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Procedia Structural Integrity 37 (2022) 722–729

Structural Integrity Procedia

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ICSI 2021 The 4th International Conference on Structural Integrity

Adherend effect on the peel strength of a brittle adhesive

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Abstract

Peel tests are widely used to characterize the peel strength of bonded joints and control adhesion quality. There are various configurations of peel test, such as the T-peel test, peel testing at 180°, the floating roller peel test, and the climbing drum peel test. These methods have been widely used mainly in the aeronautical industry, as a way of assessing the peel strength of metallic joints. However, with the growing use of composite materials in industry, it is necessary to characterize bonded joints with these materials when subjected to peeling loads. In this research, the adherend effect on the peel strength of a brittle adhesive is experimentally studied using the floating roller peel test with the aim of evaluating how adherend changes affect adhesion properties of brittle adhesives and also to assess the viability of using the floating roller peel test in composite-to-composite and composite-to-aluminum joints, as well as make a comparison with aluminum-aluminum joint performance. It is also intended to prove the applicability of this test for quality control of adhesion and determination of peel strength in joints with composite materials. The results show the Araldite® AV138 performance falls within the characteristic values of peel strength of other structural adhesives, particularly when composite adherends are concerned, and with reasonable repeatability considering it is a brittle adhesive.

Peer-review under responsibility of Pedro Miguel Guimaraes Pires Moreira

Keywords: Adhesive joints; Structural adhesive; Peel strength; Peel tests; Floating roller peel test

1. Introduction

Due to its wide range of possibilities in material bonding, the use of adhesive joints is increasing in industrial applications (Pocius and Dillard 2002). This method offers multiple advantages comparing to others more traditional

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Peer-review under responsibility of Pedro Miguel Guimaraes Pires Moreira
10.1016/j.prostr.2022.02.002

methods (fastened, welded and riveted joints) since it avoids drilling holes and fasteners, which are often the source of stress concentration and weight increase. More uniform distribution of stresses, ease of manufacture, possibility of joining different materials and low cost are the main advantages of adhesive joints. Furthermore, the replacement of fasteners by adhesive joints also reduces time and means required for the assembly process. The main disadvantages are related to the requirement of surface preparation, low peel strength and difficulties in quality control (de Moura et al. 2005, da Silva et al. 2007, Banea et al. 2011). The aeronautical, naval, automotive, and aerospace industries are good examples where adhesive joints are widely applied. With the also growing use of composite materials in these industries, sometimes it is necessary to bond composite materials or even to bond joints of metallic and composite adherends (da Silva et al. 2007). Adhesive joints have increasingly become a solution when the objective is to bond different materials, even those most susceptible to develop galvanic corrosion.

1.1. Quality control of adhesive bonding

For metal bonding, a widely accepted industrial test to secure a proper bond is the floating roller peel test (ASTM 2004). Due to its simplicity of concept and geometry, this peel test is widely used in industry to evaluate the adhesion properties of metal-bonded structures. There has been significant research using peel test for metal bonding with a variety of objectives, from adhesives' screening, effect of surface pre-treatments, bond durability, among others (Bishopp et al. 1988, Hart-Smith and adhesives 1999, Sargent and Adhesives 2005). Some studies have also investigated the effect of the adherends in the floating roller peel test, and it was found that the measured peel strength is a combination of the interface adhesion strength plus the work expended in the plastic deformation of the flexible adherend. Thus, the mechanical properties of the flexible adherend have a much bigger effect on the test results when comparing to the mechanical properties of the stiff adherend (Crocombe and Adams 1982, Kim et al. 1989, Wei and Hutchinson 1998). During this test, if a cohesive failure occurs when peeling the flexible adherend, it is ensured that the adherends are properly bonded and that this bond will endure. The most important result to evaluate the peel performance is the failure mode and peel loads can only be compared if using the same flexible adherend.

For composite bonding, such a test is yet to be developed, as the standard test methods are optimized for metal bonding, but for both adhesively bonded composite joints and composite-to-aluminum joints, the same adhesion requirements need to be satisfied. In the work of Riul et al. (2012), peel tests are used to compare the interlaminar strength of composite laminates with different manufacturing process. This study showed the potential of peel tests, not limited to secondary bonding applications but extended to co-cured composite laminates. A rapid test method (RAT) for adhesion has been suggested by Van Voast et al. (2013) and Flinn et al. (2008), in order to evaluate surface preparation of composite adherends. This test is a version of the floating roller peel test, where the stiff adherend made of aluminum is bonded to a flexible composite adherend. The results were promising, although this test used hybrids joints. The effect of different adherends might be a limitation to this test as it doesn't represent a real composite-to-composite bond. In more recent studies (de Freitas and Sinke 2014), the adhesion properties of bonded composite-to-aluminum joints were evaluated using the floating roller peel test. The aim was to investigate the viability of using this peel tests in bonded composite-to-aluminum and composite-to-composite joints and how to assess their adhesion properties from the peel test results. The results showed that the floating roller peel test is suitable to assess the adhesion properties of both composite bonding and composite-to-aluminum bonding, that the peel load gives a direct indication of the failure mode, and the results are much more affected by the nature of the flexible adherend. In this study, due to the use of composite adherends, a third failure mechanism occurred: intralaminar failure of the composite adherend (ILFC). This type of failure mode indicates a good adhesion and that the intralaminar strength of the composite adherend is lower than the debonding strength of the adhesive. In another research (de Freitas and Sinke 2015), the same authors used a new composite peel test (CPT), based on the floating roller peel test, to assess the interface adhesion of composite adherends, the effect of environmental temperature and adhesive material on the loads and failure mechanism, comparing the results to the standard floating roller peel test. The results showed that, in most cases of good adhesion, increasing the temperature favors cohesive failure of the adhesive in detriment of intra-laminar failure of the composite. Moreover, the difference between the peel strengths obtained from floating roller peel tests and composite peel tests for joints with the same failure modes are due to the differences in stiffness and ductility of the flexible adherend and not due to the difference in bond quality.

1.2. Objective of present work

In this research, the adherend effect on the peel strength of a brittle adhesive is experimentally studied using the floating roller peel test with the aim of investigate how the adherend changes affects the adhesion properties on brittle adhesives and also to investigate the viability of using the floating roller peel test in composite-to-composite and composite-to-aluminum joints and perform the respective comparison with aluminum-aluminum joints. On the other hand, it is also intended to prove the applicability of this test for quality control of adhesion and determination of peel strength in joints with composite materials.

2. Materials and methods

2.1. Materials

The adherends were composed of two different materials: aluminum and carbon fiber-reinforced polymer (CFRP) laminates. The AW 6082-T651 aluminum alloy was used (*cf.* Table 1). Surface preparation involved abrading with sandpaper and then wiping clean with an acetone-soaked cloth. The flexible aluminum sheets were 0.6 mm thick and the rigid aluminum sheets were 3 mm thick. The CFRP panels were prepared from unidirectional thin-prepreg consisting of HexPly 8552 epoxy matrix reinforced with AS4 carbon fibers (*cf.* Table 2). A 24-ply laminate layup - $[0°_3/90°/0°_3/90°/0°_3/90°]_s$ - was used to produce the rigid composite adherend with approximately 1.8 mm of thickness. For the flexible adherend a layup of four plies - $[0°/90°]_s$ - was used to obtain a thickness of 0.3 mm.

Table 1. Mechanical properties of the aluminum alloy AW 6082-T651 (manufacturer data) (Kolarik et al. 2011).

Young's modulus, *E* (GPa)	Tensile strength, σ_r (MPa)	Tensile yield stress, σ_y (MPa)	Tensile failure strain, ε	Brinell hardness, *HB*
70	350	305	0.11	105

Table 2. Mechanical properties of the unidirectional AS4/HexPly 8552 prepreg (Corporation 2013).

Laminate property	Laminate fiber direction	
	0°	90°
Young's modulus *E* (GPa)	141	10
Tensile yield stress σ_y (MPa)	2207	81

The prepreg laminates were fabricated in a hot-plate press at pressure of 7 bar, according to the recommended manufacturer cured cycle which included an initial 1 h long curing stage at 110 °C, followed by a final 2 h curing stage. Surface preparation of the cured CFRP laminates was identical to the one used for aluminum substrates. The structural adhesive studied was the Araldite® AV138, characterized by its brittle behavior, and recommended to bond different materials together, including composite-to-metal bonding. It's a thermoset two-component epoxy-based adhesive - AV138 resin and HV998 hardener. This adhesive had its mechanical and fracture properties previously evaluated (Neto et al. 2012), which are summarized in Table 3.

Table 3. Properties of the adhesive Araldite® AV138 (Neto et al. 2012).

Properties	Value	Properties	Value
Young's modulus *E* [GPa]	4.89 ± 0.81	Shear yield stress τ_y [MPa]	25.1 ± 0.33
Poisson's ration ν	0.35a	Shear failure strength τ_f [MPa]	30.2 ± 0.40
Tensile yield stress σ_y [MPa]	36.49 ± 2.47	Shear failure strain γ_f [%]	7.8 ± 0.7
Tensile failure strength σ_f [MPa]	39.45 ± 3.18	Toughness in tension G_{IC} [N/mm]	0.20b
Tensile failure strain ε_f [%]	1.21 ± 0.10	Toughness in shear G_{IIC} [N/mm]	0.38b
Shear modulus *G* [GPa]	1.56 ± 0.01		

[a] Manufacturer's data, [b] Estimated in Campilho et al. (2011).

2.2. Joint specimen details

The floating roller peel test configuration was selected for the peel strength estimation of the adhesive Araldite® AV138 under different adherend specifications. The specimens' width was 20 mm for or 15 mm for specimens with rigid aluminum or composite adherend, respectively. The specimen geometry is presented in Fig. 1 - it is composed of two adherends of different length: a shorter rigid adherend and a lengthier flexible adherend, whose deformation during the load application induces a peel load in the adhesive layer.

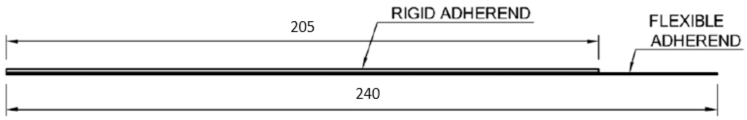

Fig. 1. Floating roller peel test specimen configurations.

The effective bond length is shorter than the rigid adherend length by 10 mm due to the use of steel spacers during joint fabrication, to control bond thickness – a 0.2 mm thick bond line was studied. In the tested specimen configurations, the designation begins with the rigid adherend type, in capital letter (A - aluminum or C - composite), followed by the flexible adherend type in small letter (a - aluminum or c - composite). In the case of flexible composite adherends, the designation ends with the fiber direction of the outer plies - (0° or 90° - with respect to specimen length.

2.3. Test procedure

Tensile peeling tests of the joints was performed using a Shimadzu AG-1 electromechanical universal testing tensile testing machine with a with a 10 kN load cell. Tests were performed at room temperature, at a test speed of 125 mm/min. The displacement imposed to the specimens was applied until complete failure, *i.e.*, until total separation of the adherends. In the floating roller peel test, the specimens are manually fixed to the tensile testing machine. The test accessory, whose geometry dimensions and test setup according to the standard ASTM D3167, are schematically shown in Fig. 2a, is attached to the upper grip of the machine. The flexible adherend of the specimen is attached to the lower grip. Since there may be lateral movement of the specimen during crack growth, it is important to maintain its alignment by rotating the fixture relative to the support point in the testing machine (Da Silva et al. 2012). Although the movement of the testing machine is that of a conventional tensile test, it is possible to simulate the peeling phenomenon due to employed fixture, and joint. An example of peel test and setup is shown in Fig. 2b.

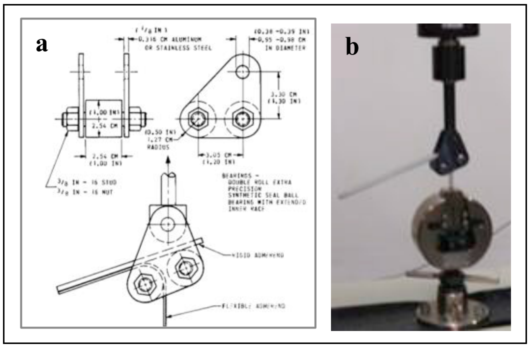

Fig. 2. (a) Geometry, dimensions, and test setup according to ASTM D3167, and (b) floating roller peel test of an A-a adhesive joint configuration.

3. Results and discussion

This section presents and discusses the obtained test results. The following analysis and discussion includes a comparison of the present results, for the different configurations, with results reported in the literature.

3.1. Peel loads and failure mechanisms

According to ASTM D3167 (ASTM 2004), at the end of the floating roller peel test, the following data must be reported: average peel load during the test (P_{avg}), maximum peel load (P_{max}), minimum peel load (P_{min}), peel strength defined as the peel load per unit specimen width (*P/b*), and failure mode. To determine these quantities, the standard specifies the first 25.4 mm of crack propagation must be ignored, and that at least 76.2 mm of crack propagation must be analyzed. In this work, the defined interval to obtain the results spanned between 25.4 mm and 105 mm of crack propagation. This interval complied with the recommendations of the standard and was applicable to almost all tested specimens. The two exceptions were one specimen for configuration A-a and one specimen for configuration C-a, for which a catastrophic failure was observed, preventing the crack growth readings from reaching 105 mm. This phenomenon may be related to the brittle nature of the adhesive. For these two specimens, the measurement range ranged from 25.4 mm to the start of catastrophic crack propagation.

The estimation of *P/b*, designated in the relevant literature as peel strength (Da Silva et al. 2012), was calculated by averaging the measured *P* during the tests by the specimen width *b*, at each instant in the measurement range from 25.4 to 105 mm. The graphical representation of these values, in the selected measurement range, will also be presented as *P/b vs.* propagation displacement of the adhesive crack. Three types of failure mode were observed: cohesive in the adhesive layer, adhesive, interlaminar of flexible composite adherend, as well as mixed failures.

The *P/b* curves for two sample joint configurations - A-c-0 (a) and C-c-90 (b) – are presented in Fig. 3, showing the repeatability and degree of agreement between specimens observed for all tested configurations. Nevertheless, the P_m values vary highly between adherend material combinations, as further discussed in this work. Actually, the curves for all specimens show an identical tendency, with oscillations relating to the brittle failure process for this adhesive, but clearly having an identical fracture behavior throughout the test. Slight P_{m} differences were found between specimens of the same joint configurations, and some specimens' curves do not span over the expected propagation length due to premature abrupt failures. All average values will be discussed in the next section.

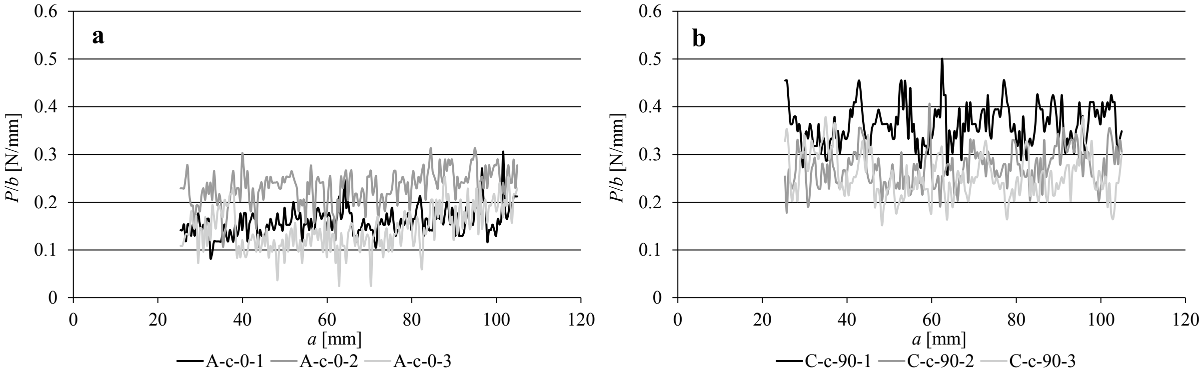

Fig. 3. Experimental *P/b* curves for configurations (a) A-c-0 (a) and (b) C-c-90.

The failure modes were highly dependent on the joint configuration, *i.e.*, adherend material combinations. Thus, failures ranged from practically 100% cohesive in the adhesive layer, to mixed cohesive in the adhesive/interlaminar in composite, or mixed cohesive in the adhesive/adhesive (at adherend/adhesive interface). Although the failure modes will be further discussed in the following sub-section, example failure modes are now presented: Fig. 4a shows essentially cohesive failures in the adhesive laver for the configuration A-c-0. In this case, through careful observation of the failed surfaces, it is possible to conclude the predominant failure mode was by cohesion in the adhesive layer. Residual areas where there is no adhesive in both adherends are nearly nil. Thus, it is concluded that the bond strength between the adhesive and the adherends is superior to the internal resistance of the adhesive itself, which also demonstrates a good surface preparation of the substrates prior to bonding. Fig. 4b reports an example for mixed cohesive in the adhesive/interlaminar failure in the flexible adherend, for the A-c-90 configuration.

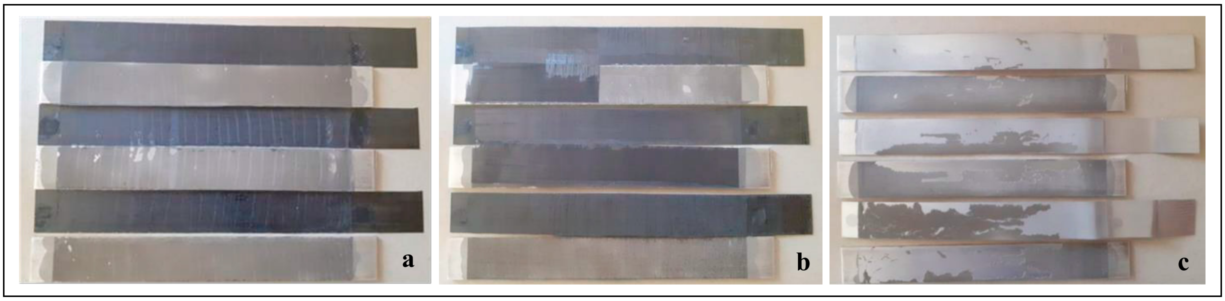

Fig. 4. Failure modes for configurations (a) A-c-0, (b) A-c-90, and (c) A-a.

Here, there was a phenomenon of delamination in the second layer of the stacking of the flexible adherend (between the two layers with fiber orientation parallel to the specimen length). The phenomenon of delamination indicates that the strength of the adhesive and its adhesion to the aluminum substrate is superior to the interlaminar resistance of the composite adherend. This change in failure mode by simply rotating the relative fiber orientation of the laminate ply is probably related to the much lower flexural stiffness of the c-90° substrate relative to c-0°, which in turn results in a diverse distribution of peel and cleavage stresses in both situations. This underlines the standard recommendation that this method should be used for comparison of different adhesives while keeping the specimen construction and test conditions identical. The case of the A-a configuration, in Fig. 4c, provides the example of mixed cohesive in the adhesive/adhesive failure. When compared with the cohesive failure depicted in Fig. 4a, surface regions are clearly found with little evidence of adhesive, showing either deficiencies of the surface preparation process or inadequacy of the adhesive for the bonded materials. Nonetheless, the resulting *P/b* values are not representative of the adhesive properties, but instead, of a weaker interface.

3.2. Adherend effect

With a few exceptions, failures were mostly cohesive in the adhesive layer. In the case of the C-c-90 and A-c-90 configurations, interlaminar failure mode was always present, suggesting that the cohesive strength of the adhesive is higher than the interlaminar strength of the CFRP adherends that had the outer layers with fiber direction at 90º relative to the specimen length direction. On the other hand, the A-a configuration specimens showed minor spots of adhesive failures, but they were mostly cohesive. For the remaining configurations, cohesive failure was always obtained, which is the desired result, indicating a good substrate surface preparation, and that the bond strength between the adhesive and the substrate is higher than the internal strength of the adhesive. Thus, it can be concluded that the Araldite® AV138 presents a good performance in most of the studied configurations.

In Fig. 5, the average *P/b* values and respective standard deviations are shown for the six tested configurations. The minimum P/b is 0.178 N/mm for the A-c-0 configuration, and the maximum is 0.375 N/mm for the A-a configuration. A similarity in average *P/b* values was found for configurations C-c-0 and C-c-90, giving average peel strengths of 0.281 N/mm and 0.297 N/mm, respectively. The coefficients of variation were calculated as 2% for the C-c-0 configuration and 5% for the C-c-90 configuration, which is indicative of high repeatability. The higher coefficient of variation in the C-c-90 configuration may be related to the occurrence of interlaminar failure in two of the specimens for this configuration. This may also be the explanation for obtaining a higher *P/b* value in specimens with CFRP flexible adherend where the fiber direction of the bonded ply is at 90º relative to the longitudinal specimen direction, when compared to specimens with flexible CFRP adherend having outer fibers parallel to the specimen length. The presence of the interlaminar failure mode prevents the *P*/*b* result from being exclusively associated with the adhesive's peel strength, requiring also the analysis of the composite's interlaminar strength (de Freitas and Sinke 2014). The various configurations showed a large diversity of results: the A-c-0 configuration presented a markedly lower peel strength value when compared to the A-c-90 configuration - 0.178 N/mm compared to 0.341 N/mm, respectively. As noted in the previous section, the failure modes were also quite distinctive, and it can be anticipated that the stiffer c-0 adherend generates higher cleavage stresses in addition to peeling stresses, whereas with the much more flexible c-90, peel stresses dominate. The C-a configuration with a *P*/*b* value of 0.210 N/mm is clearly exceeded by the performance of de A-a specimens, with 0.375 N/mm, which showed adhesive failure. In this case it is clear that under the test conditions used throughout this research, the aluminum rigid substrate results in higher peel strengths, even if the failure mode is unsatisfactory. This result suggests the A-a configuration could still have its performance improved with a more effective surface preparation. The observed diversity of results adds again to the standard recommendation that the method should be used while keeping similar specimen construction conditions. Therefore, the reported differences in P/b values for different flexible adherends cannot be solely assigned to worse adherence. Peeling strength values should only be compared between specimens with the same adherends, particularly, with the same flexible adherend. For a comparison of the quality of adhesion, the failure modes, and flexible adherend bending properties, should also be considered (de Freitas and Sinke 2014).

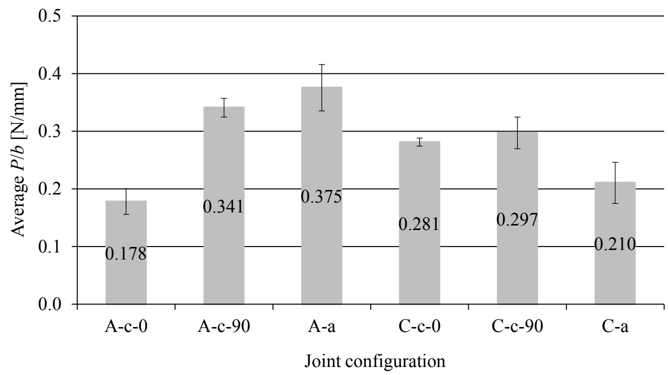

Fig. 5. Comparison of average P/b and standard deviations obtained for the six tested configurations.

Comparing the obtained values in this work with those reported in the literature, it is possible to verify that the selected adhesive has a lower peel strength, particularly in the adhesion to aluminum substrates. In a study by de Freitas and Sinke (2014), carried out with the epoxy film adhesives FM 73 (Cytec Eng. Mat.) and EA9695 (Henkel) for the same configurations, the peel strength with the A-a configuration was 11 N/mm for FM 73 and 2.08 N/mm for EA 9695. With the C-c configuration, however, the differences were not so noticeable: the FM-73 adhesive produced a value of 0.8 N/mm, and the EA 9695 adhesive resulted in 0.56 N/mm. In the present work, the obtained P/b values were 0.281 N/mm (C-c-0) and 0.297 N/mm (C-c-90). The difference is less significant in the aluminum-CFRP configurations, where we obtained a peel strength value of 0.341 N/mm (A-c-90), while in the reference work, 0.68 N/mm and 0.56 N/mm were found for the FM 73 and EA 9695, respectively. These differences may be related to the fact that the Araldite® AV138 is a brittle adhesive, and both FM 73 and EA 9695 are flexible toughened adhesives developed for the aerospace industry. In another study (de Freitas and Sinke 2015), where peel strengths of various adhesives in specimens with carbon and aluminum adherends were compared, the results obtained for the aluminum specimens were of the same magnitude as in the previous study, and thus higher than those obtained in this work for Araldite® AV138. However, for specimens with CFRP substrates several adhesives produced peel strengths in the order of 0.28 to 0.40 N/mm, which agree with values obtained in this study for the C-c-90 and C-c-0 configurations.

4. Conclusions

This work aimed at studying the structural adhesive Araldite® AV138 under peel loading in composite - aluminum, composite - composite and aluminum - aluminum joints, using a floating roller peel test method according to ASTM D3167 standard. The influence of fiber direction of the adhesively bonded ply in the flexible composite adherend was also considered, i.e., parallel (c-0°) or perpendicular (c-90°) to specimen length. This test procedure has been widely used as a quality control test and to determine the peel strength of metal joints, namely in the aeronautical industry. This work also aimed to verify its applicability as a test for quality control of adhesion and determination of peel strength in joints with composite materials and composite - metal hybrid joints. The test results showed significant differences in peeling strengths depending on the joint configuration. In addition, different failure modes, e.g., composite adherend interlaminar failures instead of adhesive cohesive failures were observed for some joint configurations, namely with flexible c-90º adherends. Overall, the best peel strength results were found for the aluminum - aluminum configuration (0.375 N/mm) and the worst for the rigid aluminum - flexible c-0° configuration (0.178 N/mm). Concerning repeatability, assessed through coefficient of variation, the worst value was 16.9% and it occurred in the composite – flexible aluminum configuration, and the best value was 2.47% for the composite – flexible c-0° configuration, indicating an overall reasonable repeatability. Comparison with literature results shows that peel strength values obtained for the configurations with CFRP flexible adherends agree with some results obtained in other investigations with structural adhesives. However, configurations with flexible aluminum adherends resulted in peel strengths generally lower than those found in the literature, which can be explained by the brittle nature of Araldite® AV138 deteriorating its performance when subjected to peel loading. In fact, the floating roller peel test although common for quality control, has still limited research literature, and the available research focuses mainly on toughened flexible adhesives.

The comparison between peel test results of must also focus on comparing the failure modes, particularly when the adherends are a parameter under study, and special attention should concentrate on the flexible adherend. The standard clearly states the method is intended for the comparison of different adhesives while keeping specimen construction and test conditions identical. The obtained failure modes are critical in the analysis of results, and ours indicate an overall good adhesion quality in all configurations. In summary, considering its brittle nature, Araldite® AV138 has a good performance in different configurations, and the floating roller peel test can be used to assess adhesion quality both with metallic, polymer composite, or metal – composite hybrid joints.

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