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# The Chemical Resistance of Epoxy Adhesive Joints Exposed to Aviation Fuel and its Additives

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## **ABSTRACT**

Adhesively bonded structure and bonded composite repairs are often exposed to aviation fuel during service. A relatively limited number of studies examining the influence of fuel and the many additives it contains have been made. The current report details experimental studies that have examined the influence of fuel and common additives such as lubricity and deicing agents on the fracture toughness of metal and composite bonded adhesive joints. The studies aimed to determine if a series of reference fuels containing each of the additives may affect adhesive bond durability. Current results suggest that the bond durability of adhesive joints exposed for several thousands hours to fuel environments may reduce. Concentrated mixtures of water and deicing agent, which were prepared in order to simulate the environment in the bottom of fuel tanks, may also reduce joint fracture toughness. The present studies highlight the complexity of examining the influence of fuel environments on adhesive joints and the need for further research to more quantifiably assess possible mechanisms of bond degradation in typical aircraft operating environments.

## **RELEASE LIMITATION**

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# The Chemical Resistance of Epoxy Adhesive Joints Exposed to Aviation Fuel and its Additives

## Executive Summary

Adhesively bonded and composite structure is exposed to a range of hostile chemical environments during service. Aviation fuel is one of the most commonly encountered fluids that adhesive bonded and composite structure may be exposed to. The fuel is a complex mixture of saturated and unsaturated hydrocarbons that contains a range of additives which are used to improve its environmental, chemical and thermal stability during storage and use. A limited number of studies are available in the open literature that specifically examine the influence of the various fuel additives on the epoxy resin based materials that are used widely in composite and bonded structure, as well as, composite repairs. Based on data provided by adhesives manufacturers, it may be expected that epoxy based composite and adhesively bonded structure should not exhibit any significant deterioration when exposed to fuel and its additives.

In contrast to work reported in the literature by adhesives manufacturers, some Royal Australian Air Force (RAAF) experiences suggest that fuel may cause deterioration of epoxy based materials. In Air Vehicles Division, Chalkley and Geddes examined disbonds of boron-epoxy doublers exposed to F-111 fuel over a number of years and were unable to rule out the possibility that fuel or its additives may have contributed to degradation of adhesive mechanical properties. Research in the Sealants group in Maritime Platforms Division investigated the effect of diethylene glycol monoethyl ether (DGME) on sealants, coatings and corrosion. DGME caused blistering of epoxy based paints and destruction of the mechanical properties of polysulfide sealants. DGME was also found to depress the glass transition temperature of an epoxy barrier layer by 12°C, causing softening and loss of barrier properties.

The present report details studies to determine whether fuel and its potentially reactive additives could degrade the durability of adhesively bonded metallic and composite structure. Double cantilever beam specimens of aluminium and boron-epoxy composite were bonded with FM73 adhesive and the fracture toughness determined in room temperature environments of fuel and its additives. Tensile fatigue resistance of boron-epoxy bonded joints conditioned in model fuel mixtures was also examined.

Epoxy adhesive to metal bonded samples showed a reduction in durability when exposed to DGME, a deicing agent, and water or a reference fuel containing cumene hydroperoxide. The corrosive nature of the DGME and water environment may be responsible for the observed degradation in bonding and would simulate the conditions a repair at the bottom of a fuel

tank may experience. Cumene hydroperoxide simulates a fuel degradation product present in aging fuel and there was some indication that bond degradation was accelerated through corrosion of the metallic substrate. Extended exposure in reference fuel comprising dodecane and xylene suggested epoxy to metal bonds continued to degrade with time either as a result of deterioration of adhesive bonds or the adhesive.

Boron double cantilever bonded specimens also showed degradation in bond durability when exposed to the DGME and water and reference fuel containing cumene hydroperoxide environments. The complex fracture modes observed for the composite samples, however, made definitive conclusions regarding degradation mechanisms difficult. A similar trend in the durability of the adhesive to metal and composite-composite bonded samples exposed to the fuel environments may, however, suggest that processes affecting the adhesive properties are playing a role in the degradation mechanisms.

The apparent increase in the fatigue resistance of boron-metal skin doubler specimens exposed to the reference fuel may indicate an increase in fracture toughness of the adhesive, caused by fuel uptake, is affecting the peel stresses at the patch termination.

Whilst the results presented in this report provide an indication that surface treatment quality may be sensitive to fuel and its additives, further experimental work is required to relate these results to likely service performance.

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# 1. Introduction

Adhesively bonded and composite structure is exposed to a range of hostile chemical environments during service. Aviation fuel is one of the most commonly encountered fluids that adhesive bonded and composite structure may be exposed to. The fuel is a complex mixture of saturated and unsaturated hydrocarbons that contains a range of additives which are used to improve its environmental, chemical and thermal stability during storage and use. A limited number of studies are available in the open literature that specifically examine the influence of the various fuel additives on the epoxy resin based materials that are used widely in composite and bonded structure, as well as, composite repairs. In work reported by Sala [1] the effects of water, Skydrol, fuel, de-icing fluid and dichloroethane on carbon-epoxy mechanical properties were examined. Generally, fuel and de-icing fluid was not found to affect the mechanical properties significantly. Sala's work is consistent with data sheets provided by epoxy adhesives manufacturers [2]. The tensile shear properties of bonded joints immersed in water, JP-4 fuel, anti-icing fluid, hydraulic oil and type III hydrocarbon fluid at room temperature for seven days typically show no change from the normal condition. Epoxy resins are regularly used to line shipping containers used to transport gasoline and jet fuel due to their excellent resistance to petroleum products[3]. Therefore, it may be expected that epoxy based composite and adhesively bonded structure should not exhibit any significant deterioration when exposed to fuel and its additives.

In contrast to work reported by Sala and adhesives manufacturers, some Royal Australian Air Force (RAAF) experiences suggest that fuel may cause deterioration of epoxy based materials. In Air Vehicles Division, Chalkley and Geddes [4] examined disbonds of boron-epoxy doublers exposed to F-111 fuel over a number of years and were unable to rule out the possibility that fuel or its additives may have contributed to degradation of adhesive mechanical properties. Research in the Sealants group in Maritime Platforms Division [5] investigated the effect of diethylene glycol monoethyl ether (DGME) on sealants, coatings and corrosion. DGME is a deicing fluid used in F-111 fuel. It was found that while DGME was a more effective icing inhibitor and anti-microbial agent than ethylene glycol monoethyl ether (EGME), EGME did not adversely affect epoxy based paints or sealants. In contrast, DGME caused blistering of epoxy based paints and destruction of the mechanical properties of polysulfide sealants. DGME was also found to depress the glass transition temperature of an epoxy barrier layer by 12°C, causing softening and loss of barrier properties.

A brief investigation was undertaken in 1997 by Geddes [6] to examine the effect of fuel additives on adhesive durability. This investigation had the objective of qualitatively identifying which fuel additives may adversely affect FM73 adhesive. Eleven chemicals, which represented the main components of F-34 fuel, and which were most likely to cause adhesive degradation were selected. Samples of FM73 cured onto a glass plate cleaned with MEK were immersed in the pure chemicals for 48 hours. The results are presented in the Appendix A: . DGME, m-xylene, DCI-4A and cumene hydroperoxide were the most reactive, while EGME had no noticeable effect. These results are consistent with those reported by Wake [5] and suggest that DGME may potentially degrade epoxy adhesives. Cumene hydroperoxide is an aging by-product that can occur in old fuel at parts per million concentrations, whilst m-xylene is a large aromatic hydrocarbon fraction found in F-34 fuel.

DCI-4A is used as a lubricity additive. Qualitative indications suggest that these chemicals should be examined further to determine if they can degrade adhesive bonded structure.

The following report details studies to determine whether fuel and its potentially reactive additives can degrade the durability of adhesively bonded metallic and composite structure. Double cantilever beam specimens of aluminium and boron-epoxy composite were bonded with FM73 adhesive and the fracture toughness determined in room temperature environments of fuel and its additives. Tensile fatigue resistance of boron-epoxy bonded joints conditioned in model fuel mixtures was also examined. Conclusions regarding the relative effect of fuel and its additives are made, as well as, recommendations for future experimentation.

## 2. Experimental

### 2.1 Model Fuel Mixtures and Additives

The complex nature of the hydrocarbon mixtures and additives, which comprise jet fuel, complicate fundamental studies designed to examine the effect of fuel environments on material properties. Two approaches can be adopted, either a fundamental approach in which fuel components are isolated and tested individually or field fuel is used as a representative example. The benefit of testing the isolated components is that the specific additives in the fuel can be identified and the mechanisms by which the material is degraded can be elucidated. The downside of the approach is that due to the complexity and variability of fuel, a potentially reactive component of the fuel may not be included in the study. Fortunately, previous studies reported above have provided likely candidates in the fuel mixtures that should be considered in examining potential sources promoting adhesive degradation. Clearly isolating the fuel additives may prevent synergistic effects being identified, but the approach provides a good first level screening. The mixtures and their constituents used in the durability studies are indicated in Table 1. Typically, concentrations of around four times greater than normal levels found in field fuel were used in an attempt to accelerate or amplify any adhesive degradation processes. Cumene hydroperoxide was used at levels similar those found in aged fuel based on the practical case that levels higher than this change the viscosity of the fuel and would make it unusable in practical service situations. On this basis higher concentrations of the peroxide were considered to be unrealistic and any degradation effects may provide an unreliable indication of potential damage this by-product would cause.

Table 1 Fuel mixtures used to examine the chemical resistance of boron composite and metallic bonded structure.

Mixture	Constituents	Comments
Reference fuel	75% dodecane, 25% toluene	Model fuel without additives
DCI-4A	0.1% in reference fuel, lubricity additive	Normally present at 25ppm, long chain carboxylic acid mixed with benzene and xylene also aids in corrosion protection
DGME	1% in reference fuel	Icing inhibitor and anti-microbial agent used normally at 0.15%
DGME +H <sub>2</sub> O	25% DGME in distilled water	Icing inhibitor in water to simulate concentration of the additive in the bottom of fuel tanks where water tends to separate from the fuel and absorb the hydrophilic additives
Cumene Hydroperoxide	3 ppm cumene hydroperoxide in reference fuel	By-product of aging fuel
JP8+100	JP-8 aviation fuel and +100 additive at 1%	+100 is a thermal stability additive containing a antioxidant, deactivating additive (MDA) and a detergent normally present at 250ppm

## 2.2 Materials

The long crack extension durability specimen (Figure 1) had adherends that were made from unclad Al-2024 T3 adherends. Two layers of Cytec FM-73® structural adhesive were cured under recommended conditions of 1 hour at 120°C under 45 psi pressure. A 1% aqueous solution of Dow Corning Z-6040®  $\gamma$ -glycidoxypropyltrimethoxysilane (epoxy silane) from Sigma-Aldrich was used as an adhesion promoter.

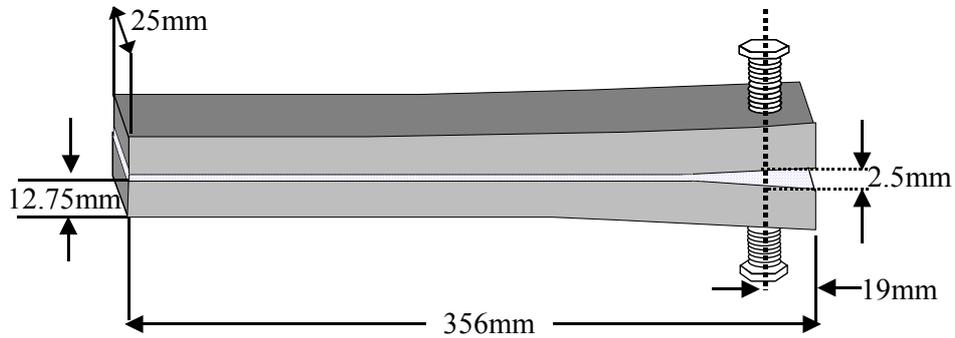


Figure 1 Dimensions of the long crack extension (LCE) test used to examine the chemical resistance of the FM73 to Al-2024T3 aluminium adhesive bond.

Boron composite double cantilever beam specimens (Figure 2) were made from precured laminates that were manufactured using 8 plies of pre-preg boron/epoxy, type 5521/4 from Textron Speciality Materials. Each laminate was cocured with a single layer of FM73 adhesive and the laminates were then bonded with a further 2 layers of FM73 adhesive.

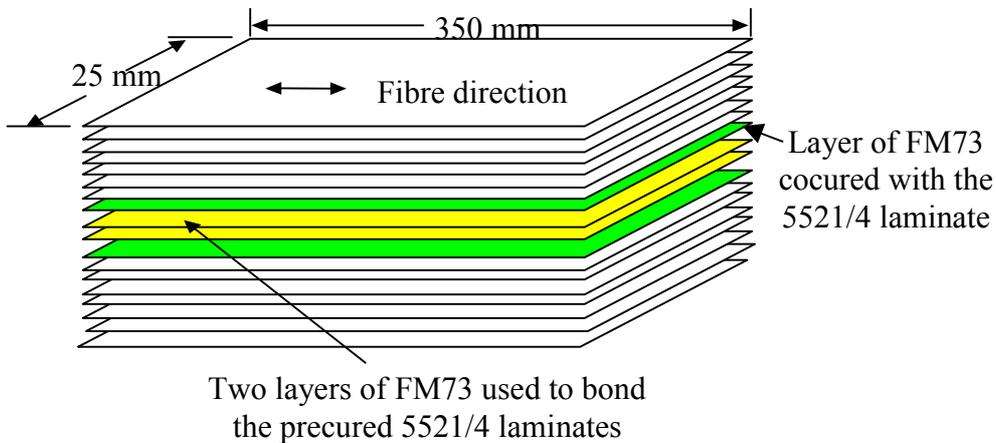


Figure 2 Diagrammatic representation of the boron-epoxy double cantilever beam specimens.

### 2.3 Surface preparation

The surface preparation of the aluminium alloy involved using the standard RAAF procedure[7]. Briefly, the aluminium was degreased with AR grade MEK solvent and water abrasion and wiping, followed by grit-blasting at 450kPa using dry 50 µm alumina grit and dry nitrogen propellant. The samples were treated with a 1% epoxy silane solution for 15 minutes and dried at 110°C for 60 minutes prior to bonding with FM73.

Following peel ply removal, boron laminate surfaces were solvent cleaned and abraded using AR grade MEK followed by abrading and water wiping using distilled water, water break testing and drying for 60 minutes at 110°C. Following drying, the bonding surfaces were lightly gritblasted and two layers of FM73 were applied to each surface prior to curing in a platen press using the standard conditions of 40 psi .

## 2.4 Mechanical testing

### 2.4.1 Long Crack Extension (LCE) Test

A Lockheed standard (derived from ASTM D3433 [8], ASTM D3762 [9] and Boeing Spec BSS 7208 [10]) was employed to determine fracture toughness in the different fuel and fuel additive environments,  $G_{Ic}$ , for the adhesive joints bonded with FM73. The dimensions of the test specimen are provided in Figure 1. The thick adherend enables fracture to propagate in the adhesive without problems associated with plastic yielding of the aluminium that may be encountered with thinner specimens. A longer initial crack-length also reduces errors in calculation of  $G_I$  [11]. A 3 mm thick adherend, as used in wedge tests, may deform plastically due to the high fracture toughness of the FM73 adhesive [12]. After bolt loading the LCE specimen, crack-growth was allowed to equilibrate at ambient conditions over several hours prior to insertion in the different fuel environments.

### 2.4.2 Boron Composite Double Cantilever Beam Specimens (BDCB)

A standard laminate of 8 plies of 5521/4 was cured using the recommended conditions of 120°C and 50psi for 60 minutes. Fingers of 25mm x 350mm dimension (Figure 2) were cut from the boron laminate and then bonded in pairs using two layers of FM73 after surface treatment using the process detailed in section 2.3. Wedges of 1.2mm thickness were inserted slowly using a drill press.

### 2.4.3 Boron Composite-Aluminium Fatigue Specimens

The skin symmetrical doubler specimens (SDS) were prepared by bonding 10 ply composite doublers to a 20mm wide Al-2024T3 alloy central adherend of 6.4mm thickness. The taper step-off rate of 25 to 1 was achieved by reducing the length of each successive ply by 3mm and applying each ply in a reverse wedding cake arrangement. The boron doublers were co-cured with a layer of FM73 prior to being bonded with a further two layers of FM73 to the aluminium adherend using the surface treatment detailed in section 2.3. Three specimens were immersed in the reference fuel described in Table 1 for a period of 11 months prior to tensile fatigue testing. Tests were conducted on a 100 kN Instron test machine using a 3Hz tensile cyclic (sinusoidal) load. The initiation and growth of cracks from the edge of the adhesive layer were monitored using the strain gauges bonded on the specimens.

The loading procedure and strain gauge technique to determine disbonding initiation load were adopted from reference [13]:

- (1) Apply 50000 cycles at a certain load amplitude and monitor strain gauge measurement every 100 cycles;
- (2) If none of the strain gauge amplitudes drops by 10% then increase the amplitude of the cyclic load by 2kN and go to step (1), otherwise terminate the test. Disbond initiation was defined to have occurred when the strain gauge amplitude had dropped by 10%.

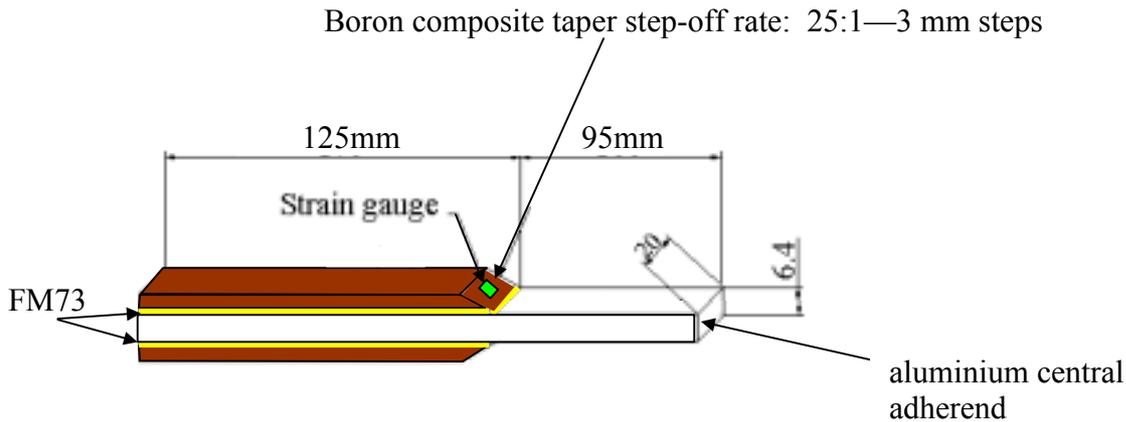


Figure 3 Skin symmetrical doubler specimen (SDS) used to examine fatigue strength of composite-metal adhesive bonded joints.

## 2.5 Environmental exposure

Both specimen types, LCE and BDCB, were exposed to the fuel environments (Table 1) in the laboratory that was typically 20°C and 50% relative humidity. Crack-length as a function of time was measured for a period of up to 1000 hours to establish relative durability performance and equilibrium fracture toughness in the humid environment,  $G_{Isc}$ . Two of the SDS specimens were immersed in reference fuel for 283 and 304 days.

## 2.6 Failure Analysis

Failed LCE, BDCB and symmetrical doubler specimens were analysed and fracture surfaces were photographed using a Cannon EOS D60 digital camera with a 100mm macro lens, operating with aperture priority (AE) mode and an aperture setting of 16 to provide sufficient depth of field for the surfaces being examined. Exposure time, using the natural fluorescent lighting of the laboratory, was typically 6 seconds.

## 3. Results

### 3.1 LCE samples exposed to fuel and fuel additives

Figure 4 indicates the crack length as a function of root time for LCE specimens exposed to the reference fuel at room temperature for approximately 1000 hours. Sample 2 actually shows a small increase in crack length, suggesting some change in the fracture toughness of the sample. In relative terms, the average crack growth is probably not significantly greater than would be expected for samples exposed to a low humidity, room temperature environment. The major growth is simply a result of adhesive creep caused by the aggressive loading mode used for the LCE specimens. Figure 5 indicates the relative performance of the samples exposed to the reference fuel containing DCI-4A. Crack-growth is similar or reduced compared with the reference fuel environment. Figure 6 shows that addition of 1% DGME to the reference fuel has no substantial influence on the crack growth of specimens. Figure 7 indicates crack growth for samples immersed in water containing 25% DGME in distilled water. Two of the three samples exhibit significantly higher crack growth at extended exposure times relative to the standard and the sample which performs at a similar level to the reference specimen also exhibits an obvious increase in crack growth at the latter stages of the exposure. These results suggest that the water DGME solution may be affecting the bond line in some manner.

Figure 8 indicates the crack growth of LCE samples exposed to fuel containing cumene hydroperoxide. As can be seen, sample 2 has a higher crack length compared to sample 1 and the reference sample, possibly indicating some effect. The performance of LCE samples exposed to JP-8 fuel containing 1% of the +100 additive is significantly different with small initial crack length and little crack growth for specimen 1 and slightly higher growth for sample 2, that is similar to the reference fuel. The reason for the small crack length of sample 1 is not clear, however, the results from both samples suggest the additive has little effect on the adhesive joint fracture toughness.

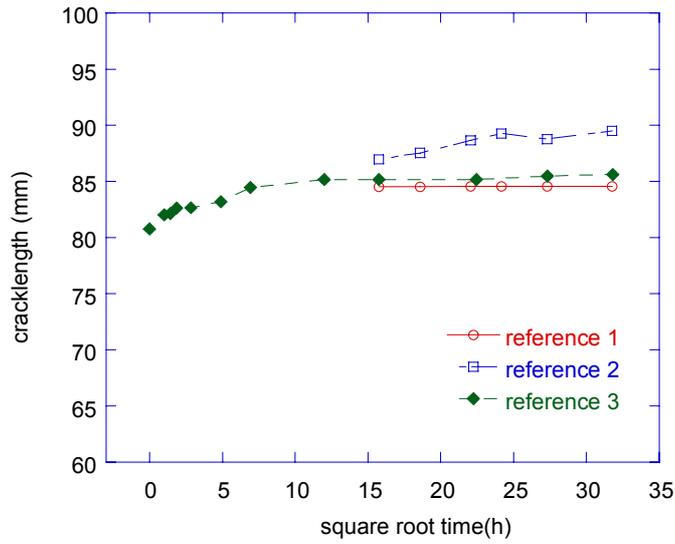


Figure 4 Crack length (mm) as a function of root time ( $h^{1/2}$ ) for LCE specimens exposed to reference fuel at room temperature.

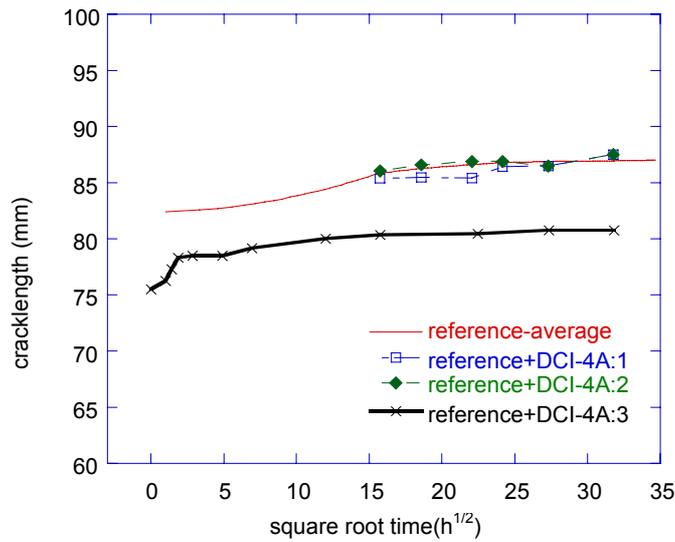


Figure 5 Crack length (mm) as a function of root time ( $h^{1/2}$ ) for LCE specimens exposed to reference fuel containing 0.1% DCI-4A at room temperature. Performance relative to the reference fuel crack growth (Figure 4) is shown.

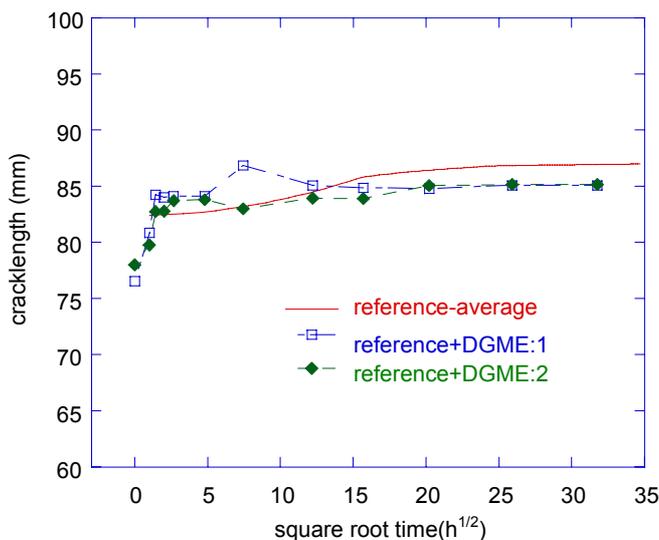


Figure 6 Crack length (mm) as a function of root time ( $h^{1/2}$ ) for LCE specimens exposed to reference fuel containing 1.0% DGME at room temperature. Performance relative to the reference fuel crack growth (Figure 4) is shown.

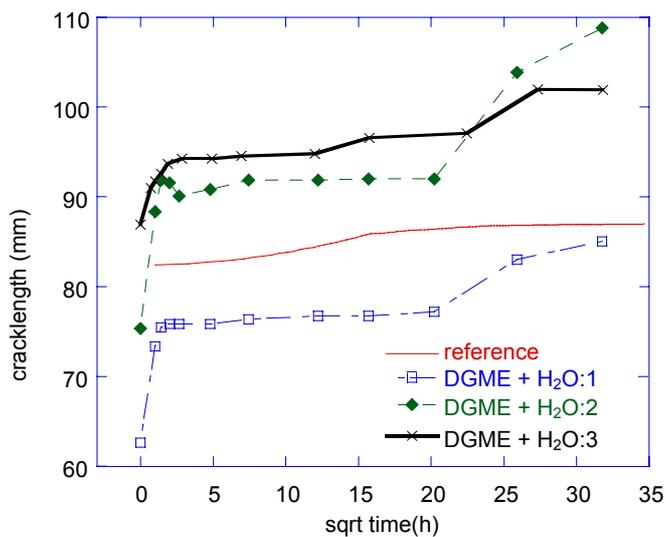


Figure 7 Crack length (mm) as a function of root time ( $h^{1/2}$ ) for LCE specimens exposed to 25.0% aqueous DGME solutions at room temperature. Performance relative to the reference fuel crack growth (Figure 4) is shown.

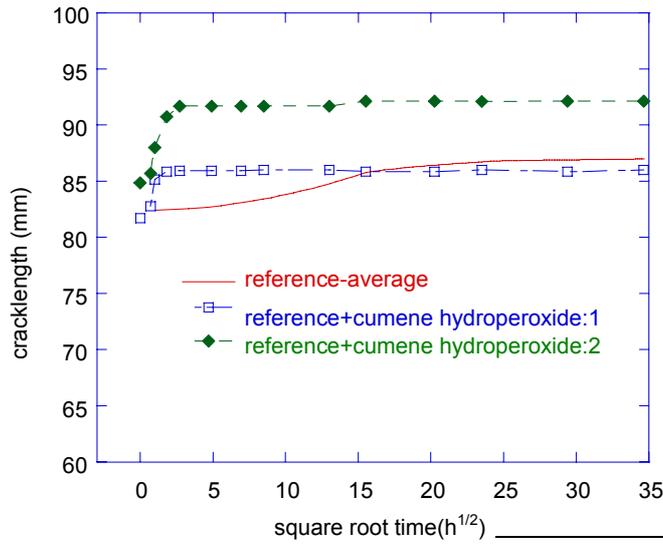


Figure 8 Crack length (mm) as a function of root time ( $h^{1/2}$ ) for LCE specimens exposed to reference fuel containing 3ppm of cumene hydroperoxide at room temperature. Performance relative to the reference fuel crack growth (Figure 4) is shown.

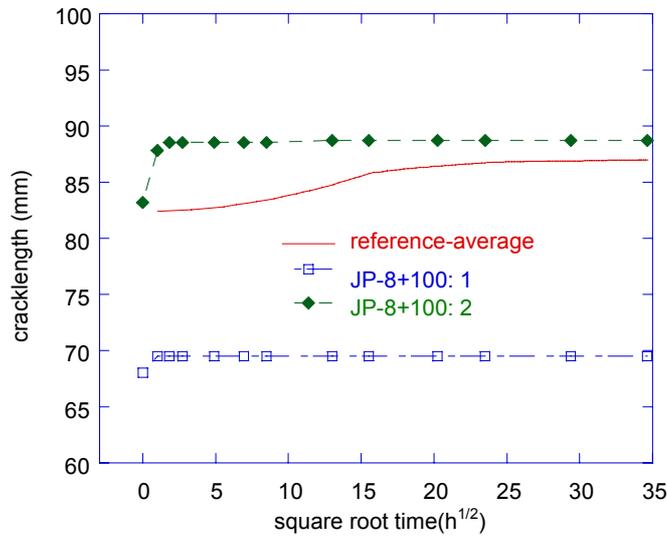


Figure 9 Crack length (mm) as a function of root time ( $h^{1/2}$ ) for LCE specimens exposed to JP-8 fuel containing 1.0% +100 at room temperature. Performance relative to the reference fuel crack growth (Figure 4) is shown.

### 3.2 Mode I fracture toughness for LCE specimens exposed to fuel and fuel additives for 1000 hours at room temperature

The fracture toughness,  $G_{Ic}$  ( $J/m^2$ ), was calculated from the crack length measured after approximately 1000 hours of immersion in the fuel and fuel and additive mixtures using equation 1[14]:

$$G_{Ic} = \frac{3Eh^3\delta^2}{16L^4} \frac{I}{\left(1 + \frac{\lambda_0}{\lambda} 0.64 \frac{h}{L}\right)^4} \quad (1)$$

where

$$\lambda = \left(\frac{3k}{Ebh^3}\right)^{\frac{1}{4}} \text{ and } \lambda_0 = \left(\frac{3k_0}{Ebh^3}\right)^{\frac{1}{4}} \text{ and } k = \frac{I}{\frac{1}{k_0} + \frac{1}{k_a}}$$

$$\text{with } k_0 = \frac{2Eb}{h} \text{ and } k_a = \frac{E_a b}{h_a}$$

$E$  is Young's modulus of the aluminium, 72.4 GPa,  $h$  is the adherend thickness (12.75mm),  $\delta$  is the crack opening displacement, 2.5mm, and  $L$  is the crack length (mm).

$E_a$  is the modulus of the FM73, 2.27GPa, and  $h_a$  is the thickness of the FM73 bondline, 0.2mm, and  $b$  is the width of the adherend, 25.4mm.

Table 2 indicates the  $G_{Ic}$  values measured for the different fuel exposures. The results suggest that the fracture toughness of the FM73 is around 2100  $J/m^2$ . The only samples which exhibited notable decreases in the fracture toughness were the samples exposed to the 25% aqueous solution of DGME and a lower value observed for one of the fuel samples containing the cumene hydroperoxide. A value around 4000 $J/m^2$  for one of the JP8+100 specimens suggests some error in the experimental measurement, however, careful examination of sample dimensions and crack length measurements failed to reveal any significant variation from the recorded values.

Table 2 Fracture toughness for LCE specimens exposed to reference fuel and fuel mixtures at room temperature after approximately 1000 hours.

Exposure Environment	Exposure Time (h)	$G_{Ic}$ ( $J/m^2$ )	$G_{Ic}$ -Average ( $J/m^2$ )
Reference	1008	2410	2126
	1008	1904	
	1011	2064	
Reference+DCI-4A	1008	2098	2264
	1008	2131	
	1011	2564	
Reference+DGME	1008	2142	2081
	1008	2019	
DGME+H <sub>2</sub> O	1008	2108	1408
	1008	899	
	1011	1217	
JP8+100	1200	4037	2951
	1200	1864	
Reference + Cumene hydroperoxide	1200	2228	1939
	1200	1649	

### 3.3 Failure Surface investigation of LCE samples exposed to fuel and fuel additives

Figure 10 shows the failure surfaces resulting from the three samples exposed to the reference fuel for 1000 and 2000 hours exposures. Whilst samples 1 and 3 exhibit predominantly cohesive failure within the FM73 layer, sample 2 shows that exposure for an additional 1000 hours actually results in extended crack growth. The extended crack growth in sample 2 also appears to propagate interfacially, between the adhesive and metal, as indicated by the metallic appearance of the left hand side sample.

Figure 11 shows the failure surfaces for the LCE samples exposed to the reference fuel + 0.1% DCI-4A for 1000 and 2000 hours indicating the crack length after 1000 hours. Generally, there appears to be less "adhesion" failure on these samples, although sample 1 shows some interfacial failure particularly in the area where cracking has occurred in the second 1000 hours, similar to the trend observed with the reference fuel.

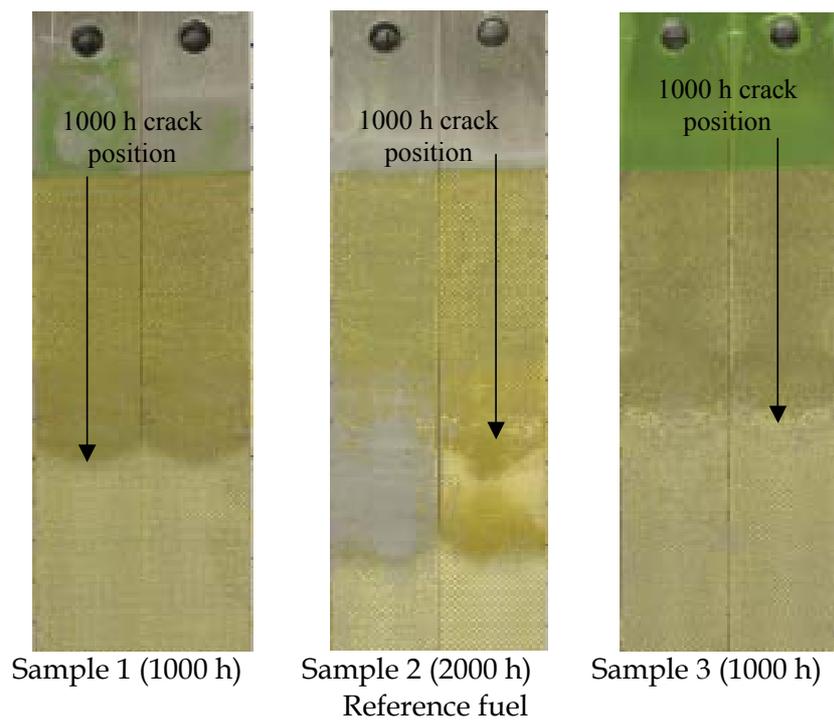


Figure 10 Failure surfaces of LCE samples exposed to the reference fuel for 1000 and 2000 hours indicating the crack length after 1000 hours.

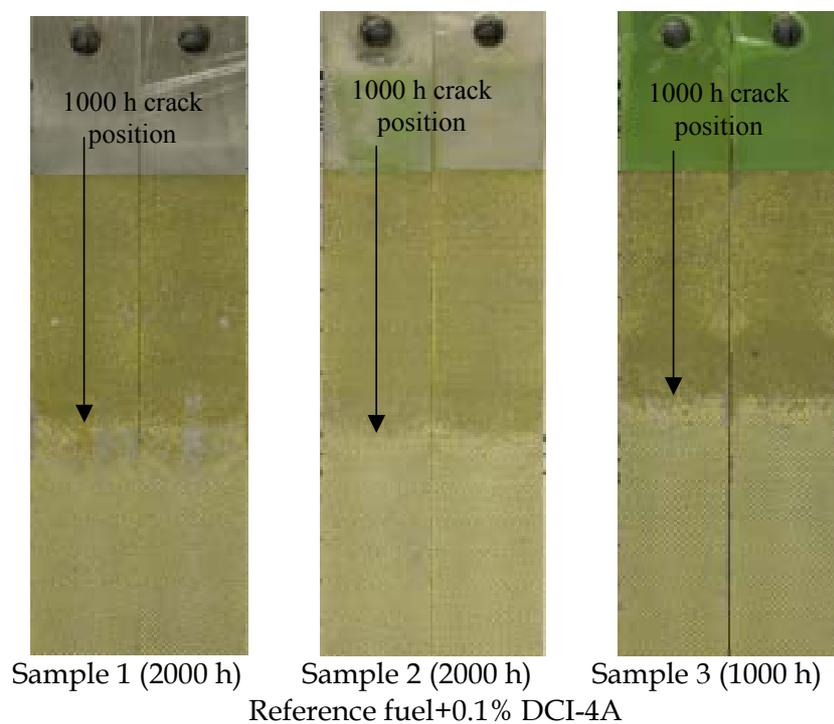


Figure 11 Failure surfaces of LCE samples exposed to the reference fuel + 0.1% DCI-4A for 1000 and 2000 hours indicating the crack length after 1000 hours.

Figure 12 shows the failure surfaces for the LCE samples exposed to the reference fuel + 0.1% DGME for 1000 hours, indicating the crack length after 1000 hours. Generally, there appears to be cohesive failure within the FM73 layer for these samples.

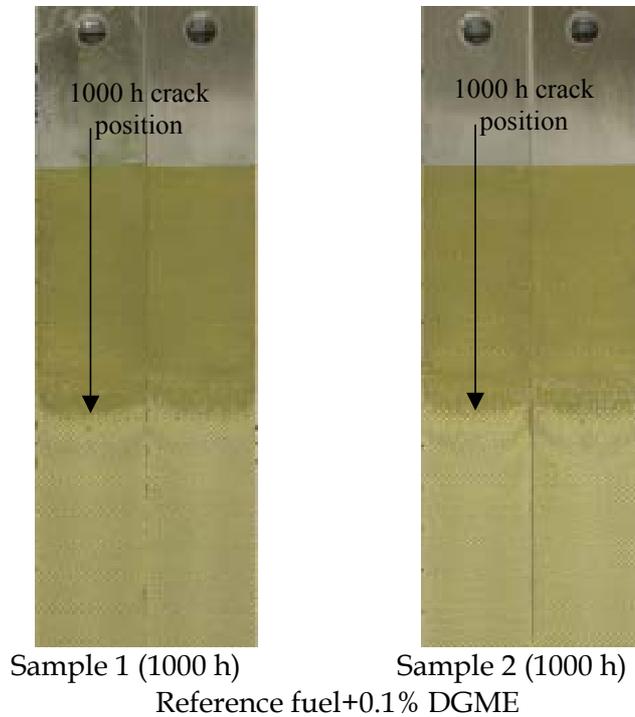


Figure 12 Failure surfaces of LCE samples exposed to the reference fuel + 0.1% DGME for 1000 hours, indicating the crack length after 1000 hours.

Figure 13 shows the failure surfaces of LCE samples exposed to a 25% aqueous solution of DGME for 1000 hours, indicating the crack length after 1000 hours. Samples 2 and 3 show obvious regions of failure at the adhesive and metal interface, suggesting that cracking has proceeded in this region due to degradation of the adhesive bonds. The first sample contains less evidence of the interfacial failure in the cracking region, although some adhesion failure has occurred.

Figure 14 shows the failure surfaces of LCE samples exposed to the JP-8+100 for 1000 hours, indicating the crack length after 1000 hours. Neither sample indicates any substantial regions of adhesion failure, however, the crack length of the two samples is substantially different. As discussed in section 3.2 there appears to be no obvious reason for the differences in crack length of the two specimens.

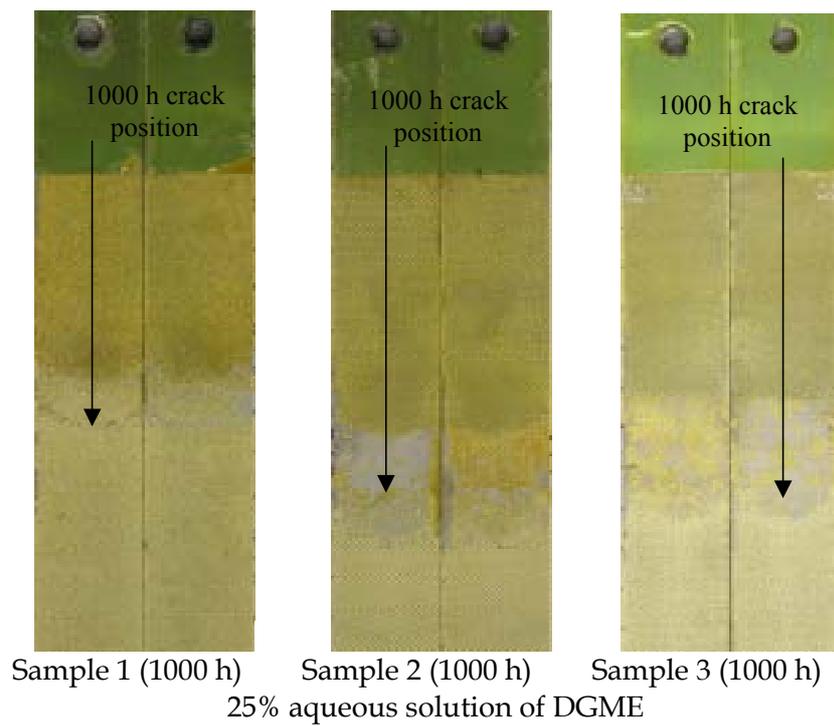


Figure 13 Failure surfaces of LCE samples exposed to the 25% aqueous solution of DGME for 1000 hours, indicating the crack length after 1000 hours.

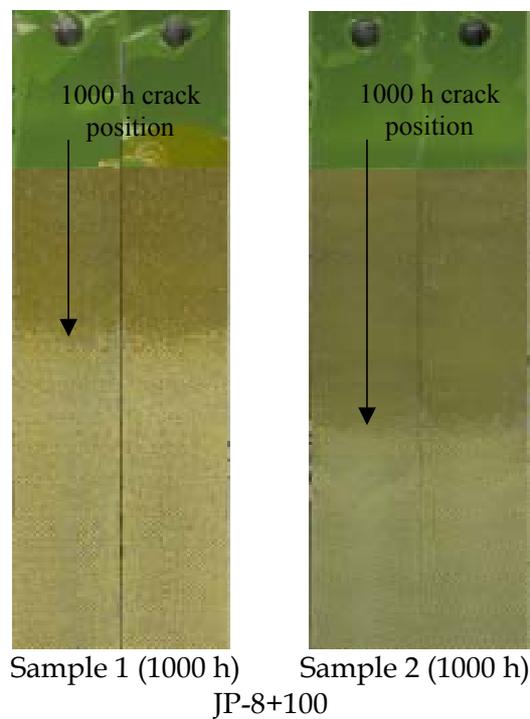


Figure 14 Failure surfaces of LCE samples exposed to the JP-8+100 for 1000 hours, indicating the crack length after 1000 hours.

Figure 15 shows the failure surfaces of LCE samples exposed to the reference fuel + 2.8ppm cumene hydroperoxide for 1000 hours, indicating the crack length after 1000 hours. Neither sample indicates any substantial regions of adhesion failure, although there appear to be a number of smaller regions where the metal has been attacked and pulled out during fracture. A higher magnification area of such an area is shown in Figure 16. Whilst similar areas of corrosion were observed in some of the other samples, the samples exposed to the reference fuel + 2.8ppm cumene hydroperoxide appeared to have a greater number of such regions.

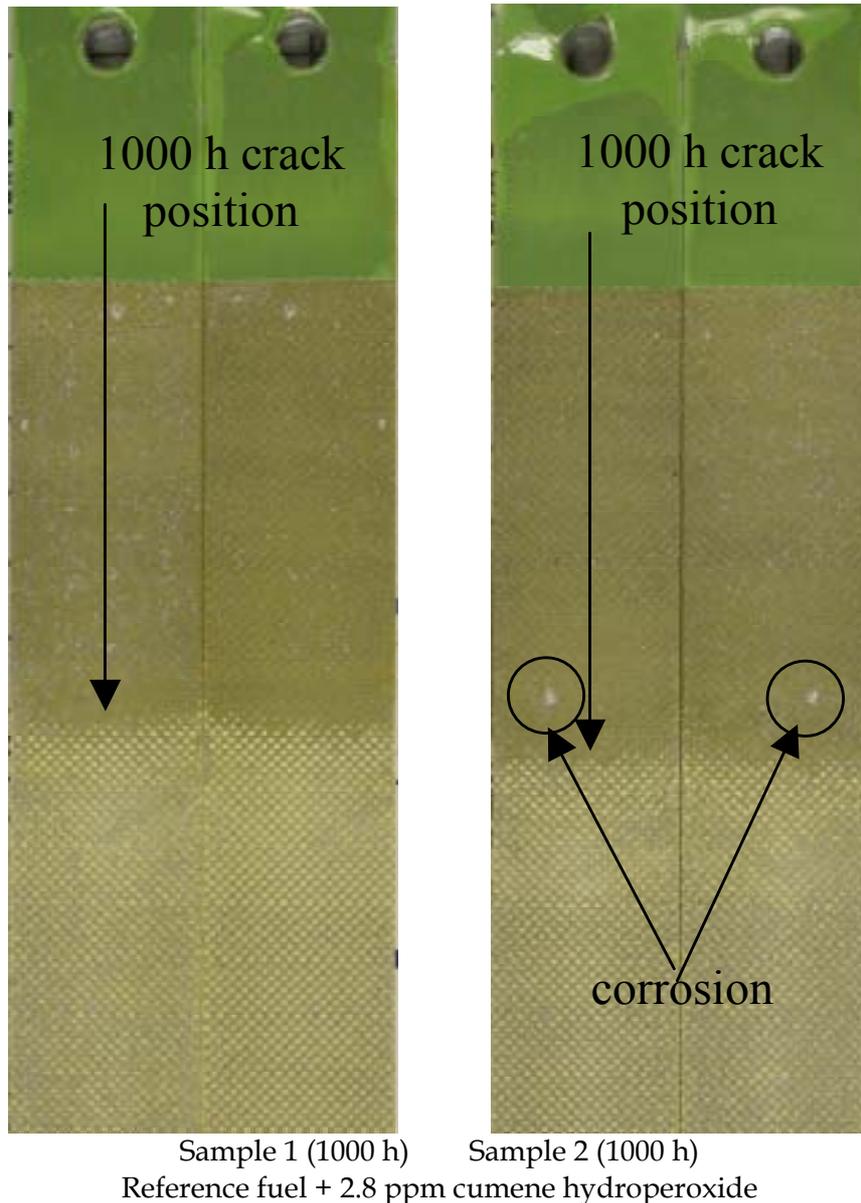


Figure 15 Failure surfaces of LCE samples exposed to the reference fuel + 2.8ppm cumene hydroperoxide for 1000 hours, indicating the crack length after 1000 hours.

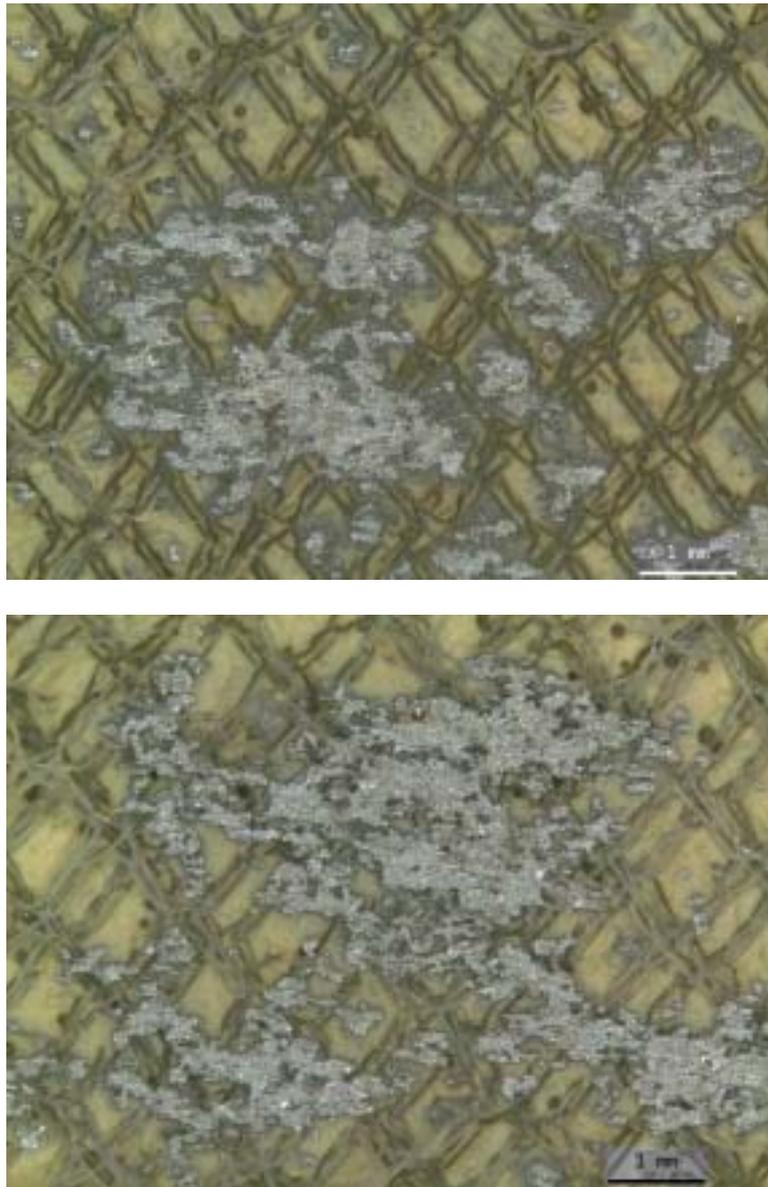


Figure 16 High magnification image of the corrosion region highlighted in Figure 15 for the LCE samples exposed to the reference fuel + 2.8ppm cumene hydroperoxide.

### 3.4 BDCB samples exposed to fuel and fuel additives

Figure 17 indicates the crack length as a function of root time for boron double cantilever beam specimens exposed to the reference fuel and mixtures at room temperature for approximately 1000 hours. The water and DGME exposed sample actually indicates an increase in crack growth relative to the reference sample and the sample exposed to the cumene hydroperoxide and fuel mixture shows a small increase in crack growth towards the end of the exposure time. The remaining samples, exposed to fuel containing DCI-4A and DGME, showed very little crack growth. The trend in these results is very similar to those observed for the aluminium specimens in section 3.1.

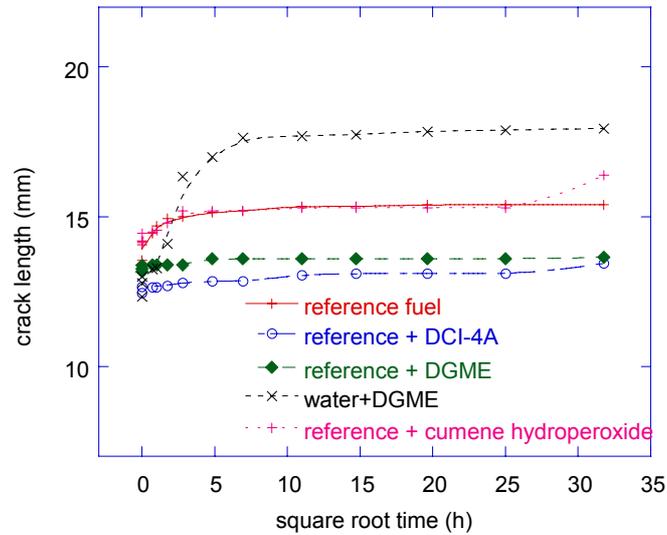


Figure 17 Crack length as a function of exposure time for boron double cantilever beam specimens exposed to fuel and fuel additives for 1000 hours.

### 3.5 Mode I fracture toughness for boron double cantilever beam (BDCB) specimens exposed to fuel and fuel additives for 1000 hours at room temperature

The fracture toughness,  $G_{Ic}$  ( $J/m^2$ ), was calculated from the crack length measured for times up to 1000 hours of immersion in the fuel and fuel and additive mixtures using equation 1 (refer section 3.2). Values of the variables used in equation 1 are provided in Table 3.

Table 3 Values used to calculate the fracture toughness of boron double cantilever beam specimens exposed to fuel and fuel mixtures for 1000 hours[15].

Variable	Value
$E$ , Young's Modulus of Boron	207 GPa
$E_a$ , Young's Modulus of FM73	2.27 GPa
$h$ , BDCB adherend thickness	1.8 mm
$h_a$ , thickness of the FM73 bondline	0.9 mm
$\delta$ , crack opening displacement	1.2 mm
$b$ , BDCB adherend width	25.5mm

Table 4 indicates the  $G_{Ic}$  values measured for the different fuel exposures. The results suggest that the fracture toughness of the FM73 is around  $2500 J/m^2$  for the reference fuel after 1000 hours exposure. Slightly higher values are observed after 1000 hours for the reference fuels containing DCI-4A and DGME. The samples immersed in the 25% aqueous DGME solution showed almost a 50% decrease from the reference sample. The samples exposed to fuel containing cumene hydroperoxide showed approximately 20% decrease in fracture toughness relative to the reference samples at 1000 hours exposure time. These trends are very similar to those observed for the LCE specimens (Table 2). Some variation in the calculated  $G_{Ic}$  values

may be expected relative to those presented in Table 2. The reason for the variation include the shorter crack length measurements in the boron samples, resulting in greater potential error in the calculated fracture toughness. Additionally, the calculation of the adhesive thickness was based on measuring the difference in thickness of the laminates and the bonded specimens. During cure the FM73 will co-cure with the boron laminate and produce a layer near the FM73 and resin interface that is a mixture of properties of the two layers. Further complicating calculations is a mixed mode of fracture observed for the samples, which is discussed further in section 3.6.

Table 4 Fracture toughness for boron double cantilever beam specimens exposed to reference fuel and fuel mixtures at room temperature for 1000 hours.

Time (h)	$G_{Ic}$ -Average(J/m <sup>2</sup> )				
	Sample				
	Reference	Reference + DCI-4A	Reference + DGME	DGME+H <sub>2</sub> O	Reference + Cumene hydroperoxide
0	3698	3193	3746	4517	3353
0	3286	2718	3554	4021	3331
0	3187	3368	3670	3820	3134
0.5	3014	3204	3542	3596	3100
1	2853	3204	3542	3554	3065
3	2702	3165	3542	2949	2901
7.8	2673	3088	3542	1820	2660
23.3	2589	3051	3379	1599	2660
48	2561	3051	3379	1411	2660
121	2481	2906	3379	1398	2604
216	2481	2872	3379	1385	2604
385	2455	2872	3379	1359	2604
625	2455	2872	3379	1346	2604
1009	2455	2642	3340	1334	2074

### 3.6 Failure surface investigation of BDCB samples exposed to fuel and fuel additives

Figure 18 to Figure 22 show the failure surfaces for the boron double cantilever beam specimens exposed to the reference fuel and additives for 1000 hours. In all samples the fracture appears to propagate either between the FM73 layers or within the boron composite resin layer adjacent to the FM73 layers. The support scrim in the 5521/4 resin has been exposed during fracture supporting the conclusion that fracture has propagated through the composite resin. The relative percentage of failure in the resin appears to increase from Figure 18 to Figure 22, with the cumene hydroperoxide sample showing almost 100% failure through the 5521/4 layer.

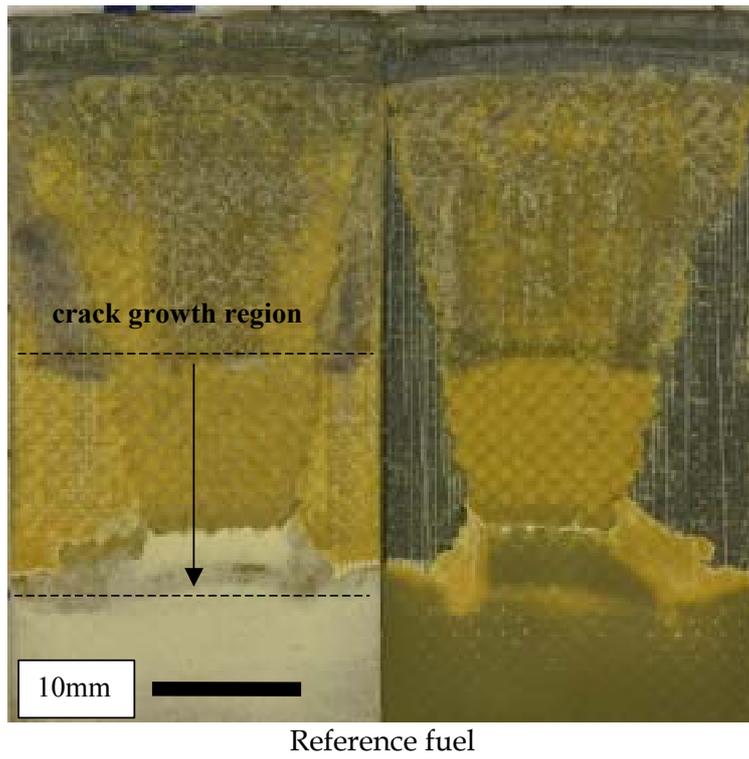


Figure 18 Failure surfaces of boron samples exposed to the reference fuel for 1000 hours indicating the crack growth region.

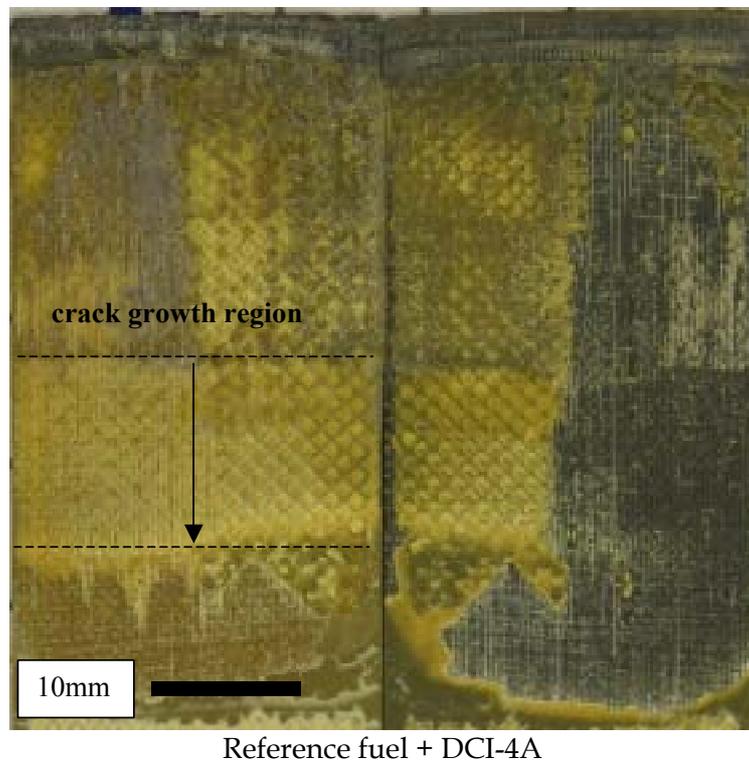
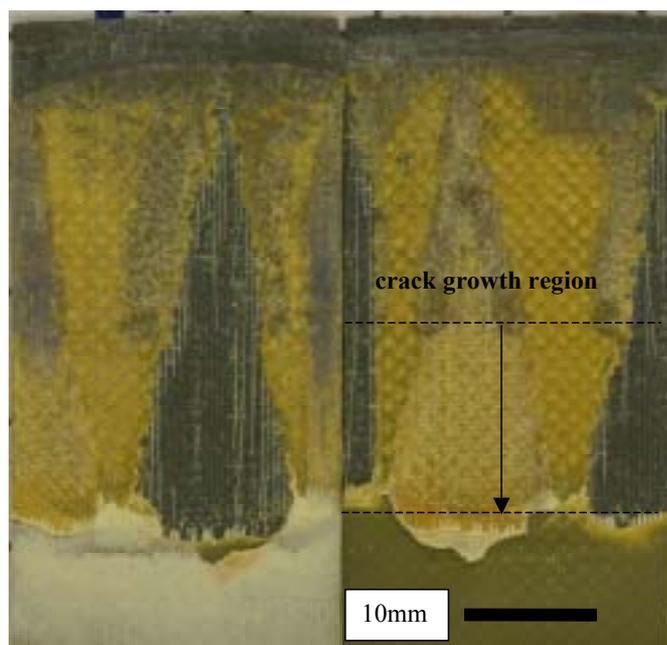
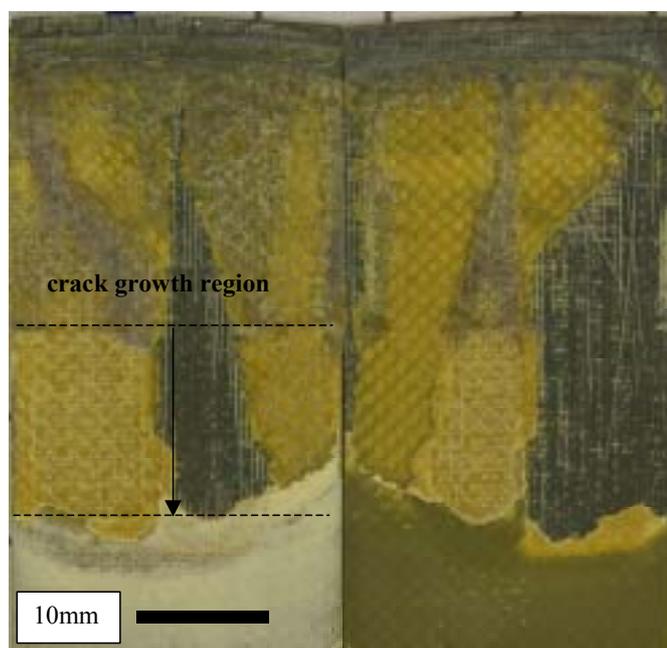


Figure 19 Failure surfaces of boron samples exposed to the reference fuel + DCI-4A for 1000 hours indicating the crack growth region.



Reference fuel + DGME

Figure 20 Failure surfaces of boron samples exposed to the reference fuel + DGME for 1000 hours indicating the crack growth region.



25% aqueous DGME solution

Figure 21 Failure surfaces of boron samples exposed to the 25% aqueous DGME solution for 1000 hours indicating the crack growth region.

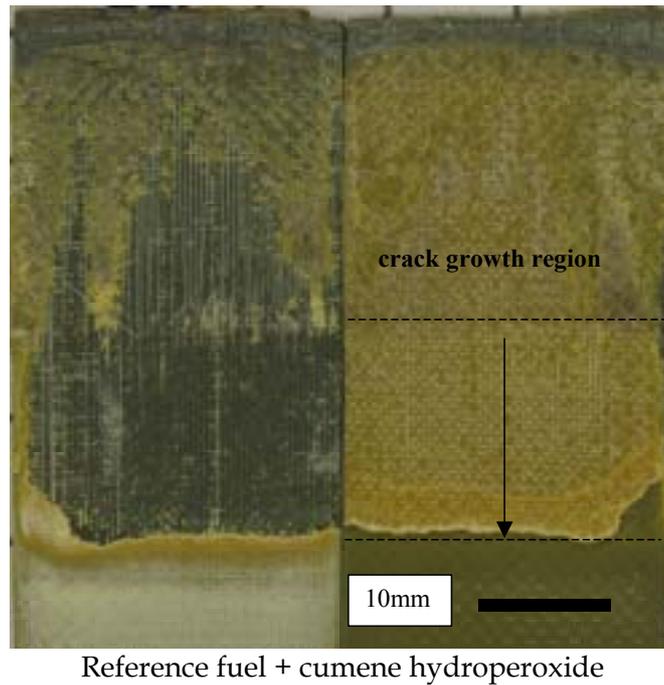


Figure 22 Failure surfaces of boron samples exposed to the reference fuel + cumene hydroperoxide for 1000 hours indicating the crack growth region.

### 3.7 Boron composite-aluminium fatigue specimens

Figure 23 graphs the strain gauge reading for the boron-aluminium fatigue specimens after exposure to the reference fuel for 283 (sample 1) and 304 days (sample 2). Both samples indicate delamination occurs at the start of the 29kN loading cycle. A third specimen was also tested, however, the strain gauge failed just prior to the onset of delamination, which occurred towards the end of the 29kN loading cycle.

Figure 24 shows the failed surfaces produced by the fatigued boron-aluminium sample immersed in the reference fuel for 283 days prior to testing. The fracture has propagated from the corner and close to the FM73 and metal interface. This locus of failure was also observed for similar specimens that were unconditioned. However, the unconditioned samples also failed at a lower load, around 26kN[16].

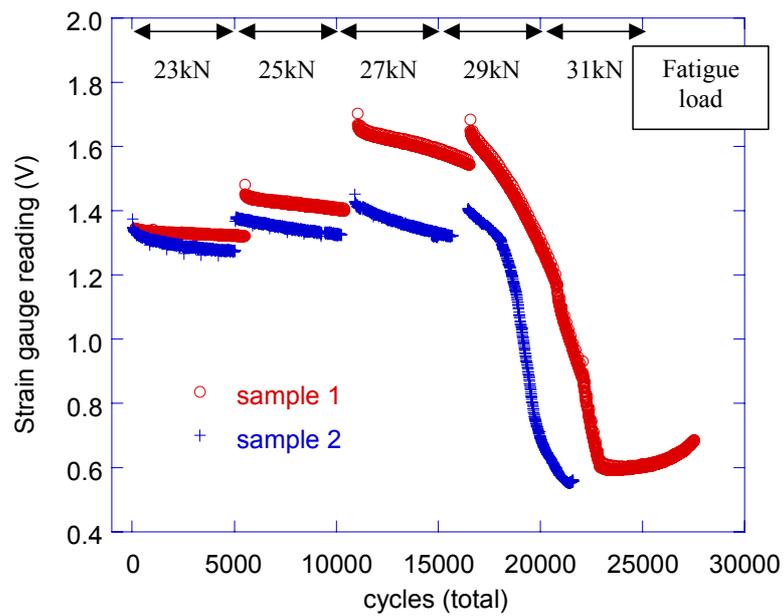


Figure 23 Strain gauge reading for boron-aluminium SDS fatigue specimens indicating the point at which delamination occurs after fatigue loading is carried out in 2kN, 50,000 cycles increments.

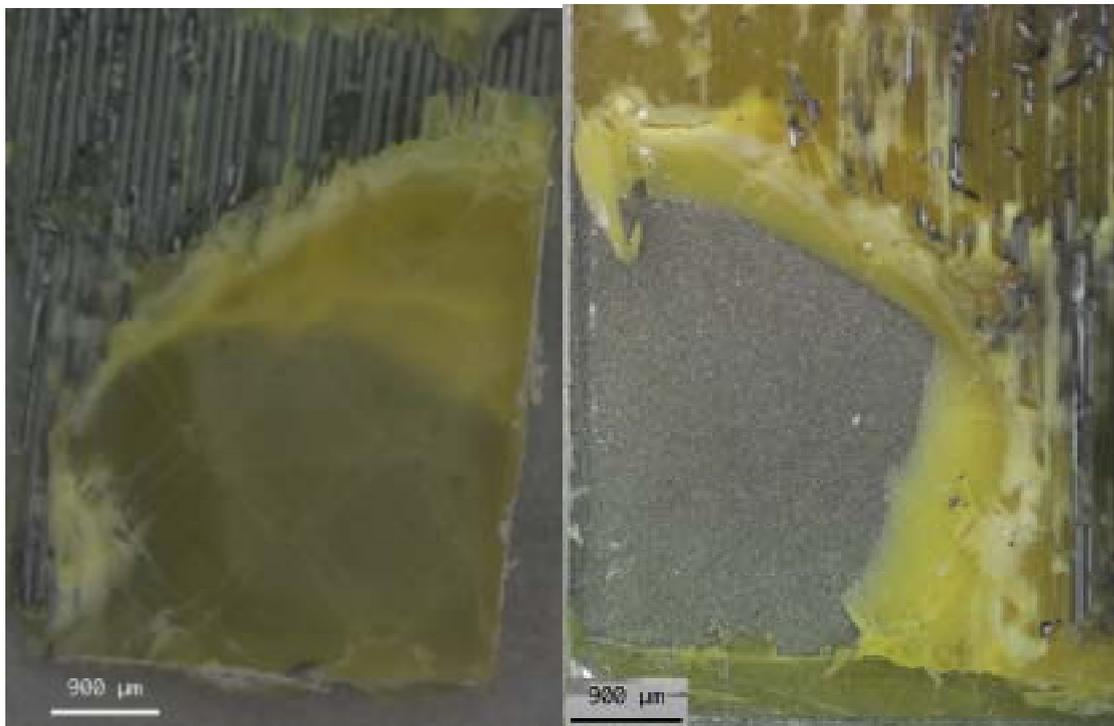


Figure 24 Failure surfaces of the fatigued boron-epoxy SDS immersed in the reference fuel for 283 days prior to testing.

## 4. Discussion

### 4.1 LCE samples exposed to fuel and fuel additives

The results presented in Figure 5 to Figure 9 indicate the deviation in average crack growth relative to the LCE samples exposed to the reference fuel (Figure 4). The LCE samples exposed to the fuel containing the DCI-4A, DGME and the JP8 fuel containing +100 all show no obvious deterioration relative to the reference fuel. In contrast, some deterioration in bond durability was observed for the LCE samples exposed to the DGME and water mixture and the reference fuel containing cumene hydroperoxide.

Deterioration of bond durability for the samples exposed to the DGME and water mixture (Figure 7) may be expected given water, typically, is responsible for most environmental degradation of epoxy-metal adhesive systems. The case where bonded repairs or structure may experience an environment of concentrated DGME and water are likely to be realised in the bottom of fuel tanks. Moisture contained in the fuel will accumulate in these regions due to immiscibility of the fuel and water and the greater density of the water. As DGME will have a far greater compatibility with water than fuel, it is also likely that the water at the bottom of fuel tanks would contain high concentrations of the icing inhibitor. The addition of DGME to water is likely to increase the corrosive nature of the environment the adhesive joint is exposed to and this may explain the degradation in joint strength observed. Based on the values provided in Table 2, there is approximately a 35% reduction in the fracture toughness from the reference condition after 1000 hours exposure. This result suggests that the DGME and water environment needs to be considered in design of bonded repairs and structure.

The degradation in bond strength caused by exposure to fuel containing cumene hydroperoxide is not as definitive as the DGME and water mixture exposure. One sample showed a 25% reduction in the fracture toughness after 1200 hours exposure, however, the second sample showed no reduction in strength relative to the reference sample. All samples indicated a variability in the  $G_{Ic}$  values after 1000 hours exposure, but the variability appeared to be greatest for the DGME/water and the fuel/cumene hydroperoxide mixtures. The increase in variability may be a sign that these environments are degrading the adhesive joint properties. The inconsistency in the results may represent a susceptibility in the bonded system that varies dependent on the quality of the materials and processes applied in joint fabrication.

Failure surface inspection of the LCE samples (Figure 10 to Figure 15) also revealed a correlation between the level of degradation in joint fracture toughness and the level of interfacial failure between the adhesive and metallic layers. The DGME/water exposed samples (Figure 13) showed high levels of adhesion failure in the samples which exhibited the highest levels of crack growth. The cumene hydroperoxide samples (Figure 15) showed areas where corrosion of the underlying aluminium has progressed to a greater extent on the sample with the greater crack growth. Interestingly, the reference fuel sample (Figure 10) that was exposed for 2000 hours showed a substantial region of apparent interfacial failure that had occurred during the second two thousand hours. Similarly, the DCI-4A and fuel exposed sample (Figure 11) also showed an increase in interfacial failure during the second two

thousand hours of exposure. These results may indicate that the reference fuel alone potentially can alter the interfacial chemistry of the epoxy-metal region.

In the absence of further failure analysis, which could more accurately establish the locus of fracture of these samples, it is not possible to establish whether the adhesive or adhesive to metal bonding is being affected. Potentially, the rubber toughened phase of the epoxy adhesive may be more susceptible to the fuel than the epoxy component and alteration in the rubber mechanical properties may lead to changes in the joint toughness. Clearly, further fracture studies would be needed to support this possibility. In the case of the cumene hydroperoxide samples, the decrease in fracture toughness may be due to the combined effect of the fuel affecting the adhesive and the peroxide reacting with the substrate in a corrosion mechanism (Figure 16). The variability in failures observed for the LCE samples may provide an indication of the complexity of the processes involved in the degradation of the epoxy-metal joints exposed to fuel that may be expected in service environments.

## 4.2 BDCB samples exposed to fuel and fuel additives

The trend in degradation of joint fracture toughness observed for the boron double cantilever beam (BDCB) samples (Table 4), was similar to the results for the LCE samples. The DGME/water exposed sample reduced by 45% relative to the reference sample and the cumene hydroperoxide sample reduced by 15%. The similar trend in  $G_{Ic}$  values for the BDCB and LCE samples may suggest that the reduction in fracture toughness observed for the DGME/water and cumene hydroperoxide exposed samples is due to a change in the FM73 properties, given the metallic surface is absent with these specimens.

The BDCB samples fracture analysis was complicated by mixed modes of fracture observed for all samples. Failure appeared to propagate either within the FM73 layers or within the 5521/4 layer adjacent to the FM73 layer. The brittle 5521/4 resin matrix is susceptible to fracture when cleavage stresses are induced during the wedge style test and it is difficult to correlate failure modes with fracture toughness values calculated in Table 4. The fracture toughness calculation is also dependent on the adhesive modulus and as fracture propagates into the resin layer this value clearly changes from that used for FM73 [15]. Visual examination of the failure surfaces (section 3.6) suggests that the DGME/water and cumene hydroperoxide/ reference fuel samples have the highest amount of failure within the resin layer. However, the fracture toughness of the BDCB specimens, in general, does not appear to be directly related to percentage of failure within the 5521/4 layer.

## 4.3 Boron composite-aluminium fatigue specimens

To date only boron composite-aluminium fatigue samples conditioned in fuel have been tested. The results, however, suggest that there is an increase in the fatigue resistance of the skin symmetrical doubler (SDS) specimens. In contrast with the LCE samples, the SDS specimens were exposed for more than 40 weeks. Given the LCE samples exposed to the reference fuel exhibited an increase in interfacial failure after only 12 weeks (Figure 10), further degradation in the fracture toughness of the SDS samples may have been expected. It isn't entirely clear why a reduction in fracture toughness of the adhesive bond would translate to an increase in fatigue resistance.

Previous work on SDS specimens manufactured using metallic patches[17] provided a similar failure mode to that observed for the boron SDS specimens (Figure 24). In the metallic patched SDS samples, theoretical modelling indicated that peel stresses played a significant role in influencing the fatigue resistance of the adhesive joints loaded in tension. Substantial improvements in fatigue resistance could be achieved by reducing the peel stresses of the SDS specimens by decreasing the taper angle of the patch. In the case of boron SDS specimens, an increase in the fracture toughness of the adhesive, caused by fuel uptake, may lead to a similar effect in helping to reduce peel stresses.

## 5. Conclusions

The results presented in this report rely in part on the use of an aggressive accelerated test method. Mode I loading of an adhesive joint is the worst possible case and typically repair design would minimise peel stresses. As a result, small changes in fracture toughness observed for exposure of adhesive joints exposed to fuel in these studies is likely to be representative of a field repair that would provide highly durable performance over a prolonged service life. Nevertheless, some proposals for the use of mode I style tests to qualify the environmental durability of adhesive bonds would also necessitate testing the performance of such specimens in probable aircraft operating environments such as the fuel and its additives studied in these experiments. On the basis of the limited testing conducted in this current work, it may be reasonable to conclude that bond quality may be sensitive to environments containing fuel and its additives. The average crack growth for the mode I metallic adherend samples exposed to fuel showed relatively small decreases in fracture toughness from the dry condition and it would be unlikely that the long term durability of the adhesive bond would be affected by fuel if a good quality surface treatment was employed.

Epoxy adhesive to metal bonded samples exposed to DGME and water and reference fuel containing cumene hydroperoxide showed a reduction in durability. The corrosive nature of the DGME and water environment may be responsible for the degradation in bonding observed and would simulate the conditions a repair in the bottom of a fuel tank may experience. These conditions are not improbable and the corrosive nature of the environment may need to be considered when designing repairs in certain locations. Cumene hydroperoxide simulates a fuel degradation product present in aging fuel and there is some indication bond degradation occurs through corrosion of the metallic substrate. The decrease in fracture toughness is not substantial and once again the long term durability of a practical repair would be unlikely to be significantly affected by this fuel by-product. Extended exposure in reference fuel, comprising dodecane and xylene, suggested that epoxy to metal bonds continue to degrade with time, either as a result of deterioration of adhesive bonds or the adhesive. This deterioration was only observed for a sample exposed for 2000 hours, which is a significantly longer period than the 48 hour test used by the RAAF in qualifying bonding technicians.

Boron double cantilever bonded specimens also showed degradation in bond durability when exposed to the DGME and water and reference fuel containing cumene hydroperoxide environments. The complex fracture modes observed for the composite samples, however, made definitive conclusions regarding degradation mechanisms difficult. A similar trend in

the durability of the adhesive to metal and composite-composite bonded samples exposed to the fuel environments may, however, suggest that processes affecting the adhesive properties are playing a role in the degradation mechanisms. The relative decrease in fracture toughness for these tests, however, suggests that the composite and adhesive materials are reasonably resistant to these type of environments.

The apparent increase in the fatigue resistance of boron-metal skin doubler specimens exposed to the reference fuel may indicate an increase in fracture toughness of the adhesive is helping to reduce peel stresses at the patch termination.

## 6. Recommendations

The results presented in this report provide an indication that surface treatment quality may be sensitive to environmental exposure to fuel and its additives. However, further experimental work is required to quantify the magnitude of these effects accurately. Mode I style tests are particularly aggressive and, whilst useful as a quality control tool for screening surface treatments, may not be ideal for simulating loads experienced by correctly designed adhesively bonded repairs. The following work should be considered as a part of a future research program:

- 1) Bulk adhesive samples should be conditioned for extended periods in the fuel and its additives and tested to determine if the mechanical properties are affected.
- 2) More detailed fracture analysis should be conducted on the adhesive samples exposed to the fuel and its additives to establish whether the resin or rubber phases are being affected.
- 3) Boron composite bonded samples need to be redesigned to more effectively interrogate the boron to adhesive interface exposed to the fuel and its additives and obviate fracture within the brittle resin layer.
- 4) Design of composite bonded repairs exposed to fuel should consider the location of the repair as well as the fuel additives.

## 7. Acknowledgements

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## Appendix A: A Qualitative Investigation into the Compatibility Effects of F-34 Fuel Additives with Cured FM73 Adhesive - R.C. Geddes

COMPONENT	USE	EFFECT ON FM73	COMMENT
<i>STADIS 450</i>	static dissipator	Contacted area softens to a rubbery state within 48 hours	
<i>DCI-4A FSII</i>	lubricity additive and icing inhibitor	Softens to rubbery state after 48 hours	
<i>1-7 Octadine</i>	alkene (fuel)	No noticeable effect. Evaporates within 48 hours	
<i>1-Methylnaphthalene</i>	diaromatic (fuel)	No noticeable effect. Evaporates within 48 hours.	
<i>HLCO TSR 350 (400)</i>	oil exposed to elevated temperatures	No noticeable effect. Left stain on surface of adhesive.	
<i>M-Xylene</i>	aromatic (fuel)	Causes massive disbonds (up to 65% of surface area). Softens adhesive.	
<i>DCI-4A</i>	lubricity additive	Causes massive disbonds (up to 50% of surface area). Softens adhesive.	Disbonds originated from edge applications of additive
<i>EGME (Ethylene Glycol Monomethyl Ether)</i>	icing inhibitor	No noticeable effect	
<i>DGME (Diethylene Glycol Monomethyl Ether)</i>	icing inhibitor	Causes massive disbonds (up to 75% of surface area). Softens adhesive.	
<i>HITEC E515</i>	corrosion inhibitor	No noticeable effect	
<i>Cumene Hydroperoxide, tech.</i>	peroxide, a degradation product	Very aggressive. Softens Adhesive within 48 hrs, and will cause disbonds.	All removed easily from glass at conclusion of test.

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19. ABSTRACT Adhesively bonded structure and bonded composite repairs are often exposed to aviation fuel during service. A relatively limited number of studies examining the influence of fuel and the many additives it contains have been made. The current report details experimental studies that have examined the influence of fuel and common additives such as lubricity and deicing agents on the fracture toughness of metal and composite bonded adhesive joints. The studies aimed to determine if a series of reference fuels containing each of the additives may affect adhesive bond durability. The present results suggest that the bond durability of adhesive joints exposed for several thousands hours to fuel environments may reduce. Concentrated mixtures of water and deicing agent, which were prepared in order to simulate the environment in the bottom of fuel tanks, may also reduce joint fracture toughness. The present studies highlight the complexity of examining the influence of fuel environments on adhesive joints and the need for further research to more quantifiably assess possible mechanisms of bond degradation in typical aircraft operating environments.					