DURABILITY OF ADHESIVELY BONDED JOINTS FOR AIRCRAFT STRUCTURES

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ABSTRACT

Although significant gains have been made in recent years towards understanding the science of adhesively bonded metallic and composite joints, a better understanding of the role of processing parameters and material characteristics on the long-term durability on bonded structures is desired. One such need involves improvements to a commonly used test method for assessing bond durability, the ASTM D3762 metal wedge crack durability test. While the test is considered to be a useful method for investigating bond durability, the existing standard provides little guidance regarding specifics on test conditions and requirements that constitute an acceptable metal bonded joint. Thus the objective of this research investigation is to revisit and revise the ASTM D 3762 metal wedge crack durability test. Following a review of the literature and discussions with identified stakeholders, a listing of potential issues with the current wedge test method was prepared. Several aspects of the ASTM D3762 wedge test were identified for experimental investigation, including methods of specimen manufacturing, testing procedures, accounting for the failure mode produced (cohesion vs. adhesion), environmental conditions during testing, and the need for an improved acceptance criterion. Those aspects associated with specimen manufacturing and the initial test procedure have been investigated first. Two issues associated with the wedge specimen manufacturing that were investigated are controlling the bondline thickness and proper machining of the specimens from the test panel. Additionally, three issues associated with the initial testing procedures were also investigated concurrently: the method of wedge insertion, measurement of the initial crack length; and the specimen orientation during testing. Testing was performed using 2024-T3 aluminum specimens bonded using AF 163-2K adhesive. Expected benefits to aviation include an improved adhesive bond durability test method for use in assessing the reliability of adhesively bonded aircraft structures as well as an FAA Technical Center report to provide additional guidance for aviation industry users.
INTRODUCTION

Significant gains have been made in recent years towards the understanding of the science of adhesively bonded metallic and composite joints. However, a better understanding of the role of processing parameters and material characteristics on the short-term quality and subsequent long-term durability on bonded structures is desired. One such need involves a commonly used test method for assessing bond durability, the ASTM D3762 metal wedge crack durability test\(^1\). In this test, a double cantilever beam specimen is loaded by forcing a wedge between the beams (rather than pulling them apart in a tensile testing machine) as shown in Figure 1. The wedge is retained in the specimen, and the assembly is placed into a test environment, typically an aqueous environment at elevated temperature. Further crack growth is measured by inspection following a prescribed time period. While the test is considered to be a useful method for investigating bond durability, the existing standard provides little guidance regarding specifics on test conditions and requirements that constitute an acceptable metal bonded joint. Of particular concern is the reduction in strength of the bonded metal joint over time due to moisture. Moisture absorption of the adhesive can lead to a reduction in bond strength through hydration\(^2\).

Figure 1: ASTM D3762 wedge test for assessing bond durability\(^3\).
While the existing metal wedge crack durability test, ASTM D3762, is considered to be suitable for assessing bond durability, the acceptance criteria stated in the standard is not as specific as desired. Thus a need exists to assess candidate acceptance criteria and revise the existing test standard such that it provides specific guidance on how to determine acceptance using the wedge crack durability test. The objective of this research project is to develop and implement improvements in the ASTM D3762 wedge test.

Following a review of the literature and discussions with identified stakeholders, a listing of potential issues with the current wedge test method was prepared. Several aspects of the ASTM D3762 wedge test were subsequently identified for experimental investigation, including methods of specimen manufacturing, testing procedures, accounting for the failure mode produced (cohesion vs. adhesion), environmental conditions during testing, and the need for an improved acceptance criterion.

**FINDINGS FROM THE LITERATURE**

The first task of this research investigation was to review published studies that had been performed using the ASTM D3762 wedge test. It was found that pertinent topics from the literature could be grouped into three main categories. These topics, which will be discussed below, are: bond degradation, test applications, and test variations.

**Bond Degradation**

A primary cause of bond degradation in aluminum joints is hydration\(^2,4-6\). As explained by Davis and McGregor\(^2\), aluminum when exposed to oxygen forms an aluminum oxide surface layer. Hydration occurs when this oxide layer is exposed to water. This type of bond degradation typically results in a transition from a reliable cohesive failure to significantly weaker and less predictable mixed-mode failure. This transition results in a weakening of the bond that is not predictable, quantifiable, or easily detectable. Bond degradation can be
prevented or at least mitigated by proper surface preparation. Prebond preparation of the surface is, therefore, crucial to long term environmental durability.

Test Application

The test method described in ASTM D3762 can be used in a qualitative manner for predicting the environmental durability of an adhesive joint. As a result, this test method may be used to compare adhesive systems and surface preparations, assess environmental severity, and establish acceptance of adhesive joint environmental durability. Each of these uses is described briefly in this section.

The wedge test is useful for comparing the durability of different adhesives and surface preparations. Armstrong compared the durability of nine types of adhesive along with four different surface preparations. It was determined that some adhesives where more susceptible to environmental degradation than others and that the rate of degradation also varied. It was also determined that chromic acid anodized adherends preformed better than glass-paper abraded and deoxidine 202 treated surfaces for all types of adhesives. McMillian compared the durability of bonds with substrates made of 2024-T3, 2024-T3 clad, 7075-T6, and 7075-T6 clad aluminum alloys. These alloys were prepared with a phosphoric acid anodize, a chromic acid anodize, and an optimized FPL etch. Adhesive bonding was performed using “old” and “new” technology adhesives with CIAP primer. Similarly, Cotter and Kohler compared the durability of bonds made of BS3L70, BS3L72 (Clad BS3L70), 2024-T3 clad, and 7075-T6 clad alloy substrates with three types of adhesive, three surface preparations and two environments. It was found that the surfaces treated with a phosphoric acid anodize exhibited less crack extension than chromic acid anodize and that both anodizing processes exhibited less crack extension than chromic-sulphuric acid etch when exposed to high humidity at 50°C. This same hierarchy did not hold true for immersion in salt solution at 50°C: the phosphoric acid anodized surfaces
showed the greatest crack extension while chromic acid anodize showed the least. It was also shown that 2024 aluminum alloy demonstrated greater crack extension and therefore less durability than 7075 aluminum alloy when prepared with a chromic acid anodize and exposed to salt solution at 50°C. It was also shown that non-clad alloys proved to be more durable that their clad counterparts.

In addition to aluminum, the durability of steel adherends have also been evaluated using the wedge test\textsuperscript{5,9,10}. Bistac et al.\textsuperscript{10} compared the durability of bonds made from mild steel substrates with both no surface treatment and a phosphatized surface. The specimens were bonded with ethylene-vinyl acetate copolymer and subjected to four environments. It was found that the steel with a phosphatized surface exhibited greater durability than the steel with no surface treatment in all environments. Adams et al.\textsuperscript{5} also used steel adherends to show that a moisture-rich environment caused a greater reduction in fracture energy than a dry environment. It was also shown that increased temperature in addition to wet environments could facilitate faster degradation, a result of both an increased moisture diffusion rate and the rate at which hydration reactions occur\textsuperscript{5,7}.

While not directly related to the above studies, another area in which an improved standard may be beneficial is investigating coupling agents such as silanes, which are commonly used to enhance adhesive joint durability. There is considerable evidence that with such compounds having organofunctional groups, one end of the molecule reacts with the organic adhesive and the other with the substrate. As a result, chemical/physical bonds are developed between the adhesive and the adherends\textsuperscript{11,12}. Silanes have been shown to significantly increase the environmental durability of adhesive joints for a number of different materials\textsuperscript{11,13}. Since the wedge test has been used to test the effect of coupling agents on environmental durability of bonded joints, it is expected that improvements in this standard will also be useful for studying the effectiveness of coupling agents.
In summary, a review of the literature suggests that adhesive durability results are highly dependant upon the adherend, surface preparation, adhesive/primer system, and environment. These dependencies make the correlation of results to service performance difficult. Despite this complexity, McMillan successfully correlated results from the wedge test to service performance. The author reports:

“...the majority of components that experienced disbond in service will produced adhesive crack extension in excess of 1 in. in 1hr at 49°C and 100% relative humidity, and that all panels that disbonded in service produced test crack lengths greater than 0.3 in.”

From this correlation, an acceptance criterion was established:

“A control limit between 0.5 and 1.0 inch would eliminate the majority of in-service disbonds and a 0.75 inch control limit was arbitrarily established…”

Sargent asserts that the level of detection of the wedge test is low relative to other testing methods, but that it is capable of detecting potentially poor adhesive joint performance. This is echoed by TTCP AG13, in that the wedge test is believed to be the most discriminating of tests for surface preparation evaluation.

**Test Variations**

A review of the literature revealed that variations to the ASTM D3762 wedge test are commonly used. Such test variations have included methods of crack length measurement as well as changes to the specimen geometry. Examples of test method variations are provided in this section.

Based on the standardized test method, the measurement of the crack length appears to be a simple procedure. The standard states,

“Using 5 to 30-power magnification and adequate illumination, locate the tip of the crack... For additional accuracy, take and average readings on both side of the specimen”.
But as noted by Sargent\textsuperscript{14}, Popineau et al.\textsuperscript{15}, and Jumel and Shanahan\textsuperscript{16}, edge effects, anticlastic bending, and root rotations quickly add complexities to crack measurement. Anticlastic bending and to a lesser extent the transition from plane strain to plane stress at the edges of the specimen cause concave curvature of the crack front. Popineau et al.\textsuperscript{15} notes that this curved crack front implies a crack length that varies with thickness and that not taking the curvature into account when measuring crack length can cause up to 40\% error in $G_{lc}$ calculations. Because of this, Popineau et al.\textsuperscript{15} employed speckle interferometry to accurately measure the length of the crack at the centerline of the specimen. Jumel and Shanahan\textsuperscript{16} suggest an improvement of the classical beam model that applies plate theory to account for the curvature. Budzik et al.\textsuperscript{17} used an instrumented wedge test. By applying a series of strain gauges to the back surface of the substrate, the authors were able to accurately and continuously monitor crack extension.

In addition to modifying crack measurement methods, various specimen geometries have been used. Sargent\textsuperscript{14} used a specimen width of 4 mm in place of the standard 1 in. (25 mm). This reduced width facilitated the accelerated arrival of a uniform moisture gradient in the adhesive and reduced the crack front curvature. Bistac et al.\textsuperscript{10} modified both the length and width of steel specimens to 40 mm and 10 mm, respectively. Also, the thickness of their adherends was reduced from the standard 3.2 mm to 1 mm. It is noted that the variation of adherend thickness is permitted in the standard for the purpose of avoiding plastic deformation\textsuperscript{1} and can be derived from basic beam theory. However, the equation presented for addressing adherend thickness have been the source of some confusion, which will be addressed in a later section.

**PRIMARY AREAS OF INVESTIGATION**

Several recommendations for improvement of ASTM D3762 have been presented in the literature. Following the literature review and discussions with identified stakeholders, a listing of potential issues with the current wedge test
method was prepared. These issues can be grouped into three areas: specimen preparation, test procedure, and interpretation of results.

**Specimen Preparation**

During an initial review of the ASTM D3762 standard, several issues dealing with the preparation of the specimens were identified. Topics that required clarification included: specimen geometry, load definition, order of procedures, bond line thickness control, and specimen cutting methods.

It was noted that the specimen size was not clearly defined. Whereas the text of the standard defines the adhered dimensions as 152 mm x 203 mm x 3.2 mm (6 in. x 8 in. x 0.125 in.), the drawings in the standard show that the dimensions as 152 mm x 152 mm x 3.2 mm (6 in. x 6 in. x 0.125-in). Upon further investigation it was determined that the consensus within the community is that the dimensions should be 152 mm x 152 mm x 3.2 mm (6 in. x 6 in. x 0.125 in). It was determined that the figures in the standard could also be improved in order to clarify dimensions, callouts, and definitions of regions such as the test area.

Adams et al., suggests clarification of the load $T$ referred to in the standard for use in determining the thickness of the adherends. ASTM D3762 defines $T$ as “150% of the maximum load to start the crack in the adhesive bond, N (or lbf).” Adams states that:

“The source of this equation needs some investigation. It relies upon previous experiments having been carried out and is ambiguous as to which load $T$ should be used. There are two possibilities; is it the force required to insert the wedge to cause the first crack or is it the force required to separate the adherends…”

Upon further investigation, the current authors determined that this equation is also used in ASTM D3433, Standard Test Method for Fracture Strength in Cleavage of Adhesives in Bonded Metal Joint. Based on a review of ASTM D3433, it became clear that the load $T$ is the force required to separate the
adherends. As a result, a clarification of the origin and determination of the load $T$ should be considered in a revision of ASTM D3762.

In the Procedure section, and more specifically in Section 9.2 of ASTM D3762, the prescribed order of operations needs to be revised. Currently the standard states:

“Prime the faying surface of each panel, apply the adhesive, assemble the panels, and cure the adhesive as required by the appropriate specification. Insert a 50.8 by 152 by 0.10 mm (2 by 6 by 0.004-mm) separation film along one of the 152-mm wide edges of the assembly as shown in Fig. 1 to omit the adhesive from between the separation film and the aluminum surface”.

However, the separation film should be inserted prior to the curing of the adhesive. Further clarification could also be provided as to how the separation film should be inserted. While the separation film is listed as 50.8 mm by 152 mm by 0.10 mm (2 in. x 6 in. x 0.004 in.) only the first 19.1 mm (0.75 in.) should be placed between the panels as shown in Figure 1 of the standard. Finally, the incorrect use of mm in the parenthesis when describing the separation film size should be corrected.

**Test Procedure**

As with the topic of specimen preparation, several issues dealing with the test procedure were identified following a detailed review of the ASTM D3762 standard. These issues included the method of wedge insertion, crack measurement schedule, specimen orientation during conditioning and the selection of environment.

ASTM D3762 provides limited guidance on how the wedge is to be inserted in to the specimen. Section 9.5 of the standard states:

“Open the end of the test specimen that contains the separation film, and insert the wedge. Position the wedge so the end and sides are approximately flush with the sides of the specimen. (In any use of an auxiliary tool to open
This description fails to address the many different methods and rates of wedge insertion. Of particular concern is the effect of wedge insertion rate on the resulting initial crack length and subsequent crack growth. For example, one laboratory may insert the wedges by hammering them into the specimen whereas another laboratory may insert the wedges slowly using a pressing operation. Additionally, while removal of the separation film is mentioned, it is not included in the test procedure, possibly providing confusion regarding whether such removal is required.

While the method of crack measurement is clearly defined, the time at which the initial crack length should be measured following wedge insertion is not specified. Although a majority of the initial crack length is produced during wedge insertion, some additional crack growth may occur following wedge insertion but before environmental exposure\textsuperscript{14}. Therefore, an initial crack measurement made immediately following wedge insertion may differ from a measurement made one or more hours thereafter. While ASTM D3762 does not prescribe a time at which the initial crack length should be measured, TTCP AG13 suggests that the initial crack length should be measured one hour after wedge insertion\textsuperscript{4}, thus allowing the initial crack length to stabilize.

TTCP AG13 also suggests that the orientation of the wedged specimens during environmental exposure should be specified\textsuperscript{4}. Figure 2 shows four possible specimen orientations. It is suspected that some orientations could permit condensation to accumulate at the crack tip, thus varying the effect of environmental exposure among the different orientations. The effect of specimen orientation was examined experimentally as part of this investigation and will be discussed in this paper.
Interpretation of Results

Another recommendation from the literature is that the acceptance criterion as currently stated in ASTM D3762 are in need of revision. Currently, the example acceptance criterion stated in ASTM D 3762 is:

“...no individual specimen having a crack extension, $\Delta a$, exceeding 19 mm (0.75 in.) with the average of all specimens not over 6.3 mm (0.25 in.), when placed in 50°C (122°F) condensing humidity for 1 h”.¹

The current acceptance criteria put heavy emphasis on the amount of crack extension, $\Delta a$, that occurs during environmental exposure. However, a recommended acceptance criterion takes into account not only the role of environmental crack extension, but also the role of initial crack length and failure mode in the test area. TTCP AG13⁴ suggests that a more appropriate set of criteria would be;

- Tests are to be performed at 50°C, 95% humidity and noncondensing.
- Specimen orientation to be specified.
- Initial crack lengths are to be measured one hour after insertion of the wedge while exposed in a laboratory environment. The crack length measured must not exceed 1.2 times the crack length obtained from
specimens prepared using the same adhesive to bond surfaces prepared using phosphoric acid anodizing to BAC 5555.

- **In all cases, the Initial crack length must not exceed 50.8 mm (2in).**
- **The crack growth rate on average must not exceed 5.08 mm (0.2 in.) in 24 hrs exposure and also must not exceed 6.35 mm (0.25 in.) in 48 hrs exposure.**
- **The surface generated during exposure must not exhibit greater than 10% adhesion (interfacial) failure.**

In addition to maximum and average crack extension, the criteria in TTCP AG13 take into account such things as specimen orientation, initial crack length, failure mode, environment, initial crack length measurement, and extended exposure⁴.

**EXPERIMENTAL PROGRAM**

Several aspects of the ASTM D3762 wedge test were identified for experimental investigation, including methods of specimen manufacturing, testing procedures, accounting for the failure mode produced (cohesion vs. adhesion), environmental conditions during testing, and the need for an improved acceptance criterion. Those aspects associated with specimen manufacturing and the initial test procedure have been investigated first. Additionally, three issues associated with the initial testing procedures were also investigated concurrently: the method of wedge insertion, measurement of the initial crack length; and the specimen orientation during testing.

To investigate the effect of the method of wedge insertion, measurement of initial crack length, and specimen orientation on the results of the wedge test, an experimental program was initiated. A series of 2024-T3 aluminum alloy adherends were prepared using three surface preparations. The first surface preparation was a phosphoric acid anodize with a BR 6747-1 bond primer. This surface preparation was considered an “ideal” case because it falls in line with best practices for aluminum bonding as described by ASTM D2651 and BAC
In addition to the “ideal” case surface preparation, two intentionally “weak” surface preparation methods were used. The first “weak” method was a grit blasting process with BR 6747-1 bond primer applied. The second method was a phosphoric acid anodized surface like the “ideal” case but without the use of the bond primer. Specimens with the three surface preparations were all assembled in the same manner using AF 163-2k film adhesive and a hot press cure.

Following the curing of the adhesive, five individual 25 mm (1 in.) wide by 152 mm (6 in.) long specimens were cut from the 152 mm by 152 mm (6 in. by 6 in.) bonded panel using a two-step process. First, the specimens were cut slightly oversized using a band saw. Second, the band sawed surfaces were milled to the specifications described in the standard of both width and surface finish.

**Method of Wedge Insertion**

As mentioned previously, the method of wedge insertion is not discussed in the test standard and thus can vary among test laboratories. In order to quantify the effect of various methods of wedge insertion on initial crack length and crack growth, an experimental program was performed to examine the effects of wedge insertion method.

Following the assembly of the panels, curing of the adhesive, and machining of the specimens, wedges were introduced into the specimens using two different methods. The first method, a high rate insertion, was accomplished by gripping the tail end of the specimen in a vice and hammering the wedge into the specimen in one strike. The second method, a low rate insertion, was performed by gripping the tail end of the specimen in a vice and pressing the wedge into the specimen using a drill press. The chuck of the drill press was slowly lowered using the feed handle to press the wedge into the specimen at a rate of approximately 5 mm/sec (0.2 in/sec). From each bonded panel, five specimens were produced. Of these five specimens, the first two were hammered and the last two were pressed. The remaining specimen was alternately hammered or presses so that an even number of each insertion
method was produced. This division of hammered and pressed specimens out of the same panel was deemed important because of the possible panel-to-panel variability.

The initial crack length for each of the specimens was measured by the method described in Section 9 of the test standard immediately following wedge insertion. The initial crack lengths for the PAA and primed or “ideal” bonded specimens are shown in Figure 3. The hammered and pressed specimens were found to have average initial crack lengths of 33.84 mm and 34.00 mm respectively. The standard deviations were also determined to be respectively 0.69 mm and 1.12 mm. Thus for the ideal bonding case, that the difference between the hammered and pressed specimens was not statistically significant.

Figure 3: Initial crack length of “ideal” bonded specimens.

The same tests were performed for the two surface preparations designed to produce “weak” bonds. Initial crack lengths were again measured and are shown in Figure 4. Specimens 161–185 received the grit blast and primer surface
treatment whereas specimens 191–215 received only a PAA treatment. The grit blasted and primed specimens had average initial crack lengths of 32.61 mm and 34.50 mm for hammering and pressing, respectively, with standard deviations of 1.27 mm and 1.17 mm. The PAA treated specimens showed a similar relation, with initial crack lengths of 33.41 mm and 34.93 mm for hammering and pressing and standard deviations of 1.14 mm and 1.34 mm.

Upon further inspection of the data, it was determined that all three types of surface preparation produce the same trend. The hammered specimens on average exhibit shorter initial crack lengths than their pressed counterparts. For the “ideal” bonded specimens this variation was not statistically significant. For the intentionally “weak” bonded specimens, however, this variation was statistically significant. These results suggest that the method of wedge insertion can produce an effect on the initial crack length produced in the specimens.

Figure 4: Initial crack length of “weak” bonded specimens.
Measurement of Initial Crack

Following the investigation on the effect of wedge insertion rate on the initial crack length, an additional investigation was undertaken to determine the effect of wedge insertion rate on the time between wedge insertion and initial crack length measurement. In this investigation, crack lengths were monitored in ambient conditions (lab air) for five days following wedge insertion. These experiments were performed to characterize the crack growth of both hammered and pressed specimens and to determine the ongoing effects of wedge insertion rate. The results for the “weak” bonded specimens are shown in Figure 5. The left plot in Figure 5 shows that not only were the crack lengths for the pressed specimens longer than the hammered specimens immediately following wedge insertion for both types of “weak” specimens, they remained longer following five days in ambient conditions. The right plot of Figure 5, which shows the crack growth following wedge insertion, also illustrates an interesting aspect of the rate of wedge insertion. For both types of “weak” bonds, the hammered specimens exhibited nearly twice as much growth as the pressed specimens. These trends were observed for all three types of bonds, but were only statistically significant for the “weak” bonds.

Figure 5: Crack length and crack growth of the “weak” bonded specimens during the first five days following wedge insertion in lab air.
Specimen Orientation

The effect of specimen orientation on crack growth during environmental exposure was also investigated. As mentioned above, it was suspected that some orientations could cause condensation to accumulate at the crack tip, thus producing variations among orientations in the resulting crack growth. An environment of 50°C and 100% RH was chosen based on the adhesive system used. Once the specimens were placed in the environment, crack lengths were measured at one and two hours of exposure and every day for seven days. Figure 6 shows the results for the "ideal" bonding case. A similar investigation was carried out for the two “weak” bonded cases. These results are shown in Figure 7.

Figure 6: Crack lengths for the "ideal" bonding specimens during environmental exposure.
Both the “ideal” bonded and the “weak” bonded specimens that received the PAA surface treatment performed similarly while the specimens that received the grit blast and prime treatment experienced additional crack extension. While the difference between surface preparations was very discernible, the variation caused by specimen orientation did not show any recurring trend. This result also held true for crack growth corresponding to all three surface preparations as shown in Figures 8 and 9.
Figure 8: Crack growth for the "ideal" bonding specimens during environmental exposure.

Figure 9: Crack growth for the “weak” bonding specimens during environmental exposure.
SUMMARY OF FINDINGS TO DATE

Following the initial literature review, several aspects of the ASTM D3762 wedge test were identified for further investigation. Three issues selected for initial investigation focused on the initial stage of the test procedure; the method of wedge insertion, the initial crack length measurement, and the specimen orientation during testing. Test results showed that the method of wedge insertion does affect the initial crack length, especially for the “weak” bonded specimens. Not only were the initial crack lengths affected by the method of wedge insertion, but the crack growth and resulting crack length from five days in ambient air were also affected. While crack growth and length during environmental exposure varied with surface preparation, specimen orientation caused no recurring trend in any of the three surface preparation methods tested to date.

CURRENT RESEARCH

Current research is focusing on assessing the effects of bond line thickness on test results. In one set of experiments, test panels will be prepared with a thickness gradient across the width of the panel. In a second set of experiments, multiple panels will be prepared, each with a different adhesive thickness. Items of investigation include the effect on initial crack length, crack growth during exposure and failure mode.

As this research project progresses, test results and proposed additions and revisions to the ASTM D3762 standard will continue to be communicated regularly to ASTM Committee D14 on adhesives. In addition to proposing revisions to this standardized test method, research results from this investigation will be disseminated through an FAA technical report and journal publications. Expected benefits to aviation include an improved adhesive bond durability test method for use in assessing the reliability of adhesively bonded aircraft structures.
REFERENCES


