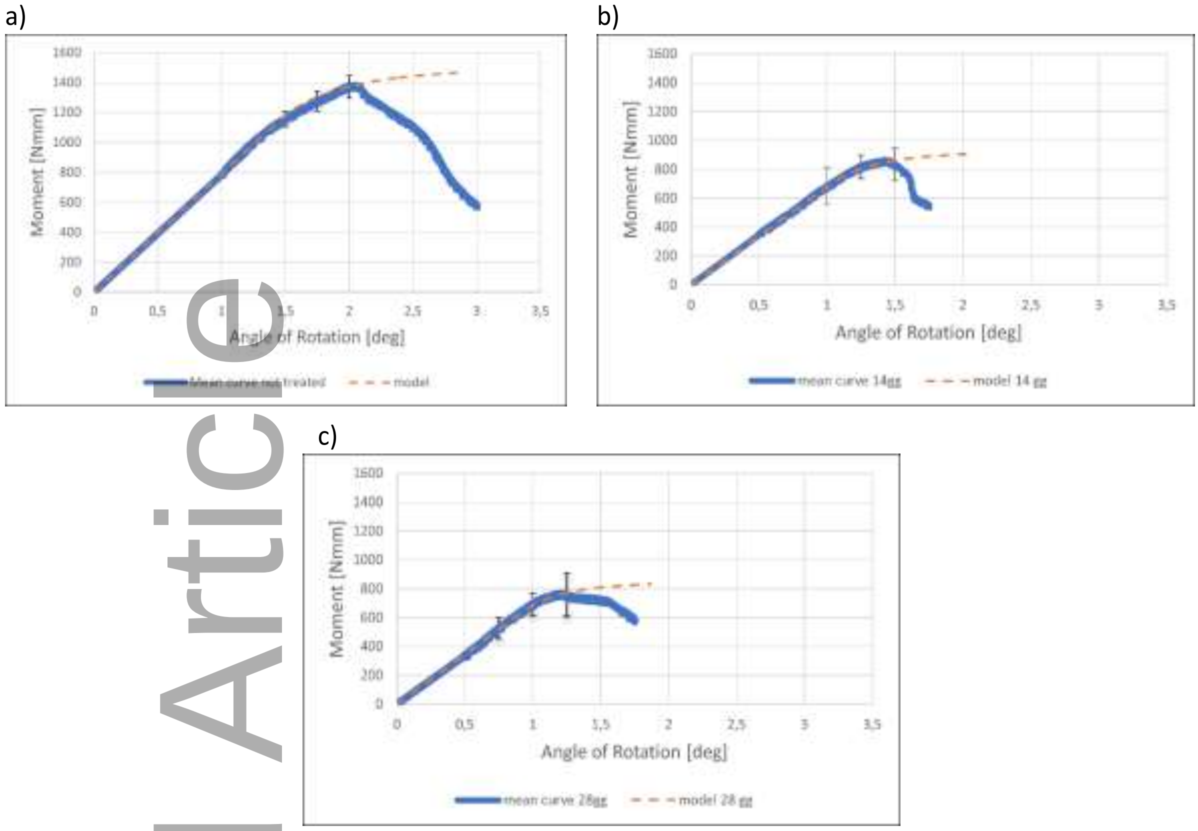

Figure 3. a) the mean curve and corresponding analytical model for the as-cured specimen; b) the mean curve and corresponding model for the specimens that have been submitted to 14 days of salt spray treatment and c) the mean experimental curve and corresponding model for the specimens submitted to 28 days of salt spray treatment. In each mean curve, the characteristic standard deviations have been reported.

A good correlation between the experimental and the analytical model has been obtained for all three conditions. The failure was not analytically modeled because the purpose of this model was to establish the values of the elastic and maximum shear stresses. The maximum shear strength and elastic shear strength have a similar decreasing behavior moving from the as cured joints to a 28-day treated ones. The decreasing behavior of the mechanical properties is well visible in Figure 4.

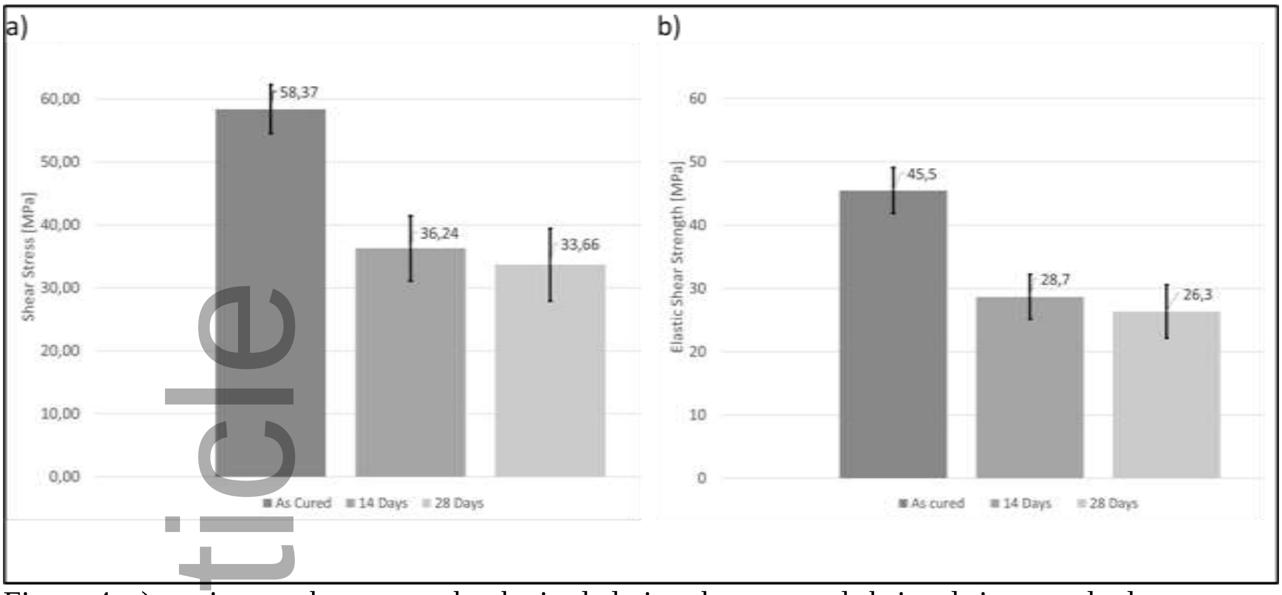

Figure 4. a) maximum shear strength obtained during the tests and their relative standard deviations; b) maximum elastic shears strength estimated through the analytical model.

For both the mechanical properties (τ_{max} and τ_{el}) there is an evident drop in the first 14 days of treatment while they seem to stabilize in the next days of treatment. This means that the degradation, in terms of mechanical properties, takes place in the first period of exposure to the salt environment. In particular, there is a decrease of around 38% and 37% of τ_{max} and τ_{el} respectively in the first 14 days of exposure, then a further decrease of 7% and 8.5% in the following 14 days of τ_{max} and τ_{el} respectively.

3.2. Fracture Modes

After torsion tests, all the fractured surfaces displayed a combined adhesive/cohesive fracture pattern, where a portion of the adhesive material was observed on both surfaces post-failure. These representative fracture surfaces are depicted in Figure 5. Notably, upon image analysis, it was found that in all cases, the percentage of adhesive present in both substrates ranged between 33% to 67%.

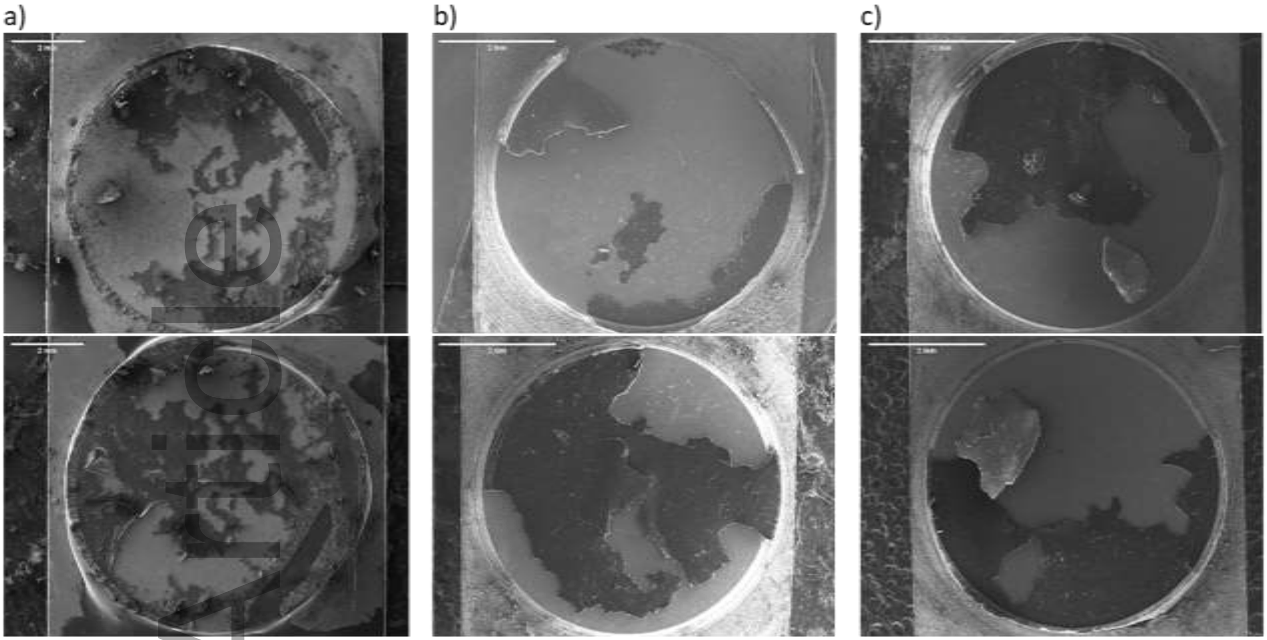

Figure 5. Typical mixed (adhesive/cohesive) fracture surfaces after torsion tests for: a) as cured sample; b) samples treated in the salt spray for 14 days and c) for 28 days.

Since all fractures exhibited a mixed adhesive/cohesive nature with similar adhesive distribution across both substrates, the investigation into the decrease in mechanical properties focused on their cross-sections. This examination aimed to identify potential corrosion between the adhesive and the steel substrates following salt spray tests. Figure 6 reports the fracture surfaces representative cross sections of the as-cured sample and the one after 28 days of salt spray treatment.

The compositional analysis done on the adhesive by SEM and EDS on fracture surfaces (figure 5) and in the cross-sections (figure 6) after torsion tests showed no sign of adhesive degradation in saline environment after 14 and 28 days: the adhesive composition is the same one before and after saline ageing, and there is no sign of corrosion or oxidation reactions inside the adhesive.

Even in the 28-day treated sample, there is no observable corrosion in all the observed cross sections, even at higher magnification (figure 6 b), while in the metal substrate not covered by the adhesive, the corrosion is quite evident and the corrosion products were analyzed by EDS analysis, as in figure 6c.

Actually, the adhesive is acting as a protection layer for steel: in all joined areas where steel is in contact with the adhesive, there is no sign of corrosion (figures 5,6 and7), both after 14 and 28 days of saline ageing.

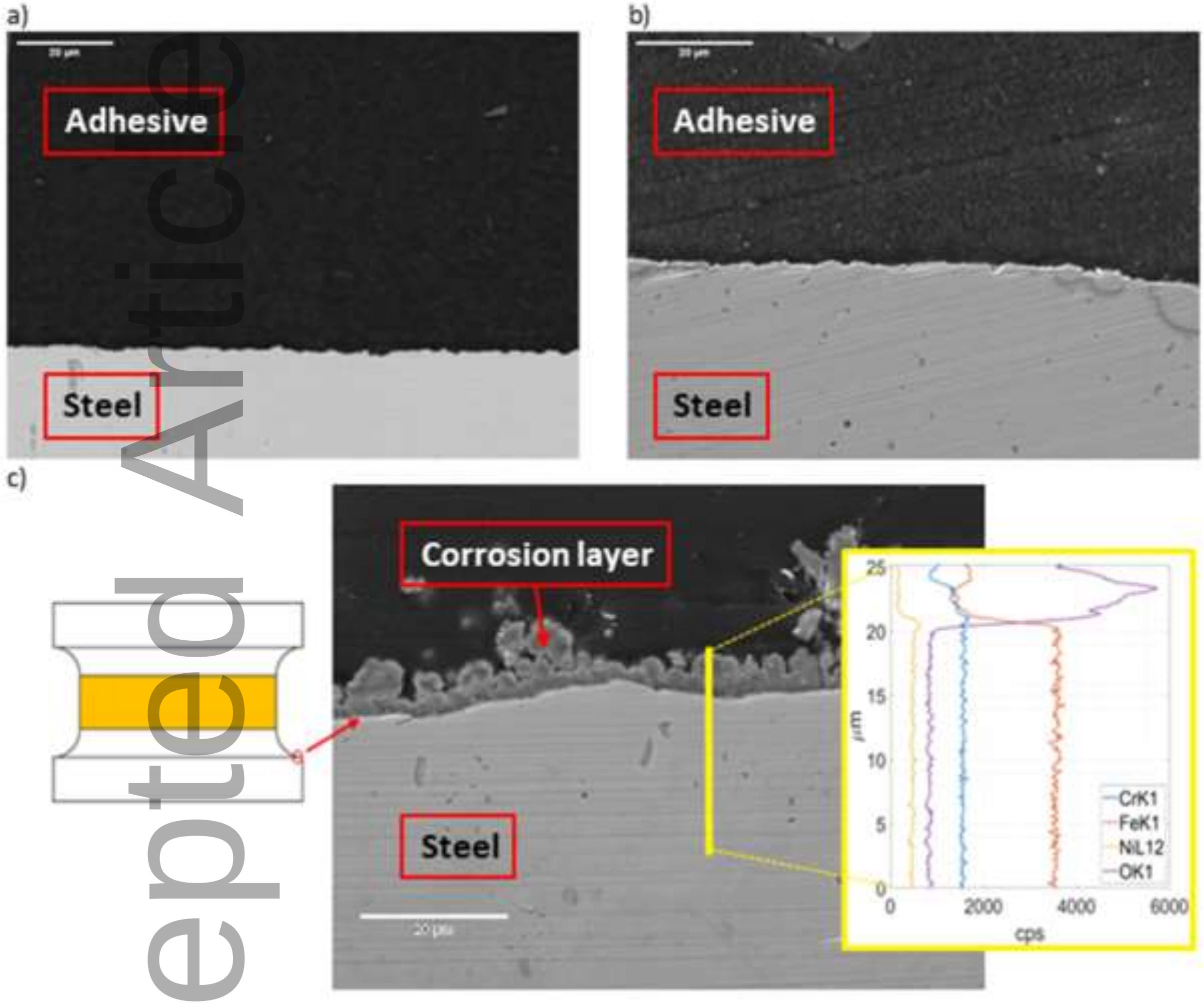

Figure 6. Representative cross sections of: a) as cured samples; b) treated sample for 28 days and c) corrosion layer with its EDS analysis on the steel surface exposed to the salt spray treatment.

On the contrary, the steel corrosion is evident outside the joined area (figure 6 c, 7) both after 14 and 28 days treatments.

After further observation of the joint external perimeter of all samples, the presence of some corrosion zones was noted (figure 7). This could result in the premature failure of joints that are exposed to a salt environment, primarily due to corrosion occurring at the perimeter of the torsion hourglass where stress is at its peak, thus initiating the failure process. In Figure 7 the corrosion in the metal substrate close to the joint perimeter has been highlighted.

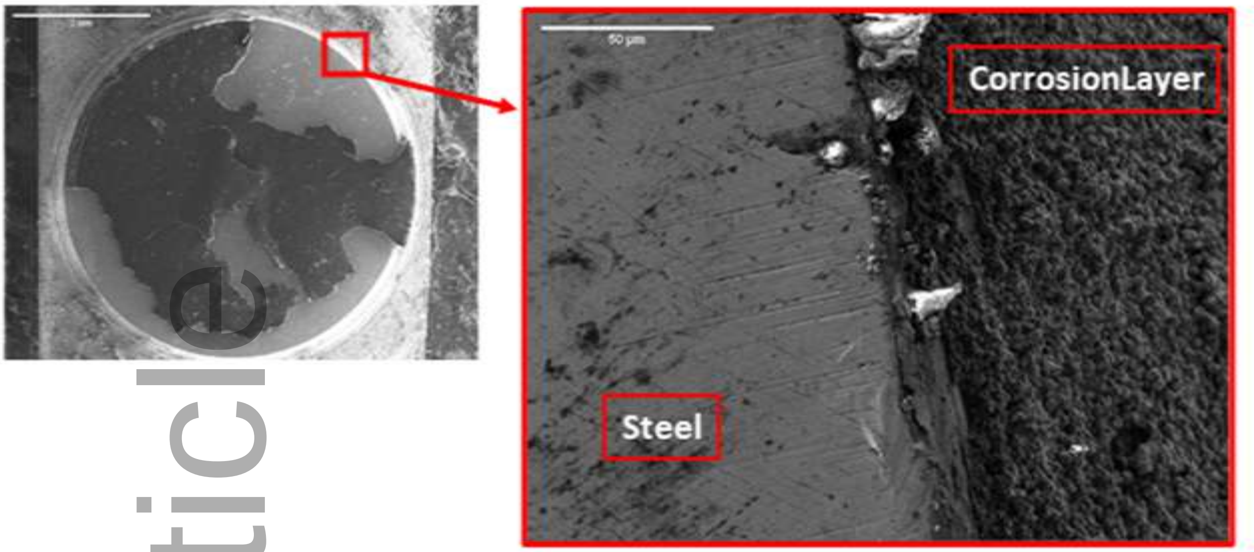

Figure 7. Higher magnification of the fracture surface in the external perimeter zone of the hourglass treated for 14 days in the salt spray environment.

Since the corrosion is close to the joined area, there is a measurable effect on the joined samples shear strength after saline ageing.

Steel corrosion products are present at the perimeter of the joined region (figure 7): consequently, a lower shear strength has been measured by torsion test on the joined samples after 14 and 28 days in saline environment.

The corrosion affected the steel-adhesive interface along the joined region perimeter, where stress is at its peak during the torsion test: thus, the corrosion acts as a crack initiator along the joined perimeter.

Since the steel corrosion increases with time, a lower shear strength has been measured by torsion tests after 28 days treatment, compared to what measured after 14 days.

4. Conclusions

The combination of a certain corrosion in the perimeter zone of the joint and possible chemical degradation of the adhesive's mechanical properties when exposed to a harsh environment leads to a significant reduction in the strength of the adhesive joint, even within a relatively short timeframe. This evidence suggests that safeguarding the bonded area, such as through the use of an additional adhesive or paint, could prove beneficial in extending the lifespan of components joined with adhesives and exposed to saline environments.

Additionally, it is important to highlight that this torsion test not only assesses the pure shear strength of adhesive joints but is also highly sensitive to even minor defects that may arise in the perimeter zone of the bonded sample. This sensitivity can be of great utility in predicting the durability of adhesively joined components operating in hostile environments.

An improvement in shear strength could also be obtained by an interlocked structure between substrate and adhesive, which could improve the as cured shear strength. However, the effect of the saline environment on such an interlocked structure is still unknown.

Further development of this research will investigate: -the increase of exposure time to the saline environment until a possible stabilization of the joints mechanical properties is reached; -the application of protective coatings on steel outside the joined area to protect steel from corrosion; -to age and test real components as in [6]: -to test different substrates and adhesive in harsh environments.

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Table of Contents

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In contemporary industrial applications like automotive, aerospace, and construction, adhesives play a crucial role in maintaining structural integrity. This study examines the effects of ASTM B117-simulated saline conditions on epoxy-bonded AISI304 steel. Torsional shear strength is presently being assessed through a 14- and 28-day salt spray test, utilizing analytical modeling to extrapolate shear strength. These findings offer current insights for developing resilient structural designs in challenging environments.