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Optimization in Autoclave Process to Produce Durable Aluminium Composite

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ABSTRACT

The purpose of this paper is to characterize adhesive bonding performance through the fracture evaluation. Failure modes correlate to the bond strength, in which the weak bond of adhesion will be considered unacceptable in the aircraft manufacture certification, whereas the cohesion bond as strong as adhesive itself is preferably accepted. The final quality of adhesive bonding process depends on several key variables during manufacturing. A good anodizing treatment is properly maintained in order to eliminate possible bond failures in the long term bond durability in service. This paper described the method to improve an adhesive joining for durable bonds through optimizing the process variable during autoclave curing. The process simulation utilized the drum peel test to describe the cohesive fracture phenomenon in which applicable on a daily load basis in commercial application.

Keywords*: *Aluminium Composite*, *Adhesive Bonding*, *Failure Mode*, *Cohesive Failure

1. Introduction

Adhesive bonded panel is formed by an adhesive joining process between skins, its doublers and honeycomb core using a film adhesive which undergoes a physical or chemical hardening reaction. This reaction causes the parts to join together through adherence and cohesion strength. The final quality of adhesive bonding process depends on the several key variables during manufacturing. The honeycomb core must sufficiently compact with adhesive layer to join its cover skins and doublers.

The application of metal bonding technology using a film adhesive in any case has some advantages compare to others metal joining concept. The main advantage of the adhesive joining compared to the welding, riveting, brazing and screw fastening is that the adhesive bonded load is distributed uniformly to the loading direction [1]. However, employing primer and film adhesive requires a proper handling and storage of these materials. Handling system of sensitive materials include the packaging in sealed bag, the supporting tool to maintain roll condition, its transportation to customer shop, and how to manage the time life of these materials.

Adhesive bonded panel is widely used for primary composite structure in commercial aircrafts. The effect of surface preparation procedures and material systems on the aluminium surface chemistry subsequently correlates to the bond performance. A good anodizing process ensures a high adherence grade between the aluminium skin surface and the cured primer coat. A number of characterization techniques to evaluate adhesive bonding quality include the surface appearance, the surface chemistry, the surface energy, and the fracture evaluation.

In the fracture evaluation, the only way to measure bond quality empowers the standard specimens for lap shear [2] and drum peel tests [3]. Most of bonding shops utilize these strength tests to qualify the bonding processes, meanwhile some researchers [4-9] focused on the efforts to improve quality of the adhesive materials. However, there is not an effective solution to provide a method to differentiate between the bond strength and the bond durability practically in commercial application.

Failure mode correlates to the bond strength in which the weak bond of adhesion will be considered unacceptable in the aircraft manufacture certification, whereas the cohesion bond as strong as adhesive itself and also the inter laminar (structure) bond as strong as laminate itself are preferably accepted. For practical purposes, some researchers proposed a modification of metal to metal peel test called as a "rapid adhesion test" method that is a quick to assess the adhesion in which the backing adherent clamped to while the peeling adherent is removed.

The failure mode represents poor bond of adhesion failure and strong bond of cohesive failure [10].

A good anodizing treatment as the main process variable mostly eliminates bond failures in a long term bond durability in service. The aluminium surface does not only require clean, but also chemically active surface that is resistant to hydration. The possible bond failure modes are classified in the form of the adherents outside the joint, the cohesion failure of the adhesive, or the adhesion failure of the interface. Failure of the adherents outside the joint may be achieved while using moderately thin adherent materials. The cohesion failure may be caused by an inadequate overlap length, or the presence of thermal stresses or void defects. While, the adhesion failure of the interface can be caused by an inadequate or ineffective surface preparation process.

One important factor to improve adhesive bonds performance is a comprehensively effort to develop both the bond strength and the bond durability. The bond durability depends on the resistance of the adhesive to adherent interface against to water ingress. The resistance to hydration is established by the process used to prepare the surface of the adherents for bonding. The adhesive bond durability becomes an important topic since the publication of the recommendation of Amendment FAR Section 25.605 proposed by Directorate General Technical Airworthiness of Royal Australian Air Force [11]. The regulation is:

1) The methods of fabrication used must produce a consistently sound and durable structure. If a fabrication process (such as gluing, spot welding, or heat treating) requires close control to reach this objective, the process must be performed under an approved process specification that has been demonstrated to produce a structure that is strong and durable.

2) Each new aircraft fabrication method must be substantiated by a test program that demonstrates that the process used is capable of producing a structure that is strong and durable.

Some researchers argue that lap shear specimen is not capable to validate long term bond durability. The service history statistically describes that lap shear testing can not distinguish between a good and a bad processes. The metal bond durability can be validated through the wedge test that tolerates the specimen crack growth of an average of 0.50 inch and a maximum of 0.75 inch in one hour exposure to 60˚C and 95% RH [12]. However, a further recommendation offered an acceptance criteria requires more stringent than broad consensus where a crack growth length should be less 0.20 inch/24 hrs and 0.25 inch/48 hrs, and also <5% adhesion failure [13].

Although it is confirmed that durable bonds of adhesive joining meet a wedge test criteria, however the wedge test is still considered less practical to be applied in daily load commercially to accompany the speed of the production rate. Actually, the wedge test is being applied when producing the first article or if any major chemical replenishment or solution dumping for revalidation of the surface preparation process. In a commercial application, it is not easy to anticipate crack propagation less 0.15 inch/1.25 hour consistently in a climatic chamber at 95% RH.

The wedge specimen does not only require long sequential steps and enough processing period, but also requires a high care especially during specimen cutting to eliminate any vibration impact to the subsequent result anomalies indicated by an improper crack propagation length in this specimen. In daily load basis, practically, the preferably commercial test to validate the failure mode analysis is the drum peel specimen to configure the durability characteristics of the actual aluminium composite panels. This paper describes one of tactically improvement for durable bonds of adhesive joining through optimizing the process variable during autoclave curing.

2. Key Variables in the Adhesive Bonding Process

2.1. Setting Single Parts Prior to Integration

Setting or pre-fitting activity integrates precisely between honeycomb core and all required aluminium skins prior to surface treatment of the skins and all related single parts. The work of this sequent refers to the detail drawing and depends on the operator hands. The operators should ensure that all single parts have been completely pre-fitted. Commonly it requires the additional thickness allowance in the range of 0.30 mm until 0.50 mm during this pre-integration step.

This additional thickness allowance is sufficiently required to anticipate pressurization impact through the vacuum bagging of an aluminium composite panel. The utilization of the cover on the stopper contributes to maintain the final thickness and the surface uniformity or the smoothness of the aluminium composite panels. The required thermocouples are positioned at the leading and lagging point on the surface of the tool is to ease the control during autoclave curing. The key indicator of the success of this pre-fitting is when the honeycomb core is capable to adhere to the film adhesive perfectly.

2.2. Environmental Factor to Materials

The film adhesive and other related adhesives in the form of foam and primer are classified as materials that sensitive to time and temperature. These materials require a proper handling and storage system to provide the physical mechanical properties for manufacturing the adhesive bonded structure. The sensitive materials are stored in a cold storage to eliminate potentially polymerization before lay up process. Prior to cut and applied through dry lamination process, these materials should be conditioned to reach the lay up room condition between 18˚C until 24˚C and required relative humidity in the range of 55% to 75%.

The adhesion failure is indicated by the absence of adhesive on one of the bonding surfaces. It may occur due to hydration of the chemical bonds which form in the molecular link between the film adhesive and the bonding surface. The adhesive bond between aluminiums will fail only if the anodized layer converts to the hydrated and causes the aluminium surface-to-adhesive chemical bonds to dissociate leading to non bond. The oven heating after primer application ensures the adherence of primer on to anodized aluminium surface and enhances the adhesive adherence. Adhesive joining which is formed on surfaces which are chemically active and resistant to hydration will be durable in service.

2.3. Anodizing to Activate the Aluminium Surface

In the view of human aspect, the causes of adhesion failure should be anticipated through the well understanding of an appropriate surface preparation technique which is able to produce a chemically active surface resistant to hydration. The first preparation of aluminium skin before anodizing is manually cleaned to remove any anti corrosion coating oil. Basically, aluminium surface treatment provides good bond durability involves a number of steps, namely: to degrease the whole surface through emulsion or alkaline cleaning, to remove the existing surface layer through deoxidizing and to establish an active surface in the anodizing bath which will form hydration resistant bonds with the adhesive or primer.

The key indicator of the successful of this surface treatment and the primer application is that the primer will adhere perfectly on to the surface of the anodized aluminium skin. In contrary words, the inappropriate anodizing process will not provide a strong bond between the primer layer to the anodized skin surface. The anodizing process is essential and must be performed sequentially to establish a durable bond. Many process specifications, reference books and repair manuals do not contain completely procedures to conform complete sequence and consequently do not produce durable bond [14].

2.4. Adhesive Bonding Technique

Adhesive bond does not tolerate any contamination on aluminium surfaces prior to bonding. Any adhesion failure which occurs in service is a direct result of the manufacturing process. Certain types of gloves (such as nitrile gloves) reduce the surface energy after contact on aluminium surfaces and subsequently reduce bond strength. In some cases the low surface energy is reflected in the reduced average fracture toughness and the change of the mode of failure. Both without gloves and contaminated gloves change the failure mode of the drum peel specimens [10].

In case of bonding on tight surfaces, the super thin fabric reduces tacky problem to ease adhesive bonding. Actually, this super thin fabric reinforces the film adhesive with a typical thickness of 0.010 inch (0.250 mm) such as FM-73M.OST.06 Cytec or thicker. The super thin fabric usually does not require a thinner film adhesive for metal-to-metal bonding. The contaminated super thin fabric will remain spots or specific smell and these are easily detected. The super thin fabric should be stored in dry sealed bag and free from any contamination.

2.5. Process Control during Autoclave Curing

The autoclave facility is operated referred to a number of process parameters such as heat up rate, holding time and temperature, cooling down rate and the end temperature. The vacuum bagging is checked from any vacuum leakage and maintained in a partial vacuum just before an application of the autoclave pressure. Partial vacuum is vacuum condition in which less to the maximum value of vacuum capacity. Meanwhile, vacuum indicator will drop if there is a leakage in the sealed bag. The lagging thermocouple that controls the slowest heating location of the bagging system should be placed in the proper place.

The historical process control is recorded on the autoclave recorder. The controlled parameters include the vacuum and pressure values. If any vacuum leakage is detected during the process, it will be indicated through the vacuum graph that moves abruptly to the edge side of rolling recorder and move to roll direction consistently at zero scale. The perfectly sealed fully or partially vacuum should be maintained at a constant value during curing cycle.

The manufacturing adhesive bonded panel requires different pressure parameters for metal to metal bonding and sandwich panels. The metal to metal configuration requires a cure pressure of 3.0 bars, whereas the sandwich metals needs a lower pressure around 1.7 to 2.0 bars. The drum peel at this range of results a consistently high strength for adhesive bond, but the historical data do not show consistently a perfect cohesive bonding especially for sandwich structure.

3. Experimental Procedures

3.1. Methodology

The experiment evaluated the performance of a crack wedge compared to non crack wedge tests. The evaluation of non crack wedge test involved sequentially work steps by varying the process variables to examine its possible impact against to the cohesive failure phenomenon. The sequentially experiment were performed to determine different types of roots dominantly cause the cohesive failure phenomenon in the aluminium composite panels. A number of steps involved in the experimental procedure as followed:

1) Evaluate the test specimen types to validate the most critical the bonding adherence variables among the peel, shear and drum peel specimens. The first step of this study utilized the expired and new film adhesive to select the most representative test to examine the aluminium composite panels.

2) Identify how far the process parameter variation would induce the mechanical properties through destructtive test specimen by using the new film adhesive. The process variables include: a) incomplete cleaning in alkaline bath and drying at room temperature, b) application of multi layer adhesive, and c) application of interlayer of aramid.

3) Identify the impact of slightly higher autoclave pressure against to the mechanical properties of the sandwich panels. The process variables included: a) single film adhesive, b) interlayer of super thin fabric, c) used primed skin, and 4) interlayer of super thin fabric.

3.2. Cohesive Failure Criteria

This study employed destructive specimens to explain the adhesive bonding phenomenon in the aluminium composite panels. The drum peeling specimens was chosen to measure the confidence level of the quality of this adhesive bonding strength. The specimens were firstly treated in an anodizing line and the primer adhesive of BR-127 Cytec was applied within 4-8 μm thickness. Further, this primed skin was fully dried in an oven at 120˚C during one hour, prior to bonding lay up using the film adhesive FM-73M.OST.06 Cytec at 0.010 inch (0.250 mm) thickness [15].

The drum peeling specimens were prepared using the cladded aluminium alloy LP-3140-T3. The upper skin (300 mm × 75 mm) and lower skin (240 mm × 75 mm) of specimen was approximately 0.5 mm thickness. The thickness of metal core 7.9-1/4-4ON was 12 mm [13]. The laboratory testing was conducted by Instron machine.

The bonding surface was also observed visually to check cohesively grade of film adhesive. The possibility of any cohesive failure area was checked with refer of the bonding surface profile of the destructive specimens using drum peel testing. The best adhesive bonding should be 100% cohesively bonded.

4. Results and Discussion

4.1. Crack Wedge Test

The validation of metal bond durability based on the wedge test ASTM D3762-03 that tolerated the crack wedge propagation length until 0.50 inch in one hour exposure to 60˚C and 95% RH. This validation was dif- ficult to be fulfilled consistently. Even, a further recommendation called out an acceptance criterion more stringent less 0.15 inch/hour. In **Figure 1** the bare aluminium tended to show inconsistency result compare to clad aluminium (**Figure 2**). However, the root cause of this failure might be depend- ing on the specimen cutting prior to laboratory test, rather than the surface treatment procedure.

This crack wedge specimen applied stringent process variable of internal pressure at 6 bars during autoclave curing to obtain the adherence quality of the metal bond. The basic handicap executed this test type in the production line, if applied in daily load. The difficulty is how to maintain production speed when requires re-test procedure to pass the criteria tolerance. The possible re-test

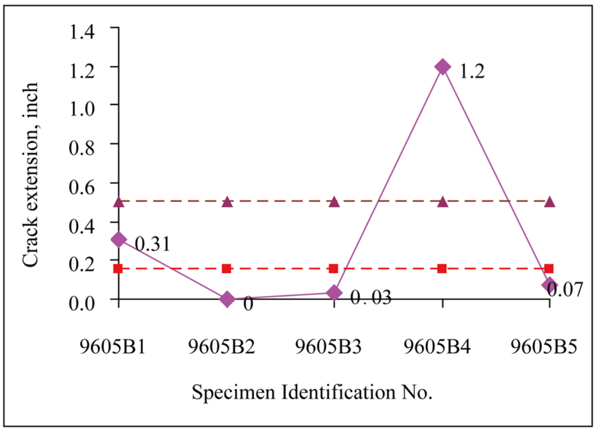


Figure 1. Crack wedge length of bare Al.

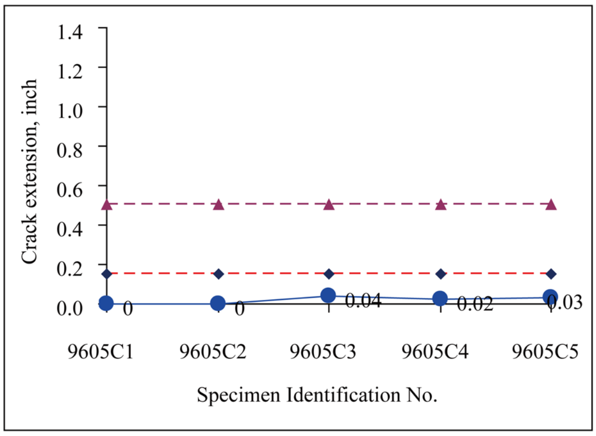


Figure 2. Crack wedge length of clad Al.

may frequently be performed due to the result inconsistency. However, the application of this method either for processing the first article and revalidation after totally chemical replenishment was acceptable to ensure the whole control of a surface treatment prior to metal bonding.

4.2. Non Crack Wedge Test

The tactically stratification intended to identify the most critical test to determine the adhesive bonding performance. By utilizing the expired and new film adhesive FM-73M OST.06 Cytec, the critical test type was subsequently selected. **Table 1** herein shows the values of three standard test specimens in which the drum peel test method became the most critical performance among the others.

This table also shows that the peel and shear specimens still provide exciting values than the drum peel specimens although using the expired adhesive film. The utilization of new film adhesive results a higher strength value through the drum peel test compared to the peel test. It shows that the expired film adhesive indicates out tolerance in performance based on the drum peel test rather than peel and shear test. The configuration of the peel and shear specimens represent metal to metal bonding in which the upper skin will press uniformly over bond surface. The utilization of the film adhesive FM-73M.OST.06 with the initial thickness of 0.25 mm tends to reach a bonding thickness between 0.050 mm to 0.200 mm in adhesive bonded panels after an autoclave curing process. However, the metal to metal bonding sufficiently utilizes the film adhesive FM-73M.OST.03 with an initial thickness of 0.125 mm.

In this matter, the study focused on the drum peel test to examine a relationship between the process parameter and its failure mode characteristic of aluminium sandwich panels. The experiment utilized the new adhesive film and in the same time with the surface preparation was simulated to do an improper cleaning in the alkaline bath at lower temperature around 25˚C. The standard process in alkaline cleaning should be conducted at 65˚C in 'Turco' solution, and the anodized aluminium required an air drying at 60˚C. The 'Turco' solution used in this experiment actually was removed and replaced by a new non-chlorofluorocarbon and at a low temperature, NCLT solution operating in room temperature condition. The curing process utilized the autoclave internal pressure between 1.7 to 2.0 bars to cure these specimens. All sandwich specimens fulfilled minimum value of drum peeling strength of 400 N minimum.

The double layers of film adhesive FM-73M OST.06 Cytec drastically proved higher value of mechanical strength in sandwich panels. In practical application, double layer of film adhesive was intended for rework purposes to fill a gap between core and cover skins. Unfortunately, by accommodating the interlayer such as aramid pre-impregnated between two film adhesive layers dropped its mechanical properties until 238 N, far less than the practical requirement of 400 N minimum (**Table 2**).

Set of operating parameters were completely fulfilled during surface preparation to ensure the higher bond characteristic between the film adhesive and the primed aluminium skins. The autoclave operated with an internal pressure of 1.7 to 2.0 bars, and the drum peel specimen consistently passed the practical requirement, however the fracture characteristic after peeling test did not guarantee absolutely free of cohesive failure.

The trial experiment was conducted applying higher pressure to the specimen by mean of the table press at around 3.0 bars and showed excellent adhesive bond without crash on the honeycomb core 7.9-1/4-4ON of 12 mm thickness. With refer to this parameter, then autoclave operation was prepared to higher pressure than the common practice at 1.7 to 2.0 bars to gain higher adhesive bond to avoid adhesive failure.

Further step was prepared the drum peel specimen using the new film adhesive to be subsequently polymerized in the autoclave at the higher internal pressure between 2.5 to 3.0 bars. Other variable was simulated for example an additional super thin fabric and trial used primed skin to evaluate each of adhesive bond performance. The applied pressure at 2.5 bars to 3.0 bars resulted clearly characteristic of peeling fracture. These peel specimen showed consistently 100% cohesive than

Table 1. Identification of test specimens performance.

Specimen ID No.	1102A	2502A	2303A
Film adhesive configuration and specification	1 layer FM-73M.OST.06	1 layer FM-73M.OST.06	1 layer FM-73M.OST.06
Film adhesive life time	expired	expired	new
Peel strength, (>170 N)	247	330	342
Shear strength, (>27 MPa)	42	40	39
Drum peel strength, (>400 N)	299	66	520

Table 2. Test specimens comparison at standard autoclave internal pressure.

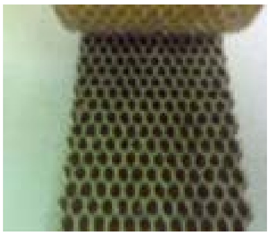
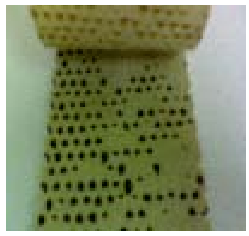
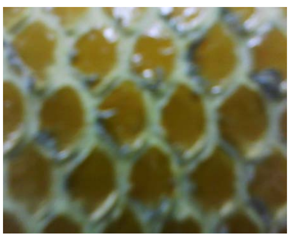
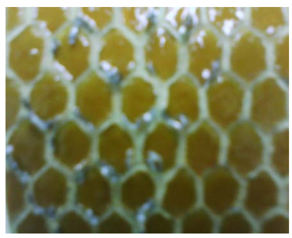
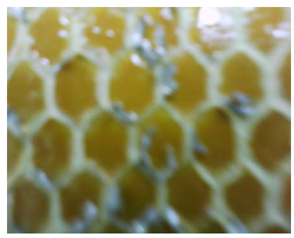
Specimen ID No.	1103A	1103B	1103C
Film adhesive configuration and specification	1 layer FM-73M.OST.06	2 layers FM-73M.OST.06	1 layer FM-73M.OST.06 + aramid
Film adhesive life time	new	new	new
Process variables in preparation before bonding	Incomplete cleaning in alkaline and drying at room temperature	multi layer adhesive	fully interlayer of aramid
Cut section photograph			
Drum peel strength, (> 400 N)	446	1722	238 (not comply)
Autoclave internal pressure, bars	1.7 to 2.0	1.7 to 2.0	1.7 to 2.0
A-scan ultrasonic test	Completely bonded	Completely bonded	Completely bonded
Cohesive failure determination	Not consistent	Nearly 100% cohesive	Nearly 100% cohesive

Table 3. Test specimens comparison at higher autoclave pressure.

Specimen ID No.	0604A	0604B	0604C
Film adhesive configuration and specification	FM-73M.OST.06/original skin	FM-73M.OST.06 +Cerec +original skin	FM-73M.OST.06 / used skin
Film adhesive life time	new	new	new
Process variables in bonding lay up	Standard	Interlayer of super thin fabric	Used primed skin
Cut section photograph			
Drum peel strength, (> 400 N)	471	520	495
Autoclave internal pressure, bars	2.5 to 3.0	2.5 to 3.0	2.5 to 3.0
A-scan ultrasonic test	Completely bonded	Completely bonded	Completely bonded
Failure mode determination	100% cohesive/inter laminar bond	100% cohesive/inter laminar bond	100% cohesive/inter laminar bond

the common practice less than 2.0 bars (**Table 3**).

Additionally, the super thin fabric that was manually laid on film adhesive improved its mechanical properties up to 10% compared to the standard adhesive film. The super thin layer should be placed on the tacky side of the adhesive layer FM-73M.OST.06 Cytec at 0.010 inch (0.250 mm) thickness to ease the bonding application during manual lay up.

In the other case, the used primed skin with the sufficiently peel strength at 495 N provided higher confidence to ensure the rework process. In this case, the rework configured one side skin removal and partially core replacement. In this experiment, the used primed skin was treated in the similar anodizing process prior to bonding application utilizing film adhesive FM-73M.OST.06.

5. Conclusions

The first important step to provide excellent bond durability involves the surface cleaning, the deoxidizing surface layer and the activating surface to form hydration resistant bonds with the primer and film adhesive. In fracture evaluation recently, the optimal measurement of adhesive bond still depends on lap shear and drum peel. Failure mode of the accepted adhesive bond preferably indicates cohesion bond as strong as adhesive itself or inter laminar bond as strong as laminate itself.

The crack wedge extension to validate adhesive bond durability is considered less practical commercially if conducted on a daily load basis, and usually being applied to produce the first article or to revalidate the surface preparation process. Practically, drum peel specimen configures bond durability characteristics. The applied slightly higher autoclave pressure between 2.5 bars until 3.0 bars proves clearly characteristic of 100% cohesive peeling fracture than the common practice less than 2.0 bars.

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