Adhesion properties of bonded composite-to-aluminium joints using peel tests

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Abstract

In this research, the adhesion properties of bonded composite-to-aluminium joints is evaluated using floating roller peel tests. Tests were performed using two different adhesives and different adherend layups: composite-to-aluminium, composite-to-composite and aluminium-to-aluminium. The results show that floating roller peel tests, widely used in metal bonding, can also be used to assess adhesion properties of composite bonding and composite-to-aluminium bonding. However, attention should be paid on which results are important to take from the peel tests. In adhesion tests the failure mode is more important than the failure load. The peel load can only be compared when using exactly the same type of flexible adherend. Even when the adhesion properties are good (cohesive failure), the peel load value can decrease up to a factor of ten when peeling of a composite flexible adherend instead of an aluminium flexible adherend.

Keywords: peel tests, composite-to-aluminium joints, adhesion properties

1 Introduction

With the increasing use of composite materials in aerospace industry, composite parts often need to be joined either with other composite parts or aluminium parts. Adhesive bonding offers advantages for joining materials, since it avoids drilling holes and fasteners, which are often sources of stress concentration and weight increase. Moreover, the replacement of fasteners by adhesive joints also reduces time and effort required for the assembly process.

Most of the available research in bonded composite-to-aluminium joints is focused in determining the lap shear strength or the fracture characteristics of the adhesive joint. For the fracture analysis, double cantilever beam (DCB) specimens are mainly used to characterize the crack propagation behavior and giving input data for fracture mechanics [1, 2]. Single- and double-lap joints (SLJ and DLJ) have been used to evaluate the shear strength of bonded composite-to-aluminium joints [3, 4, 5, 6]. Analytical and numerical studies have been also performed in order to predict the lap shear strength of bonded joints with dissimilar adherends [7, 8, 9]. A special focus has been given on the thermal stresses that can derive from bonding two materials with different coefficients of thermal expansion. Some solutions have been suggested, such as dual adhesive joints (combination of high-temperature adhesive with a low temperature adhesive), to minimize the possible decrease in strength due to residual thermal stresses [10, 11].

There is, however, limited research available on evaluating the adhesion properties of composite-to-aluminium joints: are the adherends properly bonded together? And will this bond endure? These type of questions are extremely important to avoid in-service interfacial failure of adhesive joints. Lap shear test coupons give very little information about the adhesion of the joints. Adhesion tests should be added to the lap shear test results in order to ensure the integrity of an adhesive joint.

Some authors combine SLJ with DCB or T-peel tests in order to evaluate the durability of bonded composite-to-aluminium joints, for example against humidity, or the effect of different surface pre-treatment [12, 13]. Nevertheless, both studies were more focused on evaluating the effect on the mechanical properties rather than on evaluating the adhesion properties of the joints.

Established standard tests are currently being used for testing adhesion in bonded metal joints. Nowadays, floating roller peels tests or wedge tests are used in industry together with lap shear tests to ensure the integrity of bonded metal joints. For adhesively bonded composite joints and composite-to-aluminium joints, the same adhesion requirements need to be satisfied.

However, no standard tests have been established yet for testing adhesion properties to composite adherends. Also in bonded composite joints, the research has been focused on DCB and SLJ [14, 15, 16, 17]. A simple and straight forward test coupon that reveal the adhesion of composite bonding and composite-to-aluminium bonding has not been established yet [18, 19].

In this research, the adhesion properties of bonded composite-to-aluminium joints are evaluated using floating roller peel tests. 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. The aim is to investigate the viability of using such peel tests in bonded composite-to-aluminium and composite-to-composite joints and how to assess their adhesion properties from the peel tests results.

2 Materials and Specimens

Floating roller peel test specimens were produced by adhesive bonding a thin-flexible adherend to a thick-rigid adherend. During testing, the flexible adherend is peeled off from the rigid adherend.

2.1 Materials

Peel test specimens were produced using clad Aluminium alloy 2024. The aluminium surfaces were pre-treated with chromic acid anodizing and primed with BR 127 (Cytec Engineered Materials, Tempe, Arizona, USA). The flexible aluminium sheets were 0.5 mm thick and the rigid aluminium sheets were 1.6 mm thick.

The Carbon Fiber Reinforced Polymer (CFRP) panels were prepared from unidirectional pre-preg consisting of HexPly 8552 epoxy matrix in combination with AS4 carbon fiber (Hexcel Corporation, Stamford, Connecticut, USA). A lay up of ten plies $[0°/90°/0°/90°/0°]_s$ was used to produce the rigid composite adherend with approximately 2.4 mm thickness. A lay up of two plies [0°/90°] was used to produce the flexible composite adherend with approximately 0.37 mm thickness. After some trials, this lay up was the best in achieving enough flexibility for the flexible composite adherend. Only one ply 0° lacked transverse support and two plies at 0° was more stiff and, therefore, more difficult to bend than the two plies [0°/90°]. The plies were autoclave cured at 180 °C for 120 min, under 6 bar. The cured CFRP panels were abraded with sand paper and then wiped clean with an acetone-soaked cloth.

Two epoxy film adhesives were used on the experimental program – FM 73M.060 (Cytec Engineered Materials, Tempe, Arizona, USA) and EA 9695 M.060 (Henkel, Düsseldorf, Germany). Both film adhesives were carried on a polyester mat. The main difference between the two is that FM 73 film adhesive is formulated to have excellent performance in metal bonding while EA 9695 is especially suited to composite bonding. Table 1 shows the peel performance metal-to-metal of both adhesives and recommended curing cycle, according to the manufacturers technical data sheet (TDS). On both cases the peel performance was determined using standard ASTM D3167 (floating roller peel tests) [20] using aluminium alloy sheets.

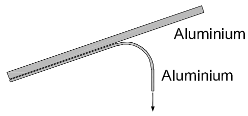
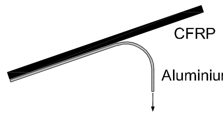
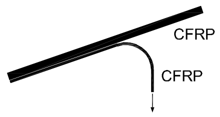
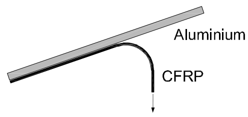
Table 1: Adhesives curing cycle and peel performance on aluminium-to-aluminium, in accordance with manufacturers technical data sheets (TDS).

Adhesive	Peel performance	Cure Cycle (temperature,time)
FM 73	280 N/25 mm	120 °C, 60 min
EA 9695	90 N/25 mm	120 °C, 60 min

2.2 Specimens

Four types of specimens were produced for each adhesive: aluminium-rigid-to-aluminium-flexible (standard), composite-rigid-to-composite-flexible, aluminium-rigid-to-composite-flexible and composite-rigid-to-aluminium-flexible. Table 2 shows the nomenclature used to reference the four types of specimens.

Table 2: Nomenclature of the specimens (t – thickness).

Nomenclature	Rigid adherend	Flexible adherend	Figure
A–a	Aluminium (t=1.6 mm)	Aluminium (t=0.5 mm)	
C–a	CFRP $[0°/90°/0°/90°/0°]_s$ (t=2.4 mm)	Aluminium (t=0.5 mm)	
C–c	CFRP $[0°/90°/0°/90°/0°]_s$ (t=2.4 mm)	CFRP [0°/90°] (t=0.37 mm)	
A–c	Aluminium (t=1.6 mm)	CFRP [0°/90°] (t=0.37 mm)	

The adherends surfaces were laid up with the adhesive films. A thin Teflon tape was placed at the beginning of the bondline. The adhesives were autoclave cured at 120 °C for 60 min under 3 bars pressure. Each bonded panel was 100 mm wide by 300 mm long. After cured, the panels were water jet cut into three 25 mm wide specimens. Figure 1 shows a drawing of the bonded panels. The dimension of the specimens were measured after cutting. The final adhesive thickness was determined by subtracting from the total thickness, the thicknesses of the adherends. The average±standard deviation bondline thickness was 0.11 ± 0.05 mm in the specimens of FM 73 and 0.21 ± 0.06 mm in the specimens of EA 9695.

3 Experimental Procedure

The experimental procedure was based on the standard test method for floating roller peel tests described in ASTM D3167 [20]. Testing was carried out using an electromechanic Zwick machine (Ulm, Germany) with maximum capacity of 20 kN, coupled with a load cell of 1 kN. The testing speed was 125 mm/min. Figure 2 shows the tests set up of the floating roller peel test. The tests were performed at room temperature. A total of three specimens were tested in each test condition. During tests, the load and the cross head displacement were recorded every 0.1 s and 0.1 mm.

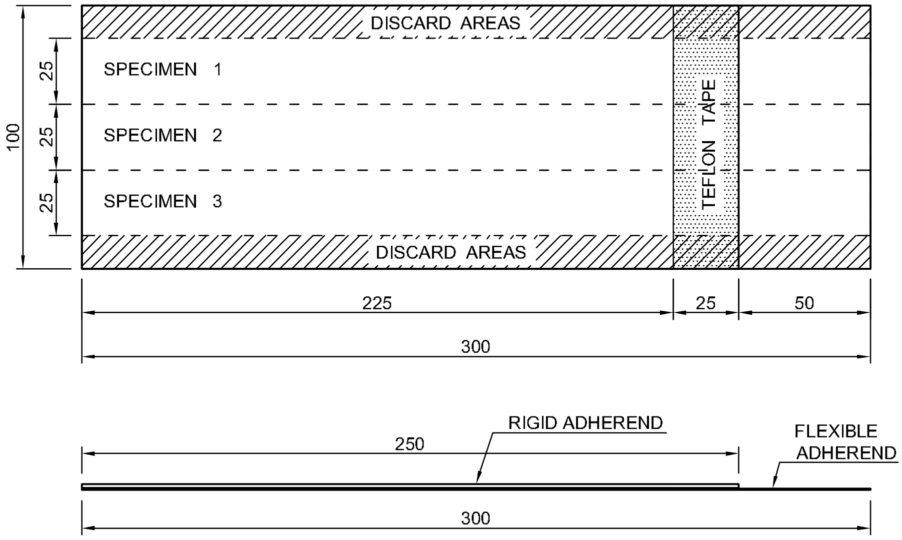
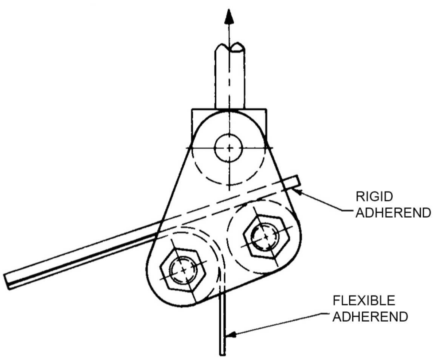


Figure 1: Peel test panels.



(a)

(b)

Figure 2: Floating roller peel test [20].

4 Results

The average peel load and the failure mechanism of the specimens tested are given in Table 3. The average peel load values are shown as the mean±standard deviation of the three specimens tested in each test condition. Three types of failure mechanism were observed, cohesive failure within the adhesive (CF), adhesive failure (AF) and intralaminar failure of the composite adherend (ILFC). The % of failure is an estimate based on visual observation of the specimens failure surface after testing. Figure 3 shows examples of load displacement curves measured during peel tests. The average peel load was determined for 150 mm of peeling, disregarding the first 15 mm after a first peak (decrease of about 5%).

Table 3: Average peel loads and failure mechanisms.

	FM 73				EA 9695			
	F_{ave} (N/25mm)	Failure mode			F_{ave} (N/25mm)	Failure mode		
		CF	AF	ILFC		CF	AF	ILFC
A–a	275 ± 13	100%	–	–	52 ± 8	55%	45%	–
C–a	220 ± 10	95%	–	5%	83 ± 2	90%	10%	–
C–c	20 ± 5	50%	5%	45%	14 ± 2	80%	5%	15%
A–c	17 ± 1	35%	5%	60%	14 ± 2	50%	20%	30%

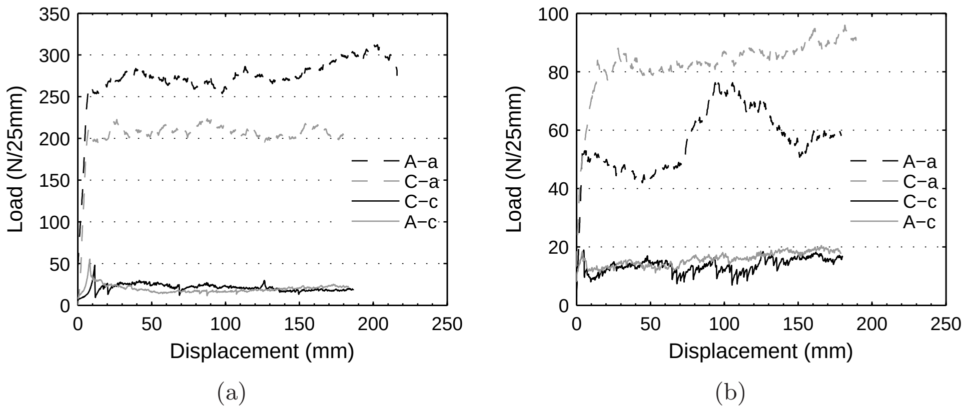


Figure 3: Examples of load displacement graphs measured during peel tests. (a) FM 73 and (b) EA9695.

Looking at the results for the standard test condition aluminium-to-aluminium (A-a), FM 73 has a good peel performance (100% CF), and in agreement with the manufacturer's TDS (Table 1). Although is not specifically mentioned in the TDS, it is assumed that the performance peel load given is for 100% CF. On the contrary, EA9695 performed less well than expected. The peel load is lower than the one given in TDS, most probably due to 45% of bad adhesion (AF) observed in the specimens.

When instead of peeling a flexible aluminium sheet from an aluminium rigid sheet (A-a), one peels a flexible aluminium sheet from a CFRP rigid panel (C-a), the results are comparable with the standard condition. The FM73 peel load decreases about 20% on C-a when compared to A-a, which might be caused by the slight decrease on the % of cohesion failure (from 100% to 95%). Taking into account that both specimens A-a and C-a have comparable % of CF, the fact that the coefficient of thermal expansion of the aluminium is significantly different from the CFRP can also play a role on the peel load. The residual stresses build up in the adhesive bondline during cooling down can decrease the peel load from A-a tests to C-a tests.

On the contrary, in the EA9695 A-a to C-a specimens, the peel load increased approximately 60%, from 52 N/25mm (A-a) to 83 N/25 mm (C-a), most probably due to an increase from 55% to 90% of cohesive failure area. The results of the EA9695 C-a are actually closer to what was expected for this adhesive, according to the TDS (90 N/25 mm), most probably because the % of cohesive failure is closer to 100% (90%CF).

The scenario changes significantly when peeling of a CFRP flexible sheet (C-c and A-c), instead of a aluminium flexible sheet (A-a and C-a). On both adhesives, there is a very significant decrease of the peel load when peeling off the CFRP (C-c and A-c) instead of the aluminium sheet (A-a and C-a). The peel load decreases about 93% in the FM 73 specimens and about 80% in the EA9695 specimens. In the graphs of Figure 3, the change in the order of magnitude of the peel load from A-a or C-a specimens to C-c or A-c specimens can be clearly seen.

The difference between peeling of the flexible CFRP from an aluminium or from a rigid CFRP is almost negligible. For FM 73 adhesive, the peel load is 15% lower on the A-c specimens when compared to C-c specimens. For EA9695 adhesive, the difference in the peel load is actually none (14 N/25mm on both specimens type).

5 Discussion

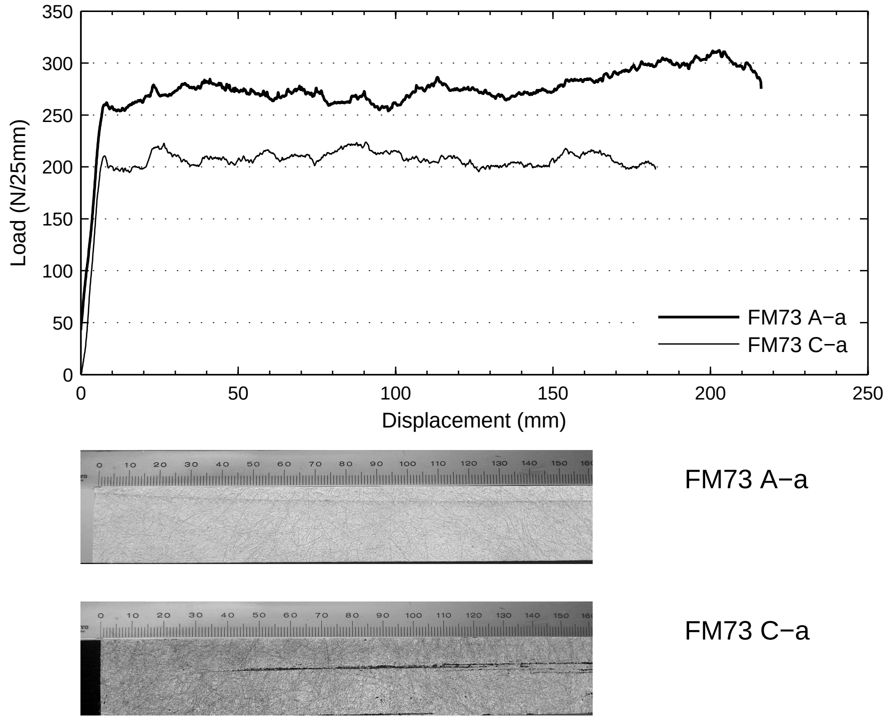
In order to better understand the results of the peel tests, four different parameters are discussed: fracture surfaces, flexible adherend, rigid adherend and adhesives. The aim is to understand their influence on the test results in order to correctly evaluate the adhesives peel performance to both materials, aluminium and CFRP.

5.1 Peel loads vs. fracture surfaces

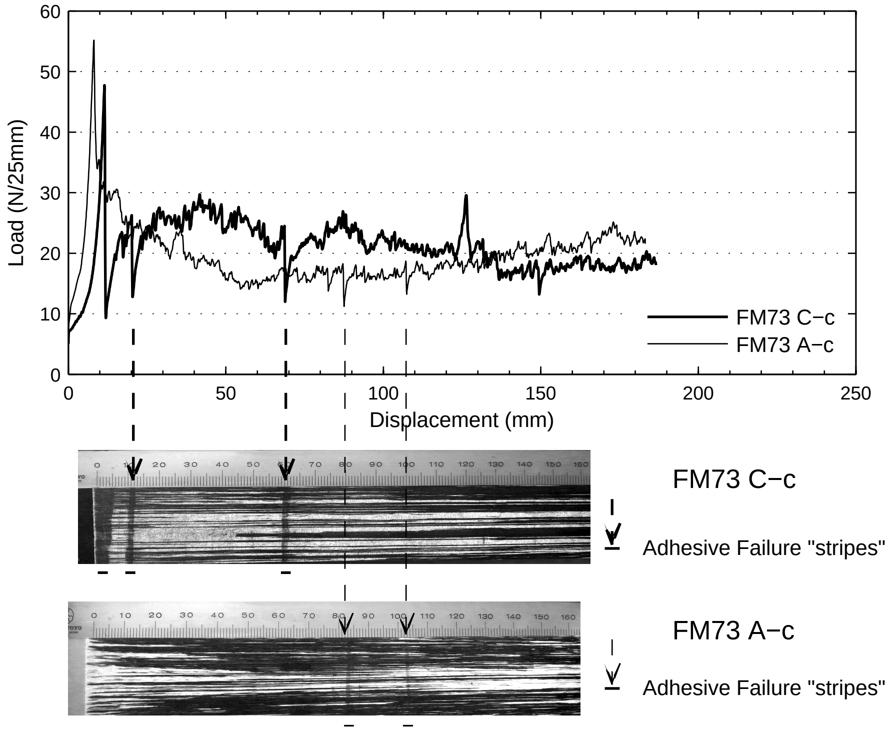
In order to better understand the influence of the failure mechanism on the peel loads, Figure 4 shows an example of FM73 specimens' load displacement curves and their corresponding fracture surfaces. All fracture surfaces shown in Figure 4 are from the rigid adherend.

Figure 4(a) compares the results of FM 73 specimens when peeling off an aluminium adherend from an aluminium (A-a) and from a CFRP (C-a). The peel loads are significantly high, between 200 N/25 mm and 300 N/25mm and within comparable order of magnitude. For both, the major failure mechanism is cohesive along the complete debonding length. There is neither significant changes of failure mechanism along the debonding length of the specimens nor of the peel load along the displacement.

The results when peeling off the CFRP adherend (flexible) are significantly different (Figure 4(b)). The peel loads are between 10 N/25mm and 30 N/25mm, ten times lower than when peeling off aluminium (A-a or C-a). In the fracture surfaces of Figure 4(b), three failure mechanisms can be observed, cohesive failure,



(a)



(b)

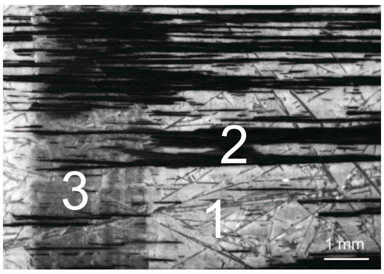
Figure 4: Load displacement curves of FM 73 specimens and correspondent failure surfaces of the rigid adherends (a) when peeling off the aluminium and (b) when peeling off the composite .

intralaminar failure of the composite and adhesive failure. A close up of the three fracture surfaces is shown in Figure 5. When adhesive failure occurs, it is spread along the complete cross section width (adhesive failure "stripes")

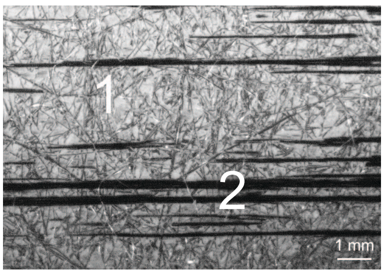
Figure 6 shows the fracture surfaces of both adherends (flexible and rigid) of FM73 C-c specimen close to the adhesive failure "stripe". As it can be observed in Figure 6(a), at the adhesive failure "stripe", there is no adhesive attached to the flexible member, the complete adhesive layer is attached to the rigid member (zone 3), which proves that this is indeed adhesive failure. As opposite to that, in the zone of cohesive failure (zone 1), the adhesive is attached to both surfaces, on the flexible adherend and on the rigid adherend. A zoom in of the adhesive failure (zone 3) and cohesive failure (zone 1) on both adherends is shown in Figure 6(b). Furthermore, looking to the load-displacement curves, the peel load drops suddenly at the adhesive failure "stripes". One can directly correspond the load drops with the adhesive failure "stripes", as indicated in the graph by the arrows. The distance between the adhesive failure "stripes" along the debonding length is the same as the displacement difference between the peel load drops. There is a direct relation between the debonding length and the cross head displacement. The sudden drops of the peel load show that adhesive failure corresponds to the lowest peel load, as expected.

In the areas where there is a combination of CF and ILFC these sudden peel drops do not occur. However even in the areas where adhesive failure does not occur, the peel load remains below 30 N/25 mm. In the areas where the cohesive failure is the major failure mechanism, as for example in specimens C-c between 20 mm and 40 mm of debonding length, the peel load remains ten times lower than A-a peel load with 100% CF. This shows that the significant decrease of the peel load from C-c to A-a is not caused by the type of failure mechanism but mainly by the type of flexible adherend.

Looking at the peel load between 20 mm displacement and 70 mm displacement in Figure 4(b), the peel load of C-c is higher than A-c. If looking at the fracture surface between that area, the C-c fracture surface has relatively more %CF than %ILFC and the other way around for the A-c. On the contrary after 150 mm displacement, the peel load of C-c is lower than the A-c, since the %CF decreased in the C-c and increased in the A-c. This might indicate that the adhesive's peel strength (cohesive failure) is higher than the CFRP intralaminar strength.



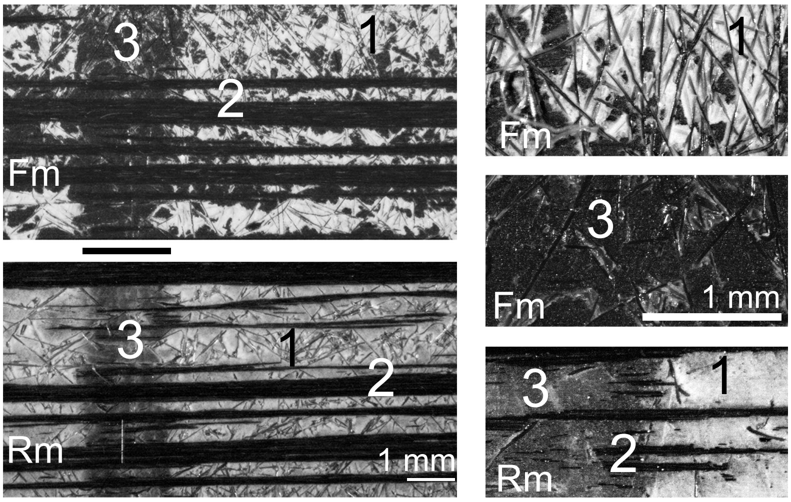
(a)



(b)

Figure 5: Fracture surfaces at the rigid adherend of FM73 specimens when peeling off (a) the composite from the aluminium (A-c) and (b) the composite from the composite (C-c): 1 – cohesive failure, 2 – intralaminar failure of the composite and 3 – adhesive failure.

Figure 7 shows examples of load displacement curves and corresponding fracture



(a) (b)

Figure 6: Fracture surface of both sides of FM 73 C-c specimen, flexible member (Fm) and rigid member (Rm) (a) close to an adhesive failure "stripe" and (b) zoom on both sides: 1 – cohesive failure, 2 – intralaminar failure of the composite and 3 – adhesive failure.

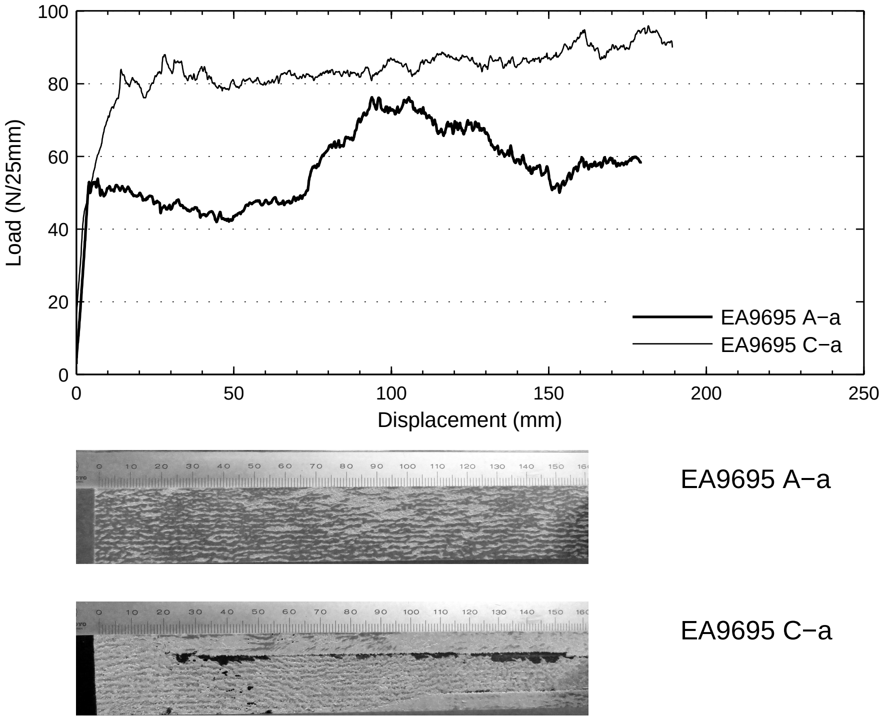
surfaces of EA9695 specimens.

As described in the results, EA9695 A-a specimens had a lower peel performance than C-a specimens. Looking to the fracture surface of the A-a specimen, two types of failure surfaces can be identified: cohesive failure and adhesive failure. A close up of the two types of failure can be seen in Figure 8(a). Indeed, the adhesive failure has a smooth fracture surface in contrast with the cohesive failure. In the A-a load displacement curve the peel load is around 50 N/25 mm up to 60 to 70 mm displacement. Around 100 mm displacement, the peel load has a peak of approximately 70-75 N/25 mm. This peak corresponds to an increase of % CF around 90 mm of debonding length. This increase of % of CF is the reason for the increase of the peel load. This comparison indicates that the cohesive failure leads to higher peel loads than the adhesive failure, as expected. However in this case there is no adhesive stripes as described before, neither the sudden drops. The adhesive areas are in combination with the cohesive failure in the same cross sections.

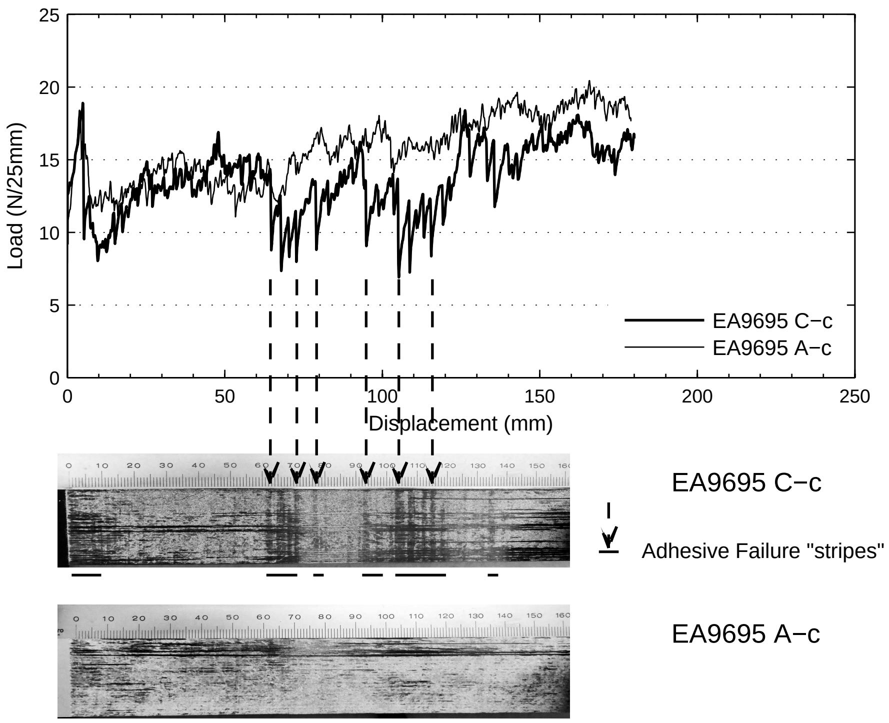
For the specimen C-a, the peel load is more constant along the displacement. The major failure mechanism is cohesive with some small areas of adhesive failure (approximately 10%). A close up of the fracture surface in this specimen is given in Figure 8(b). In contrast with the previous examples where the adhesive failure was always at the interface with the flexible adherend, in this case AF also occurs at the interface with the rigid adherend (CFRP).

The peel load of C-a specimens is always higher than on the A-a specimens, because the % of CF is always higher in the former than in the latter. The peel loads of both specimens get closer to each other when the % of CF in A-a specimens increases. Both peel loads are below what was expected because none of them had 100%CF.

Figure 7(b) shows the results of EA9695 when peeling off a flexible CFRP. Similar to what was observed in FM73, the peel loads are considerably lower than when



(a)

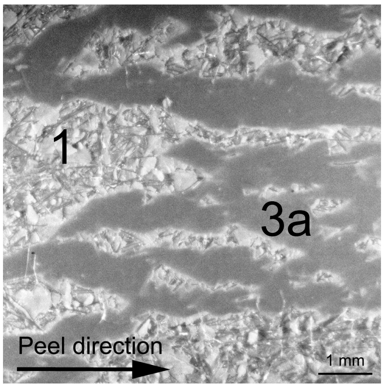


(b)

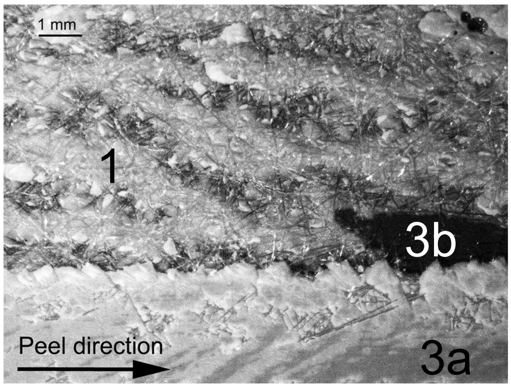
Figure 7: Load displacement graphs of EA 9695 peel specimens and correspondent failure surfaces of the rigid adherends (a) when peeling off the aluminium and (b) when peeling off the composite.

peeling off an aluminium sheet. This indicates that these decrease is not related with the type of adhesive.

The adhesive failure "stripes" also correspond to sudden drops in the peel load, as shown in C-c specimen. There is also a direct correspondence between the load-displacement measured and the failure-debonding length observed. Even in the area where the cohesive failure is clearly the major failure type, like between 20 to 50 mm displacement in C-c specimen or after 130 mm displacement in A-c specimen, the peel load is never as high as for the A-a or C-a specimens. Even when the % of CF is higher in C-c or A-c specimens than in A-a or C-a specimens, the peel load is always significantly lower when peeling off a composite than when peeling of an aluminium. This proves once more that the major decrease of the peel load between A-a (or C-a) specimens and C-c (or A-c) specimens is related to the type of flexible adherend and not to the type of fracture surface.



(a)



(b)

Figure 8: Close up of the fracture surfaces at the rigid adherend of EA9695 specimens when peeling off (a) the aluminium from the aluminium (A-a) and (b) the aluminium from the composite (C-a): 1 – cohesive failure, 3a – adhesive failure at the aluminium interface, 3b – adhesive failure at the composite interface.

5.2 Effect of the flexible adherend

The flexible adherend has a major influence on the peel tests results.

Firstly, due to the asymmetry of the specimen, the failure path is more likely to occur at the interface close to the flexible adherend than close to the rigid adherend. This has been observed in previous research on similar peel tests, in which a composite flexible member was peeled off from a concrete rigid member [21]. The numerical simulations showed that the crack propagation along the interface between the composite strip and adhesive layer yielded the lowest strain energy release rate, and therefore is the most likely failure path. Unless the adhesion at the interface between the rigid member and the adhesive layer is very weak, the adhesive joint will most probably fail at the interface between the flexible member and the adhesive layer.

Secondly, the type of material and thickness of the flexible member have a major influence on the peel load value. Previous research showed that, if plastic deformation takes place in the flexible member during testing, the peel force includes the force required to deform the flexible adherend plastically as well as the decohesive force of the interface [22, 23]. The contribution of the plastic deformation of the thin film to the peel load can be sometimes of the order of 100 times higher than the interface adhesion [24, 25, 26].

As described previously in this paper, even with similar failure mechanism, the peel loads are significantly different when using composite or aluminium flexible members. One can easily observe the typical residual curvature of the thin flexible aluminium member after testing. This residual curvature is not observed in the composite flexible member since this material does not deform plastically.

Using different flexible adherends, aluminium or CFRP, is the main cause for the major decrease of peel load from A-a (or C-a) specimens to C-c (or A-c) specimens. The decrease of the peel load does not mean that the adhesion is worse on the latter case than on the former. One can only compare peel loads of different adhesives within the same type of specimens, and specially when peeling off exactly the same flexible members. The thickness, Young's modulus, yield strength and ductility of the flexible adherend play a major contribution to the measured peel strength [23].

5.3 Effect of the rigid adherend

The rigid adherend has less influence in the peel tests results than the flexible adherend. Firstly, it has less influence on the failure mechanism than the flexible adherend as it was described previously. Secondly, because the type of material and thickness also has almost no influence on the peel load. This has been proven in the test results when comparing different rigid adherends. The results are comparable, if considering the same failure mechanism.

5.4 Adhesive effect

The adhesive has also a major influence on the peel tests results. When peeling off aluminium sheets, FM 73 has higher peel load than EA9695. The same can be observed when peeling of the composite, FM 73 has also higher peel loads than EA9695. This actually means that if the adhesion is good between the adhesive and adherends (no adhesive failure), and cohesive failure is the major failure mechanism, the comparison between different adhesives is similar even when peeling off different materials. EA9695 peel load is lower than FM 73 peel load both when peeling off the aluminium or peeling off the composite.

6 Conclusions

The adhesion properties of bonded composite-to-aluminium joints were evaluated using floating roller peel tests. Peel tests were performed using two different adhesives and different adherend layups: composite-to-aluminium, composite-to-composite and aluminium-to-aluminium. Results show significantly different failure mechanisms and peel loads.

The study shows that floating roller peel tests are suitable tests coupons to assess the adhesion properties of both composite bonding and composite-to-aluminium bonding. However, attention should be paid on which results are important to take from the peel tests. The tendency is to put too much emphasis on the peel load and not enough on the failure mode, which is actually what determines the adhesion properties on both metal and composite bonding. The most important result in order to evaluate the peel performances of adhesive joints using peel tests is the failure mode. In general terms, cohesive failure means good adhesion and adhesive failure means bad adhesion.

The results also show that the peel load gives a direct indication of the failure mode, however the order of magnitude of the peel load is much more affected by the type of material of the flexible member than by the failure mode. There is a decrease of almost a factor of ten in the peel load when peeling off a thin CFRP sheet instead of a thin aluminium sheet, even if both joints fail cohesively. Therefore, peel loads can only be compared if using exactly the same type of flexible adherend (peeling off member). Only then, peel loads can be used as an indication of adhesion properties. The effect of the rigid member is less significant.

When using peel tests for composite bonding, a third failure mechanism can occur, intralaminar failure of the composite (ILFC). This type of failure mode indicates a good adhesion. Furthermore it also means that the intralaminar strength of the composite adherend is lower than the debonding strength of the adhesive. The downside of obtaining ILFC is that it deviates the results from characterizing the adhesive peel performance to the composites.

The type of adhesive material has also a significant effect on the peel load as long as the failure mode is cohesive failure. If the adhesion is good between the adhesive and adherends (no adhesive failure), and cohesive failure is the major failure mechanism, the relative comparison between different adhesives is actually similar even when peeling off different materials.

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