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COB-2019-2259 CREEP BEHAVIOR OF METAL BONDED JOINTS

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Abstract. Numerous advantages presented by the use of adhesive bonded joints have been noted, leading to an increased use of this type of joining of materials in place of traditional methods. Despite this growth, little information is found in the literature on the behavior of these unions under creep, due to the high execution time of these tests when compared with other destructive tests. With the aid of the Creep Pneumatic Equipment (CPE) created by the Adhesion and Adherence Laboratory (AAL / UERJ / IPRJ), is able to test up to 10 joints at the same time. An analysis of creep behavior of single lap joints (SLJ) composed of metallic substrates and bonded with an epoxy based adhesive (NVT 210E) was performed. These joints were manufactured according to ASTM D 1002 and RT TMEC n. 003/08/PETROBRAS bonding procedure. Joint tensile tests were performed with the Shimadzu Universal Testing Machine to obtain the rupture joint shear stress. The joints were tested under creep in the Creep Pneumatic Equipment (CPE) at the following percentages of the rupture stress: 100%, 68.4%, 59.5%, 45.6%, 39.7% and 49.6%. NVT-bonded joints exceeded 1000 hours of creep test with 45.6% of rupture stress, which means 8.1 MPa. Joints loaded with 49.6% of the rupture stress are still being tested. The fractures presented after the creep tests were mixed, both cohesive and adhesive.

Keywords: creep, bonded joints, structural adhesives, epoxy

1. INTRODUCTION

The structural adhesives used in bonded joints have gained prominence in recent years. The numerous advantages provided such as: do not need thermal input, do not concentrate stresses, besides being the best way to join different materials; are reasons for the replacement of screws, welds and rivets by these adhesives. Metal joints joined by structural adhesives have found enormous applications in industrial processes, mainly in the offshore industry. Because of this, AAL has conducted studies and has used adhesives for repairs to offshore pipelines, including the NVT adhesive created by NOVATEC in partnership with the laboratory, which is a rigid epoxy-based adhesive.

Su and Makie (1993) argue that studying the creep behavior of adhesive joints in structural applications is necessary to increase their durability and reliability. The study of the creep behavior of bonded joints is a subject of great importance for the design of structural adhesive joints, and also for the AAL, since several adhesive repairs have been performed by the laboratory, mainly with the NVT 210E. Due to this, some work was carried out in the AAL to study the creep behavior of the bonded joints. Queiroz (2013) for example, studied the creep behavior of single lap joints bonded with a rigid and a flexible adhesive.

The creep behavior of single lap joints with the structural adhesive NVT 210E had not yet been performed, this being one of the relevant items of this work.

The Creep Pneumatic Equipment (CPE) was used to perform the tests and the linear regression technique to obtain the curves representing the creep behavior of the adhesives.

2. EXPERIMENTAL ANALYSIS

2.1 Production of joints

The material of the metal sheets chosen was ASTM A36, cold-rolled, with following dimensions: 25.4 mm X 101.6 mm X 1.5 mm, according with the ASTM D 1002 standard and with the RT TMEC 003/08/PETROBRAS bonding procedure. The Figure 1 shows the specimen used according with ASTM D 1002 standard.

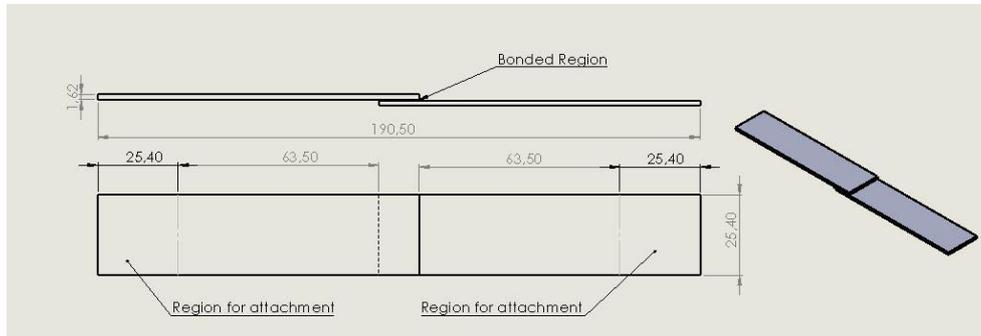


Figure 1. The specimen used in the experiment.

The standard ASTM D 1002 instituted Eq. (1), which shows the relationship between overlap length (L) and substrate thickness (t). A variation between these values may occur, provided that the force to break the specimens does not exceed the yield stress of the substrate. The overlap is the bonded region of the joint.

$$L = \sigma_{xy} \frac{t}{1.5\tau_f} \quad (1)$$

where

L = overlap length, [mm];

σ_{xy} = yield stress of substrate material, [MPa];

t = substrate thickness, [mm];

τ_f = shear fracture stress of joint, [MPa].

The template used for bonding joints restricts the variation of substrate thickness and overlap length. ASTM A36 steel was chosen as the metallic substrate, so the yield stress is fixed. Since the substrate yield stress is 250 MPa, the substrate thickness is 1.5 mm and the overlap length is 12.7 mm, so it is possible to manufacture joints whose shear stresses do not exceed 19.5 MPa.

The adhesive used was the NVT 210E, which is a semi-flexible epoxy-based structural adhesive, created by the AAL. The NVT 210E is used for: duct recovery, sheet metal bonding, surface leveling, filling of worn parts, duct seals, tanks, bonding of different types of materials, anti-corrosion coating. The thickness of the adhesive used in this work was 0.5 mm.

2.2 Surface treatments and roughness analysis

Shot blasting and silanization were performed as surface treatments prior to bonding. Shot blasting with iron blast G25, to achieve the required roughness level, which is the R_t greater than $85\mu\text{m}$, according to Annex N-9 Petrobras. Silanization with 1% hydrolyzed silane (it took 35 minutes to tap into hydrolyzed water to be prepared). The silane was sprayed and not bathed, to better simulate the situation in the field, because it is more used. The Figure 2 illustrates the silanization procedure used in this work.



Figure 2. Silanization procedure.

After these surface treatments, the roughness measurements were performed with the TalyScan 150 three-dimensional rugosimeter Taylor Hobson. With a three-dimensional rugosimeter, a speed of 1000 $\mu\text{m/s}$ was used, with an accuracy of $1 \times 10 \mu\text{m}$ x-y mesh and a measuring area of $4 \times 1 \text{ mm}$ according to ISO 4288: 1996. The Figure 3 shows an example of the three-dimensional result of surface roughness analysis made by Taylor Hobson rugosimeter.

After the silanization procedure, roughness measurements were performed again to compare with results prior to silanization, according to Tab. 1.

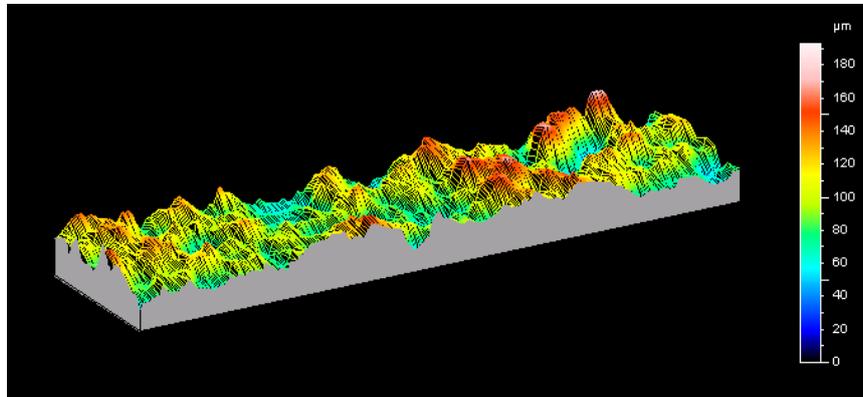


Figure 3. Three-dimensional result of surface roughness analysis.

Table 1. Roughness results before and after silanization.

Post superficial treatment		Post silanization	
Ra(μm)	Rt(μm)	Ra(μm)	Rt(μm)
10.2 \pm 1.7	73.8 \pm 13.3	10.0 \pm 1.5	72.7 \pm 13.0

It is observed that the result of roughness (Rt - distance between the highest peaks and lower valleys and Ra- an average of measured roughness) decreased slightly after silanization. This is because silanization decreases the roughness peaks, but this procedure creates micro-roughness along the material. Thus, there is a significant increase in the rupture stress of the material. According to studies by Limaverde (2008), silanization had a greater influence factor on the rupture bonded joints stress, with a degree of influence greater than 99.9%, whereas chemical attack had a degree of influence of only 18.7%. In addition, Passos (2016) reported the advantage of using hydrolysed silane primer, due to the ease of spraying method and low cost.

2.3 Bonding procedure of the specimens

For bonding, a bonding template was used (makes it possible to bond 10 joints at a time, according to ASTM D1002). Climatic conditions, such as temperature and humidity, were controlled for bonding joints in accordance with ASTM E 337. The figure 4 shows a joint bonding procedure in the bonding template. Table 2 shows the joints dimensions after bonding and cure process.



Figure 4. Joint bonding procedure.

Table 2. Joint dimensions after bonding and cure process.

Parameters	Values
Total length [mm]	192.0 (±0.0)
Width [mm]	24.9 (±0.2)
Overlap length [mm]	12.9 (±0.2)
Adhesive thickness [mm]	0.5 (±0.1)

2.4 Tests with Universal Testing Machine SHIMADZU

In order to find the maximum joint stress, tensile tests were performed on the SHIMADZU Universal Testing Machine available in TECPOL-IPRJ. These tests used a speed of 1.3 mm/min according to ASTM D 1002.

The initial length (l₀) was 140 mm, which is the total length of the joint minus 25 mm from the ends attached to the claws. The area considered for the calculation of the stress was 322.58 mm².

No extensometer was used, displacement was reported by the test machine. The initial reference corresponds to 100% load (maximum stress). The Figure 5 illustrates a test on the Universal Testing Machine and the fractured specimens.



Figure 5. Test on the Universal Testing Machine.

2.5 Tests with Creep Pneumatic Equipment

The Creep Pneumatic Equipment (CPE) converts the pneumatic inlet pressure into force applied to the specimens. This force depends on the working area and the pressure exerted, as the working area remains unchanged, the pressure is adjusted to vary the tensile stress of the test. The Figure 6 illustrates the CPE.

Two different types of tests were performed in the CPE, the tensile tests (to compare the results with the tests performed on the universal testing machine) and the creep tests.

The tensile tests performed in this work were at a speed of 47.3 kPa/s, which means 0.2 bar increments every 15 seconds, which was adequate for the tests.

For creep tests, it was necessary to adjust the desired pressure according to the percentage of stress required. Recently, Queiroz (2013) developed the Eq. (2), which converts the stress (MPa) to the pressure (bar) required for the test:

$$p \text{ (bar)} = \frac{\tau \text{ (MPa)}}{3,55 \text{ (MPa/bar)}} \quad (2)$$



Figure 6. The specimen used in the creep experiment.

The test specimens are being tested under creep in the machine by varying the stress at some percentages of the maximum stress and the results is presented in the form of graphs. These tests were carried out within a maximum time of 1000 hours, after this time, an extrapolation of the data was performed with the aid of linear regression.

3. RESULTS AND DISCUSSION

3.1 Rupture Stress

With the tensile tests performed by Universal Testing Machine (Tab.3) and Creep Pneumatic Equipment (Tab.4), an average of 17.9MPa was found as the maximum tensile stress for the joints bonded with the NVT 210E adhesive. The Figure 7 illustrates the graph of the tensile test performed on the Universal testing Machine for NVT 201E joints.

Table 3. Results of the tensile tests performed on the Universal Testing Machine for the NVT 201E.

Specimen	Rupture force [N]	Rupture stress [MPa]	Displacement [mm]	Strain [%]	Speed [mm/min]	Time [s]
1	5659.6	17.5	0.6	0.4	1.7	20.5
2	5738.2	17.8	0.6	0.4	2.3	16.2
3	5680.4	17.6	0.5	0.4	2.0	16.1
4	5997.6	18.6	0.6	0.4	1.8	18.5
5	5862.9	18.2	0.6	0.4	1.8	19.2
Average	5787.7	17.9	0.6	0.4	1.9	18.1
Standard deviation %	2.4	2.4	5.9	0.0	12.6	10.5

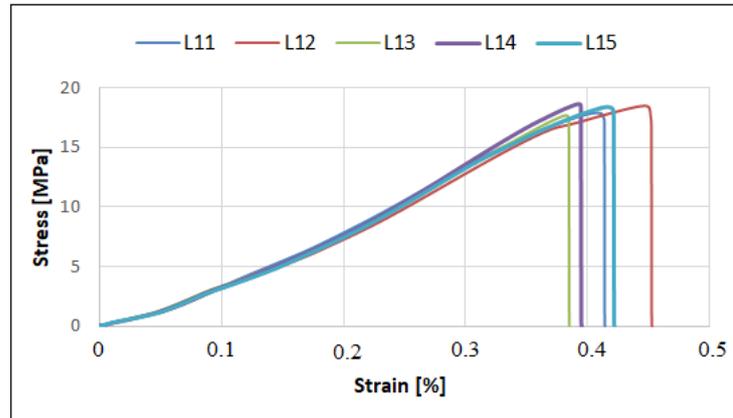


Figure 7. Graph of the tensile test performed on the Universal Testing Machine for NVT 201E joints.

Table 4. Results of the tensile tests performed on the Creep Pneumatic Equipment for the NVT 201E.

Specimen	Rupture Pressure [bar]	Rupture Stress [MPa]	Time [s]	Pressure application rate [bar/min]	Stress application rate [kPa/s]
6	4.8	17.0	360.0	0.8	47.2
7	5.0	17.8	372.0	0.8	47.8
8	5.0	17.8	380.0	0.8	46.8
9	5.3	18.8	390.0	0.8	48.2
10	5.0	17.8	383.0	0.8	46.5
Average	5.0	17.8	377.0	0.0	47.3
Standard Deviation	0.2	0.6	11.5	0.0	0.7
Deviation %	3.6	3.6	3.0	0.0	1.5

3.2 Creep Tests

The Table 5 shows the results of creep tests for the NVT 210E adhesive, the average of stress and the average of time until rupture are presented. The percentage of stress varies in 100%, 68.4%, 59.5%, 45.6%, 39.7% and 49.6% of the maximum stress . These percentages were chosen during the creep tests. The time test until rupture of 45.6 and 39.7 % of the stress exceeded 1000 hours of testing and was withdrawn at 1300 hours without failure. The joints with 49,6% of rupture stress are still testing, with more than 500 hours of test.

Table 5. Results of creep tests for the NVT adhesive.

% of Rupture Stress	Stress [MPa]		Time until rupture [hours]	
	Average	Standard deviation (%)	Average	Standard deviation (%)
100.0	17.9	2.9	0.1	3.0
68.4	12.5	0.0	1.9	71.2
59.5	10.7	0.0	69.9	47.8
45.6	8.1	0.0	>1300.0	0.0
39.7	7.2	0.0	>1300.0	0.0
49.6	8.9	0.0	TESTING	0.0

As the joint of 49.6% of the rupture stress is still under test, for the graphs it was considered a value of 300 hours, but it has already exceeded 500 hours of testing. The Figure 8 presents the stress versus average life time of the shear joints bonded with the NVT 210E adhesive.

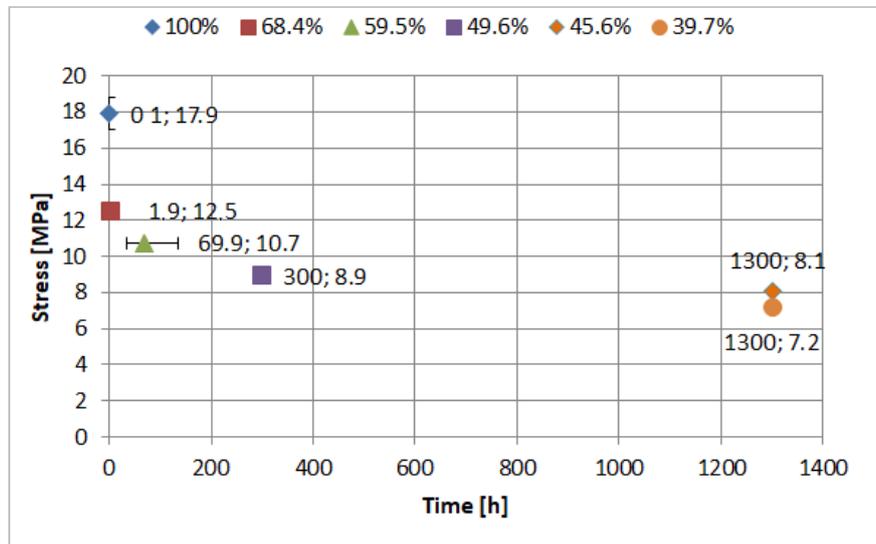


Figure 8. Stress versus time of the shear joints bonded with the NVT 210E adhesive.

3.3 Data extrapolation

The Figure 9 shows the same graphic of the Fig. 8 (stress x time life until rupture), but in logarithm scale.

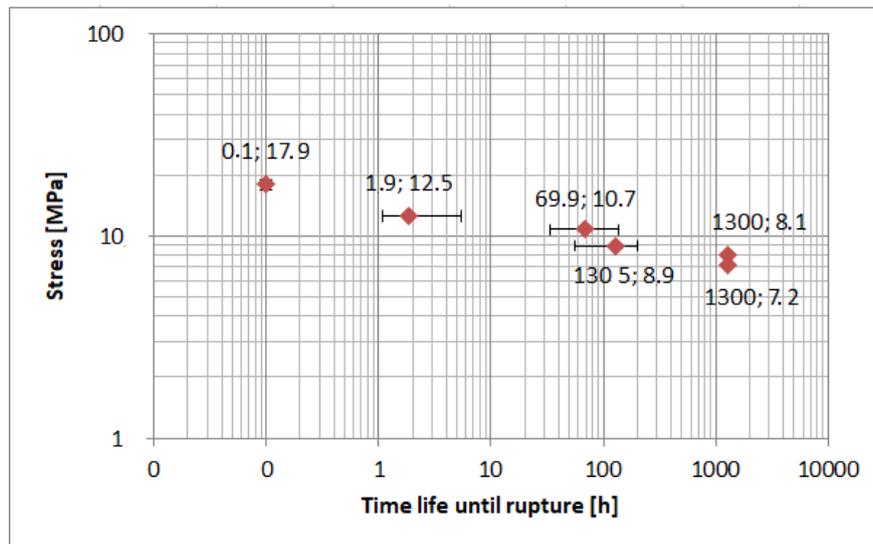


Figure 9. Log of stress versus log of time life of the shear joints bonded with the NVT 210E adhesive.

A tendency of alignment of the experimental data was observed, such that a line could be drawn, as show in the Fig. 10. For this analysis, the point (300; 8.9) was discarded because it represents the tests that are still in progress, and the points (1300; 8.1) and (1300; 7.2) that represents the points where tests were completed in 1300 hours without rupture.

The coefficients of the Eq. (3) (equation of a line written on a logarithmic scale) were determined with the aid of the linear regression technique.

$$y = kx^n \quad (3)$$

Where n is the slope of the line and k is equal to y when x is 1.

Some adaptations of the equation 2 were made. Replacement of the variable y by the joint loading stress, τ . The variable x has been replaced by the time until the rupture t_r , and the constants k_1 and n_1 in the place of k and n . The relationship between the loading stress of the joints and the lifetime of the NVT 210E adhesive is given in Eq. (4).

$$\tau = k_1 t_r^{n_1} \tag{4}$$

Where the following values were found:

$$n_1 = -0.067;$$

$$k_1 = 13.217 \text{MPa/h.}$$

The Figure 10 shows the curve generated by Eq. (3) on a logarithmic scale. It is possible to notice the approximation of the data obtained experimentally (the points of the graph) with the proposed model. And that the model curve (line of the graph) overlapped amid the limits/tolerances of each experimental point. The Figure 11 illustrates Model curve and test data of the NVT 201E in a Cartesian scale.

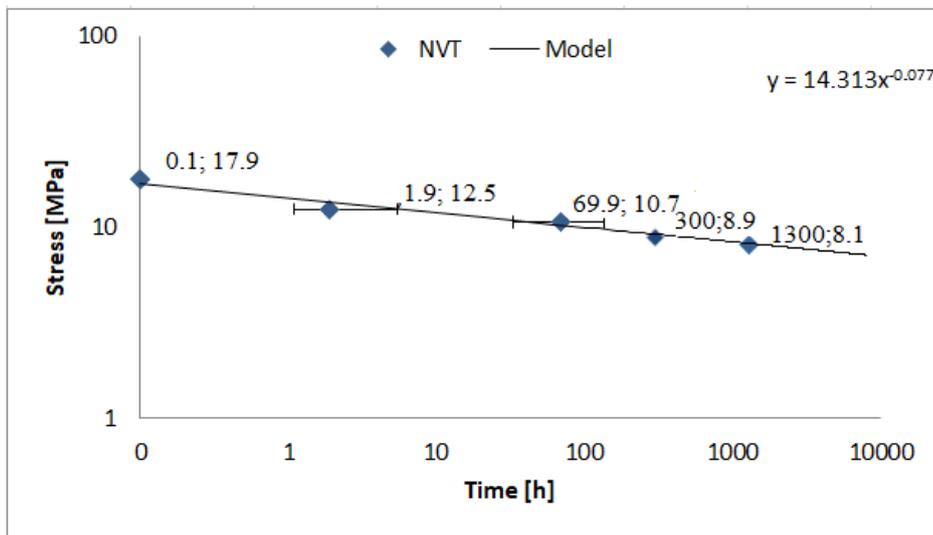


Figure 10. Model curve and test data of the NVT 201E on a logarithmic scale.

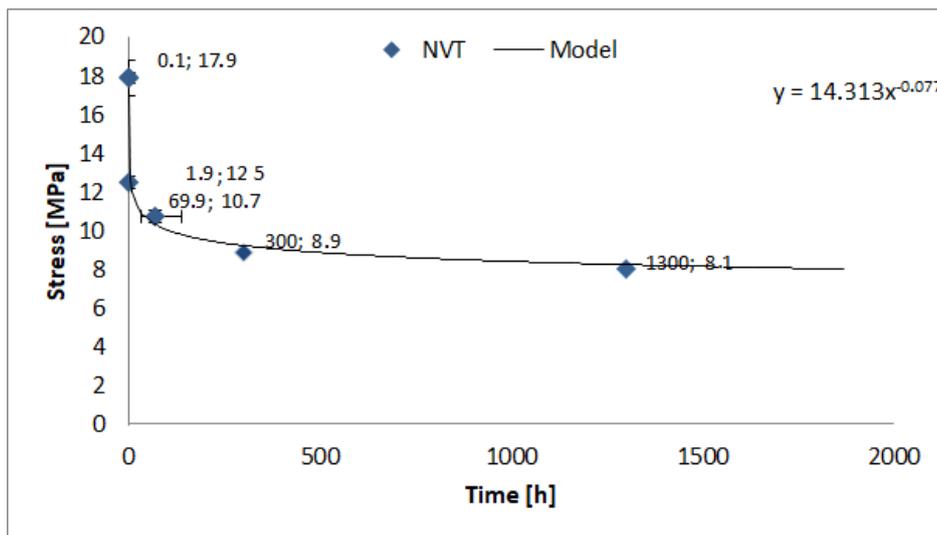


Figure 11 Model curve and test data of the NVT 201E in Cartesian scale.

By means of Eq. (5), it is possible to theoretically estimate the lifetime of the joints as a function of the applied load.

$$t_r = n_1 \sqrt{\frac{\tau}{k_1}} \quad (5)$$

Thus, by means of Eq. (4), the lifetime of the joints submitted to the loading of 8.1 MPa could be estimated, even if no rupture occurred. The result was approximately 478.35 hours, but the joints loaded with this stress level have already exceeded 500 hours in the test. Already for the tension of 8.1 MPa, a t_r of 1625.48 hours was found. For a more accurate estimate it would be necessary to use another method to compare with the theoretical results obtained by the equation, such as the Larson-Miller parameter.

3.4 Joint Fracture Analysis

Both the joints tested in the tensile and creep tests presented mixed fractures (cohesive and adhesive). Usually a cohesive fracture indicates that the adhesive bond is stronger than the internal strength of the adhesive itself. The adhesive indicates that the internal resistance of the adhesive is stronger than the adhesive bond. Adhesive fracture is often caused by improper preparation of the substrate surface and cohesive fracture is caused by good surface preparation. The Figure 12 shows the mix fracture of the joint bonded with NVT 210E adhesive.

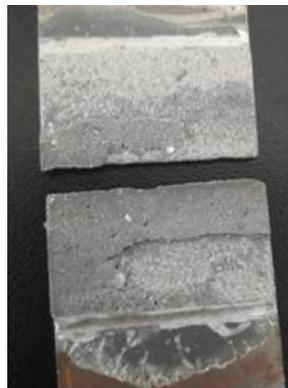


Figure 12. Joint with mix fracture.

4. CONCLUSION

This paper evaluated the creep behavior of bonded metal joints with an epoxy-based adhesive (NVT 10E). The tests are underway in the Creep Pneumatic Equipment (CPE). With the results of the CPE tests, it was possible to obtain equations that represent the creep behavior of the joints. With the aid of the linear regression technique, the long-term behavior of the joints was predicted at room temperature.

The specimens were prepared according to ASTM D 1002 standard and RT TMEC 003/08/PETROBRAS bonding procedure. Tensile tests were performed on the Universal Testing Machine and Creep Pneumatic Equipment, to obtain the average of joint rupture stress. Afterwards, creep tests were performed with variations in the percentage of rupture stress.

The joints exceeded the 1000 hour test with 45.6% of the rupture stress, which corresponds to the tension of 8.1 MPa. Tests with 49.6% of stress loading are in progress while this article was written. The fractures presented in the specimens after the tensile tests and creep tests were mixed (cohesive and adhesive).

Using the linear regression technique it was possible to calculate the rupture times for the loaded joints with 49.6% of rupture stress (which is still under test) and for the loaded joints with 45.6% of rupture stress (which exceeded the time of 1300 hours without rupture). The result for the loading of 49.6% of rupture stress, or 8.1 MPa, was 478.35 hours, but the joints loaded with this stress level have already exceeded 500 hours in the test. Already for the tension of 8.1 MPa, or 45.6% of rupture stress, a time of 1625.48 hours was found. For a more accurate estimate it would be necessary to use another method to compare with the theoretical results obtained by the equation, such as the Larson-Miller parameter.

5. ACKNOWLEDGEMENTS

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