

Environmental Exposure of Boron-Epoxy Composite Material

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DSTO-TN-0309

ABSTRACT

Boron-epoxy composite material (Textron 5521 F/4) was exposed at two tropical exposure sites within Australia for a period of about 3.5 years to evaluate the effects of outdoor exposure on mechanical properties. Specimens were exposed to three exposure types; fully exposed, covered and shaded. Moisture contents of 0.85% at Tindal N.T and 1.07% at AMRL-Q, Innisfail, QLD were found after about 3.5 years for the shaded and covered exposure conditions. Fully exposed specimens had about 20% less moisture. Mechanical tests were then performed in $\pm 45^\circ$ in-plane shear at both room temperature and 60°C to assess the effects of moisture on the matrix. The chord shear modulus was also determined in some cases. The results indicated that no significant changes in shear strength occurred at room temperature or 60°C due to outdoor exposure. The peak shear stress is about 15% lower when tested at 60°C compared to those tested at room temperature. The chord shear modulus values were subject to scatter but did not reveal any deterioration in material performance. An accelerated laboratory conditioning scheme to produce a 1% moisture level in the composite was also devised which allowed an equilibrium moisture level to be established in eight ply boron-epoxy specimens after only six weeks. This study found no evidence that absorbed moisture is likely to degrade the strength of boron-epoxy repair patches used on RAAF aircraft when tested at room temperature and 60°C.

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Published by

*DSTO Aeronautical and Maritime Research Laboratory
PO Box 4331
Melbourne Victoria 3001 Australia*

Telephone: (03) 9626 7000

Fax: (03) 9626 7999

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AR-011-602

October 2000

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Executive Summary

Polymer matrix composite materials are becoming increasingly prevalent in modern military aircraft, either as original structure (graphite-epoxy wing-skins on the F/A-18) or as modifications or repairs.

The Bonded Repair Technology pioneered by DSTO typically uses repair patches made from boron-epoxy material. These patches have proven to be very successful and some are still in service after 25 years. No study has been undertaken to determine if the strength of such patches is degraded by representative levels of absorbed moisture.

It is well known that the properties of composite materials are degraded when exposed to hot/wet conditions. A typical composite material will begin to absorb moisture from the air soon after manufacture to a level of about 1% by weight after long-term service. Combined with elevated temperature experienced during flight this hot/wet conditioning of the material can degrade matrix properties by up to 25% for supersonic aircraft and 15% for subsonic. An understanding of hot/wet performance is vital in determining the operational envelope.

This paper details the results of experimental work to determine the effects of tropical outdoor exposure on boron-epoxy composite material (Textron 5521 F/4).

The exposure of boron-epoxy composite specimens showed that the samples absorb 0.85% moisture at Tindal N.T and 1.07% at AMRL-Q, Innisfail, QLD after 3.5 years. The weather patterns at Tindal are highly influenced by the tropical wet and dry seasons which see significant drying of the composite during a dry season cycle. AMRL-Q tends to produce a more consistent hot and humid environment all year round.

Mechanical testing showed no significant contribution of the moisture in reducing the $\pm 45^\circ$ in-plane shear stress when tested at both room temperature and 60°C. The peak shear stress does however decrease by about 15% when compared to specimens tested at room temperature.

This work has successfully determined the moisture content levels produced in boron-epoxy material during tropical exposure. Such information is vital in evaluating the effects of moisture on adhesively bonded composite repairs in the laboratory. The durability of the boron-epoxy material, as proved by mechanical tests, shows that this material is likely to perform well in tropical environments over the long term and serve the RAAF well as a repair material.

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1. Introduction

The effects of the operating environment on the mechanical properties of composite materials need to be investigated to effectively use these materials in aircraft service. It is a well-known fact that the epoxy matrices of many commercially available composite materials degrade in performance on exposure to moisture. The epoxy matrix can absorb moisture up to a level determined by the relative humidity of the environment. Therefore high humidity exposure can create high levels of diffused moisture in the matrix and degrade mechanical properties such as glass transition temperature (T_g) and shear strength. Tropical operating environments, such as those experienced in the northern regions of Australia are particularly conducive to producing high levels of moisture in composites. Previous work on graphite/epoxy composites performed at AMRL showed that tropical exposure produced an increase in weight due to moisture of around 1%.

The effects of environment on Textron 5521F/4 boron epoxy were evaluated. This material is used by the RAAF for the adhesively bonded repair of metallic structures, for example the C130 wing planks and F-111 wing pivot fitting. Many thousands of boron-epoxy repairs have been applied to RAAF aircraft and it is therefore important to determine the effect that long-term environmental exposure may have on their mechanical behaviour. Previous work has examined the effects of moisture uptake in boron-epoxy composites when exposed to simulated environments in the laboratory [1].

This work extends the results of that research by exposing boron-epoxy (Textron 5521 F/4) laminates to environmental conditions at two outdoor locations, Tindal, N.T and AMRL-Q, Innisfail QLD. Laminates were exposed for a period of 3.5 years and then tested to assess any changes in mechanical properties. The mechanical tests utilised the $\pm 45^\circ$ shear test (ASTM D3518-94). The moisture content of all laminates was tracked by means of traveller coupons which were weighed on a regular basis.

In addition an identical set of laminates were conditioned in the laboratory to a moisture level equivalent to that seen at the exposure sites. This was done to determine whether laboratory based moisture conditioning could be successfully used to replicate the effects of real outdoor environmental exposure.

2. Specimen Manufacture

Specimens were laid up in $[+45/-45]_{25}$ configuration using Boron-Epoxy 5521F/4 prepreg material sourced from Textron (Batch A-182 Roll: 72 Manufactured 1/5/1996) and cured August 1996 using the manufacturer's recommended cure cycle (1 hour at 120°C , 400kPa pressure). The specimens were then painted at AMRL-Maribyrnong

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(13/8/1996) using the standard F/A-18 paint scheme (light colour). Traveller coupons were made using eight plies of unidirectional boron.

Prior to testing for shear strength (ASTM D3518-94) tabs were applied to the ends of the samples to prevent slippage. The baseline room-temperature test specimens used adhesively bonded aluminium tabs. Tabs were applied using FM73 adhesive cured at 80°C under 200kPa pressure using rubber mats and a platen press. Tabs were 38mm long with a 3° taper starting about 10mm from the tab end. All subsequent tests did not utilise bonded tabs but used 180 grit emery cloth. The latter method reduced the probability of failing the specimens in the grips since peel stresses at the ends of the bonded tapered tabs are avoided.

Laminates were cut using a diamond saw using water. Traveller coupons were cut to 110mm by 45mm and $\pm 45^\circ$ shear test specimens were cut to 250mm by 110mm before being placed in exposure racks. The $\pm 45^\circ$ test specimens were cut to 25mm by 250mm prior to mechanical testing.

3. Outdoor Exposure Locations

Two exposure locations were used Tindal, N.T and AMRL-Q, Innisfail QLD. AMRL-Q is a very wet tropical location which has an average humidity in excess of 80% R.H. Tindal represents a tropical environment but is further inland and experiences distinct dry and wet seasons.

Specimens were placed in a stainless steel fixture in such a way that all specimens experience the prevailing levels of humidity but see three different degrees of exposure to solar radiation. Specimens classed as "exposed" are fully exposed to solar radiation during the day, "covered" specimens are fully protected from direct incident radiation by a stainless steel plate, while "shaded" specimens are placed beneath a piece of aluminium honeycomb which restricts direct exposure to radiation to only a few hours at midday. Exposure conditions are summarised in Table 1.

Table 1 Summary of exposure locations and exposure types

Location	Exposure Types
AMRL-Q, QLD	Exposed, Covered, Shaded
RAAF Tindal, N.T	Exposed, Covered, Shaded

Specimens were placed at the exposure sites on 15th of August 1996 and recalled 2nd of March 2000. This represents about 3.5 years of exposure.

4. Moisture Uptake

The moisture uptake of the specimens was monitored using eight ply unidirectional laminates of dimensions 110mm by 45mm. Three specimens for each exposure type were weighed using a four-figure balance. A reference stainless steel weight was also measured to ensure calibration of the balance at all times. Moisture uptake is calculated as a percentage increase in weight of the composite. (Equation 1)

$$\frac{\text{weight}(\text{current}) - \text{weight}(\text{initial})}{\text{weight}(\text{initial})} * 100 \quad \text{Equation 1.}$$

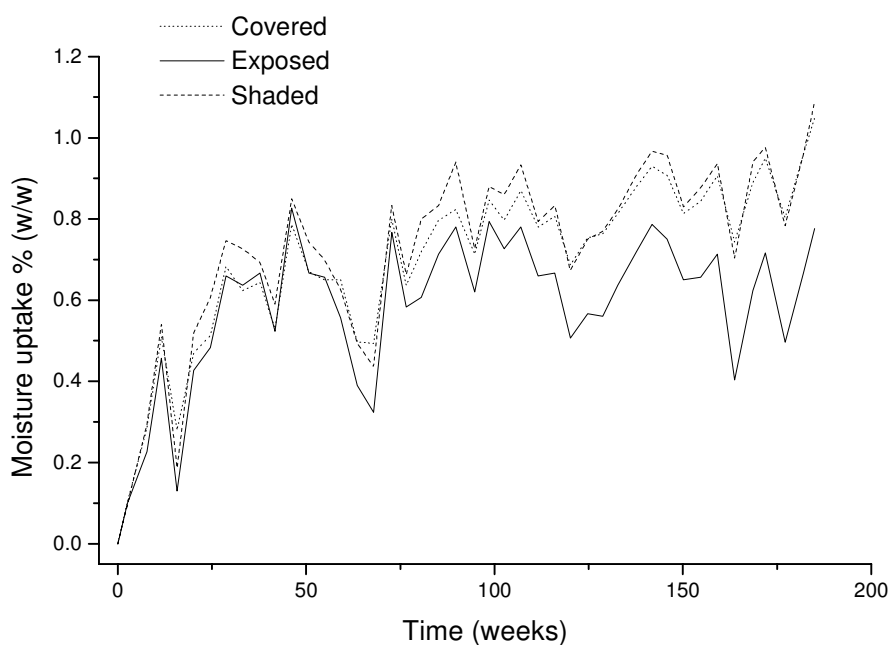


Figure 1 Weight gain of boron-epoxy composite material exposed at AMRL-Q, Innisfail, QLD.

Moisture uptake for coupons exposed at AMRL-Q is shown graphically in Figure 1. Figure 2 shows the moisture uptake for coupons exposed at Tindal, N.T.

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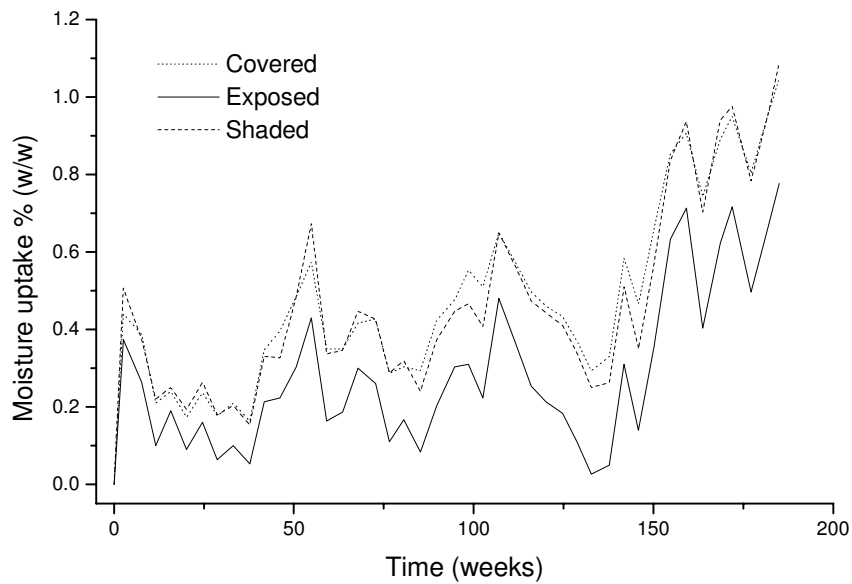


Figure 2 Weight gain of boron-epoxy composite material exposed at Tindal, N.T.

Table 2 Moisture uptake values for traveller coupons at March 2000

Exposure Site	Exposure Type	Moisture Uptake (at March 2000)
Tindal, N.T	Exposed	0.77%
Tindal, N.T	Shaded	1.09%
Tindal, N.T	Covered	1.05%
AMRL-Q, QLD	Exposed	0.63%
AMRL-Q, QLD	Shaded	0.84%
AMRL-Q, QLD	Covered	0.86%

Table 2 shows the final moisture content levels at both sites for all three exposure types. The final moisture contents were determined by recording the specimen weight gain and calculating the moisture uptake percentage using equation 1. The specimen weights were measured immediately prior to the specimens being recalled in March 2000. The specimens were recalled at the end of the tropical wet season to ensure the maximum moisture content was present in the specimens prior to mechanical testing.

5. Accelerated Laboratory-Based Moisture Conditioning

The environmental testing of composite materials can be a long process as seen by the trial conducted here which was completed in 3.5 years. Accelerated moisture conditioning of composites can be an effective alternative [2]. Such conditioning can replicate the moisture levels reached outdoors in a shorter period of time. It must be noted that such conditioning is unlikely to be able to assess any long-term degradation mechanisms. In this trial accelerated conditioning was used as a means of comparison to determine whether any reductions in shear strength were purely due to the presence of moisture or whether other mechanisms were involved.

An accelerated moisture conditioning scheme as suggested by the work of Ciriscioli et al. [3] was utilised. This scheme is endorsed by MIL-HDBK-17. To condition the specimens to a moisture content of 1% by weight the specimens were placed in a 100% R.H atmosphere (placed above a sealed container of water) at 70°C until a moisture content of 1.2% was reached. The specimens were then placed in an environmental chamber at 70°C and 65% R.H to condition to a final moisture content value of 1%. This two-stage conditioning scheme allows the final moisture content to be reached in shorter time frames. In this case the conditioning took six weeks.

Specimens of this type were tested in $\pm 45^\circ$ in-plane shear as per ASTM D3518-94.

6. Mechanical Test Results

Mechanical testing was performed on a range of specimens. These include baseline specimens (no-conditioning), outdoor exposed specimens (exposed at Tindal and AMRL-Q) and accelerated laboratory conditioned specimens.

Testing of $\pm 45^\circ$ in-plane shear was performed as per ASTM D3518-94. A strain rosette of 0/90° Micro-Measurements Type EA-03-125TM-120 Option LE gauges were utilised in only some of the test cases to calculate the chord shear modulus. Strain gauges were applied using M-Bond 200 adhesive (Micro-measurements). Specimens were tested in tension using a INSTRON 1185 testing machine using a 100 kN load cell. A 2mm/minute crosshead speed was used for all tests.

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Testing was performed at both room temperature and 60°C. One test was carried out at 80°C on an accelerated laboratory conditioned sample (1% moisture) but the matrix was found to be very ductile at this temperature. The test at 80°C lasted for over 15 minutes without failure occurring. It is also likely that the specimen would undergo significant drying (ie: moisture loss) at 80°C for this lengthy period of time making it difficult to assess the effects of moisture in the matrix. Previous work [1] has shown that a 1% moisture level reduces the glass transition temperature of the boron-epoxy material to about 95°C which would explain the ductility of the matrix during the 80°C test.

Peak load was taken at the equilibrium load level as shown in Figure 3. Peak shear stress is calculated using Equation 2.

$$\text{Peak Shear Stress} = \frac{\text{Peak Load}}{2 * \text{cross - sectional area}} \quad \text{Equation 2}$$

The chord shear modulus was calculated between 2000µε and 5000µε using Equation 3.

$$\text{Chord Shear Modulus} = \frac{\Delta \text{Shear Stress}}{\Delta \text{Strain}} \quad \text{Equation 3}$$

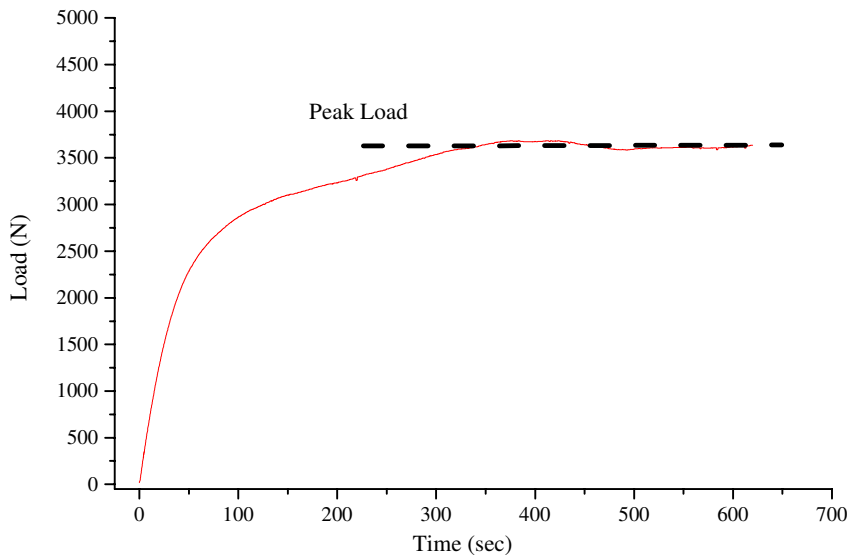


Figure 3 Typical load versus time curve showing peak load determination

6.1 Baseline Specimens

Baseline specimens (non-conditioned) were tested at both room temperature and 60°C. Chord shear modulus and peak stress were determined. Chord shear modulus results for the 60°C tests were not determined since strain gauges were not applied. Average baseline peak shear stress at room temperature is 67.1 MPa $\pm$ 2.7. Average baseline peak shear stress at 60°C is 59.2 MPa $\pm$ 0.8. Values are shown in Tables 3 and 4.

Table 3 Room temperature baseline test specimen dimensions and results

Specimen	Width (mm)	Thickness (mm)	Shear Chord Modulus (GPa)	Peak Stress (MPa)
RTB1	22.60	1.15	N/A	71.2
RTB2	23.85	1.14	6.6	65.7
RTB3	23.80	1.15	6.6	66.4
RTB4	23.80	1.15	6.6	65.3

Table 4 60°C baseline test specimen dimensions and results

Specimen	Width (mm)	Thickness (mm)	Shear Chord Modulus (GPa)	Peak Stress (MPa)
60B1	25.78	1.23	N/A	58.1
60B2	25.72	1.23	N/A	59.1
60B3	27.8	1.24	N/A	59.8
60B4	25.7	1.23	N/A	59.6

6.2 Accelerated Moisture Conditioned Samples

Accelerated moisture conditioned specimens were tested at both room temperature and 60°C. Average peak shear stress for the accelerated laboratory-conditioned samples tested at room temperature is 70.2 MPa $\pm$ 1.1. Average peak stress for the accelerated laboratory conditioned samples tested at 60°C is 55.4 MPa $\pm$ 0.7. Shear cord modulus values were not measured. Results are shown in Tables 5 and 6.

Table 5 Room temperature accelerated conditioned test specimen dimensions and results

Specimen	Width (mm)	Thickness (mm)	Shear Chord Modulus (GPa)	Peak Stress (MPa)
RTLAB1	25.73	1.23	N/A	69.8
RTLAB2	25.75	1.23	N/A	68.8
RTLAB3	25.73	1.23	N/A	70.9
RTLAB4	27.6	1.23	N/A	71.2

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Table 6 60°C accelerated laboratory conditioned test specimen dimensions and results

Specimen	Width (mm)	Thickness (mm)	Shear Chord Modulus (GPa)	Peak Stress (MPa)
60LAB2	25.68	1.26	N/A	54.8
60LAB3	25.73	1.27	N/A	56.1
60LAB4	27.82	1.26	N/A	55.2

6.3 Tindal, N.T Outdoor Conditioned Samples

Tindal outdoor conditioned specimens were tested both at room temperature and 60°C. Shear cord modulus (some cases) and peak shear shear load values were measured (Table 7).

Table 7 Results of mechanical tests for specimens exposed at Tindal, N.T

Specimen Name	Exposure Type	Test Temperature	Width (mm)	Thickness (mm)	Shear Chord Modulus (GPa)	Peak Stress (MPa)
TC60G2	Covered	60°C	1.14	25.39	7.1	64.9
TC602	Covered	60°C	1.2	25.45	N/A	61.2
TE60G2	Exposed	60°C	1.17	25.18	6.4	63.4
TE602	Exposed	60°C	1.18	25.5	N/A	63.4
TS60G1	Shaded	60°C	1.11	25.44	6.5	65.3
TS602	Shaded	60°C	1.15	25.25	N/A	63.6
TCRTG1	Covered	RT	1.26	25.42	7.3	73.1
TCRT1	Covered	RT	1.18	25.48	N/A	69.4
TERTX1	Exposed	RT	1.1	25.43	7.9	83.4
TERT1	Exposed	RT	1.2	25.47	N/A	75.6
TSRTG2	Shaded	RT	1.11	25.49	8.1	81.7
TSRT1	Shaded	RT	1.17	25.47	N/A	73.9

Average room temperature peak shear stress is 76.2 MPa $\pm$ 5.3.

Average 60°C peak shear stress is 63.6 MPa $\pm$ 1.4.

6.4 AMRL-Q, QLD Outdoor Conditioned Samples

Tindal outdoor conditioned specimens were tested at both room temperature and 60°C. Shear cord modulus (in some cases) and peak load were measured (Table 8).

Table 8 Results of mechanical tests for specimens exposed at AMRL-Q, Innisfail, QLD

Specimen Name	Exposure Type	Test Temperature	Width (mm)	Thickness (mm)	Shear Chord Modulus (GPa)	Peak Stress (MPa)
AC60G2	Covered	60°C	1.19	25.45	6.0	61.8
AC602	Covered	60°C	1.2	25.45	N/A	56.0
AE602	Exposed	60°C	1.25	24.98	N/A	55.5
AS60G2	Shaded	60°C	1.13	25.53	6.0	61.7
AS60G2b	Shaded	60°C	1.17	25.46	6.1	57.7
AS602	Shaded	60°C	1.2	25.35	N/A	58.4
ACRT1	Covered	RT	1.2	25.52	N/A	69.0
AERTG1	Exposed	RT	1.23	25.46	7.1	71.7
AERT1	Exposed	RT	1.25	25.55	N/A	69.7
ASRTB1	Shaded	RT	1.16	24.92	7.4	78.0
ASRTG1	Shaded	RT	1.34	25.21	6.0	64.7
ASRT1	Shaded	RT	1.18	25.3	N/A	72.5

Average room temperature peak shear stress is 70.9 MPa $\pm$ 4.4. Average 60°C peak shear stress is 58.5 MPa $\pm$ 2.7

6.5 Chord Shear Modulus

The chord shear modulus was determined for only some of the specimens tested. Linear curve fitting was applied over the region of 2000 $\mu\epsilon$ and 5000 $\mu\epsilon$ to determine the values. The results indicate that apart from the baseline samples there appears to be a great deal of scatter in the data. This is mainly due to the non-linearity seen in the stress strain curves. A typical stress-strain curve for a baseline specimen tested at room temperature is shown in Figure 4. Moisture conditioned specimens showed a greater degree of non-linearity as shown in Figure 5 compared to baseline specimens. There is therefore significant error produced when attempting to fit a linear function to a non-linear stress-strain curve and this is borne out in the results. For this reason it is difficult to make any significant conclusions from the data based on the use of chord shear modulus.

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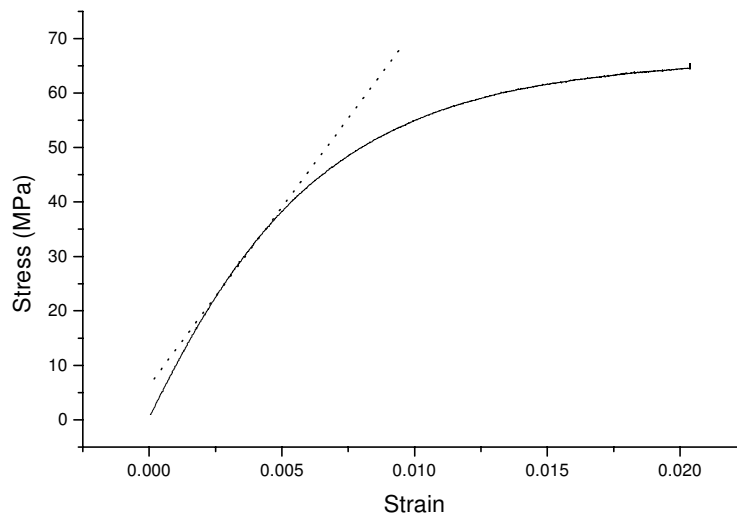


Figure 4 Typical shear stress-strain curve for baseline specimen tested at room-temperature showing linear fit for chord shear modulus

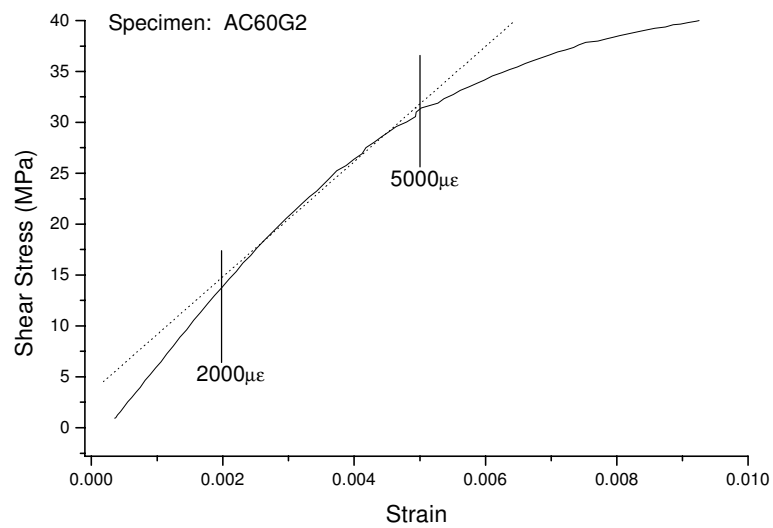


Figure 5 Shear stress versus strain curve showing linear fit for chord modulus (test at 60°C)

6.6 Summary

6.6.1 Peak Shear Stress

A summary of peak stress results is shown in Figure 6 and Figure 7. Average values are plotted for all cases. For the cases of the outdoor exposed specimens an average value was calculated using results for all three exposure types (ie: covered, shaded, exposed). From the moisture content values the covered and shaded specimens have very similar moisture content values while the exposed specimens have about 20% less moisture. There appears no clear difference between the peak shear stress values for the exposed specimens compared to the shaded/covered ones.

There appears to be no clear indication that any major changes in peak shear stress values have occurred after either accelerated-conditioning or during outdoor exposure for a given test temperature. There is however a clear reduction in peak shear stress when tests are performed at 60°C. The reduction in peak shear stress when tested at 60°C is of the order of 15%. There also seems to be a greater level of scatter for the specimens tested at room temperature.

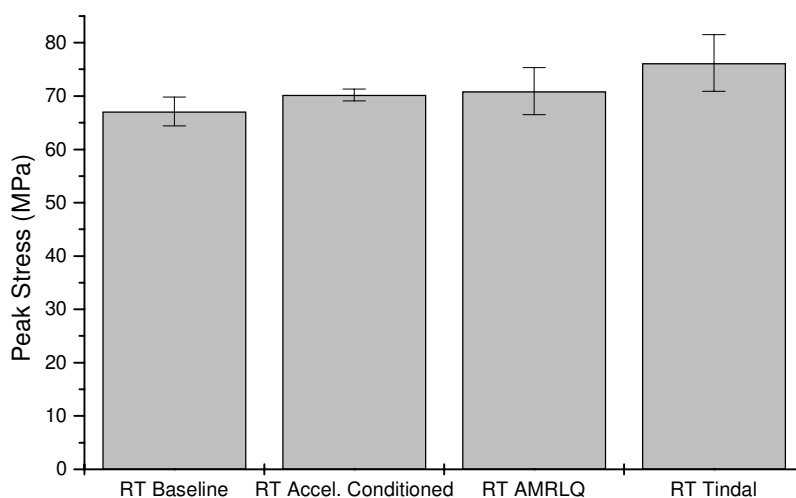


Figure 6 Average peak shear stress values for specimens tested at room temperature

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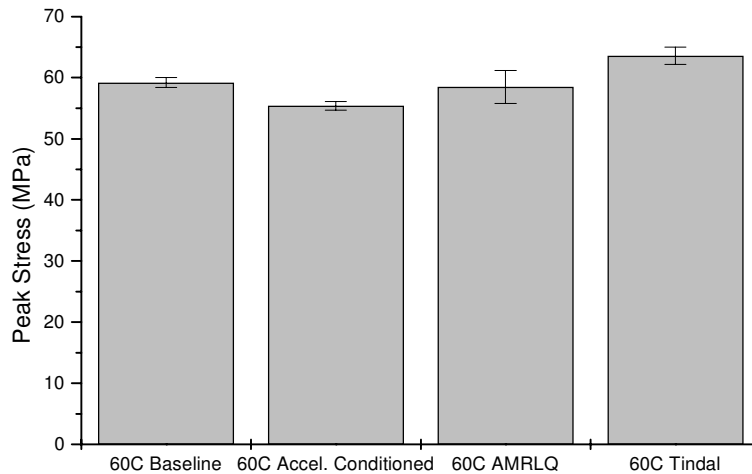


Figure 7 Average peak shear stress values for specimens tested at 60°C

6.6.2 Chord Shear Modulus

Results for the baseline room temperature specimens show a chord shear modulus of around 6.6 GPa. Other results are not clear since the curve fitting to find the chord modulus is difficult to achieve as the stress-strain curves for conditioned samples tend to be non-linear. The results do however indicate that the chord shear modulus is typically greater than or close to that of the baseline specimens. There is a clear trend towards higher chord shear modulus values (around 7 to 8 GPa) when tested at room temperature as compared to tests carried out at 60°C (around 6 to 7 GPa).

7. Discussion

The exposure of boron-epoxy specimens to two tropical environments for a period of over 3.5 years has shown that the moisture uptake varies significantly due to seasonal variations, especially at Tindal. The dry season at Tindal runs from around May to October and has a strong effect on reducing moisture content. For the exposed case the moisture content approaches zero in some years at Tindal. AMRL-Q tends to have a more stable wet weather/humid environment and produces a greater moisture uptake in the samples (0.85% for Tindal compared to 1.07% for AMRL-Q). Specimens exposed to direct solar radiation had moisture contents about 20% lower. This is due to higher levels of solar heating causing drying of the specimens.

The moisture contents seen in the boron-epoxy material are very close to those seen for AS4/3501-6 graphite/epoxy exposed at both Tindal and AMRL-Q on a previous occasion. In this study the samples at AMRL-Q showed a moisture content of about 0.9% while those at Tindal about 0.66% [4]. This suggests that the boron-epoxy may pick up more moisture over time compared to graphite/epoxy however seasonal variations could account for this difference. It is likely that a moisture content of about 1% would represent the amount of moisture likely to enter a boron-epoxy composite in service in a tropical environment.

No significant changes in mechanical properties appear to occur when the composite absorbs 1% moisture. This was verified by mechanical testing of $\pm 45^\circ$ in-plane shear specimens. There is however a trend to slightly higher peak shear stress values when the material absorbs some moisture. This is more pronounced in the room-temperature tests (Figure 6). This is likely due to the relaxation of the residual stresses present from the cure cycle. This effect has been noted by Joshi [5] when testing carbon-epoxy composites in $\pm 45^\circ$ in-plane shear.

A reduction of about 15% in peak shear stress was noted when testing was conducted at 60°C. The results of the chord shear modulus tests do not seem to be conclusive due to difficulty in making an appropriate curve fit to the data. This was particularly difficult to do for the non-baseline samples. The results do not seem to indicate significant changes in chord shear modulus after conditioning.

The use of two-stage accelerated laboratory based conditioning was found to be an effective way of producing a 1% moisture content in the samples in a short period of time. The laboratory conditioned samples showed similar properties to those subjected to outdoor exposure suggesting that changes in the specimens exposed to the outdoor environment were due to the presence of moisture only.

8. Conclusions

The exposure of boron-epoxy composite specimens showed that the samples absorb about 0.85% (w/w) moisture at Tindal N.T and 1.07% (w/w) at AMRL-Q, Innisfail, QLD after 3.5 years. The weather patterns at Tindal are highly influenced by the tropical wet and dry seasons which see significant drying of the composite during a dry season cycle. AMRL-Q tends to produce a more consistent hot and humid environment during the entire year.

Mechanical testing in shear showed no significant contribution of the moisture in reducing the peak $\pm 45^\circ$ shear stress when tested at both room temperature and 60°C. The chord shear modulus was also determined but the measurements were prone to scatter. The results do however suggest that no significant matrix changes have occurred after 3.5 years of tropical exposure.

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A two-stage conditioning technique to produce a moisture content of about 1% was achieved in the laboratory using two humidity levels and a temperature of 70°C. This produced the desired moisture content in only six weeks for eight ply boron-epoxy specimens.

9. Acknowledgments

The author wishes to acknowledge the assistance of the following people in making this project possible. Colin Grey, AMRL-Maribyrnong for painting the exposure specimens. Peter Haggart for the meticulous strain gauging of samples. Anthony Walley and Brett Nelson for assistance in mechanical testing. Thanks also go to Ed Shum and the staff at RAAF, Tindal for carefully weighing and recording traveller coupons over the three and a half year exposure period.

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Page classification: UNCLASSIFIED

DEFENCE SCIENCE AND TECHNOLOGY ORGANISATION DOCUMENT CONTROL DATA					
				1. PRIVACY MARKING/CAVEAT (OF DOCUMENT)	
2. TITLE Environmental Exposure of Boron-Epoxy Composite Material			3. SECURITY CLASSIFICATION (FOR UNCLASSIFIED REPORTS THAT ARE LIMITED RELEASE USE (L) NEXT TO DOCUMENT CLASSIFICATION) Document (U) Title (U) Abstract (U)		
4. AUTHOR(S) Roger Vodicka			5. CORPORATE AUTHOR Aeronautical and Maritime Research Laboratory PO Box 4331 Melbourne Vic 3001 Australia		
6a. DSTO NUMBER DSTO-TN-0309		6b. AR NUMBER AR-011-602		6c. TYPE OF REPORT Technical Note	
				7. DOCUMENT DATE October 2000	
8. FILE NUMBER M1/9/820		9. TASK NUMBER AIR 98/201		10. TASK SPONSOR AIR	
				11. NO. OF PAGES 14	
				12. NO. OF REFERENCES 5	
13. URL ON THE WORLD WIDE WEB http://www.dsto.defence.gov.au/corporate/reports/DSTO-TN-0309.pdf				14. RELEASE AUTHORITY Chief, Airframes and Engines Division	
15. SECONDARY RELEASE STATEMENT OF THIS DOCUMENT <i>Approved for public release</i> OVERSEAS ENQUIRIES OUTSIDE STATED LIMITATIONS SHOULD BE REFERRED THROUGH DOCUMENT EXCHANGE, PO BOX 1500, SALISBURY, SA 5108					
16. DELIBERATE ANNOUNCEMENT No Limitations					
17. CASUAL ANNOUNCEMENT Yes					
18. DEFTEST DESCRIPTORS Composite materials, Epoxy composites, Bonded composite repairs, Environmental degradation, F/A-18 aircraft, Military aircraft					
19. ABSTRACT Boron-epoxy composite material (Textron 5521 F/4) was exposed at two tropical exposure sites within Australia for a period of about 3.5 years to evaluate the effects of outdoor exposure on mechanical properties. Specimens were exposed to three exposure types; fully exposed, covered and shaded. Moisture contents of 0.85% at Tindal N.T and 1.07% at AMRL-Q, Innisfail, QLD were found after about 3.5 years for the shaded and covered exposure conditions. Fully exposed specimens had about 20% less moisture. Mechanical tests were then performed in $\pm 45^\circ$ in-plane shear at both room temperature and 60°C to assess the effects of moisture on the matrix. The chord shear modulus was also determined in some cases. The results indicated that no significant changes in shear strength occurred at room temperature or 60°C due to outdoor exposure. The peak shear stress is about 15% lower when tested at 60°C compared to those tested at room temperature. The chord shear modulus values were subject to scatter but did not reveal any deterioration in material performance. An accelerated laboratory conditioning scheme to produce a 1% moisture level in the composite was also devised which allowed an equilibrium moisture level to be established in eight ply boron-epoxy specimens after only six weeks. This study found no evidence that absorbed moisture is likely to degrade the strength of boron-epoxy repair patches used on RAAF aircraft when tested at room temperature and 60°C.					

Page classification: UNCLASSIFIED