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Effect of Saline Environment on Mechanical Properties of Adhesive Joints

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Abstract

Literature reports very few works about the effect of corrosive environments on the mechanical properties of adhesive joints. Therefore, the present study intends to contribute for a better understanding of the effect of saline solution on the mechanical properties of single-lap adhesive joints. The specimens were manufactured using Docol 1000 high strength steel plates with 1 mm of thickness and Araldite® 420 A/B epoxy adhesive. The static shear strength of the joints was influenced by the exposure time in saline solutions only up to 120 hours, and remained, after this period, nearly constant. In terms of fatigue strength, for 10^5 cycles, a decrease about 25% and 39% occurred in specimens immersed during 120 hours in deionised water and saline solution, respectively, comparatively to the control samples.

Keywords**:** Lap-shear; Durability; Fatigue; Saline environment.

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1. Introduction

Adhesive joints offer advantages relatively to conventional joining processes, namely acoustic isolation, vibration attenuation, reduction of corrosion problems, and a more uniform stress distribution. Also adhesive bonding is a cheap, fast and robust joining technique increasingly used in structural applications, namely in automotive, aeronautic, aerospace, electronics and electric industries [1; 2]. In fact, this technique has obvious advantages; however, its limited ability to withstand the aggressive environments is a considerable restriction for many applications.

The main environmental factors in climatic exposure are temperature and humidity [3]. In terms of temperature, according to Banea *et al.* [4], the most significant factors that determine the strength of an adhesive joint are: the cure shrinkage, the coefficient of thermal expansion of adhesive and the change in adhesive mechanical properties with temperature. As a consequence of the polymeric nature of the adhesives, the glass transition temperature (Tg) is a very important parameter because Tg establishes the service environment adequate for the materials' usage. At high-temperatures, for example, the load transmission capability of the adhesive joints decreases because the stiffness and strength of the adhesive decreases [5]. Additionally, adhesives suitable for high-temperatures are generally brittle at low-temperatures, giving low joint strengths at low-temperatures, while adhesives suitable for low-temperature are too weak or degrade at high temperatures [6].

Relatively to moisture, its presence in adhesive joints may not only weaken the physical and chemical properties of the adhesive itself but also the interface between the adhesive and the adherend [3]. However, the fracture behavior of the adhesive joints can be significantly affected by adherend materials [7]. For example, composite adherends can absorb water, which will affect the kinetics of water absorption into the adhesive. When a metallic joint is exposed to moisture, water enters the interface either by diffusion through the adhesive layer or by wicking along the adhesive/adherend interface [8]. According some authors [9-11], the water mainly enters the joint through diffusion and the wicking (absorption) only takes place in the presence of pre-existing microcracks or debonded areas at the interface. On the other hand, the loss in joint strength due to water uptake in bonded metal joints is often found to be caused by the degradation of the adhesive/adherend interface rather than weakening of the bulk adhesive [8].

Considering the environmental durability of adhesive joints, the most detrimental condition is hot-wet exposure [7]. In these conditions, the joint strength can dramatically be decreased as a function of exposed time. Studies developed by Ferreira *et al.* [12] and Reis *et al.* [13], on polypropylene (PP) reinforced glass fiber adhesive lap joints, showed that long immersion times promotes an important decrease in static strength of the adhesive joints when immersed in water at room temperature and at 40 ºC. This strength loss was about 30% and occurred at both temperatures. However, the behaviour for immersion times lower than 45 days depended on water temperature. At 40 ºC the degradation caused by water attack is faster than at room temperature. In the first case a sudden loss of static strength was observed after 15 days but, after this period, no influence on the strength was observed. At room temperature no influence was observed up to 15 days but, after this period, the shear stress decreased about 30% up to 45 days and remained nearly constant again. In terms of fatigue performance Ferreira *et al.* [12] compared the fatigue strength of adhesive joints subjected to 30 days immersion in water at 40 ºC and 8 days in water at 70 ºC. In the first case (water immersion at 40 ºC) only a reduced influence in fatigue strength was observed but an enormous loss of fatigue strength was observed at 70 ºC. According to studies developed by Ashcroft *et al.* [14] on lap-strap joints, there was a little effect on fatigue threshold when the samples were aged in a humid environment, until saturation, but when moisture and temperature were combined a significant effect on the fatigue threshold was observed. This can be explained by the reduction of the T_g of the adhesive. When the test temperature is very close to T_g, a sharp reduction in the mechanical properties of the adhesive occurs, which drastically reduces the fatigue resistance of the joints.

In fact, the open literature presents several studies about the effects of moisture and temperature on adhesive joints strength but there are very few works about the effect of highly corrosive environments. Prolongo and Urena [15], for example, studied the durability of epoxy–aluminium joints, with a homopolymerised epoxy resin, under a saline environment and observed that the degradation of the joint occurred on the adherends by corrosion. On the other hand, for saline environments, Del Real *et al.* [16]

showed that the durability of adhesive joints can be increased significantly with surface treatments. Therefore, this work intends to contribute for a better understanding of the effect of corrosive environments on the mechanical properties of single-lap adhesive joints. A saline environment was considered and the adhesive joints performance was analyzed by tensile and fatigue tests.

2. Materials and experimental testing

Docol 1000 high strength steel (SSAB, Borlänge, Sweden) plates with 1 mm thickness was the material used for the adherends of the single-lap joints studied. The mechanical properties were obtained from tension static tests, performed according with ASTM E 8M Standard [17], and are presented in Table 1. More details about this material can be found by Reis *et al.* [18; 19] and by Cognard *et al.* [20] for adhesive.

The specimens were manufactured as 20 mm wide strips cut from the plates and bonded with "Araldite® 420 A/B" adhesive epoxy (Huntsman Advanced Materials, Everberg, Belgium). The properties of this adhesive are shown in Table 1 and were obtained from Cognard *et al.* [21] and by Moura *et al.* [22]. The geometry and dimensions of the specimens are presented in Figure 1. An adhesive thickness (t_g) of 150 μm was used and the cure procedure, as suggested by the adhesive's supplier [23], occurred during 4 hours at 50 ºC in a climatic chamber. Careful surface preparation was taken into account in order to obtain improved adhesion. For this purpose, abrasive polishing with silicon carbide paper type P220 was used and, finally, the surface was cleaned with dry air and solvent wiping. The transverse section of the joints was observed under a optical microscope, Mitutoyo – Toolmaker's Microscope TM, (Mitutoyo Corporation, Kawasaki, Japan) and the various measurements of bond thickness were registered using a micrometric base. An average value of 5 microns was obtained without significant dispersion (standard deviation, SD ± 2.5 μm). Roughness measurements were carried out along two specimen directions (longitudinal and transversal) using a Mahr MarSurf Perthometer M2 (Mahr GmbH - Carl-Mahr, Göttingen, Germany) (R_z = 4.66 ± 0.34 μm, according [24; 25]). The mean roughness depth (R_z) is the arithmetic mean value of the single roughness depths of five consecutive sampling lengths.

“Insert Table 1”

The static strength was obtained using an electromechanical Instron Universal Testing machine (Instron, High Wycombe, UK), model 4206, at room temperature and with a displacement rate of 1 mm/min. Five specimens were tested for each test condition until the final failure of the joint. Specimen elongation was measured using an strain gauge extensometer with 50 mm reference length (Instron, model A1439-1007). The constant amplitude loading fatigue tests were carried out in tension using a Dartec 100 kN servo-hydraulic mechanical testing machine. Tests were performed at room temperature, using a sinusoidal wave load at constant amplitude with a load ratio R= 0.05 and a frequency of 20 Hz. To minimize the bending stresses during the tests, tabs with the same thickness of the sheets were used, as can be seen in Figure 1.

“Insert Figure 1”

Different hostile environmental conditions were studied, which are summarized in Table 2. The NSS specimens were subjected to corrosion conditions, in artificial and water constant atmosphere, in a chamber with neutral salt spray [26]. The WD specimens were subjected to a fully immersed deionised water and the TEMP specimens were aged at controlled temperature and humidity in a climatic chamber. After exposure, the specimens were then tested.

“Insert Table 2”

3. Results and discussion

3.1. Tensile shear tests

Figure 2 shows typical load-displacement curves obtained for single lap joints previously subjected to different environmental conditions. Figure 2a shows the effect of different environments while Figure 2b shows the effect of exposure time for the neutral saline solution. The curves show a nearly linear behaviour for relatively low load levels, while for higher values a non-linear region occurs. A similar behaviour was found by Reis *et al.* [18] for the same adhesive and adherends. On the other hand, the displacement for the failure loads depend on the environmental conditions and the lowest values occur for the adhesive joints exposed to saline solutions. According with Grant *et al.* [27] and da Silva *et al.* [28] the maximum adhesive strain has a limit and the failure occurs when it is exceeded. This limit seems to be dependent on the environmental conditions and/or adhesive/adherent interface strength.

"Insert Figure 2"

Figure 3 shows the typical failure surface morphology obtained for control samples (ND), specimens under temperature degradation (TEMP) and specimens subjected to the neutral saline solution degradation (NSS). A visual observation and optical microscopy indicated the occurrence of adhesive failure in all series with larger interface failure areas on steel adherends and agrees with Kerr *et al*. [29] and Bowditch [30]. For the control samples and specimens under temperature, failure occurred near the extremities of the joint where the stresses present the maximum values which agrees with [18; 31]. On the other hand, the samples subjected to the saline degradation present different corrosion points which promote multipoint failure initiation. This phenomenon agrees with the studies developed by Prolongo and Urena [15], where, under a saline environment, the degradation of the joint occurred by corrosion.

Figures 3c and 3d show peripheral and internal corrosion points, as a consequence of the direct contact metal/solution. In fact, for metal adherends, Ashcroft and J. Comyn [32] and Comyn [33] suggests that the water may enter joints by: a) diffusion through the adhesive; b) transport along the interface; c) capillary action through cracks and crazes in the adhesive. If it is well known that the loss of fracture strength in adhesive joints is often attributable to the presence of water, this solution (NSS) not only decrease the interface strength but promote, at same time, corrosive points with consequence drop of the adhesive strength. On the other hand, the water distribution is governed by the exposure time, water concentration in the environment and the diffusion properties of adhesives at a given temperature. This suggests that the corrosion of the adherends was accelerated by the saline solution and temperature.

"Insert Figure 3"

Figure 4 shows the average shear strength as a function of the exposure time for different environmental conditions. For the specimens tested after saline exposure, it is possible to observe that the average shear strength decreases up to 120 hours, by about 17.5% relatively to the control samples, and, after this exposure time remains practically constant. According to Figure 3, it is possible to conclude that the corrosion of the adherends appears during the first 120 hours of exposure (for Docol 1000 high strength steel) and, after this exposure time, the adhesive joint strength is independent of the contact time with this environment (saline solution at 35ºC). Despite the temperature effect on the adhesive joint strength, Figure 4 shows that during the first 24 hours the shear strength increases around 12% and, after this time, is practically constant after a slight decrease. These results suggest that a post cure of 24 hours at 35 ºC is recommended to optimize the adhesive (Araldite® 420 A/B epoxy) strength which agrees with other authors, namely Cognard *et al.* [21; 22]. On the other hand, the literature associates the temperature with significant decreases of residual strength [34-38]. In fact, only a marginal effect was observed here, which may be explained by the relatively small difference of temperatures studied (20 to 35ºC).

“Insert Figure 4”

3.2. Fatigue testing

The fatigue strength for different environmental conditions (Control samples, ND; deionised water, WD; neutral saline solution, NSS) was analyzed in terms of S-N curves. These S-N curves represent the number of cycles to failure versus maximum shear stresses. According to the static tests, the exposure time of 120 hours was determinant in terms of static strength for samples exposed to saline solutions. Therefore, the fatigue strength for samples subjected to deionised water and neutral saline solution was obtained for 120 hours of exposition. For fatigue tests, each point in Figure 5 indicates a single fatigue test.

Figure 5 presents the S-N curves, which plot the maximum nominal shear stress versus number of cycles to failure. As expected, the maximum fatigue strength was obtained for the control samples (ND). For 10^5 cycles, decreases about 25% and 39% were obtained for samples immersed in deionised water (WD) and neutral saline solution (NSS), respectively.

"Insert Figure 5"

These results agree with the literature [10; 12; 39-40] and are consequence of the hydrophilic nature of adhesives, which is caused by the polar groups needed to confer adhesive properties to polymeric materials [41; 42]. Water can enter in the adhesive, then attack it by diffusion through the adhesive/adherend and, finally, diffuse along the interface and move by capillary action through cracks in the adhesive [41]. According to Ferreira *et al.* [12], the effect of water exposure on the fatigue behaviour is mainly determined by the water temperature and to a lesser degree by the exposure time. On the other hand, saline solutions promote essentially significant damage in terms of adhesive/adherend interface strength according to Lee [43]. In fact, this phenomenon can be confirmed by the analysis of Figure 6, which represents typical fatigue failure surfaces for the different environmental expositions. Similarly to the static tests, an adhesive failure was observed for all cases confirming that the interface is the weakest region of the joint. Once again, the corrosion of the adherends can be observed in Figure 6b), for WD specimens, and in Figure 6c) for saline exposure. In the last picture are evident points of corrosion occuring for NSS environments. These points promote localized increases of stress reducing the initiation time of fatigue cracks with consequent reduction of fatigue life.

"Insert Figure 6"

The stiffness value is frequently used as a fatigue damage parameter of the joint [44]. Therefore, periodically during fatigue tests, the load and correspondent displacement of the specimens were monitored. The stiffness, E, was defined by the ratio of the axial load and the axial displacement. Figure 7 plots E/E_0 versus N/N_f, where E_0 is the initial value of E, N the current number of cycles and N_f is the number of cycles to failure. Two values of maximum axial load were analyzed: 1.4 kN on Figure 7a) and 2.0 kN on Figure 7b).

“Insert Figure 7”

A slight and stable decrease of E/E_0 until nearly final failure can be observed, for all cases. For the control samples the degradation process starts at N/N_f = 0.4 (i.e, at about 40% of the total fatigue life), which is later than for the other conditions (WD and NSS).

4. Conclusions

The present work studied the tensile static strength and fatigue strength of single-lap joints under different environmental conditions: deionised water, neutral saline solution and temperature /relative humidity controlled.

The displacement at static failure loads shows to be dependent with the environmental conditions and the exposure time, especially for the saline solutions. An adhesive failure was observed for all series, which is a consequence of the interface degradation. For saline solutions the corrosion points that occurred on the adherends have a major influence on the adhesive joint strength. In terms of tensile static strength, the saline solutions decreased the performance of adhesive joints (during the first 120 hours, remaining constant after this time), while the temperature promoted a better performance (during the first 24 hours and, after this time, remained nearly constant).

In terms of fatigue strength, the water exposition promoted a significant effect but the saline solutions decreased significantly the fatigue life. The variation reached 39% (for 10^5 cycles) in relation to the control samples. The corrosion that occurred on the adherends was determinant on the fatigue failure mechanism as observed for the static tests. Finally, the stiffness monitored during the fatigue tests decreased with the number of cycles, evidencing the fatigue damage evolution. It was evident that the degradation process is faster in severe environments than for the control samples.

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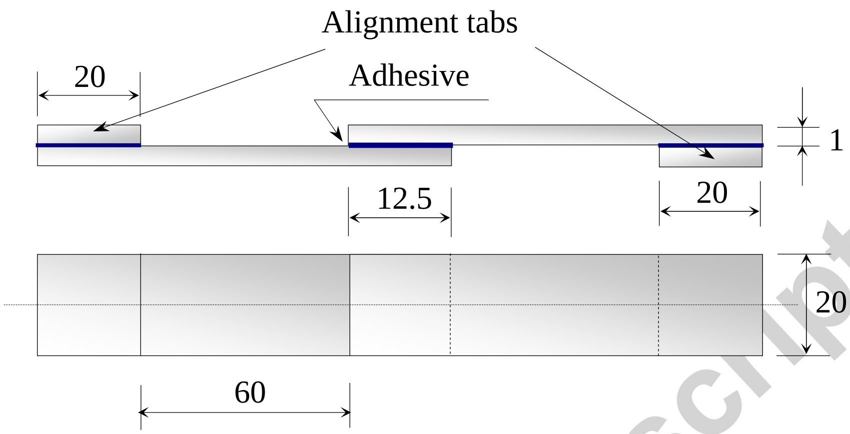
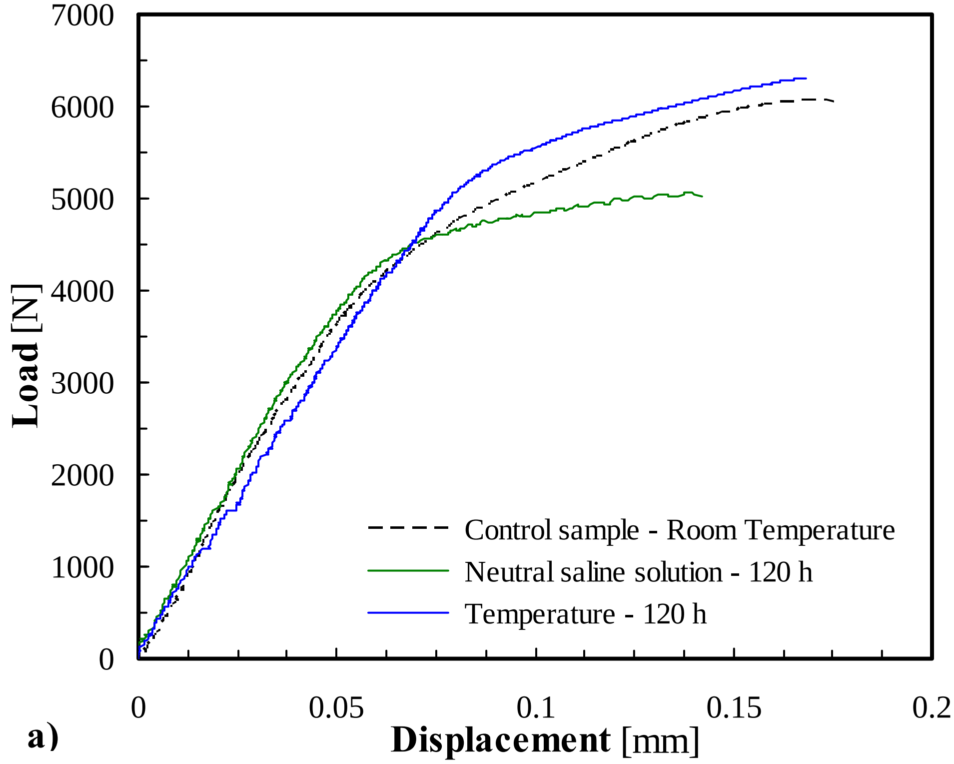
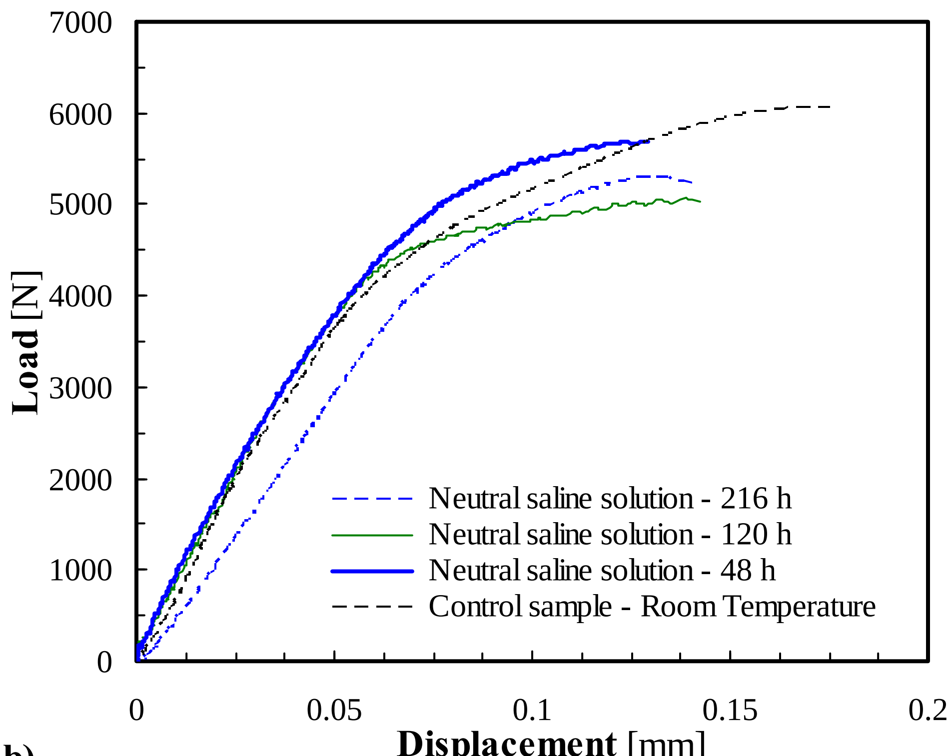


Figure 1. Specimen geometry with a 150 µm adhesive thickness (dimensions in mm).



7000
6000
5000
4000
3000
2000
1000
0
Load [N]
0
0.05
0.1
0.15
0.2
Displacement [mm]
Control sample - Room Temperature
Neutral saline solution - 120 h
Temperature - 120 h

a)



7000
6000
5000
4000
3000
2000
1000
0
Load [N]
0
0.05
0.1
0.15
0.2
Displacement [mm]
Neutral saline solution - 216 h
Neutral saline solution - 120 h
Neutral saline solution - 48 h
Control sample - Room Temperature

b)

Figure 2. Load-displacement curves. a) Joints subjected to different environmental conditions; b) Joints subjected to different time exposure to saline solutions.

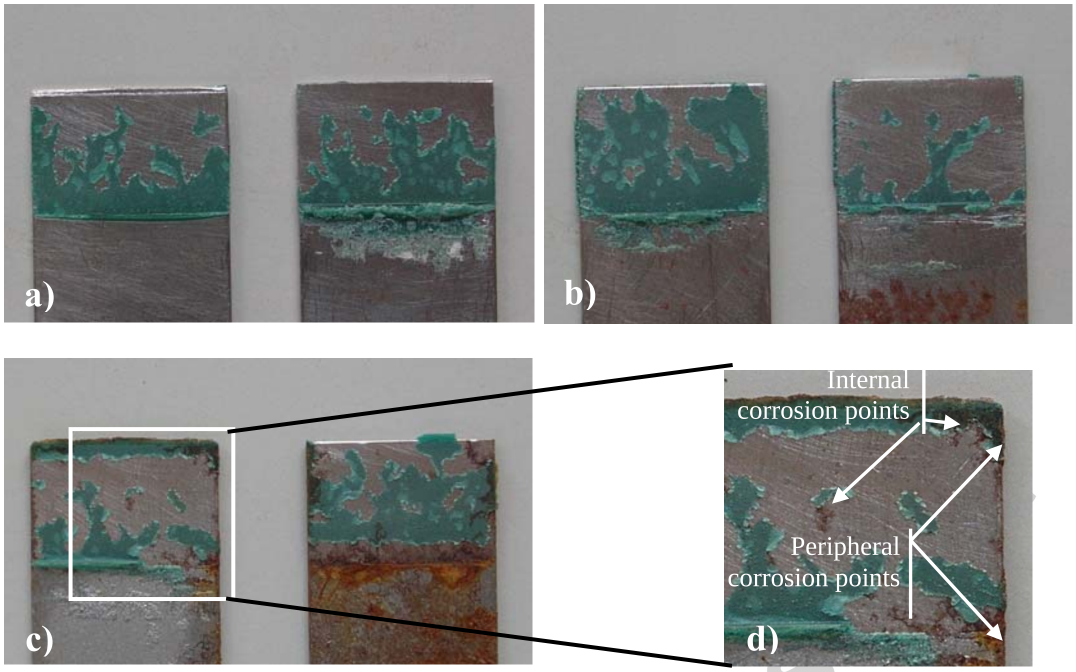


Figure 3. Typical failure surfaces for the different conditions: a) ND; b) TEMP (exposure time = 120h); c) NSS (exposure time = 120h); d) Detail of internal corrosion points and peripheral corrosion points.

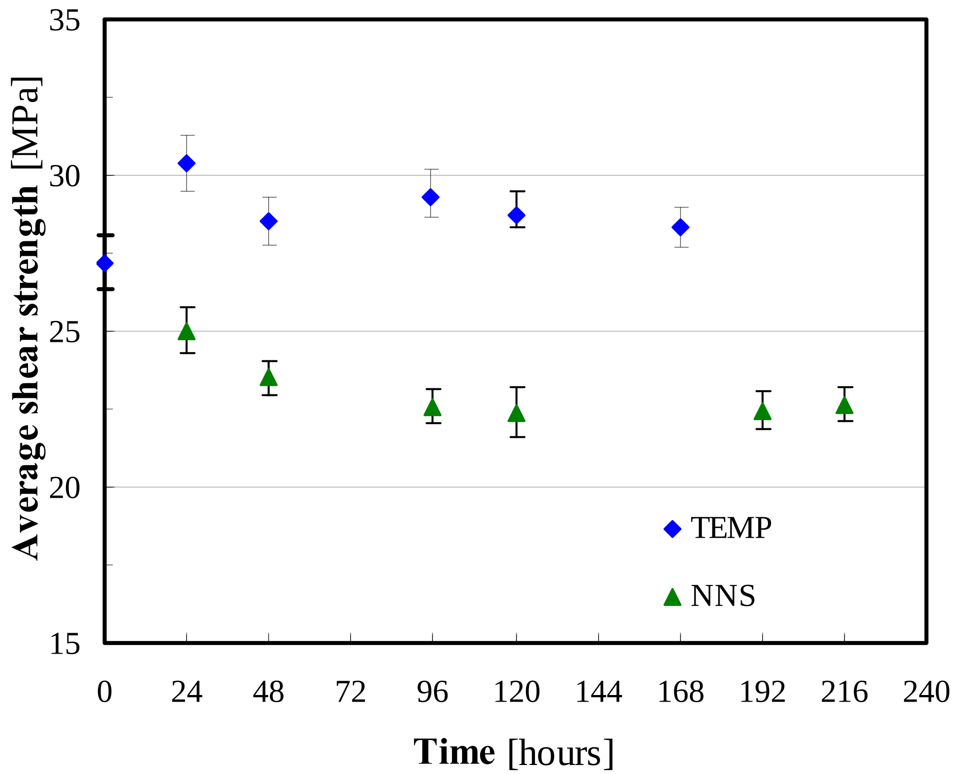


Figure 4. Average shear strength as a function of the exposure time for different environmental conditions.

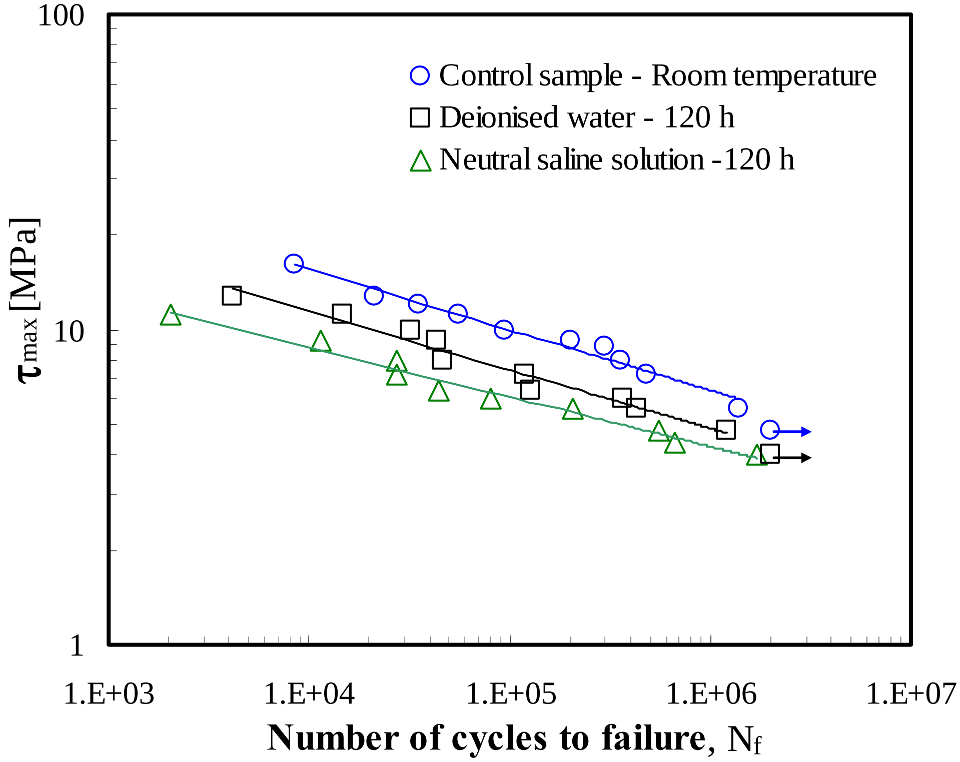


Figure 5. S-N curves for different environmental conditions (arrows indicate run-outs).

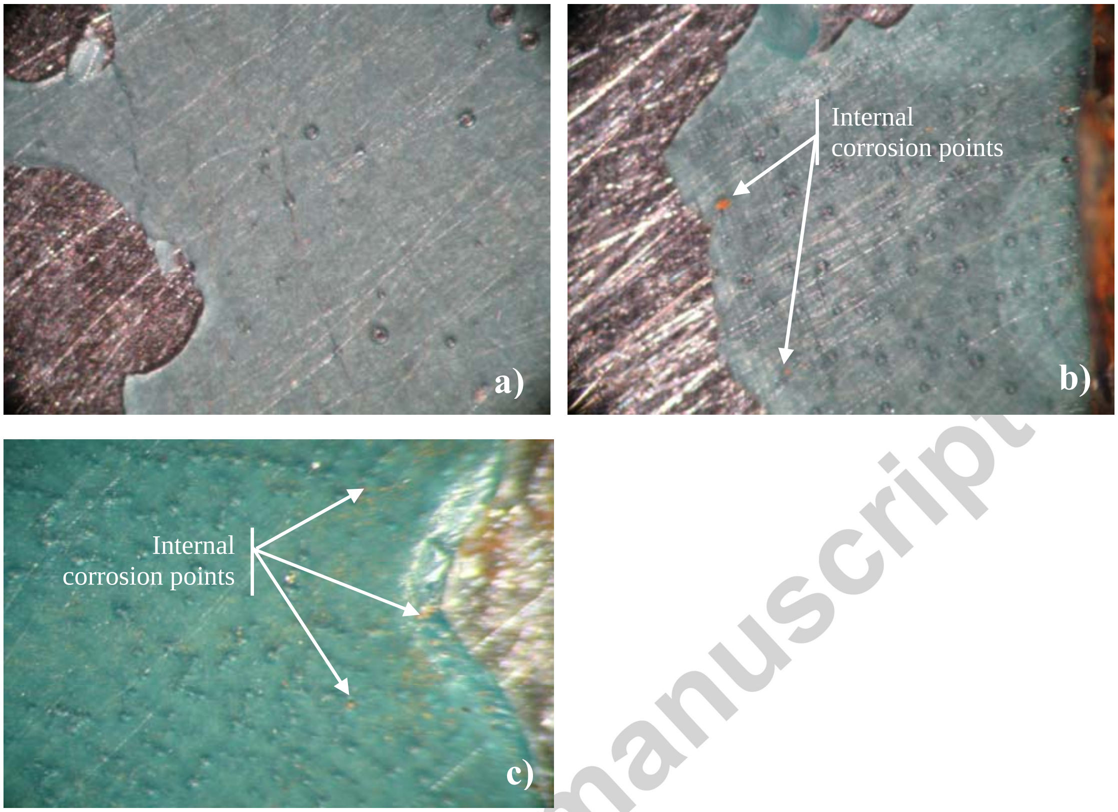
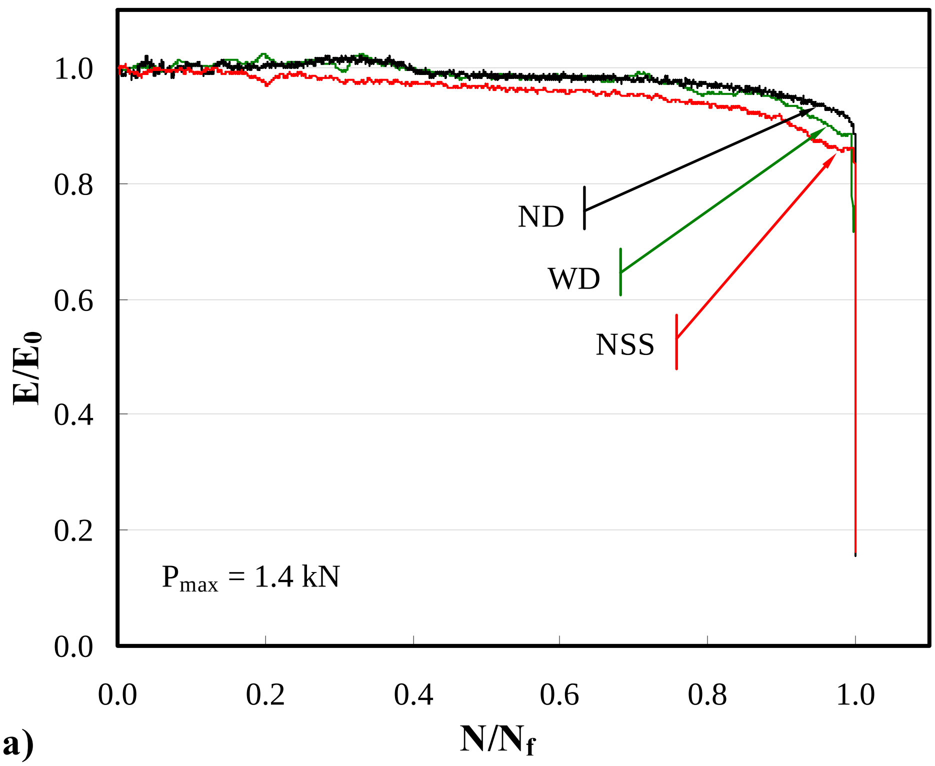
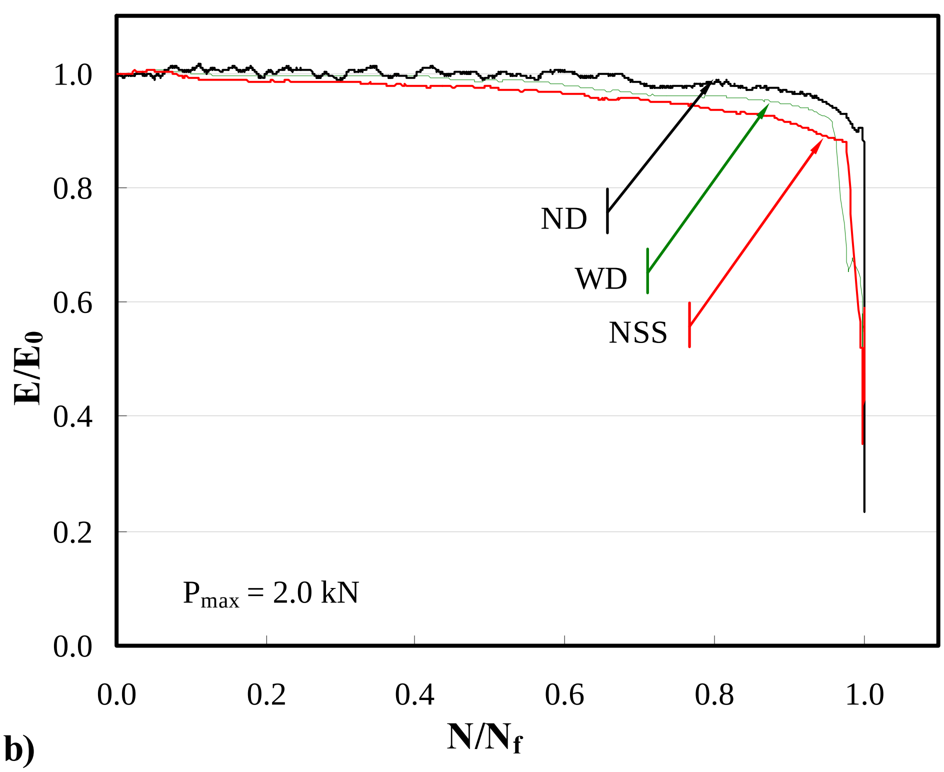


Figure 6. Micrography of typical failure surfaces of the single lap joints fatigue testing: **a)** ND exposure condition; **b)** WD exposure condition; **c)** NSS exposure condition.


1.0
0.8
0.6
0.4
0.2
0.0
E/E0
0.0
0.2
0.4
0.6
0.8
1.0
N/Nf
ND
WD
NSS
Pmax = 1.4 kN

a)


1.0
0.8
0.6
0.4
0.2
0.0
E/E0
0.0
0.2
0.4
0.6
0.8
1.0
N/Nf
ND
WD
NSS
Pmax = 2.0 kN

b)

Figure 7. E/E_0 against the normalized number of cycles N/N_f for: a) maximum load of 1.4 kN; b) maximum load of 2.0 kN.

TABLES

Table 1. Mechanical properties of the adherends and adhesive, (σ_{UTS} - tensile strength, σ_{ys} - yield strength, E - Young's modulus, ε_f - elongation at failure, ν - Poisson's ratio).

Material	σ_{UTS} [MPa]	σ_{YS} [MPa]	**E** [GPa]	ε_f [%]	ν [-]
Docol 1000 High strength steel	1052.0	502.0	205.0	11.6	-
Adhesive (Araldite® 420 A/B) [20; 22]	35.0	27.0	1.85	8.5	0.3

Table 2. Environmental conditions studied.

Series	**Environment / exposure**	**Condition**	**Exposure time** [hours]
ND	Control samples	20±2 ºC; 50±2 % HR	---
WD	Deionised water	35±2 ºC; pH: 6.7	120
NSS	Neutral saline solution [26]	35±2 ºC; pH: 7	24/48/96/120/192/216
TEMP	Temperature and relative humidity	35±2 ºC; 25±2 % HR	24/48/96/120/168