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Residual Stress Measurements and Boeing Wedge Durability Data for the Proposed 470 Bulkhead Bonded Repair

Christina Olsson-Jacques

DSTO-TN-0178



DEPARTMENT OF DEFENCE

DEFENCE SCIENCE AND TECHNOLOGY ORGANISATION

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Maritime Platforms Division Aeronautical and Maritime Research Laboratory

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ABSTRACT

Fatigue cracking problems have occurred in the F/A-18 470.5 bulkhead during initial full scale testing. The surface of this bulkhead is shot-peened to introduce compressive residual stress to increase the fatigue life of the component as part of the manufacturing and maintenance program. The Aeronautical and Maritime Research Laboratory (AMRL) is investigating the effect of applying a composite patch to reduce the critical strains in the crotch area. Boeing wedge durability tests were used to define the most suitable metal preparation procedure to apply a durable patch to a shotpeened aluminium alloy surface. The x-ray diffraction technique was used to assess any reduction in the beneficial shot-peened residual stress after typical abrasion and heat treatment stages in the preparation procedure. It was found that the abrasion and heat treatment processes used to achieve the most durable surface treatment for bonding did not significantly reduce the beneficial compressive surface stresses induced by the shot-peening process.

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Executive Summary

Fatigue cracking problems have occurred in the F/A-18 470.5 bulkhead during initial full scale testing. The surface of this bulkhead is shot-peened to introduce compressive residual stress to increase the fatigue life of the component as part of the manufacturing and maintenance program. The Aeronautical and Maritime Research Laboratory (AMRL) is investigating the effect of applying a composite patch to reduce the critical strains in the crotch area. Thus the issue of the effect of the surface needed to be examined. In addition, the quality control procedures used as part of the peening process involve organic dyes that may leave sufficient residual contaminant on the prepared surface to have an effect on the durability of the adhesive bond. Typical surface treatment procedures include abrasion and heat treatment stages. Residual stress measurements using the x-ray diffraction technique were undertaken to analyse any effects that the surface treatment stages may have on the beneficial compressive stresses left by shot-peening.

The nature and quality of the surface pre-treatment of aluminium alloys is critical to the long term environmental durability of the bonded joint¹. The results of the Boeing wedge durability test (ASTM D3762) for six different surface treatments on shotpeened aluminium alloy plates indicated that the most durable treatment included a MEK solvent clean, mild scotchbrite abrasion, alumina grit-blast, silane coupling agent and BR127 chromate primer. The adhesive used was FM73 which was cured at 120°C for 1 hour. This process satisfied the durability criteria stipulated in the RAAF Engineering Standard C5033². There was no evidence that residual contaminant from the organic dye had an effect on bond durability.

Residual stress measurements indicated that the shot-peening process induced compressive stresses into aluminium alloy surfaces of -250 to -300 MPa. The depth that the x-ray diffraction technique analysed was 75 μ m from the surface. The maximum error involved with these measurements is in the order of 50 MPa. The surface abrasion and heat treatment steps used in the bonding process gave a small reduction of 60 MPa in the surface stress. Therefore the abrasion and heat treatment processes used to achieve the most durable surface treatment for bonding does not significantly reduce the beneficial compressive surface stresses induced by the shot-peening process.

Arnott D.R., Rider A.R. Olsson-Jacques C.L., Lambrianidis L.T., Wilson A.R., Pearce P.J., Chester R.J., Baker A.A., Morris C.E.M., David M.J., Swan G., (1998) "Bond Durability Performance-The Australian Silane Surface Treatment" Proc. 21 Congress ICAS, Melb. p13-18.

^{2 &}quot;An Engineering Standard for Composite Materials and Adhesive Bonded Repairs"-Number C5033, Royal Australian Air Force HQLC Melbourne, (1996)

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1. INTRODUCTION

Composite reinforcement of components on Military Aircraft is a process developed by a team lead by Dr. Alan Baker at Aeronautical and Maritime Research Laboratory (AMRL), Defence Science and Technology Organisation. Composite reinforcements are bonded to reduce the stress intensity in regions susceptible to cracking in order to extend the fatigue life of the component.

The crotch area of the F/A-18 470.7 bulkhead has shown a tendency to cracking during full scale testing. The base surface is Aluminium (7050) which has been shot-peened to improve the fatigue life of the structure. The crack patch process includes a pre-bond surface treatment followed by bonding of a composite reinforcement with an epoxy film adhesive. This assembly is then heated to the adhesive cure temperature while under pressure to complete the bonding process.

FM73 is a relatively low temperature 120°C cure adhesive, which has been used, in a wide range of aircraft repairs and reinforcements. However the durability of bonds formed with FM73 is relatively sensitive to the pre-bond surface treatment and some effort was directed toward defining a suitable treatment. Previous research work on pre-bond surface treatments at AMRL has focused on non-peened Aluminium 2024 T3 Clad and FM73 and FM300 adhesives. A satisfactory surface treatment has been developed at AMRL to satisfy the Royal Australian Air Force (RAAF) Engineering Standard C5033² criteria with the Al2024 adherends and FM73 and FM300 adhesive systems. The procedure includes a solvent wipe, scotchbrite abrade, grit-blast and the application of an epoxy silane coupling agent. To define the optimum surface treatment application to the shot-peened bulkhead several surface treatments were trialled on shotpeened 7075 aluminium. To assess the bond durability for each trial surface treatment the Boeing wedge standard (ASTM D3762-79) is used.

The bonding process includes abrasion steps and curing at temperatures up to 120°C. In the case of the F/A-18 bulkhead there was concern that the drying and cure cycles of the pre-bond surface treatment may decrease the beneficial compressive stresses that are induced by shot-peening. The effect of these steps on the beneficial compressive stresses induced by shot-peening was assessed by measuring the relative change in residual stress in the surface of shot-peened 7050 specimens. To analyse the surface residual stresses the X-ray diffraction (XRD) Technique was used. A review of the technique is given in Appendix A.

1

2. METHOD

2.1 Trial Surface Treatments-Durability Assessment:

To identify a suitable surface treatment that is viable for the bulkhead alloy and FM73 adhesive system to be used on the bulkhead, a series of trial surface treatments was carried out on shot-peened Al7075 specimens. Shot-peening was carried out on the Boeing wedge test plates and the fatigue end specimens according to RAAF specifications. Blue ink was used as a quality tool. It was necessary to peen both sides of the Boeing wedge test plates as the shot-peening on one face induced bending of the specimens.

The surface treatments trialled were AMRL silane treatment¹, PASAGEL 105 etching and BR127 primer. The details of these treatments are tabulated in Table 2 in the results section. Further details of the methods and materials used in surface treatments can be found in Arnott et al¹. The durability was assessed using the standard ASTM D3762-79 Boeing wedge test. The RAAF Engineering Standard C5033² sets a criterion for acceptable durability in which the crack length for the Boeing wedge tests should be less than 5 mm in 24 hours and 7 mm in 48 hours.

2.2 Pre-Bond Processes-Residual Stress Assessment:

2.2.1 Specimens

Specimens for x-ray diffraction residual stress measurements were cut from the end of the specimens previously used in fatigue tests (see figure 1). These specimens were chosen for their suitability for x-ray residual stress measurements. The specimens were 6 mm thick to prevent any bending due to shot-peening and abrasion processes.



Figure 1: Specimens cut from Al7050 fatigue specimens for use in the residual stress tests.

These fatigue end specimens were designated from their original numbers KDIE15, KDIE17, KDIE22 and KDIE23 with suffixes a) and b) used to distinguish between the two ends of 15 and 23. Only one end was taken from numbers 17 and 22. Shot-peened and non-shotpeened specimens were subjected to the pre-bond abrasion and heat

treatment processes (see Table 1). X-ray diffraction was used to measure and compare the surface residual stresses.

2.2.2 Abrasion and Grit-blasting

Mechanical polishing, grinding, milling, electropolishing and abrading of a metal surface can induce residual stresses in the surface of the material. [Noyan and Cohen, 1987]³. This study is concerned with the effect of abrasive techniques used in pre-bond surface treatments on shot-peened aluminium surfaces. There are two abrasive processes involved in the pre-bond surface treatment. Scotchbrite pad with distilled water or Methyl Ethyl Ketone (MEK) solvent and grit-blasting the surface with alumina or zircon grit. Table 1 summarises the surface pre-treatment for abrasion and grit-blasting for various specimens.

	OVER A OF THE ATMENT					
SAMPLE	SURFACE I KEATMENT					
KD1E 15 (a)	Unpeened					
KDIE 15 (b)	Peened					
	<u>Mild</u> Scotchbrite; Removal = too small to accurately measure <u>Medium</u> Scotchbrite; Removal of ~12.3µm surface layer <u>Heavy</u> Scotchbrite; Removal of ~extra 25.6= total of 37.9µm Removed most of visible surface roughness. Machining marks visible.					
KDIE 17 (a)	Peened <u>FB surface treatment</u> : MEK/mild SB dry 110°C 15 mins/alumina grit. <u>Heat Treatment</u> : 120°C 1 hour, cool, 120°C for 2 hours.					
KDIE 22 (a)	Unpeened <u>FB surface treatment</u> : MEK/mild SB dry 110°C 15 mins/alumina grit- blast.					
KDIE 23 (a)	Unpeened <u>MB surface treatment</u> MEK degrease, zircon grit-blast					
KDIE 23 (b)	Peened <u>MB surface treatment:</u> MEK degrease, zircon grit-blast					

Table 1: Surface Treatment of the 7050 specimens to be analysed for residual stress.

The scotchbrite pad was used to abrade shot-peened specimens using a water or MEK solvent. In the pre-bond surface treatment a light scotchbrite abrade was used to reduce the amount of peened surface removed. The compressive stresses are expected to extend into the alloy in the order of hundreds of microns and removal of material to

this depth would remove the beneficial compressive stress produced by shot-peening. To determine if the partial removal of the peened layer had an impact on the stress in the fresh surface three grades of scotchbrite abrasion were used: A <u>mild</u> or light scotchbrite abrade, a <u>medium</u> scotchbrite and a <u>heavy</u> scotchbrite. The abrasion was carried out in the direction of the grain of the material. The mild scotchbrite involved using a light touch and abrading the length of the material approx. 20 times. The medium scotchbrite involved a fairly heavy scrub of the surface until approx. 10 μ m was removed. The heavy scotchbrite involved a very heavy pressure for about fifteen minutes that removed approximately 25 μ m. The residual stress was measured after each stage using x-ray diffraction.

Grit-blast was carried out using an automated grit-blaster manufactured at AMRL Fishermens Bend (FB). The grit used is alumina AP106. This grit is needle shaped with a maximum grit size of $50 \,\mu\text{m}$. The air pressure was $450 \,\text{kPa}$ and the grit impact density was 1 gm/cm² on the plate. This grit-blast was used for all the pre-bond surface treatments used in this report. Measurements of the thickness of the specimen before and after grit-blasting indicated that there was minimal reduction in the thickness (ie. less than $10 \,\mu\text{m}$).

Alternative grit-blast procedures are used by the adhesive bonding staff at AMRL Maribyrnong (MB). This system uses zircon grit that is spherically shaped and larger in diameter. The procedure is carried out by hand and the quality of the peening is determined visually. Previous experiments on the Boeing wedge aluminium plates have shown that this procedure induces bending of 1mm thick specimens when performed on one side of the specimen. The automated alumina grit-blasting at Fishermen's Bend in comparison does not induce bending. This indicates that the zircon grit blast induces compressive residual stress. To determine the effect of this zircon grit-blasting both the shot-peened and non-peened Al7050 specimens were treated and the residual stress measurements performed by x-ray diffraction. There was no measurable removal of material from the surface of the specimen. Measurements of the thickness of the specimen before and after grit-blasting indicated that there was minimal reduction in the thickness (ie. less than 10 μ m).

2.2.3 Heat Treatment

The pre-bond surface treatments and the cure of the adhesive both involve heating the shot-peened surfaces to 120°C for an accumulated period of approximately three hours. A paper by Bouseau et al., 1984⁴ indicates that the stresses in the surface of aluminium alloy 7075 can decrease with soaking at temperatures of 150°C. To determine if the surface compressive residual stress induced by shot-peening is effected by this heat treatment a specimen previously subjected to a mild scotchbrite and automated alumina grit-blast was heated to 120°C for 1 hour, cooled and then heated to 120°C for 2 hours. The cool down was to more closely simulate the heat treatment cycling found in the surface treatment and bond curing procedures. Table 1 summarises the heat treatment cycle for specimen KDIE17 (a).

2.2.4 X-ray Diffraction

The residual stress measurements in this report were conducted by staff (Rod Mackie) in the Physics Department, Monash University. The depth of penetration depends on the wavelength of the x-ray source and the density of the material. We used a copper x-ray source that gives an approximate analysis depth of 75 μ m in aluminium metal. The irradiated area on the surface was 4 mm in diameter. The $sin^2\psi$ technique measures the change in lattice spacing for various tilts ψ of the specimen with respect to the x-ray source and detector (See Appendix A). To optimise the shift in the lattice planes a peak at high 20 is required. The crystal lattice plane used was the 422 aluminium peak at approximately 137 degrees 20. The specimens used for this report were not subjected to any applied load during the x-ray diffraction measurements. Therefore the measurement of stress gives the average of the residual macrostresses present in the surface of the specimen irradiated by the x-ray beam. It is essential that the sample surface be accurately placed on the goniometer axis as this can also lead to a divergence from the linear relationship similar to shear stresses. A special tool is used by staff at Monash University to ensure that the specimen surface is correctly aligned to the goniometer axis.

The Monash x-ray diffraction equipment uses a position sensitive detector that measures the complete diffraction peak. In contrast to traditional x-ray diffraction laboratory equipment where the detector measures only one point per step of the diffraction profile, this position sensitive detector decreases exposure time significantly. The x-ray source and sample are located on a goniometer to obtain the θ and ψ angles required. In contrast to a conventional x-ray diffractometer, the position sensitive detector is not directly attached to the goniometer axis and remains in a constant position while the sample is tilted with respect to the x-ray beam. Consequently the exact 2 θ position cannot be measured accurately. This is not of particular concern for our purpose as it is the change in the 2 θ that is required for residual stress analysis. The detector has been calibrated and each channel is equivalent to 0.01 degrees. The shift can be measured and related to the change in strain by the following equation (see appendix A).

$$\sigma_{\phi} = \frac{E \cdot \cot\theta}{2(1+\nu)\sin^2\psi} \Delta 2\theta \tag{1}$$

The value for $\cot\theta$ is kept a constant and is taken from the value of θ when $\psi = 0$. This assumption causes little error and is accurate for small $\Delta 2\theta$. For our results this is typically less than 0.5% of the final calculated stress value.

In this report the Poisson's Ratio used was 0.29 and Young's Modulus 69 MPa for the aluminium specimens. The residual stress was measured in the longitudinal direction (sample position was vertical with respect to x-ray beam) and transverse direction (sample position was horizontal with respect to x-ray beam). This was required to show whether or not the stress was homogeneous in two directions of

the surface plane. In this report a negative stress result is called compressive stress and a positive stress result is called tensile stress. Tilts in both the negative and positive tilt directions are required as shear stresses and strong gradients with depth will depart from the linear relationship between the strain and $\sin^2\psi$. [Noyan and Cohen 1987]³ (Appendix A).

3. RESULTS

3.1 Bond Durability:

Several surface treatments were analysed using the Boeing wedge durability test. These details of these treatments together with the crack length results for 0 hr, 25 hr and 50 hr are listed in Table 2. The results from the Boeing wedge durability tests for the treatments listed in Table 2 are given in Figure 2. Standard deviations measured over the five Boeing wedge specimens for each test are approx. 1 mm.



Figure 2: Boeing wedge durability tests trial surface treatments for the Aluminium 7075 and FM73 system. The JB reference refers to surface treatments given in Table 2.

3.1.1 Shot-peening Dyeing for Quality Assurance.

The surface treatment procedures and Boeing wedge durability results for 25 hour and 50 hours are given in Table 2. Quality control for shot peening included pre-painting the surface with a blue ink. The surface is then shot peened until the ink layer has been

nearly removed. However the remaining ink that has been left on the surface or even pounded into the surface may reduce the ability of the adhesive to attach to the surface. This concern was addressed by using different procedures in the Solvent Clean and Abrasion steps (see Table 2). Where distilled water was used it is designated dH2O. It was observed visually that MEK with Kimwipes performed better than water wash and water/Kimwipes in removing the blue ink. The surface treatment in JB14 with the warm water wipe and the nylon brush silane application did not perform well when compared to the C5033 criteria (viz.: 5 mm of crack tension after 24 hours and 7 mm after 48 hours) (Table 2). Where MEK and Kimwipes were used in the solvent clean steps and the epoxy silane was applied from immersion there was no appreciable difference in the performance with shot-peened and non-shot-peened plates (Table 2: JB22, JB23, JB44).

3.1.2 Pasagel 105 Surface Treatment:

PASAGEL 105 surface treatment has been used by the RAAF in bonded repairs on aluminium non-peened surfaces as part of repair manual procedures. From the results in Table 2 and Figure 2 it can be seen that the PASAGEL 105 treatment was the least durable of our tests. It was not considered for the proposed treatment.

3.1.3 Silane coupling agent and BR127 chromate primer:

The standard AMRL pre-bond surface treatment which consists of a solvent clean, scotchbrite abrade, grit-blast and epoxy silane, although better than PASAGEL 105, also did not satisfy the C5033 criteria for bond durability. The addition of BR127 chromate primer to these treatments improved the durabilities for this system to satisfy the RAAF Engineering standard C5033 durability criteria. The two surface treatments JB20 and JB47 that include the BR127 steps form the basis of our proposed surface treatment. It should be noted that although the JB47 procedure which included the MEK solvent degrease produced better bond durability performance than the JB20 with the warm water it also had a higher initial crack length.

DIATTECT	TP1/	IB20	IB22	IB23	IB44	JB46	JB47
BW IESI	JD14	J D 20		,		Ĩ	
INO.	A17075	A17075	A17075	A17075	A17075	A17075	A17075
Adherend Shot moor	VES	YES	none	One only	none	YES	YES
Snot-peen	Warm water	Warm water	MEK and	MEK	MEK	MEK	MEK
Solvent	Kimwine	Kimwipe	Kimwipe	Kimwipe	Kimwipe	Kimwipe	Kimwipe
Clean	(d H2O)	(dH2O)	L. L.	· ·			
Abrade	Mild	Mild	Scotchbrite	Scotchbrite	Scotchbrite	Scotchbrite	Mild
	Scotchbrite	Scotchbrite	with dH2O	with dH2O	with dH2O	in MEK and	Scotcnbrite
	with dH2O	with dH2O		l		then in	with arizo
			17.	1/images	Kimuinos	Kimwines	Kimwipes
Debris	Kimwipes	Kimwipes	Kimwipes	with duo	with dH20	with dH20	with dH2O
Removal	with dH2O	with dH2O	with dH20		11000 :	11000 :	110°C in over
Dry	110°C in oven	110ºC in	110°C in	110°C in	n 110°C m	over for 15	for 15 mine
	for 15 mins	oven for 15	oven for 15	oven for 15	mine	mins	101 10 111110
		mins	mins		Auto	Auto	Auto alumina
Grit-blast	Auto alumina	Auto	Auto	alumina	alumina	alumina	grit
	grit	aiumina grit	orit	orit	grit	grit	0
	1% og eilere	1%ag silana	1%20	1%ag	1%aq	PASAGEL	1%aq silane
Coupling	nvlon bruch	nylon brush	silane.	silane,	silane,	<u>105</u> and	nylon brush
Agent	15mins	15mins	immersion	immersion	immersion	wash in	15mins
			15 minutes	15 minutes	15 minutes	dH2O until	1
				L	11000	neutral	11000
Dry	110°C in oven	110ºC in	110ºC in	110°C in	110°C in	110°C m	for 1br
	for 1hr	oven for 1hr	oven for	oven for	oven for	1 1hr	
	<u> </u>	BD107	Inr		none	none	BR127
Primer	none	BK12/	none	none	none		chromate
—	L	air 20min	none	none	none	none	air 30min
Primer	none	oven 1200C	lione	1		1	oven,120ºC
cure		30min					30min
Adhasiwa	FM73	FM73	FM73	FM73	FM73	FM73	FM73
Curo	120°C for 1hr	120ºC for 1hr	120°C for	120ºC for	120°C for 1	120°C for	120ºC for 1hr
Cure	platten press	platten press	1hr platten	1hr platten	hr platten	1hr platten	platten press
	r		press	press	press	press	
BW	29.2	31	30.0	31.1	31.1	29.8	33.6
RESULT							
(0hr)mm							1
BW Crack	20.4	5.6	16.4	11.4	11.6	22.3	5.1*
Ext. (25hr)							
mm							
BW Crack	23.6	7.2	19.7	13.2	13.8	24.5	6.2*
Ext. (50hr)					1		1
mm	1					<u> </u>	<u> </u>

Table 2: Trial Surface Treatment Procedures for A17075 and FM73 adhesive system.

*Satisfies the C5033 Standard, 5 mm after 24 hours, and 7 mm in 48 hours.

3.2 Residual Stress:

The x-ray diffraction technique essentially measures average strain through changes in the lattice spacing resulting from the applied stress as described in Appendix A. X-ray diffraction profiles were taken for nine tilts ψ of the sample surface with respect to the x-ray beam. Figure 3 shows a plot of these profiles for a shot-peened 7050 surface. To view these profiles the intensities are separated by 200 counts on the ψ axis for each tilt profile. The order of the profiles is from the bottom is ψ is 40, 34, 27, 19, 0 -19, -27, -34, -40 degrees. The shift in 2 θ (equated to channel no) is shown by the arrows which indicate the approximate peak position for $\psi = 40$ (bottom profile) and $\psi = 0$ (centre profile). The shapes of these peak profiles are very broad and the x-ray k α_1 and k α_2 are not resolved. Peaks are typically broadened by the small crystallite size or by microstrains causing a relatively large distribution in the measured lattice spacings.



Figure 3) Shot-peened Aluminium KDIE 23(b) x-ray diffraction profiles for ψ tilts. The arrows indicate the shift in 2θ from $\psi = 0$ (centre profile) and $\psi = 40$ degrees (bottom profile).

A software program to calculate the peak position of each diffraction profile has been developed by Mr. Phuong Do at Monash University. This is achieved by fitting a parabola over the top 30% of the peak. The shift in 2θ is then measured by converting the channel no into 2θ . A plot of $\Delta 2\theta$ vs. $\sin^2 \psi$ is shown in Figure 4.



Figure 4) $\Delta 2\theta$ vs. $\sin^2 \psi$ plot for diffraction profiles shown in Figure 3. This graph shows a typical linear relationship which indicates a biaxial stress state in the surface. The stress calculated for KDIE 23(b) from this plot is also given.

The gradient of the $\Delta 2\theta$ vs sin² ψ plot is measured by using the least squares method to fit the data. The stress was calculated by substituting this gradient and Young's Modulus of 69 MPa and Poisson's Ratio of 0.29 into equation 1. Note that to avoid large errors in the final residual stress calculation it is necessary to accurately measure the change in $\Delta 2\theta$ and to keep the integrity of the data to as many decimal places as possible.

3.3 Sources of Errors in the Residual stress measurements:

There are several sources of error in the measurement of the x-ray residual stress measurements. There will be instrumental errors associated with alignment of the sample to the goniometer axis and statistical errors associated with the counts, the location of the peak and with the least squares method analysis. These errors are associated with strain (lattice spacing) of the sample.

Strain is related to the stress via the use of the elastic constants. The relationship between the "true" stress in the material and the measured value depends on the accuracy of the elastic constants and the assumptions made as to the stress state present in the surface of the material. A summary of the main sources of errors is given in the section below.

3.3.1 Measurement Errors

As there was no evidence of ψ splitting in the x-ray diffraction results of the shotpeened sample in Figure 4 errors associated with instrument misalignment are considered negligible. Where the x-ray analysis was repeated on the same area of a shot-peened specimen the error was in the order of 1 MPa. For the above stress measurements of shot-peened aluminium the standard deviation determined from the method of least squares fitting is 4.5% which gives an error of approximately 14 MPa in the residual stress measurement of 322 MPa. For the specimens which had higher scatter of points or ψ spitting the standard deviation will be larger. Overall the error associated with the measurement and calculation of the residual stress for the shotpeened sample is in the order 20 MPa.

3.3.2 Elastic Constants:

The exact elastic constants for our specimens were not measured. A paper by A.A. Denton, 1966⁵ suggests that confidence limits for stress values using the x-ray diffraction technique should be of the order of 10% where the common elasticity values are used or 5% where specific calibrations are carried out. The values for Young's Modulus (E = 69 MPa) and Poisson's ratio (v = 0.29) are used by staff at Monash University as representative of the elastic constants for aluminium. The constant (1+v)/E using psi (pounds per square inch) units for a range of materials is given in Noyan and Cohen 1987³. The given range for aluminium alloys is from 11.33 to 14.09 for pure aluminium. The elastic constants used in this report give a corresponding value of 12.89. The error involved in this case is in the order of 10%. As the shot-peened results give residual stress values up to 320 MPa this error would be in the order of 30 MPa.

The sum of the errors associated with the measurement and elastic constants is in the order of 50 MPa. By keeping the analysis parameters the same and assuming that the elastic constants are similar for all of our specimens then this error can be neglected when comparing the effect of various surface treatments on the residual stress.

3.3.3 Stress homogeneity in the surface of the shot-peened specimen:

Where another area was analysed on a shot-peened specimen, with the tilt in the same direction, the error was in the order of 10 MPa across the surface. Where the sample was rotated 90 degrees to obtain the stress calculation in the transverse or longitudinal direction, the difference was in the order of 30 MPa for a shot-peened specimen (see Table 3).

3.4 Residual Stress Analysis after Abrasion and Heat Treatment:

3.4.1 Non peened Al7050

The residual stress condition of the as received Al7050 fatigue end specimens was tensile over the depth analysed by x-ray diffraction, nominally down to 75 μ m. The surface of the specimens had machining marks and the x-ray sin² ψ plot showed high scatter which indicates that the stress profile is complex and inhomogeneous. Three specimens were analysed for residual stress in the non-peened as received condition. The averaged stress over these three specimens was +300 MPa in the nominal longitudinal direction and +165 MPa in the nominal transverse direction. This difference between the longitudinal and transverse directions also indicates that the surface residual stress is inhomogeneous and not biaxial.



Figure 5) $\Delta 2\theta$ vs. $\sin^2 \psi$ plot for non-shot-peened fatigue end specimen KDIE 23(a). This profile shows a non-linear relationship which indicates that there is a non-homogeneous stress state in the surface of the specimen. As all values are negative then the stress state is tensile.

3.4.2 Shot-peened Al7050

Compared to the non-peened specimens, the three specimens that were sent away for shot-peening exhibited a highly compressive biaxial stress in the surface in the order of -200 to -300 MPa. Comparing Figure 5 with Figure 4 we can see the beneficial effect of shot-peening even on surfaces with tensile and inhomogeneous stress states.

3.4.3 Scotchbrite Abrasion.

Removal of strained material may lead to relaxation of the stress state and loss of beneficial compressive stresses. A peened 7050 specimen KDIE15 (b) was subjected to varying grades of scotchbrite abrasion. A mild scotchbrite abrade where the pad was passed over the specimen several times with a light touch indicated that there was essentially no difference in the residual stress results. A measurement of the specimen thickness indicated that very little material was removed from the surface. A medium scotchbrite where the pad was passed over the specimen with a heavier touch removed approx. 12µm from the surface. The measured residual stress was reduced. We must note that we are now measuring a deeper section of the original stress profile (ie. from 12 to 85 µm). There may also be stress relief induced by the abrading step. Removal of a stressed layer will cause a relaxation in the layers below, as the stress will decay to zero in both sections. Correction for layer removal can be approximated using equations from Noyan and Cohen 1987³. The stress relaxation equation given below is for a flat plate which contains a biaxial macro-residual stress tensor, the components of which vary only as a function of depth. (Eq. 2).

$$\sigma(z_1) - \sigma_{xmeas}(z_1) = -4\sigma_{xmeas}(H) \left(\frac{H-z_1}{H}\right) + \left[\sigma_{xmeas}(H) + 2H\frac{\partial\sigma_{xmeas}(H)}{\partial z}\right] \left(\frac{H-z_1}{H}\right)^2 + \dots \quad [2]$$

where H is the original thickness of the plate, z_1 is the distance from the lower surface to the uncovered depth of interest, $\sigma_x(z_1)$ is the true stress and σ_{xobsv} , is the measured stress value. A series correction for layer removal is possible where the integrands in the above equation are expanded in the form of Tailor's series referred to the surface values and the integration is performed term by term. For small etching depths and shallow gradients, the first terms of the series may be used (Eq.3).

$$\Delta \sigma = \sigma_{x(z_1)} - \sigma_{xmeas}(z_1) = -4\sigma_{xmeas}(H) \cdot \frac{\Delta z}{H}$$
[3]

For an estimation of this relaxation for the scotchbrite measurements H = 6.13mm = 6130 µm, dz is 12 µm, the measured stress σ_{xmeas} is approx 260MPa. Therefore $\Delta\sigma$ is -4 x 270 x 12 / 6130 = -2 MPa. for the Medium Scotchbrite.

Therefore $\Delta \sigma$ is -4 x 240 x 38 / 6130 = -6 MPa. for the Heavy Scotchbrite.

As the measured change in residual stress is approximately 30 MPa for the medium scotchbrite and 60 MPa for the heavy scotchbrite the relaxation due to layer removal is considered negligible. If we assume that the scotchbrite abrasion does not induce a separate stress in the surface then the stress reduction must be due to stress gradients normal to the surface plane. Abrading in the direction of the grain does not appear to affect the stress in one particular direction. The longitudinal and transverse stress measurements for each abrasion step are within 20 MPa. This corroborates the view that the abrasion does not induce a significant stress in the surface.

3.4.4 Grit-blast Abrasion

The results from Table 3 show that the alumina grit-blast (FB) does not appear to reduce the residual stress in the shot-peened 7050 surface in specimen KDIE17 (a). There was also no significant removal of surface material measured. The stress reduction in for both the longitudinal and transverse directions is less than 12 MPa which is within the standard deviation of the stress measurements. There does not appear to be any divergence from the biaxial stress state as shown by the $\sin^2\psi$ plot (Figure 6).

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	the construction of the second s
Table 3.	Residual stress results for A17050 specimens subjective to shot perime, etc.
1 uon 0.	to a the second beat treatment
	abrasion, grit-blasting and neat treatment.

	THE ATLANT ATLANT	RESIDUAL	RESIDUAL
SAMPLE	SURFACE TREATMENT	CTDESS MPa*	STRESS MPa*
			Tremerrorea
		Longitudinal	Transverse
	TT	+329 (Tensile)	+180 (Tensile)
KD1E 15 (a)	Unpeened		
		201	-282
KDIE 15 (b)	Peened	-501	202
	Mild Scotchbrite; Removal = too small to	-283	-293
	accurately measure		
	Medium Scotchbrite; Removal of ~12.3µm	-271	-254
	mitural over		
	surface layer	-241	-228
	Heavy ScotchDille, Kentovaror exact		
	25.6= total of 3/.9µm. Removed most of		
	visible surface roughness. Machining		
	marks visible.		260
KDIF 17 (a)	Peened	-231	-200
	FB surface treatment: MEK/mild SB dry	-220	-254
	110°C 15 mins/alumina grit.		
	Host Treatment: 120°C 1 hour, cool, 120°C	-187	-182
	fileat ficatilities into a statistic		
		+290 (Tensile)	+70 (Tensile)
KDIE 22 (a)	Unpeened	+165 (Tensile)	-46
	FB surface treatment:	100 (1000)	
	MEK/mild SB dry 110°C 15 mins /		
	alumina grit-blast.	1000 (Teme:1-)	+240 (Tensile)
KDIE 23 (a)	Unpeened	+298 (Tensile)	104
	MB surface treatment	-161	-100
	MEK degrease, zircon grit-blast		
T(DIT 02 (1))	Poened	-300	-322
KDIE 23 (b)	MP surface treatment	-263	-205
	WID Sufface meaningen grit-blast		
	MEK degrease, zircon grit-blast		

(*Elastic Constants used: Young's Modulus 69 MPa and Poisson's Ratio = 0.29)

In contrast to the shot-peened case, grit-blasting an unpeened surface KDIE22 (a) with alumina (Figure 7) decreased tensile stresses in both the longitudinal and transverse directions by approximately 130 MPa [Table 3]. This could be due to a surface deformation analogous to shot peening. There was also a more linear $\Delta 2\theta$ vs. $\sin^2 \psi$ relationship after the grit-blast in (Figure 7) compared to the typical non-peened surface (Figure 5) with less scatter in the data points.



Figure 6: $\Delta 2\theta$ vs. $\sin^2 \psi$ plot of shot-peened KDIE17(a) surface grit-blasted with the Fishermen's Bend automated unit using alumina grit.



Figure 7: $\Delta 2\theta$ vs. $\sin^2 \psi$ plot for non-peened KDIE 22(a) surface grit-blasted with the Fishermen's automated unit using alumina grit.





Figure 8: $\Delta 2\theta vs. sin^2 \psi$ plots of a) peened Al7050 KDIE 23(b) and b) nonpeened Al7050 KDIE 23(a) after grit-blast with zircon. Note the divergence from the linear relationship which indicates the presence of shear stresses.

The zircon grit-blast treatment at MB has shown that it induces an even stronger compressive stress on the unpeened surface KDIE23 (a) (Figure 8b) compared to that for the alumina grit-blast treatment (Figure 7). This indicates that it is producing a similar compressive stress effect to the shot-peening. This is expected as zircon particles are 250μ m spherical particles of higher density than the AP106 alumina.

However, when the zircon blasting was used on the shot-peened surface KDIE23 (b) there is a significant reduction in the compressive stress (Figure 8a). Unlike the Scotchbrite heavy abrasion there was no significant removal of material. Therefore there should not be any significant stress relaxation due to layer removal. There has been cases where overpeening a surface can cause relaxation and the compressive stresses are reduced [Noyan and Cohen, 1987]³. The plots for the sin² ψ of the peened and nonpeened surfaces that had been grit-blasted with zircon show a divergence away from the linear relationship indicating the presence of shear stresses. Compare Figure 8 with Figure 4. (see also Appendix A Figure 2b)

3.4.5 Heat Treatment Cycle

The specimen grit-blasted with alumina grit was heat treated to 120°C for 1 hour, cooled to room temperature and then heated to 120°C for 2 hours. This process showed a slight reduction in the stress of the specimen from 30 to 60 MPa in the longitudinal and transverse directions respectively. There appears to be a divergence from the linear relationship between the strain and $\sin^2\psi$ in both the longitudinal and transverse directions which indicates the presence of shear stress in the specimen. Compare Figure 9 with Figure 4.



Figure 9: Aluminium Al7050 KDIE 17(a) shot-peened, alumina grit-blasted and heat treated specimen. $\Delta 2\theta$ vs. $\sin^2 \psi$ plot with divergence from the linear relationship indicating the presence of shear stress.

4. DISCUSSION

4.1 Effect of Trial Surface Treatments on Bond Durability:

The Al7075 and FM73 non-peened system generally has a less durable bonding compared to the clad Al2024 and FM73 non-peened system. For Al2024 the solvent clean, Scotchbrite abrade, alumina grit-blast and silane standard procedures are sufficient to satisfy the C5033 criteria. [Olsson-Jacques C.L. et al. 1995]⁶. The same procedure for non-peened Al7075 was not sufficient to satisfy the criteria (Table 2) and therefore it was necessary to investigate further treatments to improve bond durability.

The shot-peening does not appear to have a detrimental effect on the bond durability as the results from the non-peened and one-plate peened/one non-peened Boeing wedge test are very similar. These results suggest that the MEK wipe does appear to remove any detrimental effects that the blue ink may have on bond durability. Although the ink was reported to have been water soluble visual inspection showed that the warm water wash and wipe was not sufficient to remove this ink. The durability results for the water wipe, scotchbrite abrade, alumina grit-blast, silane and BR127 treatment had a slightly faster crack growth rate than a similar treatment that replaces the water wipe with an MEK wipe. This slight difference in durability may be due to the effect of the blue ink when the adhesive joint is subjected to the humid environment.

<u>RECOMMENDATION</u>: Use MEK solvent clean not water wash to remove shotpeening ink.

Treatment of the peened surfaces with silane (brush application) JB14 had a reduced durability compared to the non-peened gritblasted specimens that were immersed in silane. (JB22 and JB44). The brush application is used in the field because immersion is not viable. The brush application has been found to produce a slight reduction in durability due to the introduction of contaminants⁷. The reduction in durability may be compounded by the presence of blue ink on the surface after the warm water solvent clean.

<u>RECOMMENDATION</u>: In the application of silane, attempt to use clean brushes that do not have components that can be dissolved in aqueous epoxy silane solutions.

The PASAGEL 105 treatment produced the least durable of all our trials. The PASAGEL 105 only treatment for this system produces a less durable bond than the silane coupling agent in either the brush or immersion applications. The PASAGEL 105 treatment is therefore <u>NOT RECOMMENDED</u>.

The addition of a BR127 chromate primer step to the grit-blast and silane (brush) procedure (JB20 and JB47) significantly improved the bond durability. The results for JB47 satisfied the RAAF Engineering Standard C5033 criteria of 5 mm in 24 hours and 7 mm in 48 hours. (Table 2). As this treatment does satisfy the criteria it is recommended for use for the Patch repair of the shot-peened Al7050 surface on the FA/18. However it was necessary to investigate the effect of abrasion and heat treatment steps in this procedure on the beneficial compressive stresses induced by the shot-peening.

4.2 Effects of Abrasion and Heat Treatment Steps on Residual Stress:

X-ray diffraction demonstrated that the stresses in the non-peened specimens were nonhomogeneous and were highly tensile. The shot-peening process overcame these high non-homogeneous tensile stresses and produced a high biaxial compressive surface stress in the aluminium alloy surface. The depth and profile of the compressive stress layer due to the ceramic bead peening was not directly measured. Clark and Clayton (1991)⁸ measured residual stress profiles with depth for glass bead peening on Aluminium 7050 T73651 using wet abrasion to remove surface layers. It is not clear from the reference what the depth of x-ray penetration was or if layer removal corrections were used. The initial zero depth removal results indicate compressive stress was in the order of -250 MPa which is similar to the results for ceramic bead peening.

The abrasion and heat treatment of shot-peened surfaces decreased the level of compressive stress. However in all cases the absolute stress value is still relatively high (ie. above 100 MPa). Although the fatigue life was not significantly affected by the stress reductions (Sharp and Byrnes 1996)⁹ the aim should be to keep the changes in the surface compressive stresses to a minimum. Therefore we should aim to use abrading and heat treatment processes which have a minimal effect on the stress profile in the surface plane and with depth.

Abrasion with Scotchbrite pad (lubricated with distilled water) should minimise surface contamination. However care must be taken not to be aggressive with this process as part of the beneficial compressive stress layer of the surface can be removed and the stress in the exposed surface can be relieved. The total reduction of stress in the surface with a medium and heavy abrasion cannot be accounted for by relaxation due surface layer removal alone (section 3.4.3). Therefore a stress gradient with depth from the surface must exist. Removal of approx 40µm due to Scotchbrite abrasion has reduced the stress by about 60 MPa. However Clark and Clayton (1991)⁸ indicate that the compressive residual stress induced by glass bead peening on aluminium 7050 increased the residual stress results to a maximum of approximately -275 MPa after 50 μ m removal with wet abrasion. A further surface layer removal of 50µm down to 100µm then significantly reduced the stress to -50 MPa. Our results indicated that the reduction occurred with less than 50µm removal which indicates that the maximum compressive stress occurs closer to the surface than for the experiments by Clark and Clayton 19918. This variation of the maximum compressive stress with depth could be due to the differences in the shot peening conditions, in stresses induced by the layer removal techniques or the pre-peening surface stress profiles of the aluminium 7050 material. As the fatigue resistance depends on the high compressive stress in the surface induced by the shot-peening process we would therefore recommend that a mild abrasion is used. RECOMMENDATION: Mild Scotchbrite abrade

There is a significant difference in the residual stress results of the Fishermen's Bend (FB) compared to the Maribyrnong (MB) grit-blast procedures on both the shot-peened and non-peened aluminium alloy surfaces. Essentially the FB grit-blast with alumina grit had less impact on the surface residual stresses than the MB procedures. This difference could be due to a number of factors in the properties of the grit and the

method of application. The 50μ m alumina grit is needle shaped whereas the zircon grit is larger, more spherical and denser than the alumina and is similar to the beads used in peening. The air pressure at which the grit-blasting is carried out is higher for the zircon than the alumina grit. The FB grit-blast was automated so that all areas are covered equally over the total area of the specimen. The zircon grit-blast was applied manually which could lead to non-homogeneous grit-blasting over the surface of the sample and excessive blasting in some areas.

As can be seen from the results the zircon grit-blast acts on the unpeened surface in much the same way as the shot-peening in that a compressive stress was induced in the surface. However it must be noted that there is evidence of shear stresses present in the surface and that the absolute value of the stress in the grit-blasted case was in the order of 150 MPa whereas the peening process induces stress in the order of 300 MPa. The peened specimen, which was then subjected to the zircon grit-blast, had a stress reduction in the order of 40 MPa in the longitudinal direction and 120 MPa in the transverse direction. This could possibly be due to an overpeening effect. There are cases where overpeening or use of too high a peening velocity can cause relaxation at the surface. [Noyan and Cohen, 1987]³. The MB grit-blasting induces shear stresses into the surface. Perhaps this could be related to the manual application such as the nozzle not being held normal to the surface and perhaps non-homogeneous grit-blasting over the surface. Further experiments would need to be carried out to investigate the full surface stress profile induced in the surface by grit-blasting procedures.

<u>RECOMMENDATION</u>: Grit-blast with alumina rather than zircon for shot-peened surfaces. Care must be taken to control the impact density and pressure of the grit delivered to the surface as well as obtaining a uniform coverage over the surface.

The heat treatment cycle of 120°C for 1 hour and 120°C for 2 hours was chosen as it represented the drying and cure heating cycles that the aircraft surface should see during the bonding procedure. Bouseau et al. 1984⁴, observed that stresses at the surface of aluminium 7075 T73 in the order of -220 MPa were slightly reduced by heat treatment at 100°C for 2 hours and significantly reduced to approx. -100 to -60 MPa for 150°C between 2 and 64 hours. The maximum compressive stress with depth was also reduced for the 150°C treatments at 4 and 64 hours. The residual stress reduction in the first 75µm of our 7050 specimen surface due to heat treatment of 120°C for 3 hours was in the order of 60 MPa with a comparative error of 25 MPa. The surface treatment procedure asks for a 110°C for 0.25 hour, 1 hour and 0.5 hour to dry the surface after the debris removal, silane application and BR127 steps respectively. The cure of the FM73 adhesive is for 120°C for 1 hour. It is expected that the surface stress reduction will not be as high for the practical application as both the temperature and time are less than the full heat treatment cycle used. Sharp and Byrnes 19969 indicated that the patch repair process causes a minor statistically insignificant reduction in the average fatigue life and concluded that the thermal/surface treatment process has no significant effect on the fatigue life of the peened part.

<u>RECOMMENDATION</u>: Do not use heat treatment temperatures and times on the surface in excess of the requirements of the proposed surface treatment.

5. CONCLUSION

For the shot-peened Aluminium 7075 and FM73 system, the surface treatment which produced the most durable result included a MEK solvent clean, a mild scotchbrite abrade, debris removal with water soaked tissues, dry at 110°C, an alumina grit-blast, an aqueous epoxy silane treatment dry 110°C for 1 hr, a spray application of BR127 primer and a cure at 120°C for 30 minutes. The PASAGEL 105 treatment produced the least durable result of all our trials and is therefore not recommended for this application.

The MEK solvent clean was necessary to remove the blue ink remaining from the shotpeening quality assurance process.

A Scotchbrite[®] with distilled water abrade was included to further reduce organic contaminant and loosely adhered oxide scale. This abrade is to be mild as the compressive residual stresses will decrease with increasingly aggressive abrasion. The distilled water wipe is included to remove debris from the surface.

An alumina grit-blast is recommended to remove any further contaminants and produce a chemically active surface to which the epoxy silane coupling agent can attach. Grit-blasting with zircon grit is not recommended as there is a significant reduction in the compressive stresses in the shot-peened surface. The zircon grit-blast is similar to the shot-peening process and damage caused by overpeening could contribute to the observed reductions in residual stress.

The silane is to be dried at 110°C for 1 hour as this has been shown in previous research to reduce voiding in the FM73 adhesive⁷.

The addition of the BR127 as a final step before bonding was required for the proposed silane treatment to meet the RAAF Engineering Standard C5033 criteria for bond durability.

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Appendix A

X-ray Diffraction Residual Stress Analysis:

The x-ray diffraction technique is non-destructive and gives a quantitative value for the average macro-residual stress in the surface of the specimen. The lattice spacing d of crystal can be measured by x-ray diffraction using Bragg's equation [1]¹.

$$n\lambda = 2d\sin\theta \tag{A-1}$$

where λ is the wavelength of the x-ray source, θ is the angle between the x-ray beam and the normal to the lattice plane.

The x-ray diffractometer with precision alignment measures 20 which is then used to calculate the lattice spacing d. The x-ray diffraction units can scan a wide 20 range and many different lattice plane spacings can be measured. Although this technique is used for the identification of polycrystalline materials and their crystallographic structures, residual stress calculations can also be made on the basis that the crystal lattice spacings act as strain gauges. The strains along a particular direction can be expressed in terms of the change in lattice spacing $d_{\varphi\psi}$ - d_0 from the unstressed state d_0 as in Equation 2.

$$(\varepsilon_{33})_{\phi\psi} = \frac{d_{\phi\psi} - d_0}{d_0}$$
[A-2]

The change in strain along a particular direction can be investigated by tilting the sample with respect to the x-ray beam. This results in a shift in the x-ray diffraction profile as the schematic representation in Figure 1. The 2θ value is related to the lattice spacing d by using Bragg's Equation [1]¹. The stress σ_{ϕ} can be related to strain by the following equation to give a working relation¹.

$$\sigma_{\phi} = \frac{d_{\varphi} - d_z}{d_z} \cdot \frac{E}{(1+\upsilon)\sin^2\psi}$$
[A-3]

where v = Poisson's Ratio, E = Young's' Modulus

Equation 3 shows the linear relationship between the strain and $\sin^2\psi$ for a biaxial surface stress. If Poisson's Ratio and Young's Modulus are known then the stress value σ_{φ} can be obtained from the gradient of the plot of strain versus $\sin^2\psi$. Typically the exact value of the unstressed lattice spacing d_0 is not known and the value of d_z where ψ is zero is substituted. This generates a minor error in the calculation of the stress in the material. Equation 3 can also be written in terms of the diffraction angle θ to give equation 4. Figure 2 shows typical behaviour from strain and $\sin^2\psi$ measurements.

$$\sigma_{\phi} = \frac{E \cot \theta}{2(1+\nu)\sin^2 \psi} \Delta 2\theta$$
 [A-4]

¹ Klug and Alexander, "X-ray Diffraction Procedures", Wiley and Son, (1954)



Figure 1: Schematic diagram of the effect of a force acting on the crystal lattice planes and the resultant shift in the x-ray diffraction profile with tilt ψ of the sample surface with respect to the x-ray beam.



Figure 2: Types of d vs $\sin^2 \psi$ plots: a) Linear relationship (biaxial stress state), b) y splitting (shear stress) and c) oscillatory behaviour (eg. anisotropic elastic constants, strong gradients with depth)²

² adapted from: Noyan I.C. and Cohen J.B., "Residual Stress", (1987), Springer Verlag. 1

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19. ABSTRACT								
Fatigue cracking problems have occurred in the $F/A-18$ 470.5 bulkhead during initial full scale testing. The surface of this bulkhead is shot-peened to introduce compressive residual stress to increase the								
fatigue life of the component as part of the manufacturing and maintenance program. The Aeronautical								
and Maritime Research Laboratory (AMRL) is investigating the effect of applying a composite patch to								
reduce the critical strains in the crotch area. Boeing wedge durability tests were used to define the most								
The x-ray diffraction technique was used to assess any reduction in the beneficial shot-peened residual								
stress after typical abrasion and heat treatment stages in the preparation procedure. It was found that the								
abrasion and heat tr	abrasion and heat treatment processes used to achieve the most durable surface treatment for bonding							
did not significantly reduce the beneficial compressive surface stresses induced by the shot-peening								

process.