Use of an In-Situ Electrochemical Sensor for Predictive Assessment of Bonded Repair

Final Report

20 September 2001

Sponsored by

Defense Advanced Research Projects Agency (DARPA)

ARPA Order K475/25

Issued by U.S. Army Aviation and Missile Command Under

Contract No. DAAH01-01-C-R

Submitted by

Guy D. Davis, Ph.D.

DACCO SCI, INC.

10260 Old Columbia Road
Columbia, MD 21046
410 381-9475
410 381-9643 FAX
davis@daccosci.com
www.daccosci.com

and

Brian J. Harkless

DynCorp Technical Services, Inc.

One Ridgmar Centre
6500 West Freeway, Suite 600
Fort Worth, TX 76116-2187
brian.harkless@dyncorp.com

20010928 022

Contract Period: 6 March 2001 to 5 September 2001

Reporting Period: 6 March 2001 to 5 September 2001

The views and conclusions contained in this document are those of the authors and should not be interpreted as representing the official policies, either express or implied, of the Defense Advanced Research Projects Agency of the U.S. Government.

Approved for public release, distribution unlimited.

Executive Summary

A series of B/epoxy-Al double lap shear specimens were prepared and exposed to high humidity and temperature. Half of these were prepared using the commercial DynCorp process used for bonded repair of aircraft. The other half was prepared using a modified process to reduce durability. Electrochemical impedance spectroscopy (EIS) measurements were taken on these specimens using a patented DACCO SCI sensor. A set of these specimens was mechanically tested each month to determine pull strength. The EIS impedance spectra were analyzed using equivalent circuit analysis. Several circuit parameters exhibited changes upon exposure to moisture. Changes in the capacitance allowed moisture uptake to be calculated. By placing the sensor electrodes in different locations, variations in moisture concentration in the bonded system can be determined. This approach provides advance warning before any significant reduction of bond strength. Thus, it has the potential to monitor adhesively bonded repair patches and allow preventative maintenance prior to any degradation of bond performance.

Table of Contents

Introduction

Adhesively Bonded Repair Patches

Aircraft structures are susceptible to corrosion caused by moisture augmented by runway or marine salt, exhaust fumes and other atmospheric pollutants, oil/gasoline/diesel fuel spills, and cleaning solvents. It is estimated that corrosion costs $4 billion annually for military aircraft systems and $9 billion for commercial and private aircraft.

The useable life of existing and future aircraft is constantly increasing as projected procurement of replacement aircraft is delayed. The age of the military fleet is dramatically increasing; over 50% of the Air Force fleet is over 20 years old. New procurement of aircraft is being reduced or stretched out and these new aircraft will be expected to remain operational for longer times.

Increased maintenance and repair is required as the aircraft ages. Most aircraft have required structural repairs because of corrosion, stress corrosion cracking (SCC), low-cycle widespread fatigue damage (WFD), high-cycle fatigue, or discrete source damage (foreign body impact, handling damage, or lightning). More of these repairs will inevitably be needed over their remaining lifetimes. Predictive assessment of the structural integrity of the repaired structure is needed to increase confidence of the repair process and reliability of the air fleet.

Traditionally, bolted or riveted repairs have been used because they are relatively simple to apply and minimize the time the aircraft is out of service (for simple repairs). However, bonded repairs for aircraft structures, such as those performed by the subcontractor, DynCorp, offer many advantages over traditionally fastened repairs and significant cost reduction compared to part replacement. The patches can be applied in a reactive manner to repair cracks or corroded areas. They can also be used proactively in critical areas to reduce stress and increase time to failure.

Ample historical experience demonstrates that, when properly designed and applied, bonded repairs are as durable as conventionally fastened approaches – sometimes more so. The best-known applications are probably those implemented in Australia by AMRL (Aeronautical and Maritime Research Laboratory) in conjunction with the RAAF (Royal Australian Air Force). Airplanes with these repairs have been flying with no crack growth or patch failures for twenty years. More recently, the USAF has extended this technology and optimized a grit blast/silane surface treatment for patching C-5 and C-141 aircraft, among others.

Bonded patches can be very cost effective. Often bonded repairs can be performed in less time than conventional repairs and allow the aircraft to be returned to service more quickly. In most cases, there is no need for aircraft disassembly.

With all these advantages and experience, some aircraft maintainers are still reluctant to consider bonded doublers as permanent structural repairs, especially on critical hardware. One reason is the lack of direct, quantifiable verification of bondline quality under the doubler. Because of these limitations, the USAF's engineering authority requires a fail-save approach for bonded repair in which fatigue cracks must be nondestructively inspected at the interval required for an unrepaired defect. Because this approach calls for expensive and time-consuming inspections to be performed at half the time for the unrepaired crack to grow to critical length, bonded repair is not economically viable for cracks approaching critical length and would not even be considered for cracks at or beyond the critical length. Reliable predictive assessment of the structural integrity of a bonded patch would allow this requirement to be relaxed and permit increased use of repaired components while current practice would require replacement.

Electrochemical Sensor

DACCO SCI, INC., has developed an *in-situ* corrosion sensor that can detect the early stages of coating degradation and substrate corrosion of painted structures. Laboratory experiments indicate that it is also capable of detecting moisture ingress into a composite and an adhesive joint and bonded area. The sensor, when coupled with a portable potentiostat, is suitable for both laboratory and field inspection.

These previous results suggested that it would be applicable to monitoring the health of a repair patch. By placing a sensor directly on a patch and making contact directly to the original structure, the presence of moisture in the patch and in the adhesive between the patch and structure can be detected. The lack of moisture would assure no bond degradation from environmental attack. The presence of moisture would indicate the potential for environmental attack. Several effects of moisture are anticipated; their relative significance will depend on the materials involved. The first is a gradual weakening of the composite resin and adhesive. In most cases, this weakening will be well within the margin of safety of the patch design. The second effect is a disruption of dispersive forces across the bondline (composite-adhesive or metal-adhesive). This can lead to bond failure if improper surface treatments have been used. The third effect is hydration of the aluminum oxide surface at the interface. The change in morphology and volume of the oxide upon hydration promotes interfacial bond failure.

Delaminations can additionally be detected by comparing sensor signals over time. Thus, the early detection before degradation occurs and threatens bond integrity allows continued monitoring of the patch and an engineering decision to be made concerning the health of the existing repair and the necessity to correct any damage. Detection of delamination provides a complementary inspection of more severe damage from any cause.

Possible actions to prevent loss of structural integrity include:

- Driving off the moisture by localized, gentle heating
- Resealing the bondline edges
- Repairing/replacing the patch.

Depending on the location of the patch and other factors, a permanent sensor could be attached to the patch. This would be especially beneficial for inaccessible locations. Wires could then be run to a convenient point for connection to the portable computer/potentiostat. In other cases, a hand-held sensor could be used whenever needed. This arrangement would be important when appearance or aerodynamic considerations would prohibit attachment of a permanent sensor.

Current NDE methods measure the quality of the bondline by appearance or after local bond failure. They include visual inspection, button pull tests, tap test, ultrasonics, and infrared thermal imagery. The last three of these techniques require the bond to have already failed and separated. X-ray techniques generally require >10% loss of material before degradation is detectable. None of the present techniques is capable of detecting an adhesive bond in the early stages of deterioration. Thus, there is a great need for an inspection procedure that can monitor bondline integrity and detect the early stages of environmentally induced degradation, invariably caused by moisture intrusion into the bondline.

This new electrochemistry-based sensor represents a new approach for inspection of bonded repair patches and other bonded structures. The early detection or assessment provided by the sensor would allow the degradation to be reversed or repaired *prior* to it affecting the reliability of the aircraft and would increase confidence in bonded repair. This approach will complement existing NDE procedures and offers several advantages:

- Reduced repair costs
- Portable, low-cost instrumentation
- Suitable for inaccessible and accessible patches and other bondlines
- Detection of moisture intrusion well before any serious environmentally induced damage occurs within the bondline
- Engineering remedies developed, planned, and scheduled for condition-based maintenance
- Increased reliability and use of bonded patches
- Increased air fleet readiness and safety
- Increased lifetime of existing aircraft.

In the Phase I effort, we evaluated the effectiveness of the sensor in detecting moisture intrusion and patch degradation in boron-epoxy patches bonded to aluminum in double lap-shear configurations. Correlations between sensor measurements and exposure time or, to a lesser extent, pull strengths were made. Approximately half the specimens were prepared according to DynCorp's qualified procedures for applying repair patches to aircraft. This involves the grit blast/silane (GBS) approach mentioned earlier. Because this process is proven to be very reliable, minimal bondline degradation was expected or actually occurred in the short Phase I period. Accordingly, the other specimens were prepared without the silane treatment. These are not as durable and were expected to exhibit deterioration in the laboratory accelerated testing. Some degradation, in fact, did occur.

Results and Discussion

Eighty double lap-shear specimens were prepared by DynCorp (Figure 1). Forty of these received the standard processing for bonded repair of aircraft. The remaining forty did not have the silane treatment and were expected to exhibit poorer performance following humidity exposure. Nine of these (four for the qualified process and five for the modified process) were mechanically tested for base line strength measurements. Five additional sets of nine specimens were placed in humidity (>95%RH, 50°C) for accelerated testing. They were removed monthly and returned to DynCorp for me-

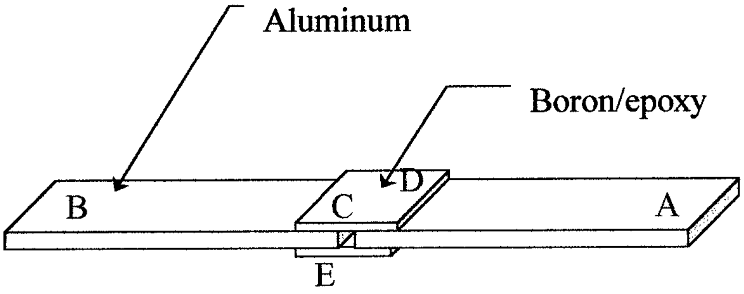


Figure 1. Schematic diagram of double lap shear specimens. The red letters indicate sensor placement.

chanical testing. The remaining specimens were reserved as spares in case they are needed and are available for early Phase II testing. They have been placed in the humidity chamber to begin accelerated aging in preparation for a Phase II program.

Prior to humidity exposure, EIS sensor measurements were taken on a selection of specimens to establish reproducibility of the measurements. Four sets of measurements were taken on each inspected specimen: aluminum-to-aluminum (A-B), boron/epoxy-to-aluminum (A-D and B-E), and same-sided boron/epoxy (C-D). (The letters correspond to locations in Figure 1.) In each case, a drop of ultrasonic couplant was used to improve the electrical contact between the electrode and the surface. It was wiped off prior to returning the specimens to the humidity chamber. Previous investigations have shown this approach to improve the signal-to-noise of the measurement. The aluminum-aluminum and same-sided boron/epoxy measurements exhibited the greatest specimen-to-specimen reproducibility as shown in Figure 2. The occasional reduction in the low-frequency impedance is believed to result from a higher conductivity path across the bondline because of inadvertent spreading of the couplant. A similar situation affects the bond/epoxy-Al measurements. Over the course of the program, greater consistency in applying the couplant was achieved and improved reproducibility was obtained so that the latest data exhibited less scatter.

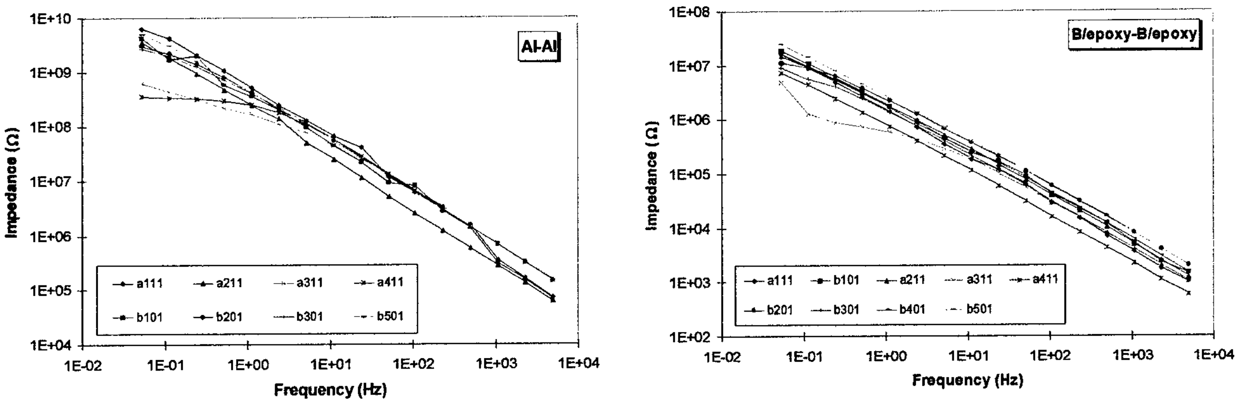


Figure 2. Impedance spectra for several specimens prior to humidity exposure. Left: Al-Al measurements. Right: same sided B/epoxy-B/epoxy measurements.

As each set of specimens was removed for mechanical testing, identical EIS measurements were taken. In addition, the final set was being tested during exposure based on the schedule: 0, 1.5, 3, 4, 6, 8, 12, 16, and 20 weeks. These data provided a longitudinal view of changes induced by moisture. The data were being modeled using the equivalent circuit of Figure 3. Previous experiments have shown that this circuit is a good model for adhesive bond data. The fit is illustrated in Figure 4. The quality of the fit suggests that this model is appropriate for the patched substrate configuration. In all but a very few anomalous spectra, the fit to the circuit was excellent.

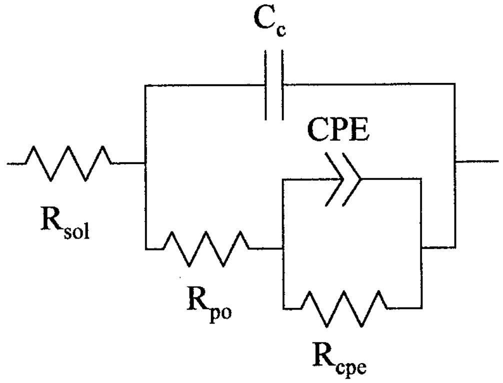


Figure 3. Equivalent circuit for boron/epoxy patched aluminum.

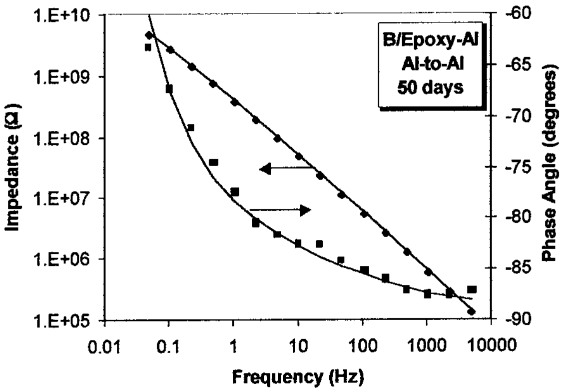


Figure 4. Example of fit using the equivalent circuit of Figure 3. The points are the data; the lines are the fit.

Upon exposure to moisture, the spectra changes. Generally the impedance decreases at both the high frequency and low frequency regions, but at different rates, as illustrated in Figure 5. The decrease at high frequencies is relatively small and is related to the increase of capacitance due to moisture absorption (see below). The decrease at low frequencies is related to decreases in the resistive elements, notably R_{cpe}, resulting from moisture increasing the conductivity of the adhesive and composite.

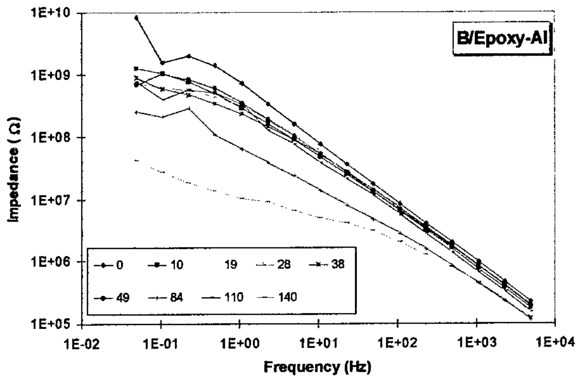


Figure 5. Impedance spectra at different exposure times.

Several circuit parameters show consistent changes with exposure time to humidity, reflecting the spectral changes of Figure 5. The capacitance values are shown in Figure 6 for the three types of measurements (Al-Al, patch-Al, and patch-patch). Note that the patch-patch values are higher than the others. We tentatively attribute this difference to the use of the ultrasonic couplant that might diffuse along the surface to couple the two electrodes. However, it is the change in capacitance, not the magnitude, that is used to determine the moisture content.

The increase of the capacitance can be explained by the increase in the dielectric constant of the composite/adhesive system due to the uptake of moisture. Analytically for a coating or adhesive, the capacitance can be expressed as

$$C = \varepsilon_0 \varepsilon A / d \qquad \textit{Equation 1}$$

where ε_0 is the permittivity of free space, ε is the dielectric constant of the coating or adhesive, d is the thickness of the adhesive, and A is the area. In the presence of moisture, this becomes

$$\mathrm{C} = \varepsilon_0 (\varepsilon_{Ad} + M(\varepsilon_W - \varepsilon_{Ad}))\mathrm{A/d} \qquad \textit{Equation 2}$$

where M is the moisture concentration, ε_{Ad} is the dielectric constant of the epoxy adhesive (3.65), ε_W is the dielectric constant of water (79.5),. The significantly larger dielectric constant of water allows very low concentrations to be readily detected. Equation 2 can be rewritten as

$$M = (C / C_0 - 1)(\varepsilon_W / \varepsilon_{Ad} - 1) \qquad \textit{Equation 3}$$

or

$$M = 74.8(C / C_0 - 1) \qquad \textit{Equation 4}$$

where C_0 is the initial capacitance before water exposure.

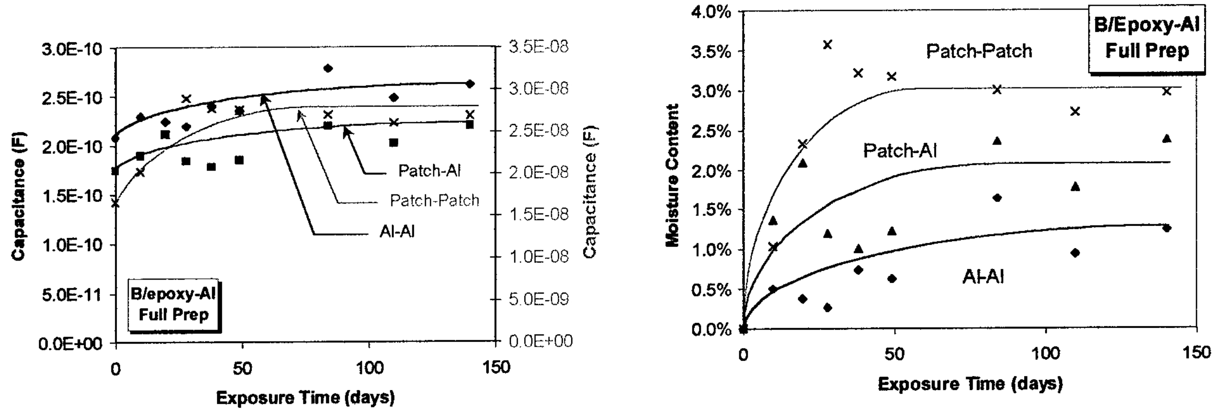


Figure 6. Left: Capacitance as a function of time for Al-Al, Patch-Al, and Patch-Patch measurements. Note the scale change for the Patch-Patch measurements. Right: Moisture concentrations calculated from the capacitance values and Equation 3.

The three different measurements give different moisture levels that likely reflect differences in moisture content in different regions of the bonded patch. The patch-patch measurements give the highest levels of moisture and saturate or level off the earliest. Since the electrodes for this measurement are placed on the surface of the composite patch, the measurements are believed to be probing the surface region of the composite. The depth of penetration or interrogation is not known. Because moisture is diffusing mainly from this surface, it is expected that the moisture level would be the highest here and saturation would occur the earliest. The patch-Al measurements interrogate the entire bond (patch plus adhesive). It would be expected to indicate a lower level of moisture and to take longer to reach saturation. This is what is observed. The interrogation volume for the aluminum-aluminum measurements is less certain for our specimen configuration. The most likely interrogation volume is the adhesive between the two Al half-specimens that are being patched. In this case, the measurement is probing the adhesive, which is expected to have the lowest moisture concentration and take the longest time for saturation. Again, this is what is observed. An alternative interrogation volume would be the entire patch system, if the current path went from Al through the adhesive to the patch, along the patch, through the adhesive to the second Al adherend. In this case, the calculated moisture level would be expected to be very similar to that of the patch-Al measurements. The clear differences between the two measurements indicate that this scenario is not likely under our measurement conditions. Thus the three different measurements suggest a moisture concentration gradient exists across the patch-adhesive system and that the sensors can qualitatively detect that difference through the use of different electrode placement. Comparisons of some of the other parameters as a function of calculated moisture concentration (see below) support the moisture gradient interpretation.

The remaining factors, CPE variables also exhibit clear trends with exposure time and moisture, but do not have analytical expressions, such as Equations 1 to 4. These are illustrated in Figure 7 and Figure 8. R_{CPE} dominates the resistive components of the circuit (Figure 3) and shows a decrease of approximately an order of magnitude for the Al-Al and Patch-Patch measurements. This behavior is very similar to those observed for coatings exposed to moisture, where resistance decreases of several orders of magnitude are often seen. This decrease in resistance is caused by moisture penetrating the pores of the polymer and increasing ionic conduction. Aside

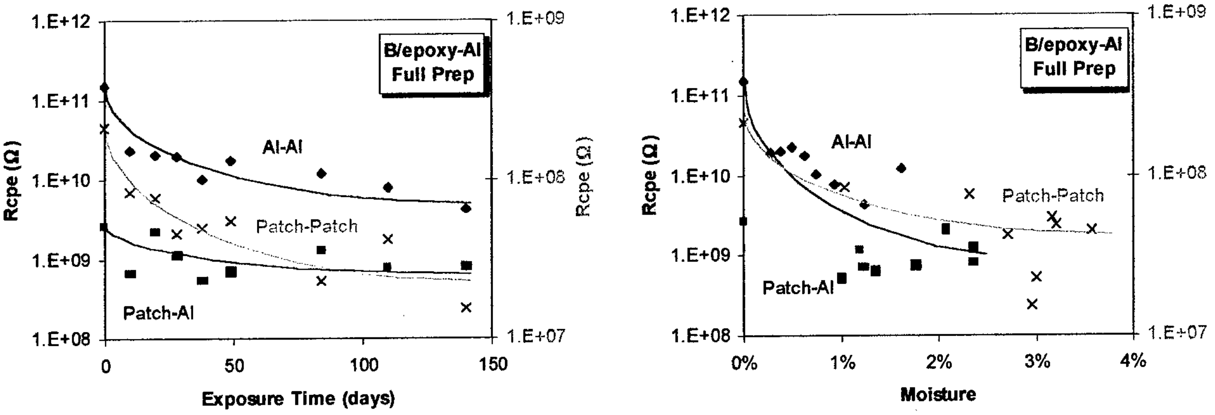


Figure 7. R_{CPE} as a function of exposure time (left) and moisture content (right). Note that the Patch-Patch values are associated with the scale to the right.

from an apparently anomalous low initial value of R_{CPE} for the Patch-Al measurements (measurements on other specimens indicate that the initial R_{CPE} values for the Patch-Al measurements shown in Figure 7 are, indeed, anomalously low), there appears to be a continuation of the R_{CPE} relationship with moisture for both the Al-Al and Patch-Al values, indicating that the adhesive properties are dominating this parameter.

A similar situation exists for *CPE* (Figure 8). There is a steady decrease of a factor of 3 with time and moisture level. Again, when plotted versus moisture, the Al-Al and Patch-Al results align except for the anomalous first point. The *CPE* is nominally an interfacial parameter. Its decrease may be indicating some reaction occurring at the interface although the silanes strengthen and stabilize the interface so that it is never the limiting region of the bond. In the case of the Patch-Patch measurement, the significance of the *CPE* parameter is less clear as the metal-adhesive interface is not being interrogated. For these measurements, it might be associated with the composite surface. The phase angle of the *CPE* also shows a trend with time and moisture. The phase angle defines the nature of the *CPE*. A phase angle of 1 makes the *CPE* a capacitor; a phase angle of 0 makes the *CPE* a resistor; and a phase angle of 05 makes the *CPE* a

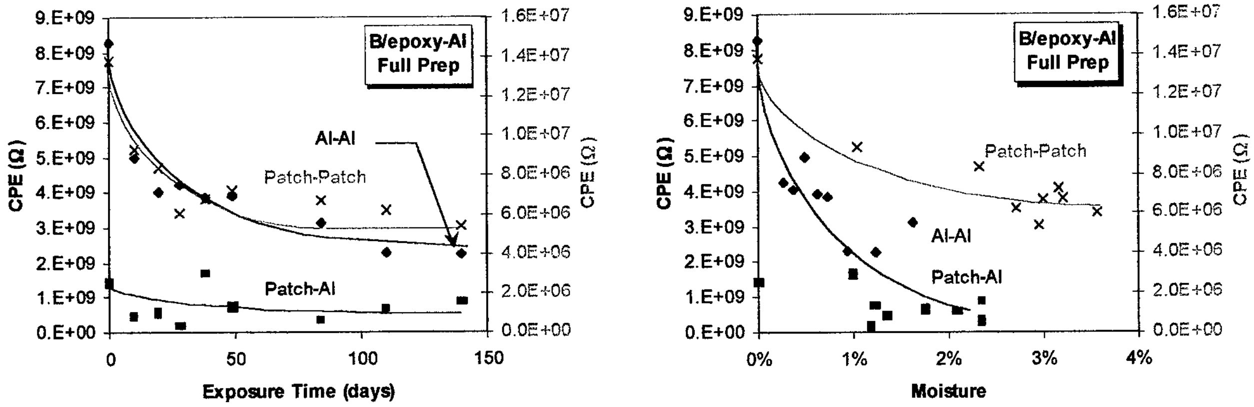


Figure 8. CPE as a function of exposure time (left) and moisture (right). Note that the Patch-Patch values are associated with the scale to the right.

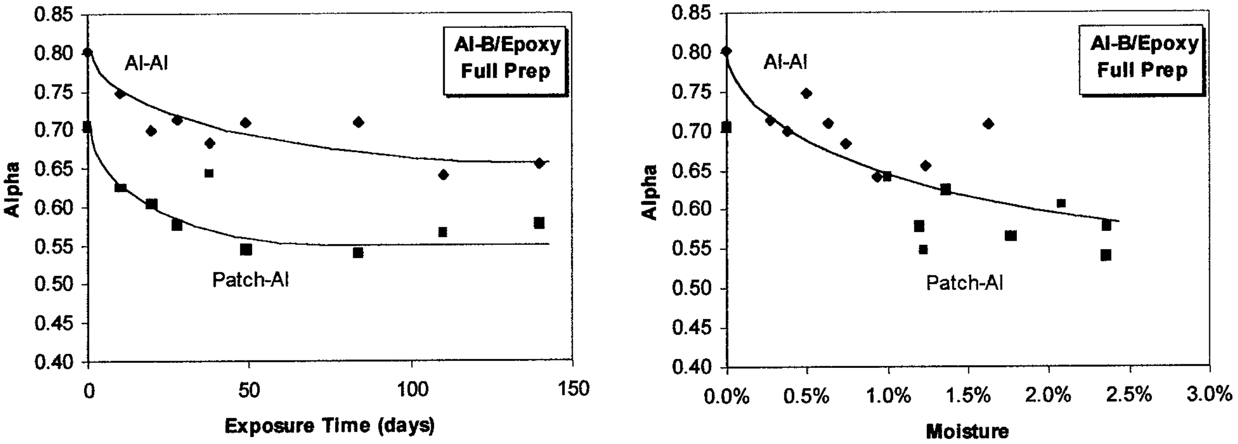
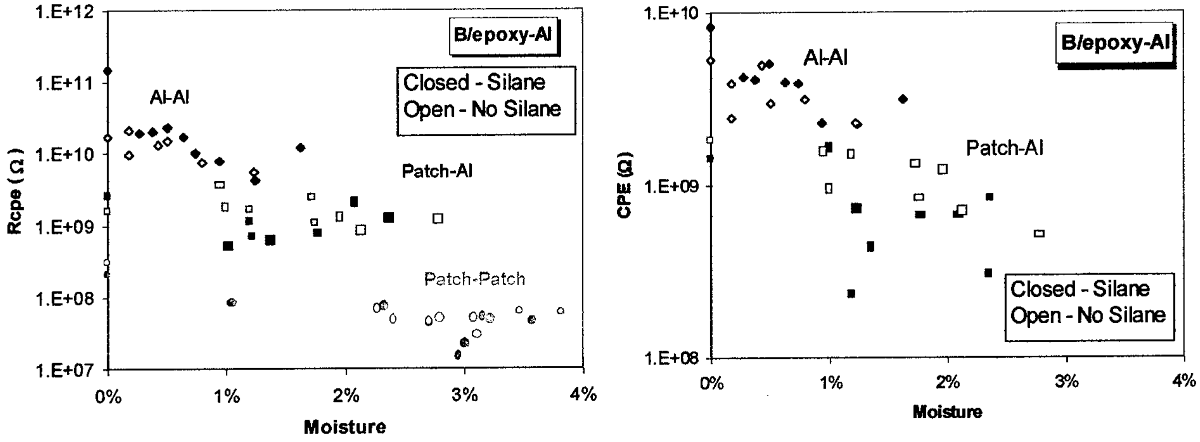


Figure 9. CPE phase angle, alpha, as a function of exposure time (left) and moisture (right).

Warburg element. The Warburg case is interesting because it indicates that diffusion processes govern interfacial or surface reactions. The phase angle of the Al-Al and Patch-Al measurements initially is close to that of a capacitor and then decreases to ~0.5 with time and moisture. Initially there is little moisture present at the metal-adhesive interface and ionic diffusion is not the limiting factor. As moisture penetrates to the interface, ionic diffusion becomes important and the phase angle decreases to that of the Warburg element. In contrast, the phase angle of the Patch-Patch measurement changes very little (0.72 to 0.78) with a slight increase with time and moisture.

Relatively little difference is seen between the EIS results for the fully prepared (silane-treated) and partially prepared (no silane) specimens as shown in Figure 10. Within the scatter of each set of data, there is no significant distinction between the two surface treatments. The resistance, R_{CPE}, is dominated by the adhesive and composite and is not expected to be dependent on the interface. Figure 10 suggests a correlation between R_{CPE} and the moisture level that holds for all three measurements although more measurements are needed, especially with low moisture content for the Patch-Patch and Patch-Al measurements using the improved data acquisition procedures to verify a correlation. Previous results on metal-metal bonds have suggested a similar relationship.

The CPE parameters (magnitude and phase angle) are nominally associated with the interface and might be expected to show a difference between the two treatments since mechanical testing (see below) shows that the metal-adhesive interface of the no-silane treatment is a limiting factor in bond performance. However, no significant difference in the two CPE parameters is seen for the two treatments (Figure 10). The reason for the lack of distinction is that no hydration or other reactions are occurring at the interface of either treatment. The interfacial failure observed for the no-silane specimens is a result of failure of physical bonds as the adhesive disengages from the grit blasted surface. It is expected that eventually hydration will occur, first for the no-silane specimens, and much later for the silane-treated surfaces. The longer time in the Phase II program and the opportunity to increase the test conditions, should allow such interfacial degradation to occur. Previous work indicates that Forest Products Laboratory (FPL)-etched aluminum surfaces that are covered with epoxy and immersed in water at the current test temperature



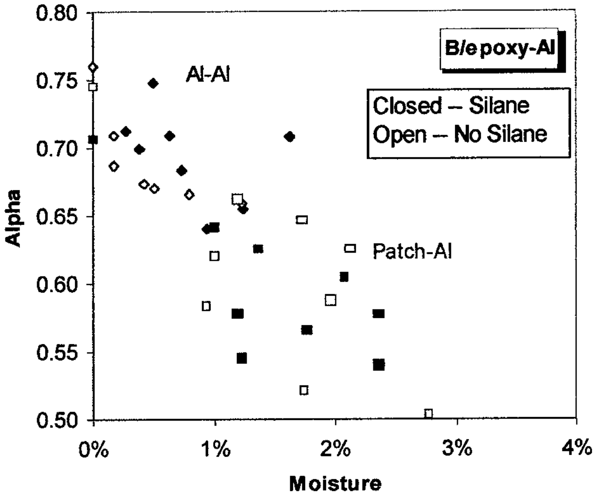


Figure 10. Comparison of silane and non-silane treated specimens. Clockwise from above: R_{CPE} for each measurement; CPE for the Al-Al and Patch-Al measurements; and Phase Angle for the Al-Al and Patch-Al measurements. Similar agreement between the silane and non-silane treatments for the CPE and Phase Angle Patch-Patch measurements were seen, but not shown.

(50°C) begin to hydrate in approximately six months. Grit blasted surfaces are expected to be comparable in hydration resistance so that hydration should be observed in the Phase II program.

Tensile pull strengths were also determined for these specimens. Figure 11 shows that the silane-treated specimens showed almost no change upon exposure. The no-silane specimens showed a 17% decrease over the same time. The silane specimens failed predominately at the composite-adhesive interface with some failure cohesive in the adhesive. There is little change in the locus of failure with time. As mentioned above, the no-silane specimens failed predominately at the metal-adhesive interface with some regions of composite-adhesive interfacial failure. Little change in the locus of failure was observed with time or bond strength. No evidence of hydration was seen on the metal surface. Rather the mechanism of failure was likely the breaking of physical bonds with the disengagement of the adhesive from the grit blasted surface.

The very small range of pull strengths make correlations of EIS parameters with strength tentative, at best. Separate correlations would be expected for the two different surface treatments based on the different mechanisms and loci of failure. Figure 12 gives examples of possible correlations for the two types of specimens. The figure suggests that R_{CPE} is very sensitive to bond strength with the resistance falling by a factor of ten with a very small decrease of bond strength. Part of the Phase II effort will include verification of these correlations over a wider range of pull strengths. As noted in the caption, the initial values for the no-silane treatment do not fall on the same line. The explanation is that the dry and moist specimens fail with different mechanisms. Although both fail predominately at the metal-adhesive interface, the difference is the presence or absence of any secondary (e.g., van der Waals) forces across the interface. Such bonds, albeit weak, contribute to the strength of the dry specimens and make disengagement of the adhesive harder. However, these bonds are very readily disrupted by even small amounts of moisture so that they do not contribute to the bond strength of humidity-exposed specimens.

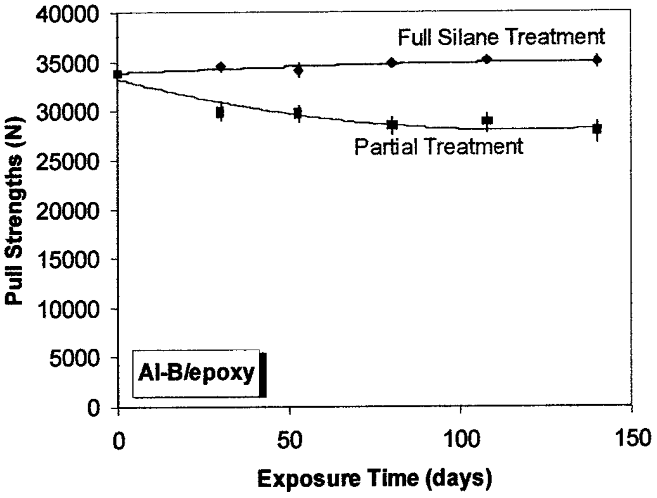


Figure 11. Lap shear pull strengths as a function of exposure time.

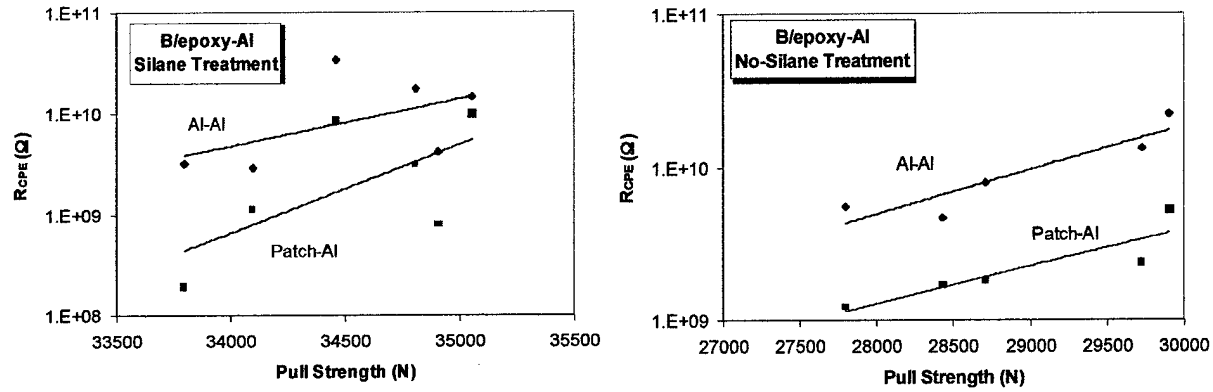


Figure 12. Correlation of R_{CPE} to bond strength for the silane-treated specimens (left) and no-silane-treated specimens (right). The no-silane results do not include the dry values, which do not correlate with the measurements following humidity exposure.

Summary and Conclusions

This Phase I SBIR program has demonstrated the feasibility of the EIS sensor to monitor the bondline of an adhesively bonded patch and track moisture ingress. Specific findings include:

- The handheld sensor allows EIS measurements of bondlines.
- Equivalent circuit modeling using the circuit of Figure 3 is appropriate for analysis of the impedance spectra.
- Several circuit parameters (C, CPE, R_{CPE}) track with moisture.

- The capacitance can be used to calculate the amount of moisture present.
- Different sensor locations probe different bondline regions.
- Relatively little dependence on surface preparation is seen to date. Interface is stable and no hydration occurred under the test conditions and duration.
- Very little change in pull strengths is seen for standard treatment; a slight decrease is seen for partial treatment. Tentative correlations between EIS parameters and bond strength were reported.

Commercialization and Other Activities

An equipment manufacturer is negotiating for an exclusive license to commercialize the EIS sensor technology. The agreement is under final review by the licensee. The license would include the patented painted, handheld, and tape sensors. Potential applications include:

- Accelerated testing of coatings in the laboratory with direct comparison to field exposure.
- Field monitoring of coated structures
- Detection of moisture in composites
- Monitoring of adhesively bonded structures.

The Air Force Research Laboratory (AFRL) is very interested in the sensor technology and is developing a small Phase III program in which they would prepare specimens similar to the ones tested in Phase I, but with their own surface preparations. We would expose the specimens to high humidity and temperature, take periodic EIS measurements, and occasionally return the specimens to them for mechanical testing.

Proposed Phase II Issues and Proposed Activities

As discussed throughout the *Results and Discussion Section*, there are several issues and challenges to be addressed in the Phase II program before the ESI sensor becomes a reliable and convenient means to monitor bonded repair patches and other adhesively bonded structures. These issues and challenges include:

- The stability of the commercial GBS interface requires long exposure for degradation. While this is very desirable from an aerospace application viewpoint, it makes it impossible to induce degradation in a short Phase I program. The longer Phase II program will help and we plan to increase the temperature slightly to accelerate degradation. We also plan to place a static load on some specimens (while being exposed to humidity) to try to accelerate degradation.
- The surface condition or wetness of a specimen can affect the EIS measurements. The use of the ultrasonic couplant improved the signal to noise ratio and facilitated data acquisition but may have also contributed to some of the data scatter. Reproducibility improved over the course of the program and an appropriate data acquisition protocol will be developed in Phase II.

- The data analysis requires a highly skilled expert. Much of the data analysis can be automated to allow a repair technician to obtain moisture levels. A data acquisition protocol will be developed.
- Separation of interface and bondline/patch properties would be desirable. Placement of electrodes in different locations (e.g., patch-patch, patch-Al, Al-Al) and circuit modeling should allow distinction of interface and bondline properties. Long enough exposure times to assure interface degradation will be needed to refine the approach and verify this distinction.
- Input for predictive assessment is needed. Moisture levels, together with time and temperature, could be used with an empirical database of hydration or other degradation or with an analytical model to predict hydration. A database of degradation of the GBS treatment (bare and epoxy-covered) will be developed similar to an existing database for FPL surfaces. Analytical expressions for CPE and R_{CPE} would also allow input to a degradation model and would facilitate separation of interface and bondline/patch properties.
- Different materials (composites, adhesives, substrates, and surface treatments) react differently to the presence of moisture and may have different EIS responses. Key materials sets can be evaluated. DynCorp uses B/epoxy, graphite/epoxy, and Al patches. Each will be tested. Some of the composite patches will have a glass/epoxy overlayer as is common practice. Some specimens will be painted to reflect actual conditions. IN addition, AFRL is interested in funding a small Phase III program to look at some of their surface preparations involving sol-gel technology.
- Once moisture is detected, preventative actions, such as sealing, may be helpful in extending a patch or bond lifetime. Such procedures would be expected to stop or slow interfacial degradation. Prioritization will be needed to determine if this task can be included in Phase II or if a separate Phase III is needed.
- Measurements on actual repair patches would be useful for demonstrations. A major advantage of this approach is its noninvasive nature. No permanent attachment of electrodes is needed. DynCorp has an F-16 section they are using for testing. It is expected to be available for evaluation of patch inspection in Phase II. Additional measurements can be made on aircraft being repaired by DynCorp to demonstrate and refine the data acquisition procedure.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
9/20/01

3. REPORT TYPE AND DATES COVERED
Final Report 6 March 2001 - 5 September 2001

4. TITLE AND SUBTITLE
Use of an In-Situ Electrochemical Sensor for Predictive Assessment of Bonded Repair

5. FUNDING NUMBERS
ARPA under K475/25
DAAH01-01-C-R

6. AUTHOR(S)
Guy D. Davis
Brian J. Harkless

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
DACCO SCI, INC.
10260 Old Columbia Rd., Columbia, MD 21046
DynCorp Technical Services, Inc.
One Ridgmar Centre, 6500 West Freeway, Suite 600
Fort Worth, TX 76116

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Defense Advanced Research Projects Agency
3701 North Fairfax Drive
Arlington, VA 222-3-1714

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

A series of B/epoxy-Al double lap shear specimens were prepared and exposed to high humidity and temperature. Half of these were prepared using the commercial DynCorp process used for bonded repair of aircraft. The other half was prepared using a modified process to reduce durability. Electrochemical impedance spectroscopy (EIS) measurements were taken on these specimens using a patented DACCO SCI sensor. A set of these specimens was mechanically tested each month to determine pull strength. The EIS impedance spectra were analyzed using equivalent circuit analysis. Several circuit parameters exhibited changes upon exposure to moisture. Changes in the capacitance allowed moisture uptake to be calculated. By placing the sensor electrodes in different locations, variations in moisture concentration in the bonded system can be determined. This approach provides advance warning before any significant reduction of bond strength. Thus, it has the potential to monitor adhesively bonded repair patches and allow preventative maintenance prior to any degradation of bond performance.

14. SUBJECT TERMS
Adhesion, Bonded Repair, Sensors, Degradation, Monitoring, Predictive Assessment, Electrochemical Impedance Spectroscopy

15. NUMBER OF PAGES
14

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	**18. SECURITY CLASSIFICATION OF THIS PAGE**	**19. SECURITY CLASSIFICATION OF ABSTRACT**	**20. LIMITATION OF ABSTRACT**
Unclassified	Unclassified	Unclassified	UL

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18
298-102