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The Durability of Epoxy Adhesive Bonds formed with Titanium Alloy

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DSTO-TR-1333

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Andrew Rider

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Platforms Sciences Laboratory

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ABSTRACT

The durability of adhesive bonds formed between titanium alloy (Ti-6Al-4V) and rubber toughened epoxy structural adhesives used for repair bonding applications was examined. Two surface treatments were studied; the grit-blast and silane method, currently employed for titanium repairs on F-111 at RAAF Amberley and the F/A-18 SRM method which uses Pasa-Jell 107, a proprietry based toxic chemical treatment. Results from wedge style durability tests indicated the grit-blast and silane pre-treatment provided notably better performance than the Pasa-Jell 107 pre-treatment for epoxy bonds formed with Ti-6Al-4V alloy. Addition of BR127 primer to the pre-treated surfaces did not offer substantial improvement in durability performance. The durability of titanium-epoxy adhesive bonds for the grit-blast and silane pre-treatment on titanium was inferior to the performance observed on aluminium alloys. Further research will be required to improve the performance of the grit-blast and silane treatment on titanium to levels observed for aluminium.

RELEASE LIMITATION

Approved for public release

AQ F03-03-0444

Published by

DSTO Platforms Sciences Laboratory
506 Lorimer St
Fishermans Bend, Victoria 3207 Australia

Telephone: (03) 9626 7000
Fax: (03) 9626 7999

AR- 012-426
August 2002

APPROVED FOR PUBLIC RELEASE

The Durability of Epoxy Adhesive Bonds formed with Titanium Alloy

Executive Summary

The "Australian Silane Treatment" is the process employed by the Royal Australian Air Force for adhesively bonded repairs to metallic aircraft structure. The process was developed in response to a need for a simple, but effective process that could be applied to metallic adherends for field and depot level repairs that would ensure a reliable and durable adhesive bond. To date a large amount of information exists which has resulted from examinations of the influence of the surface treatment on the durability of aluminium to epoxy adhesive bonds. The "Australian Silane Treatment", which involves grit-blasting the metallic adherend prior to application of an epoxy-silane primer, has been shown to be very effective for bonding to aluminium alloys typically encountered on military and civilian aircraft. In contrast, experimental data examining the durability of adhesive bonds formed with grit-blast and silane pre-treated titanium alloys is very limited. This report details durability studies conducted on titanium joints bonded with structural epoxy adhesives. The titanium surfaces were pre-treated with the grit-blast and epoxy silane treatment or the F/A-18 structural repair manual (SRM) method, which employed a process of grit-blasting followed by application of a proprietary surface treatment chemical called Pasa-Jell 107®.

The purpose of the study was to:

1) expand the database on durability data for the grit-blast silane process on non-aluminium based alloys,

2) assess whether the process was superior to the current Pasa-Jell method used on F/A-18 and

3) establish the parameters for a durable surface pre-treatment method for the Smart Patch demonstrator repair of the F/A-18 aileron hinge.

Results from wedge style durability tests indicated the grit-blast and silane pre-treatment provided notably better performance than the Pasa-Jell 107 pre-treatment for epoxy bonds formed with Ti-6Al-4V alloy. Addition of BR127 primer to the pre-treated surfaces did not offer substantial improvement in durability performance. The durability of titanium-epoxy adhesive bonds for the grit-blast and silane pre-treatment on titanium was inferior to the performance observed on aluminium alloys. Further research will be required to improve the performance of the grit-blast and silane treatment of titanium to levels observed for aluminium. It is expected that implementation of the grit-blast silane procedure for titanium bonded repairs involving epoxy adhesives will improve existing repair procedures employed by RAAF, improve maintenance efficiency and assist in reduction in costs associated with through life support life support of Australian Defence Force aircraft.

Authors

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Andrew Rider joined Airframes and Engines Division at DSTO in 1988. He has a PhD in Physical Chemistry from the University of New South Wales and is currently employed as a Research Scientist with Air Vehicles Division. He is involved in research to support RAAF maintenance of bonded structure and application of repairs used on ADF aircraft.

Contents

1. Introduction

The "Australian Silane Treatment" [1] is the process employed by the Royal Australian Air Force for adhesively bonded repairs to metallic aircraft structure. The process was developed in response to a need for a simple, but effective process that could be applied to metallic adherends for field and depot level repairs that would insure a reliable and durable adhesive bond. To date a large amount of information exists which has examined the influence of the surface treatment on the durability of aluminium to epoxy adhesive bonds [2] [3]. The "Australian Silane Treatment", which involves grit-blasting the metallic adherend prior to application of an epoxy-silane primer, has been shown to be very effective for bonding to aluminium alloys typically encountered on military and civilian aircraft. In contrast, experimental data examining the durability of adhesive bonds formed with grit-blast and silane pre-treated titanium alloys is very limited. This report details durability studies conducted on titanium joints bonded with structural epoxy adhesives. The titanium surfaces were pre-treated with the grit-blast and epoxy silane treatment or the F/A-18 structural repair manual (SRM) method [4], which employed a process of grit-blasting followed by application of a proprietary surface treatment chemical called Pasa-Jell 107® [5]. The purpose of the study was to 1) expand the data base on durability data for the grit-blast silane process on non-aluminium based alloys, 2) assess whether the process was superior to the current Pasa-Jell method used on F/A-18 and 3) establish the parameters for a durable surface pre-treatment method for the Smart Patch demonstrator repair of the F/A-18 aileron hinge [6].

2. Experimental

The long term durability of metal-epoxy adhesive bonds is typically assessed with a range of accelerated testing methods that are designed to increase the rate of bond degradation of the adhesive joint. The Wedge test is a common test method employed to assess the quality of surface treatments and provide an estimate of long-term durability [7]. Variations of the test employ thicker and longer adherends to enable longer crack growth and, therefore, more accurate fracture energy calculation and avoid plastic deformation of the adherends. The RAAF have developed their own standard for bonded repairs to metallic structures [8] and rely on strict adherence to procedures and quality control as well as qualification of technicians involved in bonding operations. Qualification of technicians also requires that a wedge specimen can be produced to certain standards prescribed in the C5033 Engineering Standard. Therefore, the assessment of the surface pre-treatment for the titanium to epoxy bonding studies using the wedge test also serves the purpose of developing guidelines for qualification of bonding technicians for bonding operations using titanium alloys.

3. Surface Pre-treatments

A detailed description of the surface pre-treatment steps employed for the titanium bonding studies is described below.

3.1.1 Grit-blast + Silane (GB+Sil)

The following steps were used in the grit-blasting and silane treatment of the titanium adherend surfaces:

a. solvent degrease with methyl ethyl ketone (MEK)

b. Scotchbrite® abrade with MEK

c. debris removal using lint free tissues wet with MEK

d. Scotchbrite® abrade with distilled water

e. debris removal using lint free tissues wet with distilled water

f. water break test

g. oven dry 15 mins at 110^0C

h. grit-blast at 60psi using 50 μm alumina, grade 240 high grade alumina

i. dip 15 mins in 1% aqueous solution of epoxy-silane, pre-hydrolysed 60 minutes

j. oven dry 1 hour at 110^0C

3.1.2 Scotchbrite® + Silane (SB+Sil)

The following steps were used in the Scotchbrite® and silane abrasion of the titanium adherend surfaces:

a. solvent degrease with methyl ethyl ketone (MEK)

b. Scotchbrite® abrade with MEK

c. debris removal using lint free tissues wet with MEK

d. Scotchbrite® abrade with distilled water

e. debris removal using lint free tissues wet with distilled water

f. water break test

g. dip 15 mins in 1% aqueous solution of epoxy-silane, pre-hydrolysed 60 minutes

h. oven dry 1 hour at 110^0C

3.1.3 Pasa-Jell® 107 (PJ)

The following steps were used in the Pasa-Jell® treatment of the titanium adherend surfaces:

a. solvent degrease (MEK)

b. grit-blast at 60psi using 1250μm alumina, grade 120 high grade alumina

c Pasa-Jell 107® applied with brush to titanium for 15 mins

e. excess removed with kimwipe tissues soaked with tap water and surface acidity checked with litmus paper

f. rinse surface with distilled water and water break test

g. oven dried 30 mins at 60^0C

3.1.4 Grit-blast + Silane + Primer (GB + Sil + BR127®)

The following steps were used in the grit-blasting, epoxy silane and priming of the titanium adherend surfaces:

a. solvent degrease with methyl ethyl ketone (MEK)

b. Scotchbrite® abrade with MEK

c. debris removal using lint free tissues wet with MEK

d. Scotchbrite® abrade with distilled water

e. debris removal using lint free tissues wet with distilled water

f. water break test

g. oven dry 15 mins at 110^0C

h. grit-blast at 60psi using 50μm alumina, grade 240 high grade alumina

i. dip 15 mins in 1% aqueous solution of epoxy-silane, pre-hydrolysed 60 minutes

j. oven dry 1 hour at 110^0C

h. spray apply BR-127® chromate primer to give yellow translucent film

i. air dry for 30 minutes at 25^0C, followed by oven dry at 120^0C, 30 minutes

3.1.5 Pasa-Jell® 107 + Primer (PJ + BR127®)

The following steps were used in the Pasa-Jell® treatment of the titanium surfaces:

a. solvent degrease (MEK)

b. grit-blast at 60psi using 1250μm alumina, grade 120 high grade alumina

c Pasa-Jell® 107 applied with brush to titanium for 15 mins

e. excess removed with Kimwipe tissues soaked with tap water and surface acidity checked with litmus paper

f. rinse surface with distilled water and water break test

g. oven dried 30 mins at 60^0C

h. spray apply BR-127® chromate primer to give yellow translucent film

i. air dry for 30 minutes at 25°C, followed by oven dry at 120°C, 30 minutes

The abbreviations used throughout the report to designate a particular pre-treatment are detailed in Table 1.

Descriptor	Adherend Processing Steps
GB+Sil	Grit-blast at 60psi + Silane (1% Z-6040® epoxy) dip 15 minutes
SB+Sil	Scotchbrite® abrade with distilled water + Silane (1% Z-6040 epoxy) dip 15 minutes
PJ	Grit-blast and Pasa-Jell® 107 applied by brush to adherend for 15 minutes
GB+Sil+BR127	Grit-blast at 60psi + Silane (1% Z-6040® epoxy) dip 15 minutes + BR127® primer
PJ+BR127	Grit-blast and Pasa-Jell® 107 applied by brush to adherend for 15 minutes + BR127® primer

Table 1 *The descriptor and details of the surface pre-treatment procedures applied to the adherends prior to bonding with paste adhesives*

3.2 Adhesives, Primers and Coupling Agent

3.2.1 FM73®

FM73® (0.085 psf) with polyester knit carrier was bonded at 40psi for 60 minutes at 120^0C using a ramp rate of 3^0C/min in a platen press using the carrier film to control the bondline thickness, which was typically 100μm. FM73® is manufactured by Cytec-Fiberite.

3.2.2 FM300®

FM300® adhesive from Cytec-Fiberite with a tight-knit tricot carrier was bonded at 40psi for 60 minutes at 175°C using a ramp rate of 3^0C/min in a platen press using the carrier film to control the bondline thickness, which was typically 100μm.

3.2.3 FM300-2K®

FM300-2K® adhesive from Cytec-Fiberite with a tight-knit tricot carrier was bonded at 40 psi for 60 minutes at 120°C using a ramp rate of 3°C/min in a platen press using the carrier film to control the bondline thickness, which was typically 100 μm.

3.2.4 BR127®

BR127® corrosion inhibiting primer from Cytec-Fiberite was applied by spray coating to produce a translucent straw coloured surface. The film was cured at room temperature for 30 minutes followed by curing at 120°C for 30 minutes prior to adhesive bonding.

3.2.5 Epoxy silane

3-glycidoxypropyltrimethoxy silane (Dow Corning Z-6040®) from Sigma-Aldrich was applied to the titanium adherend as a 1% aqueous solution using distilled water that had previously been stirred for at least 1 hour. The silane film was cured at 110°C for 60 minutes.

3.3 Titanium

Table 2 indicates the composition of the Titanium alloy used in the Wedge durability tests. The Ti-6Al-4V plate was 3mm thick and produced to the AMS 4911 specification.

<table>
<tr><th colspan="6">Composition By Weight (%)</th></tr>
<tr><th>Ti</th><th>Al</th><th>V</th><th>Fe</th><th colspan="2">O</th></tr>
<tr><td>90</td><td>6</td><td>4</td><td><0.25</td><td colspan="2"><0.2</td></tr>
<tr><th rowspan="2">Mechanical Properties</th><th>σ_y (ksi)</th><th>E (10^3 ksi)</th><th>G (10^3 ksi)</th><th>μ</th><th>Hardness (Vickers)</th></tr>
<tr><td>126</td><td>16.0</td><td>6.2</td><td>0.31</td><td>396</td></tr>
</table>

Table 2 Composition and mechanical properties of the titanium alloy used for preparation of bonded joints.

3.4 Durability Testing

Wedge style double cantilever beam specimens were prepared based on ASTM D3762-79 [7]. This involved bonding individual pairs of titanium fingers of 25.4mm x 150mm x 3.0mm dimensions that had been pre-treated using the grit-blast and silane or Pasa-Jell® 107 treatment. Each test reported is the average result from 5 test samples. A stainless wedge of 3.1mm thickness was inserted into the wedge and left in the laboratory environment for 24 hours prior to insertion in a 50°C/100% R.H. environment. Crack length measurements were performed just prior to insertion in the humid environment and at approximately 1, 2, 4, 24, 48, 100, 500 and 1000 hours using an optical microscope. The testing temperature of 50°C and 100% R.H. was chosen as these are the conditions prescribed in the RAAF Engineering Standard [8].

The fracture toughness values, G_I (J/m^2), measured from the wedge test data, were calculated using equation 1 [9], where Y is the crack opening displacement, h is the adherend thickness, E is Young's modulus and a is crack length.

$$G_I = \frac{Y^2.E.h^3\left[3\left(a+0.6h\right)^2+h^2\right)\right]}{16\left[\left(a+0.6.h\right)^3+a.h^2\right]^2} \qquad (1)$$

The calculation is valid given plastic bending of the titanium will not occur if the adherend thickness is greater than 1.3mm, according to equation (2), where G_I for undegraded FM73 adhesive is 3000J/m^2 for a 120^{0}C cured layer of 100μm thickness [10] and σ_y is the yield stress of the titanium cantilevers (Table 2).

$$h_{crit} > \frac{3.G_I.E}{\sigma_y^{\ 2}} \qquad (2)$$

3.5 Surface Roughness Measurements

The surface roughness of Ti-6V-4Al was assessed to determine the depth of indentations resulting from the grit-blasting. These parameters are of particular concern when dealing with fatigue critical components made from titanium alloys.

The roughness measurements employed an automated Laser Depth Measuring Device called a Perthometer. The instrument scans a laser beam across the surface. Measurement requires that the laser maintains focus as it traverses the surface. A rapid feed-back adjustment alters the height of the laser to maintain focus. The height adjustment is recorded and plotted as a function of the beam's horizontal translation, providing a surface profile of the roughened surface. The Perthometer provides an *average roughness*, R_A, which corresponds to the mean peak to valley height over the entire profile as well as the *maximum roughness*, R_{MAX}, which corresponds to the greatest peak to valley depth over the entire profile. Profiles were also analysed statistically to determine the average height of surface features, after correction for background slope, as well as the average angle of the surface features, with respect to the surface normal.

4. Results

4.1 Titanium Bonding to FM73

Figure 1 indicates the crack growth for titanium specimens bonded to FM73 that were pre-treated with the grit-blast+silane (GB+SIL), Scotchbrite® + silane (SB+SIL), Pasa-Jell 107(GB+PJ), grit-blast+silane+BR127 (GB+SIL+BR127) or Pasa-Jell 107 + BR127 (GB+PJ+BR127). The grit-blast +silane (GB+SIL) and grit-blast + silane + BR127 (GB+SIL+BR127) pre-treatments indicate similar performance and significantly improved durability relative to the Pasa-Jell and Scotchbrite® treatments.

Pasa-Jell treatments are quite poor and indicate significant crack growth in the initial stages of exposure. Use of the BR127 with the Pasa-Jell treatment has little effect on the final crack length. The relative performance of the grit-blast and silane and Scotchbrite® and silane treatments indicates that grit-blasting is an important part of the process with the Scotchbrite® abrasion clearly being less effective in preparing a durable surface treatment. The addition of BR127 to the grit-blast and silane treated titanium appears to have little effect on overall durability performance, which contrasts with the BR127 performance measured for Al-2024 T3 aluminium alloys [11]. This result suggests that corrosion inhibition afforded by the BR127 treatment may not be critical in improving durability between titanium and epoxy adhesives.

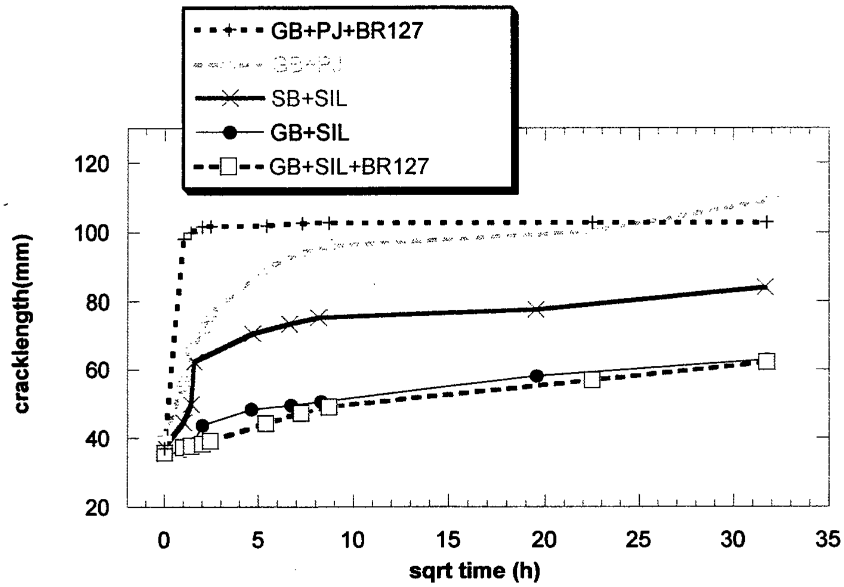


Figure 1 Crack length as a function of exposure time to a 50°C/100%R.H. environment for Ti-6Al-4V alloy bonded to FM73 adhesive. Surface pre-treatments of the titanium include: grit-blast+silane (GB+SIL), Scotchbrite®+silane(SB+SIL), Pasa-Jell 107(GB+PJ), grit-blast+silane+BR127(GB+SIL+BR127) and Pasa-Jell 107+BR127 (GB+PJ+BR127)

Figure 2 indicates the fracture toughness values calculated for the wedge test data presented in Figure 1. Table 3 indicates the G_I values for the different surface treatments calculated using equation (1) for the 0 and 1000 hour exposure times. The spread in the G_I value calculated was determined from the standard deviation in the crack length values measured for the wedge tests in Figure 1. Crack length values for the wedge tests are provided in the Appendix, Tables A1 to A5. The results suggest that the grit-blast and silane treatments provide the best dry strength and durability performance. The Pasa-Jell treatments provide substantially poorer performance both in terms of initial dry strength and durability.

The results may also be compared with bonding of FM73 adhesive to Al-2024 T3 unclad aluminium alloy [11] which were established from bolt loaded thick adherend aluminium specimens or long crack extension (LCE) specimens in Table 4. Results from the aluminium study indicated that dry strength for the FM73 to aluminium provided values of 2100 and 2800 J/m^2 for both the grit-blast + silane + BR127 and the grit-blast + silane pre-treatments, respectively. The aluminium values are similar to the titanium for the grit-blast and silane treatment but lower for the grit-blast + silane + BR127. The fracture toughness of the adhesive bonds after 1000 hours of humid exposure indicated significant differences for the silane and the silane + BR127 treatments on the aluminium. The grit-blast + silane treated aluminium gave a value of 693J/m^2 after 1000 hours humid exposure [11], which is 400J/m^2 higher than the grit-blast + silane titanium samples in Table 3. The grit-blast + silane + BR127 treated aluminium gave a value of 1029J/m^2 after 1000 hours humid exposure [11], which is 700J/m^2 greater than the grit-blast + silane + BR127 titanium samples in Table 3. This result suggests the BR127 primer mechanism for improving bond durability does not appear to be as effective for titanium based alloys and that the grit-blast + silane treatment is not as effective for titanium as aluminium alloys.

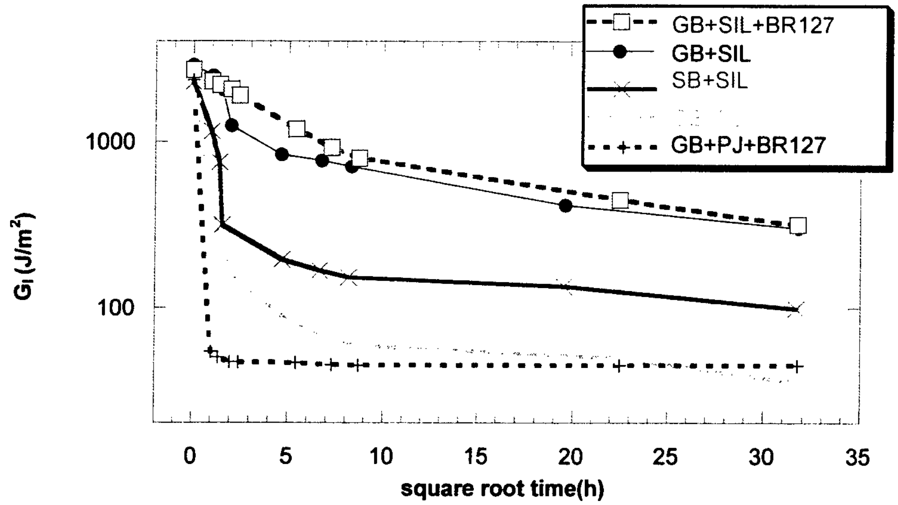


Figure 2 Elastic energy release rate (G_I(J/m^2)) as a function of exposure time to a 50^{0}C/100%R.H. environment for Ti-6Al-4V alloy bonded to FM73 adhesive. Surface pre-treatments of the titanium include: grit-blast+silane (GB+SIL), Scotchbrite®+silane(SB+SIL), Pasa-Jell 107(GB+PJ),grit-blast+silane+BR127(GB+SIL+BR127) and Pasa-Jell 107+BR127 (GB+PJ+BR127)

Treatment	G_I(J/m^2) 0 hours	S.D. (J/m^2)	G_I(J/m^2) 1000 hours	S.D. (J/m^2)
grit-blast+silane+BR127	2687	600	318	40
grit-blast+silane	2877	550	304	36
Scotchbrite®+silane	2279	470	99	9
Pasa-Jell 107 + BR127	2345	490	45	3
Pasa-Jell 107	1752	340	35	3

Table 3 G_I (J/m^2) values for FM73 bonded titanium joints pre-treated with grit-blast+silane (GB+SIL), Pasa-Jell 107 (GB+PJ), grit-blast + silane + BR127 (GB+SIL+BR127), Pasa-Jell 107 + BR127(GB+PJ+BR127) and Scotchbrite® + silane (SB+Sil) treatments. G_I values at 0 and 1000 hours exposure to the 50^{0}C/100%R.H. environment are shown, together with the Standard Deviation (S.D.) associated in the fracture energy calculation based on the standard deviation in crack growth measurement for the wedge test data.

Treatment	G_I(J/m^2) 0 hours	S.D. (J/m^2)	G_I(J/m^2) 1000 hours	S.D. (J/m^2)
grit-blast+silane+BR127	2100	200	1029	160
grit-blast+silane	2842	470	693	72

Table 4 G_I (J/m^2) values for FM73 bonded Al-2024T3 joints pre-treated with grit-blast+silane (GB+SIL) and grit-blast + silane + BR127 (GB+SIL+BR127) treatments[11]. G_I values at 0 and 1000 hours exposure to the 50^{0}C/100%R.H. environment are shown, together with the Standard Deviation (S.D.) associated in the fracture energy calculation based on the standard deviation in crack growth measurement for the Long Crack Extension test data.

4.2 Titanium Bonding to FM300 and FM300-2K

Figure 3 indicates the crack length as a function of humid exposure time for titanium bonded to FM300 and FM300-2K for the grit-blast + silane and the Pasa-Jell treatments. The relative performance of the FM300 and FM300-2K for the grit-blast + silane treatments suggests that both adhesive systems behave in a similar manner, which is expected given mechanical and physical properties of both adhesive systems are very similar. As was observed for the FM73 results in Figure 1, addition of BR127 chromate primer to the grit-blast + silane treatment has little influence on the durability performance of the titanium to FM300-2K adhesive bond. This provides further evidence that the addition of the primer to the titanium does not significantly influence the degradation mechanism of titanium-epoxy adhesive bonds. The poor durability performance of the Pasa-Jell and FM300-2K treatment is similar to that observed for the FM73 studies in Figure 1 and indicates that the Pasa-Jell treatment is far less effective than the grit-blast and silane process.

Figure 4 provides the calculated fracture toughness values for the wedge test data shown in Figure 3. In comparison with the FM73 fracture toughness values shown in Figure 2, the FM300 and FM300-2K adhesive shows lower initial dry fracture toughness, which is expected, given the more brittle nature of the adhesive. Fracture energy at extended exposure is also lower than in the FM73 case in Figure 2. Table 5 shows the elastic energy release rates for the wedge samples in Figure 3 and shows that values are approximately 100J/m^2 lower than the FM73 samples in Table 3 for the grit-blast + silane samples, but about the same for the inferior Pasa-Jell treated sample.

The results may also be compared with wedge test data for bonding between aluminium and FM300 [12]. For Al-2024T3 unclad aluminium pre-treated using the grit-blast + silane method, G_I at 0 hours exposure was 1270 ± 100J/m^2 and this decreased to 870 ± 70J/m^2 after 1000 hours exposure to 50^{o}C/100%R.H. Similar values were observed for the grit-blast + silane + BR127 aluminium pre-treated samples bonded to FM300. The titanium results in Table 5 show that the fracture toughness is approximately 600J/m^2 lower than the aluminium wedge test at 1000 hours exposure. This comparison suggests that the grit-blast + silane treatment is also notably less effective on titanium than aluminium for FM300 adhesive, although the pre-treatment is significantly better than the Pasa-Jell treatment being used for F/A-18 bonded titanium repairs.

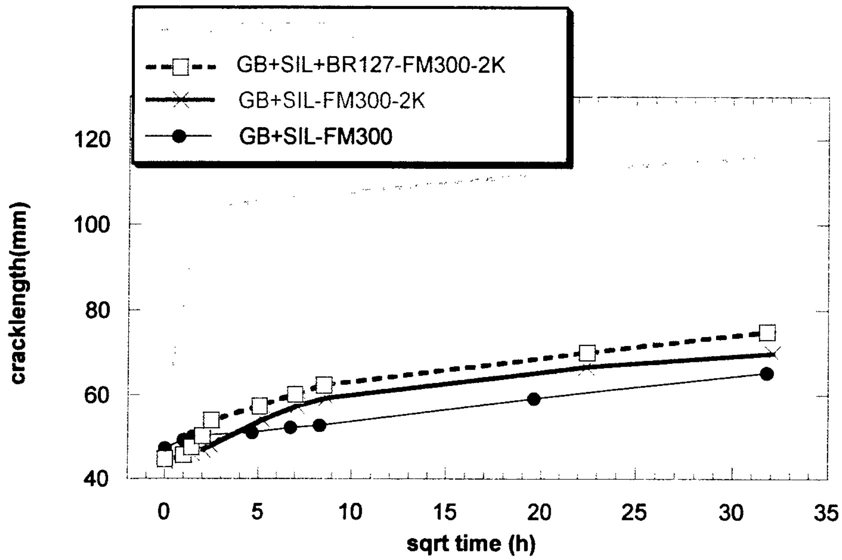


Figure 3 *Crack length as a function of exposure time to a 50°C/100%R.H. environment for Ti-6Al-4V alloy bonded to FM300-2 and FM300 adhesives. Surface pre-treatments of the titanium include: grit-blast+silane (GB+SIL), Pasa-Jell 107 (GB+PJ) and grit-blast + silane + BR127 (GB+SIL+BR127).*

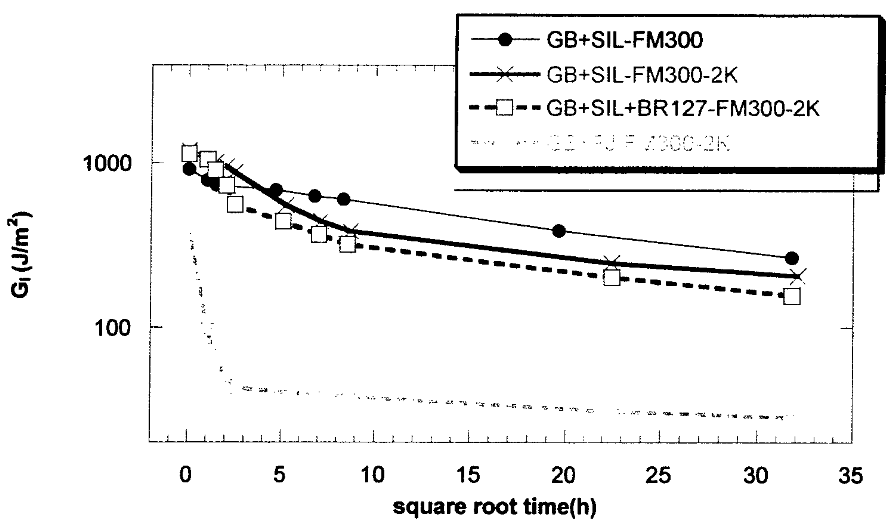


Figure 4 *Elastic energy release rate (G_I(J/m²)) as a function of exposure time to a 50°C/100%R.H. environment for Ti-6Al-4V alloy bonded to FM300-2 and FM300 adhesives. Surface pre-treatments of the titanium include: grit-blast+silane (GB+SIL), Pasa-Jell 107 (GB+PJ) and grit-blast + silane + BR127 (GB+SIL+BR127).*

Treatment	G_I(J/m^2) 0 hours	S.D. (J/m^2)	G_I(J/m^2) 1000 hours	S.D. (J/m^2)
grit-blast + silane (FM300)	922	151	266	32
grit-blast + silane (FM300-2K)	1190	209	205	23
grit-blast + silane + BR127 (FM300-2K)	1141	198	155	16
Pasa-Jell 107 (FM300-2K)	409	55	28	2

Table 5 G_I (J/m^2) values for FM300 and FM300-2K bonded titanium joints pre-treated with grit-blast+silane (GB+SIL), grit-blast + silane + BR127 (GB+SIL+BR127) and Pasa-Jell 107 (GB+PJ) treatments. G_I values at 0 and 1000 hours exposure to the 50°C/100%R.H. environment are shown, together with the Standard Deviation (S.D.) associated in the fracture energy calculation based on the standard deviation in crack growth measurement for the wedge test data.

Treatment	G_I(J/m^2) 0 hours	S.D. (J/m^2)	G_I(J/m^2) 1000 hours	S.D. (J/m^2)
grit-blast+silane+BR127	1180	90	830	80
grit-blast+silane	1270	100	870	70

Table 6 G_I (J/m^2) values for FM300 bonded Al-2024 T3 joints pre-treated with grit-blast+silane (GB+SIL), grit-blast + silane + BR127 (GB+SIL+BR127) treatments [12]. G_I values at 0 and 1000 hours exposure to the 50°C/100%R.H. environment are shown, together with the Standard Deviation (S.D.) associated in the fracture energy calculation based on the standard deviation in crack growth measurement for the wedge test data.

4.3 Surface Roughness Measurements

Surface roughness measurements conducted on titanium and aluminium alloy provides some indication of the relative hardness of the two surfaces. The hard titanium surface does not show significant differences in surface roughness parameters between the 1µm polished and Scotchbrite® abraded surface. The aluminium surface indicates significant increase in surface roughness after Scotchbrite® abrasion. A comparison of the grit-blasted aluminium and titanium surfaces indicates that there is also a notable increase in surface roughness on the aluminium sample. The trend of surface roughness results suggests that some contribution to the relative durability performance of the titanium and aluminium wedge samples may be associated with reduced roughness on the titanium surface. Clearly, differences in surface chemistry between the two metallic surfaces may also be contributing to the relative effectiveness of the silane treatment. Both the grit-blasting and silane application steps applied to titanium may need to be modified to improve the performance of the surface pre-treatment. This will involve conducting further research.

Titanium 6Al-4V Treatment	Average Roughness (μm)/S.D.	Maximum Roughness (μm)/S.D.	Average Height (μm)/S.D.	Average Angle (Degrees)
Scotchbrite® Abrade	0.56/0.04	6.7/0.4	0.62/0.7	22/19
1μm Polish	0.37/0.02	2.8/0.04	0.77/0.5	3/5
Grit-blast	1.3/0.1	13.7/2.2	2.5/1.8	43/25

Table 7 Roughness measurements performed on Ti-6Al4V alloy that was Scotchbrite® abraded or grit-blasted. A 1μm polished surface is also provided for reference.

Al-2024T3 Clad Treatment	Average Roughness (μm)/S.D.	Maximum Roughness (μm)/S.D.	Average Height (μm)/S.D.	Average Angle (Degrees)
Scotchbrite® Abrade	1.1/0.2	17.6/2.6	2.0/1.6	21/18
Grit-blast	1.6/0.1	18.5/5.2	3.7/2.5	43/25

Table 8 Roughness measurements performed on Al-2024T3 clad aluminium alloy that was Scotchbrite® abraded or grit-blasted.

4.4 Fracture Surface Examination

4.4.1 FM73 Bonded to Titanium

Figure 5 indicates the failure surfaces for the silane treated titanium surfaces bonded with FM73. In all cases the failure appears to be at the interface between the epoxy adhesive and titanium surface, although surface analysis would be required to confirm the exact location of fracture. Despite the final crack length measured for the silane and BR127 treated surfaces being similar there appears to be less adhesion failure on the BR127 treated sample. Typically, wedge tests indicate a region ahead of the crack-tip that has degraded due to moisture diffusion, but that has not failed during the test. This may have also occurred in this instance and may suggest that the BR127 treatment does provide some assistance in slightly reducing moisture diffusion rates to the interface for titanium-bonded samples. The Pasa-Jell treated titanium failure surfaces in Figure 6, with much larger adhesion failure areas also show the same trend, with the BR127 surface indicating slightly reduced total failure area.

The failure surfaces for the FM300 and FM300-2K silane treated titanium samples are provided in Figure 7. All surfaces indicate 100% adhesion failure, as with the FM73 samples. The performance of the FM300-2K does not appear to be as good as the FM300 and may suggest some subtle differences in the two adhesive systems, such as cure temperature, may be responsible for the different performance. In contrast with the FM73 case, the addition of BR127 to the silane treated titanium surface does not appear to benefit the durability performance of the titanium-FM300-2K bond. Different performance for the different adhesive systems indicates that the variables affecting titanium bonding need to be more fully characterised. The failure surface of the Pasa-Jell treated titanium bonded to FM300-2K in Figure 8 indicates that moisture diffusion ahead of the crack has led to almost 100% adhesion failure, highlighting the inadequacy of the current F/A-18 treatment being employed for bonding operations involving titanium.

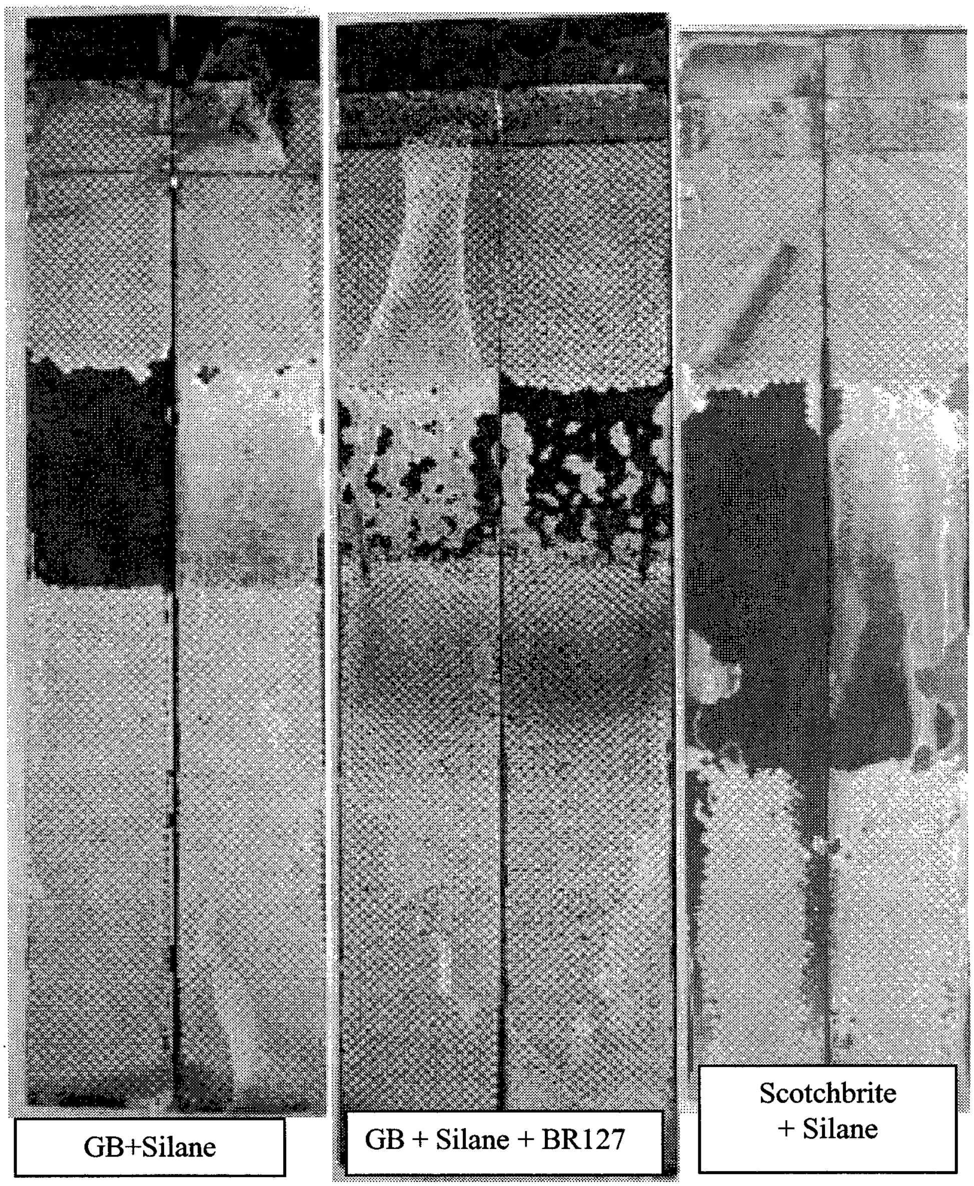
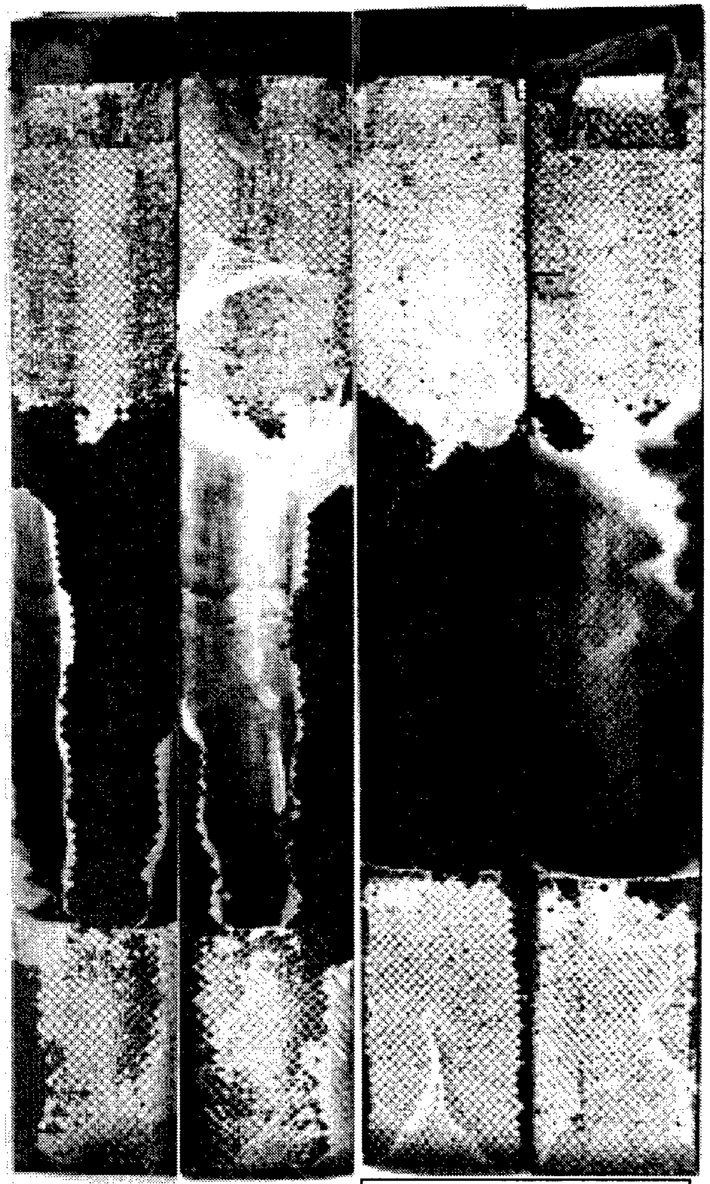


Figure 5 Fracture surfaces of failed wedge tests indicating the relative areas of adhesion failure for the FM73 bonded titanium samples pre-treated with grit-blasting or Scotchbrite® abrasion and silane.

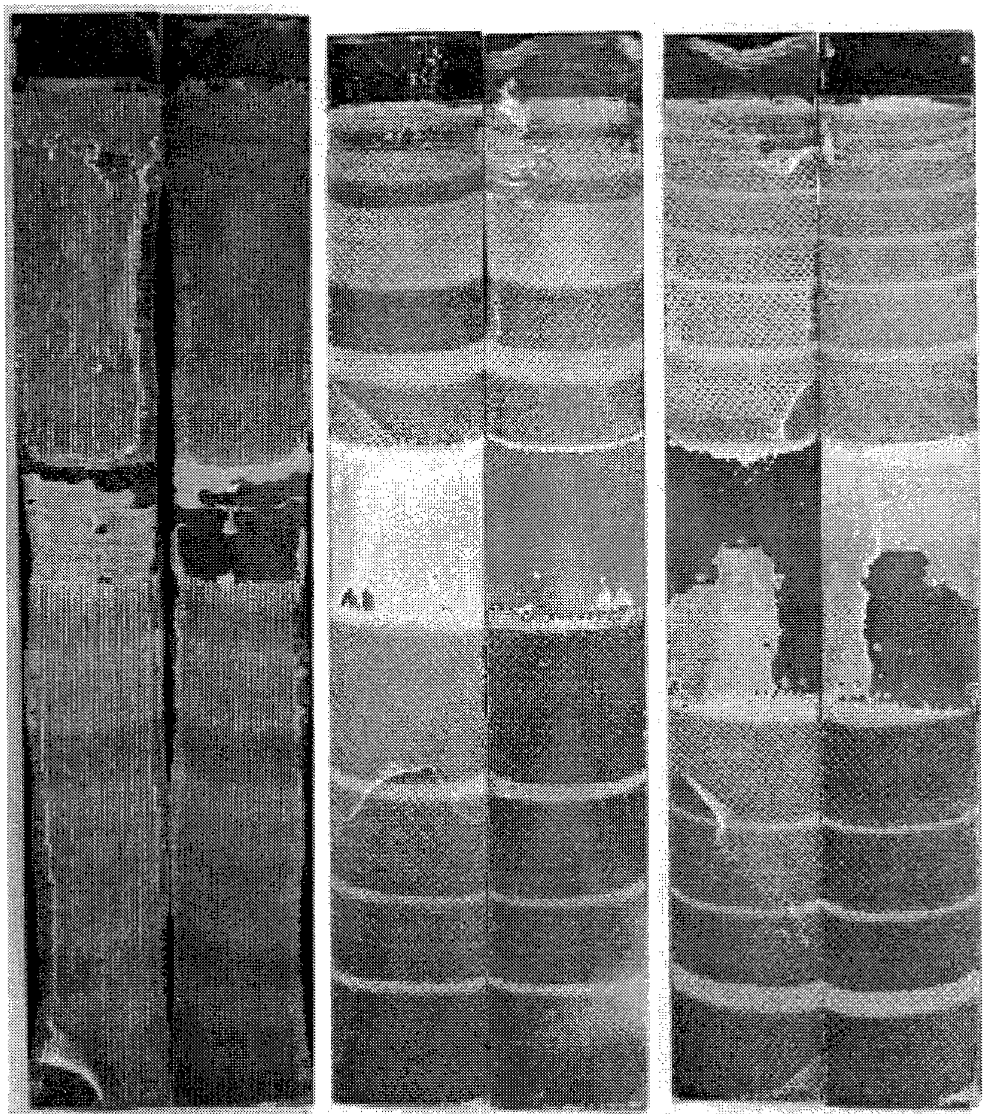


Grit-blast + Pasa-Jell

Grit-blast + Pasa-Jell + BR127

Figure 6 Fracture surfaces of failed wedge tests indicating the relative areas of adhesion failure for the FM73 bonded titanium samples pre-treated with grit-blasting and Pasa-Jell 107.

4.4.2 FM300 and FM300-2K bonded to Titanium



Grit-blast + Silane, FM300

Grit-blast + Silane, FM300-2K

Grit-blast + Silane + BR127, FM300-2K

Figure 7 Fracture surfaces of failed wedge tests indicating the relative areas of adhesion failure for the FM300 and FM300-2K bonded titanium samples pre-treated with grit-blasting and silane.

Grit-blast +
Pasa-Jell 107,
FM300-2K

Figure 8 Fracture surface of failed wedge test indicating the relative areas of adhesion failure for the FM300-2K bonded titanium samples pre-treated with grit-blasting and Pasa-Jell 107.

5. Conclusions

Studies examining two surface pre-treatments for titanium bonding to structural epoxy adhesives have indicated the following:

1) Application of the current RAAF standard for pre-treating metals, involving grit-blasting followed by application of an epoxy silane coupling agent, can improve the durability performance of adhesive bonds formed between titanium and structural epoxy adhesives typically used in aircraft bonding repair operations.

2) The grit-blast and silane surface pre-treatment produces adhesive bonds between titanium and structural epoxy adhesive bonds that are notably more durable than the Pasa-Jell treatment currently used for F/A-18 titanium bonded repairs.

3) Addition of the chromate primer BR127 to the grit-blasted and silane treated surface appears to have minimal influence on the durability of the titanium to epoxy adhesive bond.

4) Addition of the chromate primer BR127 to the grit-blasted and Pasa-Jell treated surface appears to have minimal influence on the durability of the titanium to epoxy adhesive bond.

5) The durability of bonds formed between grit-blast and silane pre-treated titanium and structural epoxy adhesives is not as good as the durability of bonds formed on grit-blast and silane treated aluminium substrates.

6) The greater hardness of titanium, in comparison with aluminium, results in reduced roughness on the titanium surface after grit-blasting and abrasion procedures.

7) Differences in the physical and chemical nature of titanium and aluminium are responsible for the different durability performance of the grit-blast and silane surface treatment for the respective metals.

8) Further research is required to modify the grit-blast and silane pre-treatment applied to titanium alloys to improve the adhesive bond durability performance relative to aluminium. Increased roughness on the titanium surface may improve the durability performance of the silane treatment.

6. Recommendations

Based on the adhesive bond durability studies detailed in this technical report the following recommendations can be made:

1) The grit-blast and silane treatment as specified in RAAF Engineering Standard C5033 [8] should be employed in preference to the current F/A-18 Pasa-Jell procedure [4] for adhesive bonding operations involving epoxy adhesives and titanium alloys.

2) Use of BR127 primer in titanium bonding operations using epoxy adhesives would not provide any substantial benefit to bond durability performance and would not, generally, be recommended for use.

3) Further research should be undertaken to improve the performance of the grit-blast and silane treatment for titanium adhesive bonding and alternative non-toxic pre-treatment procedures should also be examined.

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Appendix A: Crack Length Data from Wedge Test Specimens

A.1. Titanium Bonded to FM73

Time(h)	Sqrt time (h)	Crack length (mm)					
		Specimen 1	Specimen 2	Specimen 3	Specimen 4	Specimen 5	Average
0	0	36.05	36.45	35.75	35.7	34.55	35.7
1	1	37.2	37.9	37.45	37.2	36.35	37.22
2	1.41421	37.25	38.15	38.2	37.65	37.2	37.69
4	2	39	38.65	38.7	37.8	37.45	38.32
6	2.44949	40.75	39.15	39.4	38.05	38.5	39.17
29	5.38516	46.6	45.05	44.15	42.75	42.75	44.26
52.2	7.22496	50.05	48.25	46.6	45.95	45.5	47.27
75.5	8.68907	51.8	49.65	49.45	46.7	47.8	49.08
506.35	22.50222	58.05	56.5	58.5	56.3	55.1	56.89
1008.05	31.7498	62	67.55	62.2	60.05	59.2	62.2

Table A1 Grit-blast + silane + BR127, FM73

Time(h)	Sqrt time(h)	Crack length (mm)					
		Specimen 1	Specimen 2	Specimen 3	Specimen 4	Specimen 5	Average
0	0	36	34.9	34.55	33.75	36.1	35.06
1.1	1.04881	36.65	37.05	35.45	34.95	38.15	36.45
2.45	1.56525	39.85	37.45	37.85	38.05	38.55	38.35
4.05	2.01246	44.05	45.25	42	44.85	42.15	43.66
21.3	4.61519	52.25	48.4	47.9	47.4	46.15	48.42
45	6.7082	52.25	49.55	49.55	49.4	46.75	49.5
68.4	8.27043	52.5	50.1	50.95	50.55	48.55	50.53
382.3	19.55249	59.35	58.65	57.8	57.3	57.45	58.11
1008.05	31.7498	66.35	60.75	65.45	60.3	62.05	62.98

Table A2 Grit-blast + silane, FM73

Time(h)	Sqrt time(h)	Crack length (mm)					
		Specimen 1	Specimen 2	Specimen 3	Specimen 4	Specimen 5	Average
0	0	33.5	39.7	39.35	35.35	38.5	37.28
1	1	46.7	48.1	46.1	38.75	43.4	44.61
2	1.41421	50.85	54.85	49.35	44.95	49.05	49.81
2.5	1.58114	60.95	59.3	64.2	66.2	61.4	62.41
22	4.69042	65.55	72.85	70.15	74.1	70.65	70.66
44.1	6.64078	68.55	74	71.45	75.95	77	73.39
66.5	8.15475	72.55	74.5	75.15	77.6	77	75.36
380.5	19.50641	81.4	75.95	75.75	77.8	77.25	77.63
1002.25	31.65833	91.1	85.85	79.7	82	81.65	84.06

Table A3 *Scotchbrite + silane, FM73*

Time(h)	Sqrt time (h)	Crack length (mm)					
		Specimen 1	Specimen 2	Specimen 3	Specimen 4	Specimen 5	Average
0	0	37.2	40	41	40.45	41.1	39.95
1	1	51.75	55.15	61.4	63.4	47.85	55.91
2	1.41421	61.2	71.2	64.05	73.45	58.85	65.75
4	2	66.6	78.45	67.6	83.65	62.2	71.7
6	2.44949	70.7	82.2	70.95	91.15	67.55	76.51
26.4	5.13809	86.75	91.35	84.8	101.3	79.1	88.66
50.1	7.07814	93.3	91.75	96.1	102.2	86.65	94
75.35	8.68044	94.15	92.05	100.3	103.5	90.2	96.04
501.25	22.38861	95.2	93.65	104.4	109.2	98.55	100.2
1028.25	32.06634	118.6	99.8	111.75	115.45	101.4	109.4

Table A4 *Grit-blast + Pasa-Jell 107, FM73*

Time(h)	Sqrt time (h)	Crack length (mm)					
		Specimen 1	Specimen 2	Specimen 3	Specimen 4	Specimen 5	Average
0	0	36.4	37.95	38.45	36.1	36.1	37
1	1	91.95	107	94.6	97.65	99.35	98.11
2	1.41421	92.3	110.7	94.75	98.85	103.1	99.94
4	2	94.3	113.05	95.05	102.45	103.95	101.76
6	2.44949	94.3	113.05	95.05	102.6	103.95	101.79
29.3	5.41295	94.7	113.25	95.05	102.6	104.2	101.96
53.2	7.29383	95.35	113.8	97.05	102.95	104.2	102.67
75.3	8.67756	95.85	113.8	97.05	102.95	104.35	102.8
506.4	22.50333	95.85	113.8	97.05	102.95	104.35	102.8
1008.1	31.75059	96.25	113.8	97.05	102.95	104.35	102.88

Table A5 *Grit-blast + Pasa-Jell 107 + BR127, FM73*

A.2. Titanium Bonded to FM300 and FM300-2K

Time(h)	Sqrt time(h)	Crack length (mm)					
		Specimen 1	Specimen 2	Specimen 3	Specimen 4	Specimen 5	Average
0	0	46.65	49	46.75	47.45	46.4	47.25
1	1	49.5	51	48.75	48.4	48.65	49.26
2.15	1.46629	49.5	51.75	48.75	51.7	48.65	50.07
3.15	1.77482	49.5	51.75	49.3	51.7	49.4	50.33
21.5	4.63681	49.5	53.3	51	51.7	49.4	50.98
45.3	6.73053	51.5	54.3	51.1	52.8	51.3	52.2
69	8.30662	51.95	54.7	51.3	53.7	52.05	52.74
383.35	19.57933	58.5	61.9	57.3	60.45	57.6	59.15
1007.3	31.73799	63.6	70.15	63	66.7	62.4	65.17

Table A6 *Grit-blast + Silane, FM300*

Time(h)	Sqrt time(h)	Crack length (mm)					
		Specimen 1	Specimen 2	Specimen 3	Specimen 4	Specimen 5	Average
0	0	42.6	45.45	46.8	44.9	41.25	44.2
1	1	44.5	46.1	48.35	46	42.9	45.57
2	1.41421	46.3	46.5	48.35	46.55	43.3	46.2
4	2	46.3	46.5	48.65	48.9	43.8	46.83
6	2.44949	47.1	48.8	49.5	50.05	43.8	47.85
27.1	5.20577	54.35	56.3	55.45	52.9	50.8	53.96
50.4	7.0993	58.9	59.6	58.85	55.45	53.5	57.26
75.4	8.68332	60.2	61.2	60.95	58.85	55.3	59.3
503.35	22.43546	63.9	70.65	67.9	67.35	63.15	66.59

Table A7 Grit-blast + Silane, FM300-2K

Time(h)	Sqrt time (h)	Crack length (mm)					
		Specimen 1	Specimen 2	Specimen 3	Specimen 4	Specimen 5	Average
0	0	44.15	45.8	42.4	43.9	47.2	44.69
1	1	46.2	46.1	43.35	44.5	47.95	45.62
2	1.41421	51.1	46.75	46.55	45.2	47.95	47.51
4	2	54.15	46.75	50.9	51	48.25	50.21
6.1	2.46982	56.25	51.2	55.6	55	51.2	53.85
25.4	5.03984	57.1	55.1	57.9	58.35	57.8	57.25
49	7	59.75	57.5	59.85	61.3	61.95	60.07
73	8.544	61.85	59.1	62.2	63.1	65	62.25
504.4	22.45885	76.05	61.3	68.5	66.65	77.6	70.02
1009.55	31.77342	82.1	65.95	76.6	67.45	82	74.82

Table A8 Grit-blast + Silane + BR127, FM-300-2K

Time(h)	Sqrt time (h)	Crack length (mm)					
		Specimen 1	Specimen 2	Specimen 3	Specimen 4	Specimen 5	Average
0	0	55.7	59.35	57.45	59.8	59.25	58.31
1	1	73.3	85	85.6	86.85	85.65	83.28
2	1.41421	85.75	89.05	104.8	89.95	97.05	93.32
4	2	100.65	91.9	111.95	90.9	99.9	99.06
6	2.44949	106.1	104.15	114.2	91.95	102.7	103.82
30	5.47723	109.35	106.95	117.45	92.85	103.1	105.94
53.3	7.30068	110.3	107.7	117.45	92.85	103.95	106.45
77	8.77496	110.65	109.15	117.85	93.25	105.25	107.23
505.5	22.48333	111.05	109.15	117.85	120.8	106.5	113.07
1006.5	31.72538	112.7	112.1	120.05	125.1	108.2	115.63

Table A9 Grit-blast + Pasa-Jell 107, FM300-2K

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The Durability of Epoxy Adhesive Bonds formed with Titanium Alloy	Document (U) Title (U) Abstract (U)
4. AUTHOR(S)	5. CORPORATE AUTHOR
Andrew Rider	Aeronautical and Maritime Research Laboratory 506 Lorimer St Fishermans Bend Victoria 3207 Australia

6a. DSTO NUMBER	6b. AR NUMBER	6c. TYPE OF REPORT	7. DOCUMENT DATE
DSTO-TR-1333	AR- 012-426	Technical Report	August 2002

8. FILE NUMBER	9. TASK NUMBER	10. TASK SPONSOR	11. NO. OF PAGES	12. NO. OF REFERENCES
2002/27364	AIR 01/123	DGTA	25	12

13. URL on the World Wide	14. RELEASE AUTHORITY
http://www.dsto.defence.gov.au/corporate/reports/DSTO-TR-1333.pdf	Chief, Air Vehicles Division

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16. DELIBERATE ANNOUNCEMENT

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18. DEFTEST DESCRIPTORS

Bonded structure, adhesive bonding, surface pretreatment, titanium, repair

19. ABSTRACT

The durability of adhesive bonds formed between titanium alloy (Ti-6Al-4V) and rubber toughened epoxy structural adhesives used for repair bonding applications was examined. Two surface treatments were studied; the grit-blast and silane method, currently employed for titanium repairs on F-111 at RAAF Amberley and the F/A-18 SRM method which uses Pasa-Jell 107, a proprietry based toxic chemical treatment. Results from wedge style durability tests indicated the grit-blast and silane pre-treatment provided notably better performance than the Pasa-Jell 107 pre-treatment for epoxy bonds formed with Ti-6Al-4V alloy. Addition of BR127 primer to the pre-treated surfaces did not offer substantial improvement in durability performance. The durability of titanium-epoxy adhesive bonds for the grit-blast and silane pre-treatment on titanium was inferior to the performance observed on aluminium alloys. Further research will be required to improve the performance of the grit-blast and silane treatment on titanium to levels observed for aluminium.

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