ENVIRONMENTAL DEGRADATION OF ADHESIVELY-BONDED COMPOSITE JOINTS

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1 INTRODUCTION

Adhesive bonding of aircraft primary composite structures of large transport aircraft is seen as one of enabling technologies being investigated for next generation aircraft design and fabrication. Adhesive bonding offers many advantages as compared to riveted construction by eliminating mechanical fasteners, and better and more uniform load transfer that translates to improved fuel efficiency and more efficient aircraft design. Although certification of adhesively-bonded joints for primary structures still poses a significant challenge, there has been extensive effort to advance technologies that would ultimately contribute to advanced joint design, including emphasis on proper process control during manufacture, advancement in surface preparation, ongoing development of Non-Destructive Inspection (NDI) to monitor bond strength, advancement of environmentally-resistant adhesives and primers, and design for damage arrest amongst many other things. This massive amount of work, along with years of positive experience with adhesive bonding on aircraft, has inspired much confidence in the more widespread application of structural adhesive bonding.

It is well known that moisture has an effect on the physical, mechanical and chemical properties of polymers. Moisture, which exists in bulk polymer is either in a free or bond state [1]. Free water molecules take up the free volume among the polymer chains, causing reversible effects of plasticization and reduction in glass transition temperatures of the polymer. Bond water molecules, on the other hand, lead to irreversible damage to the polymer via hydrolysis and chain scission [2]. In addition, the significant difference in moisture absorption between the polymer and the fibres could lead to the evolution of localized stress and strain fields, leading to early nucleation of micro-cracking [3][4]. For adhesively-bonded composite joints, the bondline behaviour consisting of a substrate/adhesive interface and the adhesive becomes further complicated by the surface preparation methods of the substrates, which affect the mechanisms of environmental ageing and degradation [5]. A fundamental understanding of the environmental ageing and degradation mechanisms of adhesively-bonded composite joints and tools for quantitative prediction of the reduction in performance would enable manufacturers to make broader and more efficient use of adhesive bonding. This study focuses on the development of experimental and numerical tools to study the effects of hygrothermal degradation and fatigue loading on the Mode I and Mixed Mode I-II disbond behaviour of adhesively-bonded composite joints.

2 BACKGROUND

The conventional material strength approach is defined by applied stress and material failure strength. In this traditional approach, a design is considered sound when the anticipated applied stress is less than the material failure strength. This approach may be adequate for brittle fracture by imposing a safety factor, combined with minimal tensile elongation requirements on the material, but is not adequate for cases where discontinuities or damages propagate in a progressive manner, leading to failure of the structure. In contrast, fracture mechanics approach employs three variables: applied stress, discontinuity size and fracture toughness. Within this energy-based approach, failure occurs when the energy available for damage growth exceeds the resistance of the material. Strain energy release rate (*G*), one form of fracture toughness, is defined to be the loss of potential energy, *dU*, in the test specimen per unit of specimen width for an infinitesimal increase in delamination length, *da*, for a disbond growing under a constant displacement (1).

$$G = -\frac{1}{b}\frac{dU}{da} \tag{1}$$

where b is the width of the specimen. Analogous to the power relationship commonly used to describe the dependency of crack growth rate in metals on the stress intensity factor, K or ΔK, the fatigue delamination/disbond growth rate, *da/dN*, of a fibre-reinforced composite for a given *R*-ratio (the ratio of the minimum to maximum displacement experience during a cycle during fatigue testing) can be captured by a power relationship of the maximum strain energy release rate, G_{max}, as shown in Equation (2)

$$\frac{da}{dN} = C(G_{max})^n \tag{2}$$

where, *C* and *n* are material parameters. This is analogous to the Paris relationship for stress intensity factors.
This study aims to characterize and model quasi-static and fatigue behaviour of bonded composite specimens under Mode I Double Cantilever Beam (DCB) and Mixed Mode I/II bending (MMB) specimens that are subjected to varying mode mixities and environmental conditions. By convention, Mode I is a peeling motion while Mode II is an in-plane shearing motion. Each mode has a strain energy release rate associated with it, namely G_I and G_{II}, with mode mixity defined as:

$$\frac{G_{II}}{G} \tag{3}$$

$$G = G_I + G_{II} \tag{4}$$

When G exceeds the critical energy release rate, G_C, for the same mode mixity, the onset of delamination/disbond occurs. In the case of an existing delamination or disbond, the damage will propagate. Please note that the energy that is required for the onset of damage may not be the same as that for propagation. Thus the value of G_C for delamination/disbond onset could be different from that for propagation.

3 EXPERIMENTAL

3.1 Use of Template Specimen Design and Fabrication

The double cantilever beam (DCB) and mixed-mode bending (MMB) specimens were manufactured with two 13-ply unidirectional continuous carbon fibre-reinforced composite material, Cytec CYCOM 5276-1, bonded using two plies of 3M AF163-2K 0.06 psf adhesive, as per ASTM D 5528-1 [6] and ASTM D 6671M-06 [7] respectively. A 0.013 mm (0.0005″) thick Teflon insert was placed at the one end of the specimen between the

two plies of adhesive to introduce a pre-crack. Modifications in specimen configuration and result analysis were made to expand its application to the bonded joint configuration. The configuration of the DCB specimen is shown in Figure 1 with final dimensions of approximately 140 mm (5.5″) length, 21.5 mm (0.844″) width (b). The average of the specimen thickness ($2h$) is 3.8 mm (0.15″). Aluminium piano hinges were then adhesively-bonded to the end of the specimen. The distance between the loading point at the centre of the hinge and the edge of the Teflon insert, a_0, is 50.8 mm (2″) (see Figure 1) for the DCB specimens, and 25.4 mm (1″) for the MMB specimens (see Figure **2**Figure **2**. These specimens were used for both quasi-static and fatigue tests.

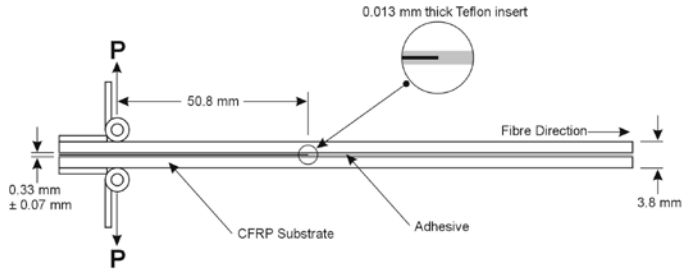

Figure 1: Schematic of the Adhesively-Bonded Mode I Double Cantilever Beam Composite Specimen.

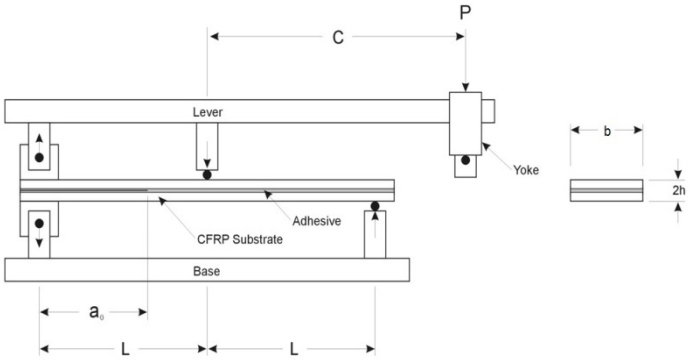

Figure 2: Schematic and image of the Mixed Mode I/II bending fixture.

3.2 Environmental Conditioning and Testing

In addition to the room temperature ambient (RTA) condition, some specimens were conditioned to other environmental conditions until they reached the equilibrium state, as listed in Table 1. These conditions include 70 °C, 95% Relatively Humidity (RH) (so called “IVWN”), as well as 82°C water immersion of 5% salinity (so called “HISN"). These two conditions were selected to simulate an upper limit and extreme conditions that an aircraft structure may be exposed to.

All specimens were conditioned in the environmental chambers in their respective environment until the equilibrium state was reached where no further weight change was observed. The IVWN specimens were conditioned in an ESPEC® PRA-4GP environmental conditioning chamber at the set temperature and humidity level for the entire duration of conditioning. The immersion temperature was maintained in a temperature controlled glass tank heated by heater strips bonded to the walls. The water temperature was maintained at ±2 ˚C of the target temperature. Water salinity was regularly measured and kept at 5% as per ASTM D1141-

98 [8]. These test specimens were weighed regularly until they reached the equilibrium state as specified in ASTM D5229 [9].

Table 1: Summary of environmental conditions.

Abbreviation	Description
RTA	Room Temperature, Ambient humidity
IVWN	Intermediate temperature (70 °C), Vapour (95% RH), Fresh Water, No freeze-thaw cycles
HISN	High temperature (82 °C), Immersion, Salt water, No freeze-thaw cycles

3.3 Experimental Setup

All the tests were conducted under displacement control at a frequency between 0.5 to 2.5 Hz. The test utilized a MTS 858 Tabletop hydraulic load frame, using a 250N or a 500N load cell with calibrations traceable to National Standards and load accuracy of 1% of reading. The data acquisition station used TestStar II Station Manager v3.4B to record the load and crosshead displacement during testing. Prior to testing, inspection and metrology were performed on each specimen. To aid in the detection of delamination/disbond, a thin layer of white lacquer paint was sprayed on the side of the specimen along with a paper scale.

All the quasi-static experiments were conducted in laboratory conditions, at a temperature and humidity level of 22±5 ℃ and 50±20% RH, respectively. All fatigue tests were performed at room temperature. While MMB fatigue tests were conducted at RTA environments, an environmental chamber, made from transparent Plexiglas, was used to maintain consistent humidity level in the conditioned specimens throughout the entire duration. Humid air generated from a cold-dispensing atomizer humidifier passed through the chamber during the test. The level of humid air intake was adjusted to ensure minimal weight change in the specimens.

3.4 Automatic Delamination/disbond Monitoring System (ADMS)

An automatic delamination/disbond monitoring system (ADMS) was developed in this study to measure disbond growth directly in a continuous manner. This was conducted by acquiring images of disbond tip positions in synchronization with load frame reading during a fatigue test, or acquiring images along with load or displacement reading from the frame during a static test. The camera system used for the study was capable of taking images up to 40 frames per second, and the image acquisition frequency can be tailored to match the rate of disbond growth. The ADMS consists of a digital camera, a 3-axis travelling base, and a Labview based-control system to analyze frame signals and trigger the camera operation. These images (see Fig. 3 for a sample) were manually examined in post-test analysis using UTHSCA ImageTool version 3.0 (see http://compdent.uthscsa.edu/dig/download.html). This non-contact system allowed for disbond length measurement as frequent as desired in an uninterrupted manner, ensuring maximum data quality and accuracy. The flexibility the system offers in terms of data density and potential for real-time determination of disbond length for test control make it an attractive technique for crack/disbond monitoring for both metallic and composite fatigue testing.

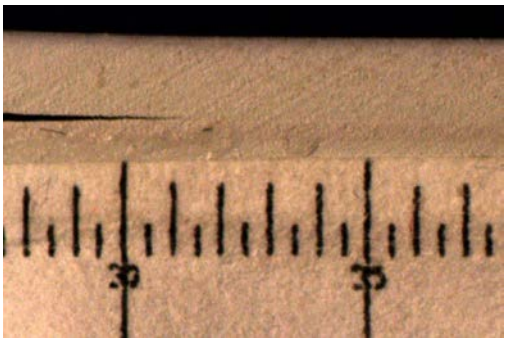

Fig. 3: A sample image acquired from the automatic delamination/disbond monitoring system (ADMS).

3.5 Test Matrix

The delamination/disbond onset and propagation critical strain energy release rate of bonded joints, G_C, was first measured under Mode I and II loading in quasi-static tests, following the procedure described in ASTM D 5528-1 [6] and ASTM D 6671M-06 [7] Fatigue tests were then conducted with a load ratio of 0.1. The mode mixity of 0 (pure Mode I), 0.2, 0.4, 0.6 and 0.8 were performed on as-made and conditioned specimens (IVHN and HIWN). Fracture toughness of the bonded joint under Mode II loading was also measured using the End of Notched Flexure (ENF) test method [10]. All the tests were conducted at room temperature ambient environment, except for Mode I DCB tests under fatigue loading, where the test could last over 1 million cycles. Table 2 shows the test matrix for the fatigue tests.

Table 2: Summary of Mode I fatigue fracture toughness tests for the study of the mode mixity and environmental effects.

Specimen condition	Type	Test condition	*R*-ratio	Mode mixity
RTA	Static/Fatigue	RTA	0.1	0, 0.2, 0.4,0.6, 0.8
IVWN	Static/Fatigue	RTA, or humidity chamber for DCB fatigue only	0.1	0, 0.2, 0.4,0.6, 0.8
HISN	Static/Fatigue	RTA, or humidity chamber for DCB fatigue only	0.1	0, 0.2, 0.4,0.6, 0.8

4 RESULTS AND DISCUSSION

4.1 Mode I and Mixed Mode I-II Quasi-static Fracture Toughness Testing

The critical energy release rates, G_c, of the as-made and conditioned specimens were measured in quasi-static tests for a range of mode mixities of 0, 0.2, 0.4, 0.6 and 0.8. However, the conditioned bonded composite specimens failed abruptly when subjected to a load mixity above 0.4, therefore no reliable data was available for comparison. Therefore, only the test results of the 0, 0.2 and 0.4 are compared for the three conditions, as shown in Table 3.

It can be seen that long-term environmental exposure led to a significant reduction in fracture toughness of the adhesively-bonded composite joints. For the Mode I, the fracture toughness of the bonded joints was reduced by as much as 34%, from 2900 J/m^2 for the as-made condition to 1917 J/m^2 for a bonded joint immersed in salt water at 80 ºC (or HISN). Such reduction in fracture toughness can also be seen on bonded joints under MMB loading of varying load mixity.

Table 3: Average values of G_c for adhesively-bonded joints exposed to different environments.

G_{II}/G	$G_{c,prop}$ (J/m^2)		
	RTA	IVHN	HISN
0	2900	2356	1917
0.2	2780	1654	2146
0.4	2709	2298	2489

4.2 Fatigue Disbond Growth

Figure 4 shows the fatigue disbond growth curves for the adhesively-bonded composite joints under Mode I with excellent repeatability. The test results also showed that the disbond growth rates followed a power relationship to the maximum strain energy release rate in the test range of 0.1 to 1.0 G_{Ic}. The disbond growth beyond the critical strain energy release rate was characterized by an unstable growth trend. No disbond threshold was observed for the test range.

4.3 Effects of Environmental Ageing

The effects of hygrothermal degradation at the conditions of 70 °C, 95% RH (IVWH) and 82 °C salt water (HISN) on Mode I fatigue disbond growth are shown in Figure 6. Compared to the unconditioned specimens, there was a slightly larger variation in the disbond growth curve from specimen to specimen. Nevertheless, the data were considered to be fairly consistent. It can be observed that the disbond growth rates for each condition followed a power relationship to the maximum strain energy release rate, G_{Imax}. On a log-log scale, the RTA specimen had the lowest slope, indicating the least dependency of the growth rate on G_{Imax}. In comparison, the disbond growth rate of the HISN specimens are the most sensitive to the change in Mode I loading. It is also interesting to observe that while environmental ageing led to a faster disbond growth rate in the higher load or G_{Imax} region, it led to a slower growth rate in the lower load region. Generally, immersion at a higher temperature led to a higher level of ageing and degradation of bondline fracture toughness of these composite joints.

A similar trend was observed for hygrothermal effects on Mixed Mode I/II fatigue delamination/ disbond growth of the bonded composites joints with a mode mixity of 0.2 and 0.4. As shown in Figure 6 and Figure 7, it appears that greater disbond propagation rate for the conditioned specimens when they are subjected to higher strain energy release rate levels. As the energy release rate reduces, the delamination/disbond behaviour among all three conditions tends to converge.

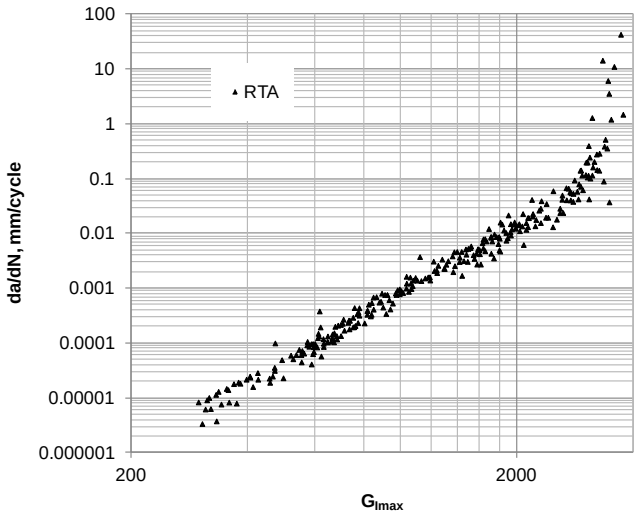

Figure 4: Mode I Fatigue disbond growth of unconditioned (RTA) adhesively-bonded composite joints.

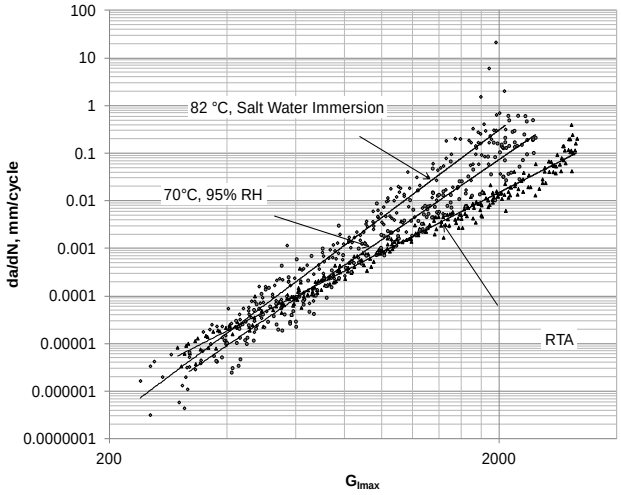

Figure 5: Steady fatigue disbond growth region of adhesively-bonded composite joints after long-term exposure to RTA, IVWH and HISN conditions.

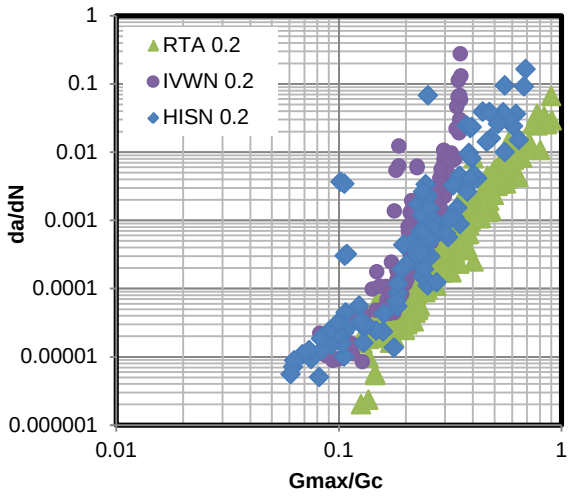

Figure 6: Delamination/disbond fatigue growth subjected to Mixed Mode I/II loading on log-log scale (mode mixity of 0.2).

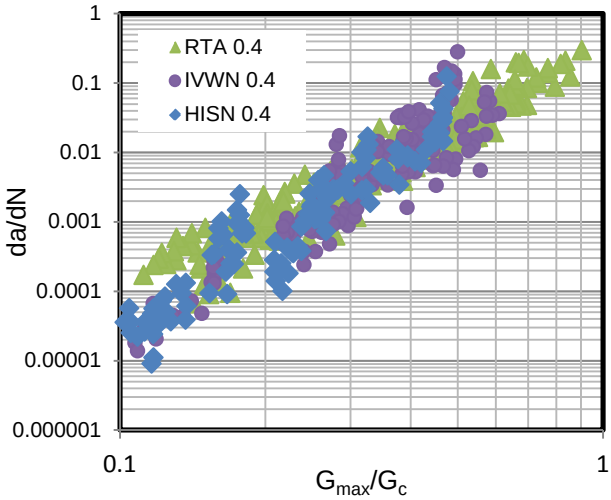

Figure 7: Delamination/disbond fatigue growth subjected to Mixed Mode I/II loading on log-log scale (mode mixity of 0.4).

While the hygrothermal effects on material behaviours such as strength and viscoleasticity can be obtained using established testing and modeling methods, there is a lack of fundamental understanding of such effects on change in failure mechanisms and predictive tools for conducting a quantitative assessments. It is conceivable that there were several competing factors associated with environmental ageing in play – viscoelasticity of polymers, change in material properties and strengths, as well as a change in failure mode. While enhanced plasticity as a result from high temperature and humidity may lead to stress relaxation, the reduction in strength of the materials as well as possibly early nucleation at the adhesive/substrate interface result in weakening of bonded joints. However, due to high scatter of delamination/disbond growth rate data for the conditioned specimens, such trend needs to be analyzed and treated carefully.

5 CONCLUSIONS

A study was conducted to investigate combined hygrothermal effect on quasi-static and fatigue performance of adhesively-bonded composite joints under Mode I and Mixed Mode I-II loading, with load mixity of 0, 0.2 and 0.4. Quasi-static DCB and MMB test results show that hygrothermal degradation led to reduction in fracture toughness of bonded composite joints under the studied loading conditions. Fatigue testing at a load ratio of 0.1 revealed that disbond growth rates for all specimens followed a Paris relationship to its maximum energy release rate, *G*, despite the mode mixity and the environmental conditions. The test results suggest that environmental degradation affected the fatigue life of the bonded joints in the highly loaded cases in a different manner from that in the lower loading cases. It is conceivable that there were multiple competing factors such as viscoelasticity of polymers and change in material properties and strengths may be responsible for these differences in materials performance.

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