ASSESSING ADHESIVE BOND FAILURES: MIXED-MODE BOND FAILURES EXPLAINED[1]

By

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Summary

This paper explains the mechanisms of formation and degradation of adhesive bonds, and how a degrading interface can cause a transition from a strong bond which fails by cohesion to a weak bond which fails by adhesion. Importantly, the mechanisms proposed also explain mixed-mode failures and the implication of these failures to the strength of adhesively bonded structures.

The paper discusses the impact of adhesion and mixed-mode failures on the regulatory framework for certification and continuing airworthiness management of adhesive bonded aircraft structure. It outlines the difficulties in quantifying mixed-mode failures and how a lack of understanding of this failure mode has led to erroneous findings.

A methodology is proposed which has the potential to prevent adhesion and mixed-mode failures from occurring.

Introduction

Adhesive bonding has been used for decades in primary aircraft structure such as helicopter main rotor blades. When such structure is involved in crash events, careful examination by safety investigators can provide assessment of the potential involvement of the adhesive bond in the failure which led to the crash.

The level of understanding of adhesive bond failures within the flight safety community is not as robust as it could be. Generally, most investigators are broadly aware of cohesion failure (fracture of the adhesive) and adhesion failure (a slick failure at the interface). They may also have some limited knowledge of common causes for bond failures such as bond-line voids, which is one cause of cohesion failure, or contamination during manufacture, which is one cause of adhesion failure.

The failure mode which is least understood is mixed-mode failure, where there is a combination of cohesion and adhesion failure within the same bond. There is minimal understanding of the mechanism of how an adhesive bond transitions from a strong bond which exhibits cohesion failure, to a weak bond which exhibits adhesion failure. This lack of understanding has led to a number of investigative reports drawing conclusions which are not sound and in some cases are in error. The classic example of inadequate knowledge of adhesive bond failures is where investigators use a Scanning Electron Microscope (SEM) to find slight traces of adhesive on a surface, and then classify that failure as cohesion and draw the conclusion that the bond demonstrated adequate strength, when in fact the bond was weak and possibly weak enough to cause the failure.

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Adhesive bonded structures are rigorously tested for static strength and fatigue performance as part of the certification basis for the aircraft, and also undergo rigorous quality assurance assessment during production. Hence it can safely be assumed that such structures leave the production line with bonds that demonstrate an adequate strength. Yet in many cases disbonds are discovered by in-service Non-Destructive Inspection (NDI). It is asserted here that, given the comprehensive rigour of certification and quality assurance, a very large proportion of these defects are either adhesion failures or mixed-mode failures due primarily to degradation/hydration of the bond interface.

This paper will outline the fundamentals of adhesive bonding and provide an explanation of the role of interfacial degradation in the development of mixed-mode and adhesion failures. It will also show that the current regulatory framework does not prevent the incorporation of defective bonds into primary aircraft structure. The paper will also show that current methods for assurance of continuing airworthiness in preventing bond failures may be ineffective in preventing failure of adhesive bonded structures.

A methodology is presented which will prevent interfacial degradation which, if adopted, will validate the current regulatory framework and assure continuing airworthiness. Recent amendments to AC-20-107 go some way to addressing this issue.

Mechanism of adhesion

To understand adhesive bond failures, it is important to understand how adhesives function. Adhesives depend upon chemical bonds formed at the interface between the adhesive and adherend at the time the adhesive is cured [1]. If chemical bonds are strong, failure will occur through the adhesive and the bond strength will be high. If the chemical bonds are weak or degraded, failure will occur through or near the interface and the bond strength will be low.

Mechanism of bond failure (metal to metal bonds[4])

The adhesive or primer forms chemical bonds at the interface during production cure of the adhesive. The bond durability in service depends directly upon the resistance of those bonds to degradation in the service environment. For metals, hydration of the surface oxides by water is the most common cause of failure. As an example, aluminium forms an oxide almost instantaneously when the pure metal is exposed to the atmosphere after etching or abrasion during the production process. The chemical bonds to the adhesive are formed with those oxides. Aluminium has an affinity for forming the hydrated oxide bohemite:

$$Al_2O_3 \rightarrow Al_2O_3 \cdot 2H_2O \qquad (1)$$

In later service, there is a potential for the adhesive bonds to dissociate so that the oxides can hydrate. This creates an interfacial failure of the adhesive bond. Moisture absorbed by the adhesive is sufficient to initiate hydration, and paints and sealants are not an adequate measure to prevent hydration because they simply slow down, not prevent, moisture absorption.

Types of adhesive bond failure

There are essentially three types of bond failure [2]; *Cohesion failure*[5], where the adhesive fractures, leaving traces of adhesive on both surfaces, *Adhesion failure*, where the bond fails at the

[4] The mechanism of degradation of adhesive bonds to composite materials is different to that for metals because of the absence of surface oxides which are susceptible to hydration.

[5] The author has advocated the use of the terms "cohesion" and "adhesion" in lieu of the more common terms "cohesive" and "adhesive" to describe adhesive bond failures, to more clearly differentiate between "adhesive failure" (failure of the interface by adhesion) and "adhesive failure" (failure of the adhesive by cohesion). This terminology has been adopted in AC-20-107B.

interface between the adhesive and the adherend and *Mixed-mode* failure which is a combination of both cohesion and adhesion failures. Mixed-mode failure is the least understood failure mode.

Cohesion failure usually occurs through the plane of the carrier cloth, which is the weakest plane in an effective bond because of the reduced surface area caused by the presence of the carrier cloth. The surface is rough and often slightly milky in appearance due to shear hackles formed by the failure (see **Figure 1**). The opposing surface is also coated in a near-continuous layer of adhesive. Bonds which fail by cohesion exhibit high strength. The causes of cohesion failure include design deficiencies such as inadequate overlap length and factors causing high peel stress or high thermal stresses. However cohesion failure can also occur from the presence of voids which reduce the available bond overlap length below a critical size.

Adhesion bond failures occur at the interface between the adhesive and the adherend, with residual adhesive remaining at any location on one surface only (see **Figure 2**). The chemical bonds at the interface become weaker than the adhesive strength at the plane of the carrier cloth. The surface of the adhesive is smooth and often replicates surface features from the adherend. Adhesion failures exhibit low strength and may occur with no applied load if degradation of the interface is complete. Causes of adhesion failure include contamination during manufacture, the use of out-of-life adhesive, or inadequate temperature control during production, however such cases should be eliminated by quality assurance tests. The remaining cause is interfacial degradation in service.

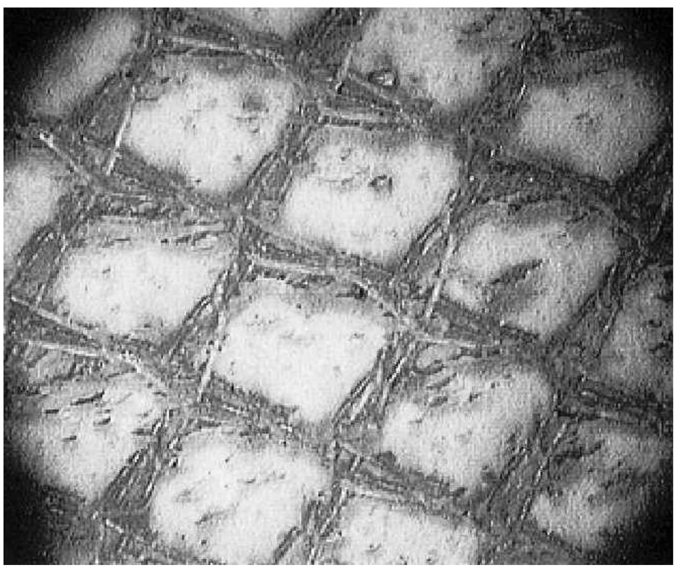


Figure 1: Cohesion bond failure example

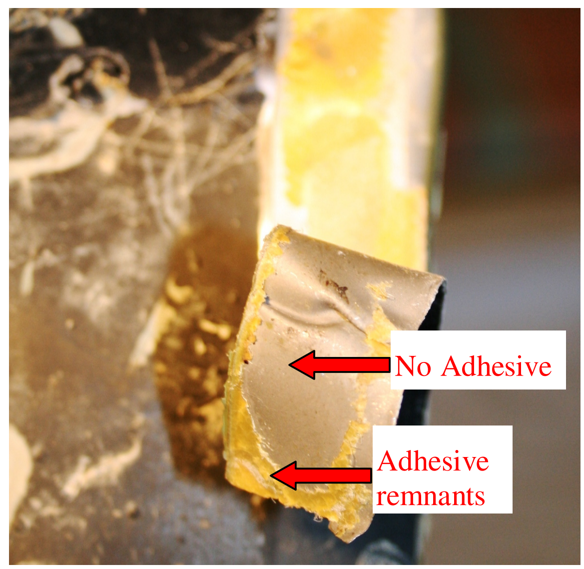


Figure 2: Adhesion bond failure example

Mixed-mode failure exhibits some cohesion failure and some adhesion failure (see **Figure 3**). This is because the interface is partially degraded. In effect mixed-mode failure is just a transitional phase between cohesion and adhesion failure. The failure exhibits areas of smooth surface as well as areas which are rough. The strength of adhesive bonds exhibiting mixed-mode failure is lower than the cohesion failure strength. The strength of the bond and the proportion of surface smoothness or roughness depend upon the level of degradation of the bond interface. It is of concern therefore that many failure investigations use a Scanning Electron Microscope (SEM) to find traces of adhesive on a failure surface so that the bond can avoid being classified as an adhesion failure. If SEM images are needed to find traces of adhesive, the failure is NOT cohesion failure, and the bond IS weaker than the original construction.

For safety investigators, mixed-mode failures are difficult to interpret because the investigator can't be sure if the bond failure caused the crash or if the bond failed as a result of the crash. It is also difficult to quantify the extent of degradation of the bond so all that is possible is to use terms such as "moderate" or "predominant" and these can only be very subjective descriptions. The only certain fact is that the bond strength was lower than at the time of manufacture.

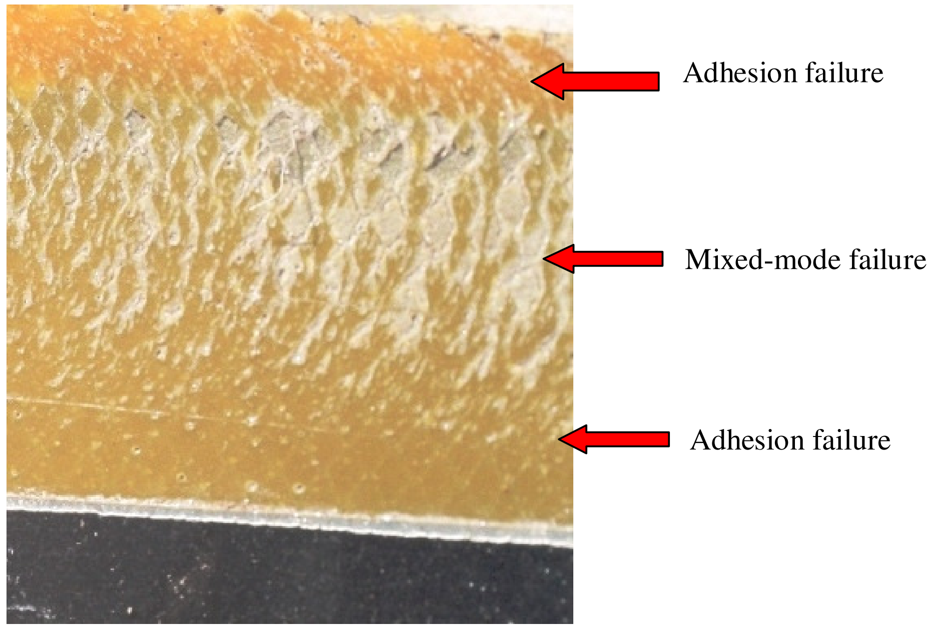


Figure 3: Mixed-mode bond failure

Fatigue may usually be excluded from the causes of mixed-mode and adhesion failures. The excellent fatigue performance of high quality adhesive bonds has been known for many years [3]. There is only one cause of mixed-mode failure: the interface produced by the bonding process was not resistant to the service environment and as a consequence, interfacial degradation was occurring.

Limitations of NDI

Current NDI methods are only generally effective at finding production voids where there is an air-gap. These are the types of defect which cause cohesion failures because the effective area of the adhesive is reduced. The ability of NDI to interrogate interfaces or even to detect weak bonds (kissing disbonds, typical of the onset of mixed-mode failure) is extremely limited. For example, surfaces bonded with double-sided adhesive tape will pass many NDI inspection methods, especially the tap-test, despite the obvious weakness of the bond compared to effective structural bonds. In effect, NDI can only tell whether or not the bond has a physical defect, it can NOT determine the strength of the bond.

With reference to NDI for inspection of in-service bonded components, NDI can therefore not detect the onset of bond strength reduction that would lead to mixed-mode failure when the load exceeded the strength of the bond. NDI is only effective AFTER a disbond has occurred either due to cohesion or adhesion failure. Hence, there is a risk that a weakened bond could propagate to a critical size before localised disbonding occurs to produce a detectable void. A critical factor relevant to continuing airworthiness of bonded structures is the fact that using current technologies, NDI can readily find cohesion failures and adhesion failures, but can not find degraded bonds which are susceptible to mixed-mode failure.

Modelling mixed-mode failure

This paper proposes a model to explain the mechanism and sequence of mixed-mode failure based on the progression of bond hydration. Consider the adhesive in a bond to be constituted of discrete columns of adhesive, with the carrier cloth embedded roughly in the middle of the bond layer, as shown in **Figure 4(a).** The plane of failure will occur through the plane of the carrier cloth because that is the plane with the least effective bond length.

Next, introduce a limited amount of interfacial degradation such that the bond in those columns is weaker than the plane of the carrier cloth, see **Figure 4(b)**. The failure will be partially through the interface and partially through the plane of the carrier cloth. As interfacial degradation progresses, see **Figure 4(c)**, the plane of failure becomes dominated by the weaker interface. Eventually once the entire interface is degraded, see **Figure 4(d)**, the failure is totally interfacial. *It is important to understand that as the plane of failure progresses towards the interface, the bond strength is decreasing*.

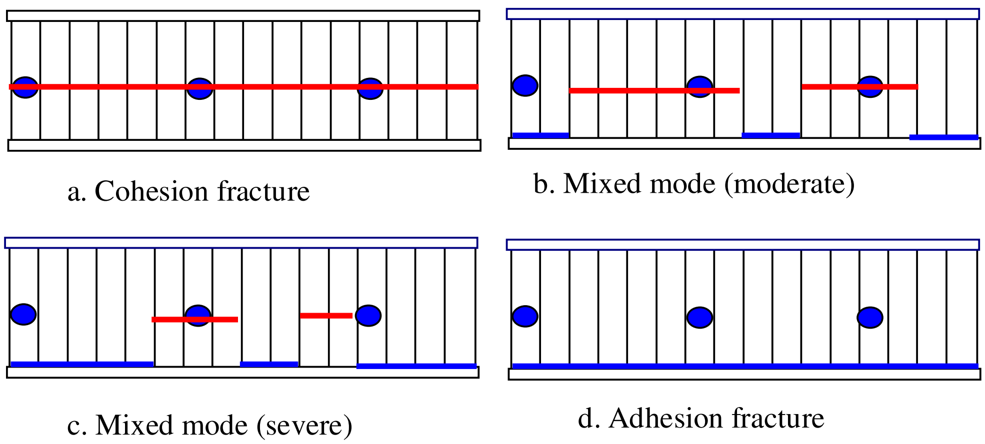


Figure 4: Model explaining how the progression of interfacial degradation changes the locus of failure of an adhesive bond

Current understanding of mixed-mode failures

It must be stated that a very large proportion of the current airworthiness system for adhesive bonded metal structures is set up on the basis that the only failure mode for adhesive bonds is cohesion failure. The Federal Aviation Regulations (FARs) require only demonstration of static strength and fatigue, and the use of processes "known to produce a sound structure". The term "sound" is subjective and is not defined. Many structures pass certification testing and quality assurance tests, including NDI, therefore one could infer that these are *sound* structures. Yet these structures may be susceptible to hydration of the interface and subsequent failure in service.

Consequently, it is possible to create a structure and certify that structure on the basis of strength and fatigue tests, provided that these tests are conducted before the bond interface has had time to hydrate.

FARs 2x.573 require demonstration of damage tolerance for aircraft structures including adhesive bonds. The FAA issued a Notification of Proposed Rule Making (NPRM) on 06 January 2010 and EASA Notice of Proposed Amendment (NPA) NO 2010-04 dated 29th April 2010 proposes to extend these requirements to rotary wing aircraft. These requirements may involve analysis or testing to demonstrate that the structure provides adequate strength in the presence of known and detectable bond defects. Testing generally involves known artificial defects implanted in the bond-line. Production acceptance criteria for tolerable bond defect sizes are established on the basis of static strength tests or analysis on the basis that the bond interface retains full strength. Even service inspection requirements assume that the bond interface retains integrity.

Analysis and testing are meaningless for defining defect acceptance criteria for small-scale adhesion or mixed-mode failures because the assumption inherent in these approaches is that the adhesive surrounding the defect maintains an acceptable level of bond strength. In reality, if a disbond occurs in service, it is almost certainly mixed-mode or adhesion failure due to degradation of the interface and such degradation is usually not necessarily confined to a localised area. The entire interface may be degrading and the bond surrounding the disbond may be weak. It is not possible to predict mixed-mode and adhesion disbond growth rates, and defects may grow without <u>any</u> flight loads.

In recent times the FAA has taken steps to address adhesion failure modes. Policy Statement PS-ACE100-2005-10038 and recent amendments to AC-20-107 to "B" status include advice that interfacial failures are unacceptable in the production process and for service defects. These amendments are to be commended, but they must eventually be followed up by appropriate NPRM/NPA action to amend the regulations to mandate bond durability testing.

Protecting the interface from hydration

Many adhesives (especially epoxies) are polar molecules which absorb moisture from the atmosphere by diffusion. That moisture will eventually find its way to the interface and so will be available for hydration of the interface. It should be noted that paints and sealants do not prevent this phenomenon from occurring; they only slow down moisture diffusion, not prevent it.

The only reliable method for the prevention of such mixed-mode and adhesion failures is to treat the surface with processes which provide resistance to hydration at the interface [4] such as that shown in Equation (1).

Testing surface preparation processes

Many current tests for process validation are based on static strength. For bonds which are susceptible to hydration, the chemical bonds at the interface are initially strong. It is not until the interface has begun to hydrate that there is a measurable loss of bond strength. Hence, short term strength or fatigue tests do NOT discriminate between processes which are susceptible to hydration and therefore cannot prevent the in-service interfacial degradation which leads to mixed-mode and adhesion failures. **Figure 5** shows schematically the effects of interfacial degradation on bond strength. For a process which is resistant to degradation, the bond strength may decrease slightly over time as absorbed moisture plasticises the adhesive. However, with careful characterisation of this effect the bond strength should remain adequate for the service life of the component. For a contaminated bond, short term strength is inadequate because the chemical bonds necessary for bond strength have been inhibited by the presence of the contamination. Such bonds should be eliminated by quality assurance testing at the time of production.

The outcome for a bond which is susceptible to hydration (or other chemical attack) at the interface is that the short term strength may be sufficient to pass certification and quality assurance tests. However, as time in service progresses and the interface gradually deteriorates, the bond strength will degrade. The long-term outcome is that the bond will eventually fail totally at the interface and this may occur in the absence of any loads.

In the intervening period if the bond experiences even moderate loads, failure may occur in a mixed-mode. This is the worst outcome, because NDI cannot detect the conditions leading to mixed-mode failures: the area of the degraded bond surface cannot be quantified, neither can the degree of adhesive bond strength reduction and therefore an estimate of the loss of strength of the bonded structural connection cannot be established. As a consequence, it is currently impossible for safety investigators to conclude if a mixed-mode failure was the cause of an accident or if the failure occurred as a consequence of the accident. The only certainty is that the bond was weaker than at the time of certification and manufacture.

As stated previously, this is significant because at the time of writing the certification regulations only require demonstration of static strength and fatigue resistance for certification of aircraft structures [5]. The FARs also require the use of processes “known to produce a sound structure”. The word “sound” is subjective and open to interpretation. It is reasonable to assert that a bonded structure which (1) has demonstrated static strength and fatigue resistance, (2) was produced in accordance with process specifications, (3) was validated by production quality assurance tests and (4) was assessed using approved NDI methods, is a sound structure. In practice none of these measures interrogate the resistance of the interface to hydration, hence it is possible to certify a bonded structure which has the potential for bond failure in later service by mixed-mode failure or even adhesion failure. Recent amendments to Advisory Circular AC-20-107 to “B” status have included the requirement to demonstrate bond durability.

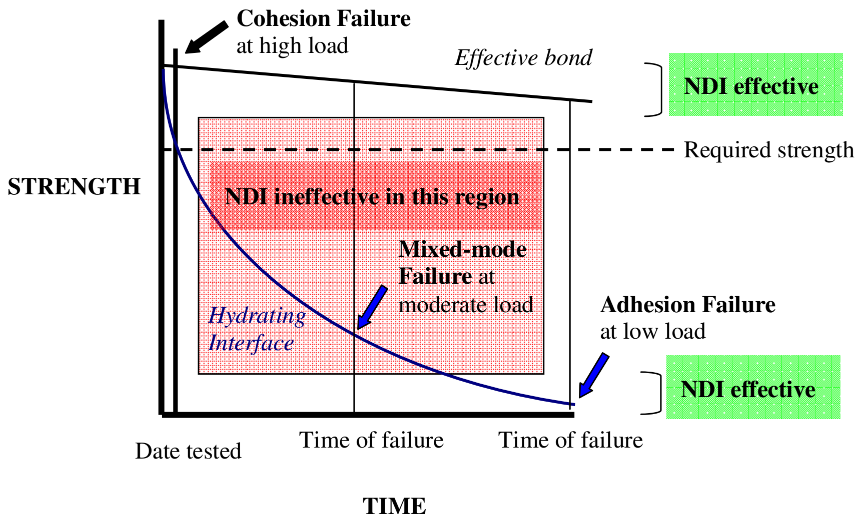


Figure 5: A schematic representation showing the transition from strong cohesion failure through weaker mixed-mode failure to weak adhesion failure due to interfacial degradation and the region where NDI can not detect insipient mixed-mode failure

The most reliable accelerated test [6] for demonstration of bond durability is the wedge test ASTM D3762. In this test, samples of the structural material are bonded together using candidate surface preparation processes. A standard wedge is driven into one end of the sample, cracking the adhesive (see **Figure 6**). The specimen is placed in a hostile environment, typically 95% RH and 50°C. The rate at which the crack grows is monitored and at the end of the test period the specimen is separated so that the locus of failure can be determined. If the failure remains within the adhesive layer, then the surface preparation may be resistant to in-service degradation, but if the failure propagates through the interface, then the process is considered ineffective in preventing interfacial degradation.

Unfortunately service experience has shown that the acceptance criteria stated in ASTM D3762 are inadequate. Fully durable bonds produce results which meet or exceed the following requirements [6, 7]:

< 0.2 inches growth in 24 hrs testing
< 0.25 inches growth in 48 hrs testing
< 10% adhesion failure in the test zone

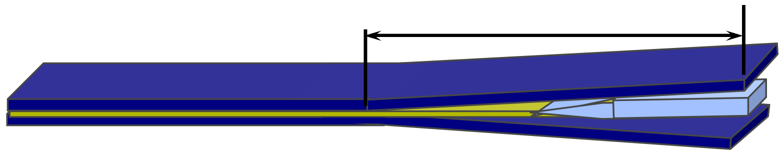


Figure 6: The wedge test specimen for bond durability testing

The reason that the wedge test is suited to evaluation of bond durability is that the adhesive and the interface are placed under extremely high tensile stresses. The initial crack arrests when the tensile stresses are just below tensile ultimate for the adhesive. That leaves the interface under extreme stresses so any degradation of the interface, such as by hydration, will result in interfacial failure. The corollary is that if the interface survives such extreme demands, then it should produce acceptable service durability and mixed-mode and adhesion failures should not occur. Anecdotal evidence supports this hypothesis. Procedures which meet these requirements have been used by the RAAF for adhesive bonded repairs on F-111 since 1992 with excellent service performance, such that the bond failure rate was reduced from 43% to less than 0.1% [8].

How to manage airworthiness of adhesive bonds

The current regulatory framework is effective for adhesive bonds if (and only if) the bond can maintain strength throughout its service life. Therefore the interface must maintain resistance to degradation for the regulations to be valid. It follows logically that one of the first steps in certification must be to assess candidate processes for production of the adhesive bond, to demonstrate resistance to in-service environmental degradation. If that step is undertaken, then damage tolerance and NDI are effective weapons against bond failure.

Previous investigations

There are a number of examples of investigations where conclusions drawn in relation to adhesive bonds could be in error[6].

In the first example, the bond failure was attributed to fatigue (see **Figure 7(a)**). This conclusion was drawn on the basis of features around micro-voids found at the disbond front as shown in the close-up image (arrowed in **Figure 7(b)**). Note that the disbond at the lower left of both images is entirely adhesion failure, yet the features in the upper portion of **Figure 7(b)** are in a region of cohesion failure in the plane of the carrier cloth. The authors contend that it is impossible for the disbond to propagate by cohesion failure through the carrier cloth whilst also producing an adhesion failure at the interface behind the disbond front. The investigator has failed to conclude that the planes of failure were different for both failure modes and has incorrectly concluded that the ductile cup-and-cone features, which occurred when the adherends were separated, were caused by fatigue.

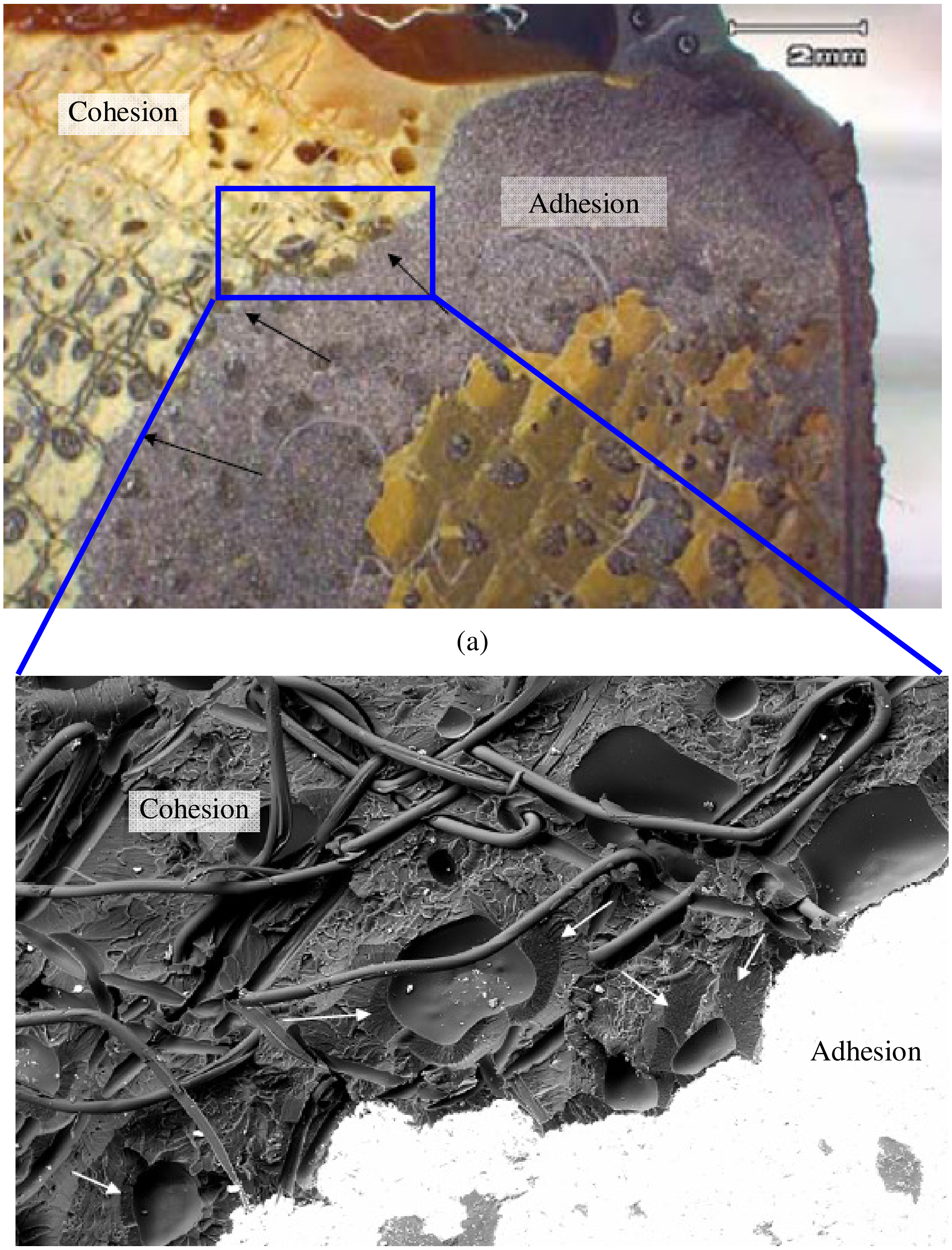
The complete adhesion failure in the in-service disbond indicates that this bond failed by hydration of the interface. This was not a fatigue failure.

In the next example (see **Figure 8(a)**) a disbond of the skin-to-spar joint occurred in flight at the outboard tip in a main rotor blade. The investigating authority reported that the disbond was caused by airborne particles and water droplets eroding the exposed adhesive at the leading edge of the skin-to-spar joint, leading to separation of the skin by aerodynamic forces. Examination of the

[6] The authors stress that reference to these examples is in no way intended to reflect on the integrity of the investigators. We intended only to show that the level of understanding of adhesive bond failure forensics within the safety community needs to be enhanced. To respect the investigators, reference to specific reports will be withheld.

surface shows predominantly adhesion failure with some mixed-mode failure, indicating that the strength of the bond was significantly compromised.

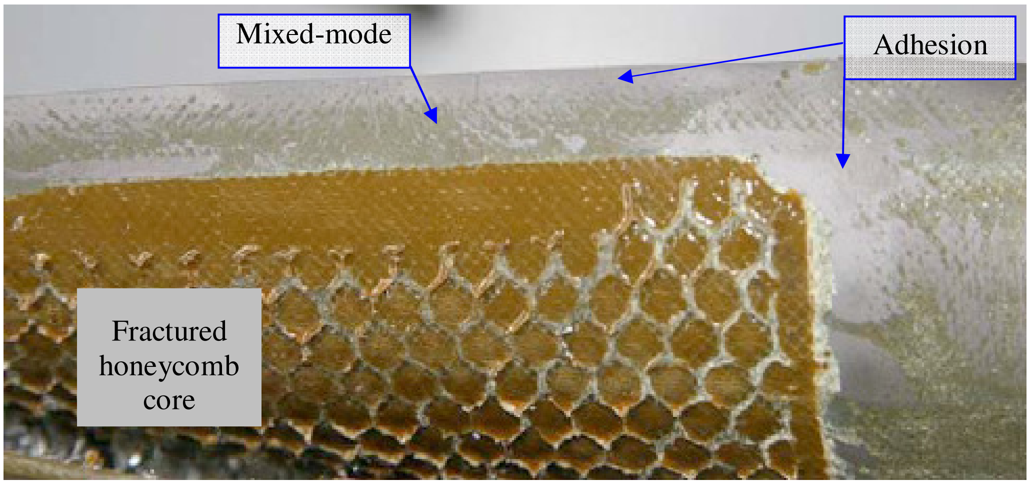
Aerodynamic forces may have initiated the failure, but failure could only propagate because hydration of the interface had degraded the bond strength to such a low level as to allow the disbond to progress under the applied load. The real problem was not erosion; it was the degraded strength of the adhesive bond.



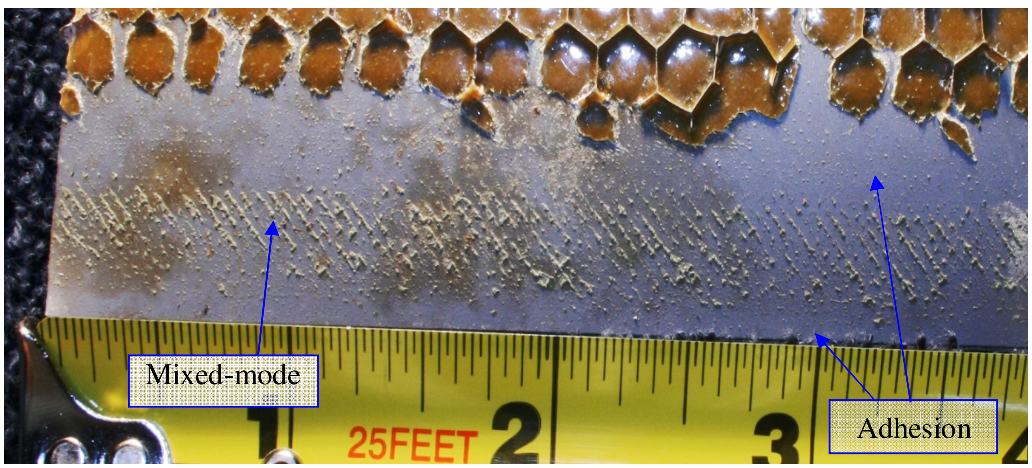
(b)

Figure 7: Photographs of a disbond showing the location of features ahead of the disbond front. View (b) is the boxed region of (a) magnified and imaged by SEM. The white portion in the lower right of (b) is bare metal.

A disbond from a point further inboard on the leading edge of a rotor blade from another helicopter which crashed after the blade had disintegrated is shown in **Figure 8(b)**. Note the similarities in the failure surfaces in **Figure 8(a)**. The partner blade to that shown in **Figure 8(b)** was found intact without any evidence of erosion along its bond line, therefore suggesting that erosion was not a factor in the demise of the blade shown in **Figure 8(b)** and suggesting instead a mixed-mode failure due to hydration at the bond interface. Its similarity to the blade in **Figure 8(a)** confirms that the disbond of the blade in **Figure 8(a)** was also due to interfacial hydration and not erosion as was officially reported.



(a)



(b)

Figure 8: (a) Disbond surface from a main rotor blade and (b) Disbond surface from a main rotor blade of a crashed aircraft

Conclusions

Only true cohesion failures indicate that the full strength of an adhesive bond was achieved (assuming that the adhesive was correctly formulated and cured). Cohesion failures in the absence of bond-line defects are caused by design inadequacies such as insufficient overlap length or failure to address thermal stresses. Adhesion or mixed-mode failures are due to degradation of the interface and indicate a reduction in bond strength caused by use of surface preparation processes which do not provide resistance to degradation, in particular to hydration of interfacial oxides. Current regulatory requirements do not adequately prevent the use of processes which produce bond interfaces with a poor resistance to degradation, however recent changes to AC-20-107-B now require demonstration of bond durability.

Of all bond failure types, mixed-mode failure is the most insidious because:

- Although NDI can detect cohesion and adhesion failures after they occur, it can not detect the bond strength reduction that can lead to mixed-mode failure.
- Mixed-mode failures are difficult to quantify in terms of the loss of strength of the interface, therefore it is impossible to assess the extent of loss of strength in the bonded component.
- The rate of progression of interfacial degradation is not currently predictable; hence it is impossible to assess failure loads for bonds which suffered interfacial degradation and consequently failed in mixed-mode.
- It is impossible for safety investigators to conclude whether a mixed-mode failure was the cause of an accident or if the failure occurred as a consequence of the accident. The only certainty is that the bond was weaker than at the time of certification and manufacture. . However, a destructive test on an intact sample of the affected component could provide useful information on the residual strength of the bond.

The use of durability and damage tolerance assessment based on NDI to monitor adhesive bond defects in service can only be effective if the surface preparation processes have a demonstrated resistance to interfacial degradation prior to construction. The wedge test (with modified acceptance criteria) is the most effective method for demonstration of bond durability.

The only effective defence against mixed-mode and adhesion failures is to validate the resistance of the interface to degradation, especially hydration. If the interface is resistant to hydration, then damage tolerance and NDI are effective weapons against bond failure and the current regulatory framework will be adequate for management of continuing airworthiness.

There is a need to increase the level of knowledge of adhesive bond forensics within the safety investigation community. If possible, structural teardown of time-expired components should be carried out to clarify the extent and severity of the conditions that lead to mixed-mode failure in service.

Safety Concerns:

Unlike fracture surface analysis for metals, bond failure analysis may not definitively prove the cause of an accident because it is impossible to exactly quantify the loss of bond strength due to hydration and mixed-mode or adhesion failure based on examination of the failure surface. When determining the cause of an accident potentially involving disbond factors, investigators can only rely on circumstantial evidence which often does not provide certainty and is expensive to collate. Therefore, the feedback loop which is normally provided to industry through the investigation process is less assured in this area of bonding science. This, together with the difficulty of in-service bond inspection, makes the task of preventing accidents due to disbond failures difficult.

The only adequate defence against adhesive bond degradation in service is to validate the resistance to hydration of the candidate surface preparation process prior to construction and certification of the bonded structure. Very few manufacturers include long-term bond durability assessment in certification programs for adhesive bonded structures because of a lack of technical understanding and also, there is currently no regulatory requirement to do so.

Many adhesively bonded Principal Structural Elements are managed using damage tolerance methodology, based on an invalid assumption that the adhesive surrounding a defect maintains full strength.

Consequently, there are many thousands of bonded structures in service (not only helicopter main rotor blades) constructed using bonding processes which have been certified on the basis of static and fatigue tests without evaluation of long-term bond durability, and therefore managed by unreliable "safety-by-inspection" programs. These rely on tests and analysis that did not consider the consequences of in-service degradation of bond strength due to hydration. This issue must be considered in conjunction with the fact that NDI can not detect the onset of incipient bond failures.

There is therefore a significant risk to continuing airworthiness of any bonded structures which have been constructed using processes which are susceptible to mixed-mode or adhesion failure.

This risk is not confined to a small number of manufacturers, and is not limited to a small number of components; it is relevant to the significant proportion of the aircraft industry that currently does not include effective environmental durability testing in adhesive bond quality assurance processes.

Acknowledgement:

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