MEMS adhesive bond degradation sensor

Alan Wilson*, Z. Sbiaa, M. Hopcroft and B.C. Laskowski.

Analatom Inc., 540 Weddell Drive Suite 9, Sunnyvale, CA 94089, USA.

*Maritime Platforms Division
Aeronautical and Maritime Research Laboratory, Defence Science & Technology Organisation
GPO Box 4331, Melbourne, Victoria 3001, Australia.
*alan.wilson@dsto.defence.gov.au

ABSTRACT

The problem addressed in this paper is the through-life, non-destructive monitoring of corrosion and disbonding damages in airframes. The concept presented here is to produce a MEMS smart sensor consisting of a number of small, independent, wireless sensors within the structure of the aircraft. The MEMS smart sensor can be installed during repair and in particular when the specific platform goes through a complete tear down during the Life Extension Program (LEP). The sensors are permanently installed and can be permanently monitored and contain bond degradation sensing elements and CMOS circuits. Each sensor has an independent address and can perform measurements and communicate over a true 2-wire bus to an external interface unit. US (09/501,798) and international (PCT/US00/03308) patent applications have been lodged for this technology.

Keywords: bond degradation, MEMS, adhesive, sensor, aluminium, aluminum

1. INTRODUCTION

The adoption of adhesive bond technology has been hampered by a lack of confidence in bonded components. This is largely due to the lack of a technique for determining the strength or integrity of a bonded joint in-service. Analatom's solution to this problem is to develop an Adhesive Bond Degradation (ABD) sensor consisting of a number of small, independent, wireless sensors that can be mounted within the adhesive bond-line. These sensors are sensitive to the condition of the metal/epoxy interface which is the principle locus of failure due to environmental degradation [1-4]. A number of the ABD sensors would be included in any bonded repair or component to monitor the condition of the whole bond. The projected size of these devices ($1mm^2$) and wireless or minimal 2-wire interface will have no impact on the strength of the bonded joint that they are included in since other studies have shown that void densities of up to 17% have little impact on the strength of a bonded joint [5]. The projected area fraction taken up be the ABD sensors is less than 0.5%.

2. CONCEPT

The ADB sensors must be located within the bond-line of the adhesive joint with a minimal number of wires exiting the bond-line to ensure little impact on the bond strength by inclusion of the sensors. An adhesive bond-line can be as thin as 80µm, determined by the thickness of an internal adhesive support/spacer scrim or a non-conductive bead spacer or other material which is introduced to control the bond-line thickness. As a consequence the sensors must be fabricated to be less than 80µm thick and contain circuitry to make measurements and to communicate, in parallel with other sensors in the bond, on a minimum wire bus. This implies internal control circuitry, preferably CMOS for low power requirements, fabricated on wafers that can be back thinned to produce less than 80µm thick devices. Fig. 1 is a schematic of a smart bonded structure with an array of ABD sensors included in the bonded joint. In the case of metal bonded to metal the sensor chips have metal studs which contact both metal plates. All power and communications are through these connections so a truly wireless sensor can be implemented.

The concept can be adapted for composite plates bonded to metal and/or composite or honeycomb with the inclusion of either a) a radio frequency power and communications interface to drive the sensor and CMOS circuits or b) a two wire interface which would produce minimal disruption to the composite structure. In the latter case the sensors would be strung one after the other, connected in parallel, along the wires. A two wire interface could be implemented use flexible circuit material and is projected to be less than 50µm thick and 500µm wide. The focus here is on metal-to-metal repairs and components, bonded using thermoset epoxy film adhesives, which include a support carrier (and spacer) scrim. However, the sensors would be suitable for a wide range of adhesive systems. The sensing elements are designed to be sensitive to the changes in the local chemical environment at the metal-to-adhesive interface as the bond degrades..

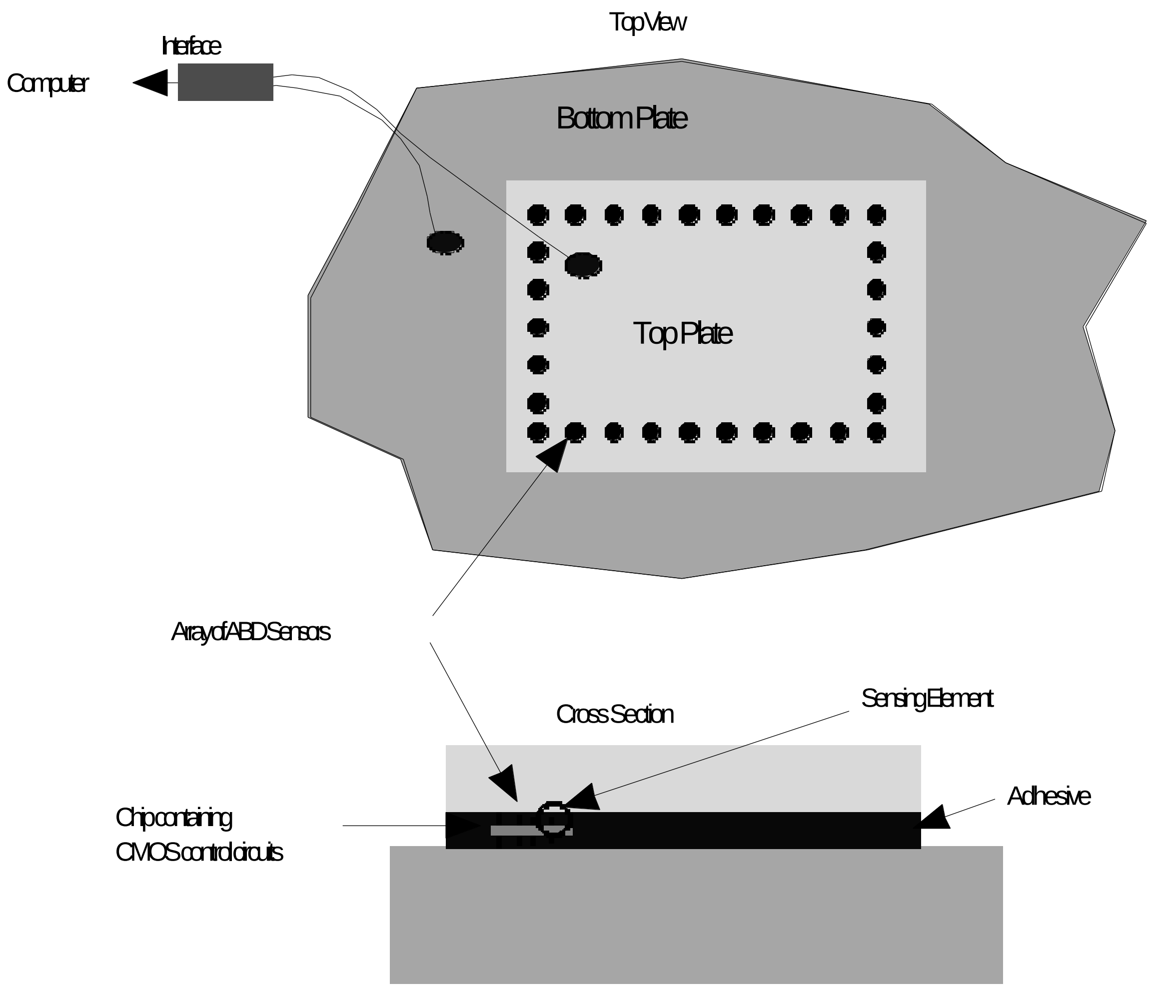

Figure 1. Example of a bonded repair with an upper metal plate (light gray) over a lower metal plate (dark gray) and an array of ABD sensors in the adhesive bond-line. In the metal/metal implementation the sensors have metal studs which contact the metal plates to make these plates the power and communications "wires".

3. ADHESIVE BOND DEGRADATION SENSOR

The Adhesive Bond Degradation (ABD) MEMS sensor contains sensing elements which measure the conductivity between the sensor and the opposing metal plate and CMOS circuitry required for the sensor to be semi-autonomous. The sensing elements consists of metal studs mounted on both the top and bottom surfaces of a silicon chip. Some of the sensing elements are in contact with the metal plates and other studs are note in contact with the plates giving the sensor two modes of operation, viz.:

- A physical sensor which detects gross disbonding and poor bonding.
- A chemical sensor which detects bond degradation prior to complete loss of bonding.

The sensor detects gross disbonding or poor bonding by loss of electrical continuity for the elements initially touching the metal plate. This loss of continuity from a particular sensor indicates the loss of bonding at this point between the upper and lower plates. Sensing elements that are not initially connected to both the top or bottom plates act as chemical ion detectors.

These elements detect the buildup of metal ions between the stud and the metal plate due to chemical activity associated with water penetration of the bond and the degradation of the metal/epoxy interface which causes the loss of bonding.

The ABD silicon chip contains CMOS circuitry for control of the environmentally sensitive probes and the communications with the outside world. Fig. 2 is a schematic showing the CMOS functions that are implemented on the sensor. Power and data are provided on the same two-wire input. Data and Clocking is input serially by voltage shifts of the power supply voltage with level detectors and digital circuits to extract Hi and Lo logic levels and separate Data and Clock information. Separated Data is clocked into a shift register. The first data word corresponds to the address of the sensor that is to be interrogated. The second data word is a command word that tells the sensor what sensing element to measure, the current to use and instructs the sensor to output the voltage measured at the sensing element as a current superimposed on the power line. Some special command words perform internal calibration actions and an overall reset action.

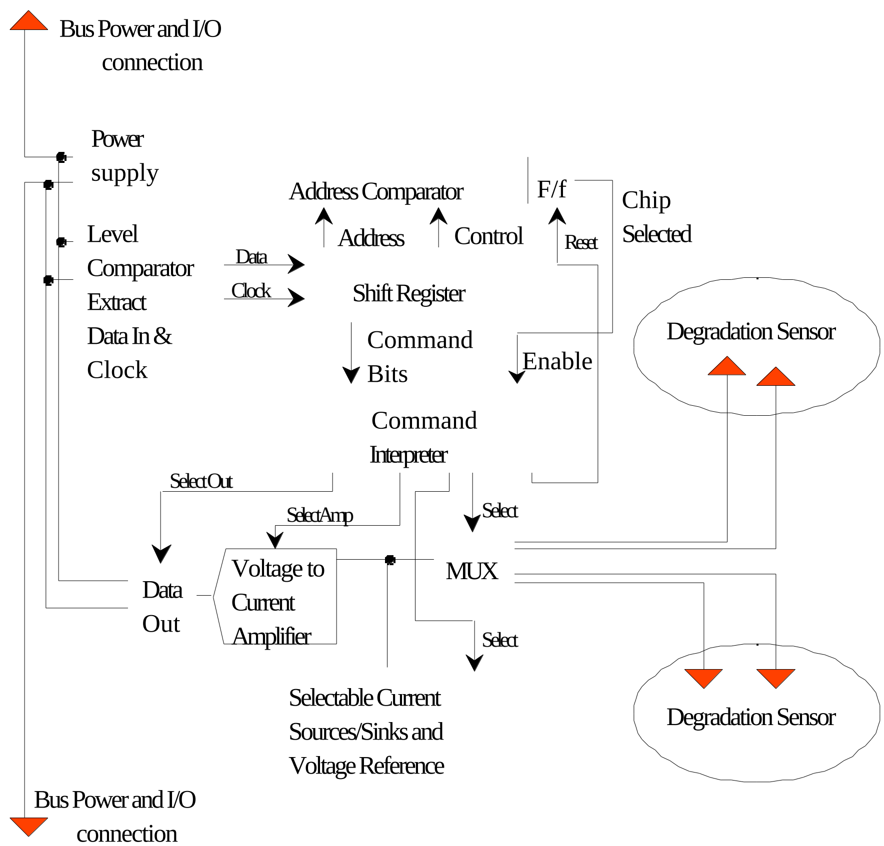

Figure 2. Schematic of CMOS circuit functions on the ABD sensor. Power and communications are provided through the Bus Power and I/O connection which are elements that contact the metal plates. One of up to 4 sensing elements for each side can be addressed and a selected current passed between the element and the plate. The voltage required to pass this current is converted to a current which is superimposed on the power and I/O bus.

The use of the Adhesive Bonding Corrosion Sensor requires the following steps:

- Initial bonding and inclusion of sensors to form a smart bonded structure.
- Interrogation of the smart bonded structure immediately after bonding using computer controlled hardware and a database to record the initial conditions of all of the sensors.
- The smart bonded structure is then interrogated by the computer controlled hardware and the database updated during regular maintenance schedules (relevant for aircraft) or continuously (if there are no weight or access

restrictions). If there is a significant change in the readings from any sensor the maintenance technicians or operators are alerted and the location of the fault specified.

4. FABRICATION PROCESS

The key to the operation of the sensor is the fabrication of the metal studs on a silicon wafer containing CMOS circuitry. The studs are made by electroplating copper onto aluminum pads fabricated on the top and bottom surfaces of a silicon wafer. Plating onto aluminum can be difficult and trial electro-plating with a cleaning and plating technique modified for CMOS computability has been performed.

4.1 Sensing Elements.

The wafers incorporated a bus-bar system which connected similar sensing element pads on each sensor to a single contact finger at the edge of the wafer. In use in a bonded joint some of the studs must be high enough to contact the metal plates and others must be shorter. Thus different currents must be supplied to different sensing element pads for the sensors on the wafer to ensure that a set of sensing elements of different heights are fabricated on each sensor. Electroplating was performed using current control with a unit specially designed to provide 6 low current sources. Cu sensing elements were electroplated onto trial wafers using the following preparation technique:

1. 35% HNO_3, for 30 seconds: cleaning.
2. Rinse and dry (distilled water and nitrogen gas dry).
3. 2% HF acid etch for 90s: this strips the Al oxide formed in the last CMOS oxygen plasma resist stripping step.
4. Rinse and dry.
5. 35% HNO_3, for 20 seconds: cleaning.
6. Thorough rinse and dry.
7. ZnO in NH_4OH, saturated solution for 5min: zincating to provide a conductive layer for the electroplating.
8. Rinse and dry.
9. 70% HNO_3, for 15s: strip first zincate.
10. Thorough rinse and dry.
11. ZnO in NH_4OH, saturated solution for 5min: second zincate step.
12. Rinse and dry.
13. Into Cu plate bath with electrodes and power connected: plated using constant current sources set to give predetermined current/unit area (maximum of 600uAmperes/mm^2).

This process is adapted form a common technique used for electroplating bulk aluminum [6]. The major difference is the use of ZnO in NH_4OH rather than NaOH since the Al being zincated here is very thin and a zincate from a NaOH solution would strip all of the Al.

4.2 Back Side Sensing Elements

The fabrication of the sensing elements on the front side of the wafer is relatively straight forward: standard Al pads produced by a normal CMOS process is all that is required. Fabrication on the backside however requires structures that will go from the front of the wafer to the back of the wafer. The process here uses Silicon On Insulator (SOI) wafers with a 20μm thick device layer, a 1μm oxide and a 500μm thick handle layer. Standard CMOS processing up to the end of the second metal is used with the exception that areas where bottom side sensing elements will be formed are clear to the bare silicon. The second metal oxide is patterned to form the mask for a tetramethyl ammonium hydroxide (TMAH) etch of the device silicon to the buried oxide layer (Fig. 4a). At this point the accumulated oxides on the back side of the wafer are also removed. After etching a low temperature oxide (LTO) is deposited to insulate the walls of the TMAH etch pit. Thick resist is then deposited and patterned for a reactive ion etch (RIE) of the buried oxide and to expose pads and connections to the second metal layer (Fig. 4b). Following the RIE a third metal layer is deposited and patterned (using thick resist again) to define the top and bottom sites for power and sensing elements. LTO is then deposited and patterned to form the top pads (Fig. 4c). The back side is then patterned with large squares in the oxides deposited by two LTO steps and Si doped or pH-controlled TMAH (which does not attach Al [7,8]) is used to etch through the handle wafer to the buried oxide layer to give a wafer with back side ribs of Si supporting squares of sensors (Fig. 4d). After this both the top and bottom sensing elements are electroplated.

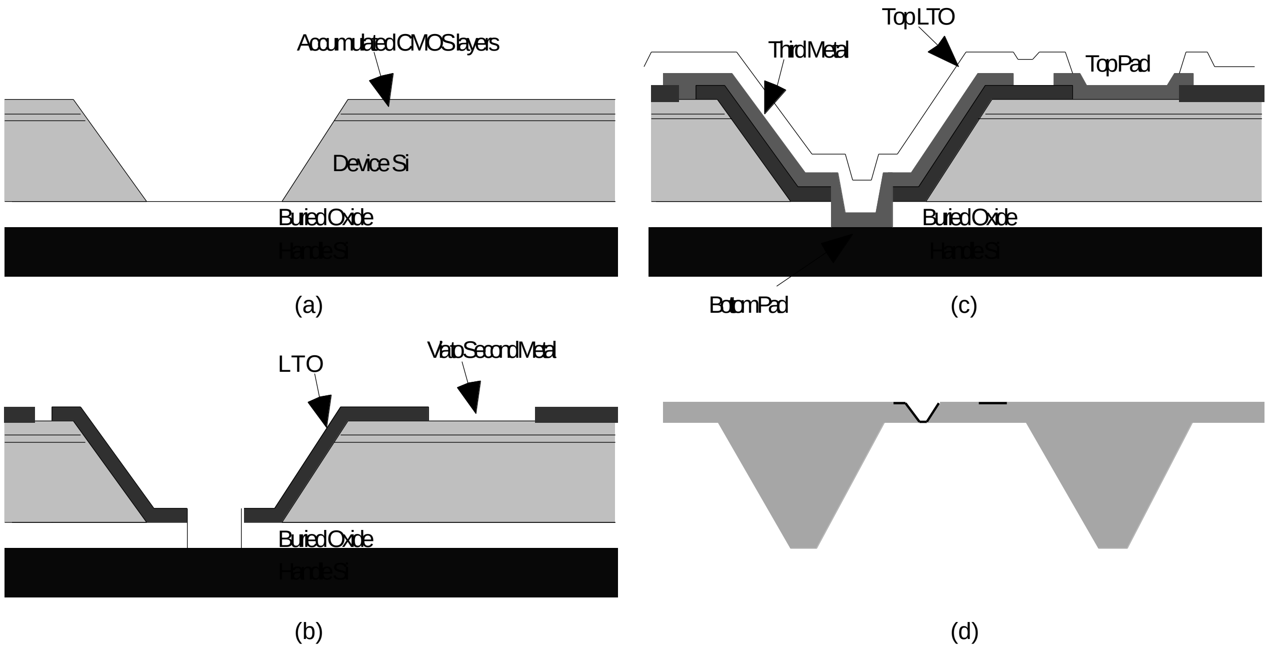

Figure 4. Schematic of processing to form the pads for electroplating the bottom (and top) sensing elements: a) TMAH etch pit in front surface, b) deposit LTO to electrically isolate the substrate and RIE etch to form Vias to the second metal layer and cut through the buried oxide, c) deposit and pattern the third metal layer and deposit and pattern the top LTO layer and d)(lower scale diagram) pattern the backside oxide and TMAH (Si doped) etch to the buried oxide layer to expose the bottom third metal layer pads.

4.3 CMOS

The CMOS circuitry was fabricated using a standard CMOS process up to the second metal deposition, i.e. all of the CMOS critical steps will be done before construction of the sensor pads. The CMOS part of the sensors are eventually projected to be 1mm x 1mm and around 20µm thick. This will provide sensors small enough to be non-intrusive within the bonding process.

5. EXPERIMENTS AND RESULTS

5.1 Adhesion of Sensing Elements

A large amount of adhesive is squeezed out of the joint during cure due to the applied external pressure. This happens at low temperatures when the adhesive is still quite viscous and the shear forces developed may be great enough to dislodge the Cu sensing elements from the Al pads on the Si. The robustness of the Cu sensing elements plated onto Al pads on a Si chip during the cure cycle of a thermoset epoxy adhesive was tested. Specimens made using the technique above were bonded between a plate of Al and a plate of glass which had another plate of Al, with a hole located over the sensor area, bonded to it as follows:

- 2 Al plates 150mm x 150mm x 3mm, prepared as per the Australian Silane treatment [3].
- 1 plate of glass 150mm x 150mm x 3mm.
- Cytec FM®300 and Cytec FM®300 U thermoset epoxy adhesive.
- Configuration: Al/FM300/Glass/FM300, FM300 U/Al.

The Cytec FM®300 had holes punched in it where the 4 test sensors were to be placed and Cytec FM®300 U (no scrim) was placed over the test sensors. The test sensors were in the middle of each edge, 15-20mm back from the edge. The sensors were placed in rebates in the bottom Al plate with plastic shims if required to ensure that the Cu studs would contact the glass. The upper plate had large holes so that the sensors could be seen during and after cure. The Cytec FM®300 was cured at 177°C for 90 minutes, with a heat up rate of 5°C/minute, in a vacuum bag, initial pressure 90mBar which fell to 60mBar during the run. After cure most of the Cu studs remained in place on the Si sensor chips (Fig. 3). A few were removed during the bonding process. This implies that the adhesion between the Cu and Al is nearly good enough but needs some improvement. Another paper in this conference [9] details work to improve the adhesion of the sensing elements.

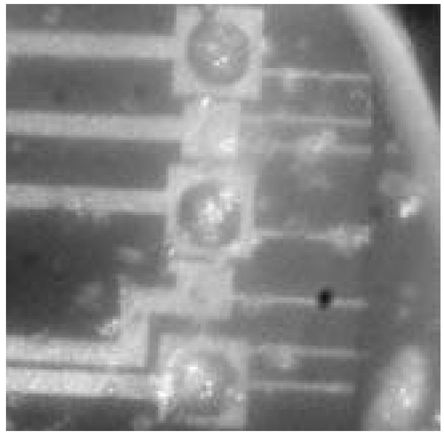

Figure 3. Image of 3 sensing studs in position after completion of the cure cycle for Cytex FM®300 epoxy film adhesive.

5.2 Response of the Sensing Elements

Sensing elements were laid up in a Boeing Wedge test (ASTM D3762-79, re-approved 1988) configuration using the Australian Silane treatment [3], placed in 50°C water and loaded at a constant slow rate [10] which ensures that the environmental degradation of the metal/epoxy interface is faster than the crack opening rate (Fig.4). The voltage required to pass +/- 100μA between the sensing element and the Al plate was measured every 4 hours after the specimen was immersed in water. The results in Fig. 5 initially show an open circuit until day 34 when the conductivity increases indicating the presence of ions at the interface. At day 36.5 the conductivity decreases until day 42 when the sensor again shows the presence of ions indicative of bond degradation due to water ingress up the metal/epoxy interface. At day 47 the conductivity is greatest and then starts to decrease a bit. This decrease is thought to be due in part to attack of the sensing element itself.

The high conductivity between days 34-36.5 and the return to lower conductivity between days 36.5-42 indicates the possible existence of two different mechanisms for producing ions at the metal/epoxy interface and/or a chemical reaction (with an approximate 2 day induction time) which then removes all ions and/or water in the process of the chemical reaction. The later could be due to the conversion of the pseudoboehmite like Al oxide/hydroxide interface to bayerite [11]. This reaction, which has an induction time of 15 hours at 60C, is a well known process that occurs to Al oxide/hydroxide surfaces in the presence of water and is considered by one of the authors to be the primary mechanism for the loss of bond integrity. Another process that is occurring in concert with any chemical degradation of the Al oxide/hydroxide is the dissolution of the silane coupling agent placed on the Al oxide/hydroxide during the pre-bond surface treatment [3]. The silane must be removed before the water can attack the Al oxide/hydroxide. One or both of these affects could result in a time when there are free ions (during silane dissolution and the induction time for the Al oxide/hydroxide conversion) leading to higher conductivity followed by a time when all available water is involved in the chemical conversion of the pseudoboehmite like Al oxide/hydroxide to bayerite leaving few free ions for conductivity. This might be the case in the water diffusion limited situation present with the bond-line with the chemical reaction proceeding faster than the diffusion of water to the reaction site. After the conversion is complete there will once again be free water to promote conductivity between the sensing element and the Al plate giving the large increase in conductivity from day 42 onwards.

The asymmetric behavior seen between the positive and negative currents (with the positive current giving higher magnitude voltages) is due to the electro-chemical formation of a (poor) copper oxide diode on the sensing element surface. The “noisy” nature of the signal is due to dynamic electro-chemical reactions, which are changing both the topologies of the sensor/Al system and the ion concentrations between the sensor and Al. More detailed studies are being performed to relate the nature and position in time along the graph above with the mechanical condition of the bonded joint.

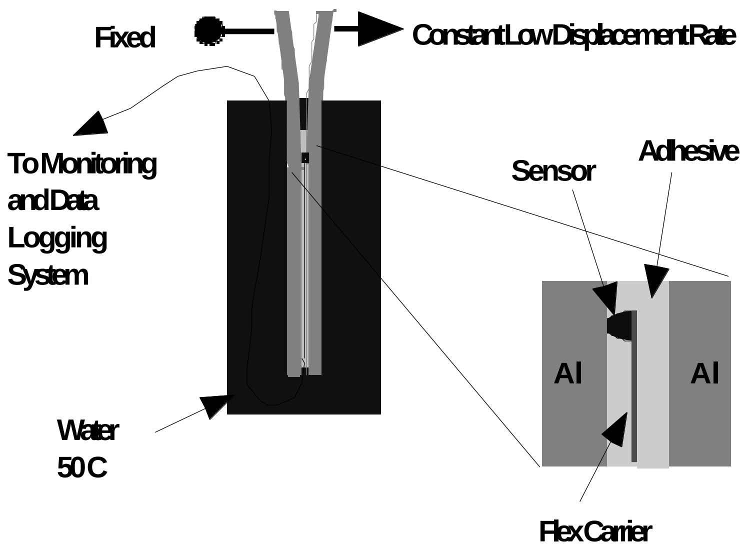

Figure 4. Lay up for accelerated ageing test of the ABD sensor.

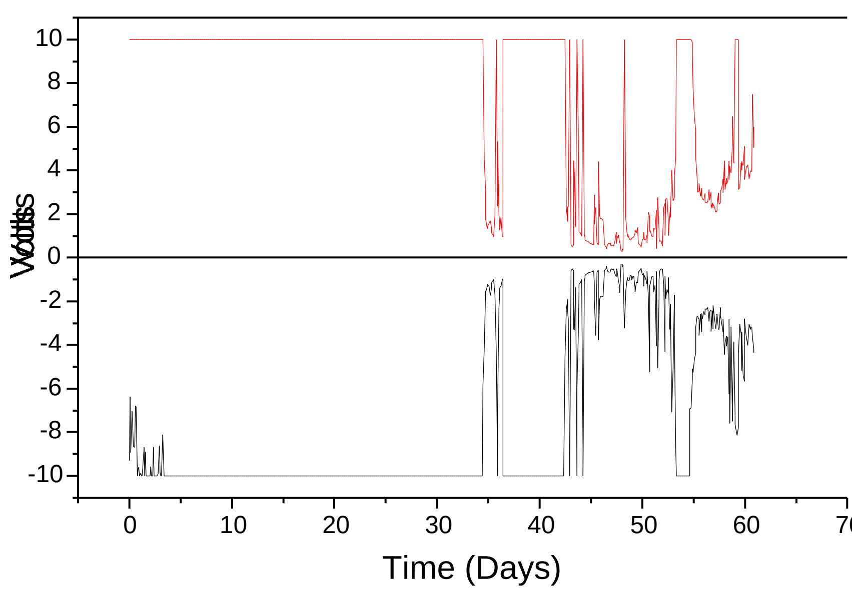

Figure 5. Bond degradation sensor signal variation with exposure to 50^{o}C water. The two data sets are the voltages required to pass +/- 100uA current through the sensing element. The +/-10V reading reflects the maximum voltage available and indicates a high impedance (undegraded) state. The noise at the start of the -100μA trace is thought to be due to the fact that the sensing element is very close to the Al.

6. CONCLUSION

The ABD sensor is sensitive to the interfacial degradation products associated with the environmental degradation of adhesively bonded components. The current fabrication process for the sensing elements gives devices which are nearly robust enough to survive the adhesive bonding lay up and curing process: other work is in hand to address this problem. The ABD sensor has the potential to be located within aircraft structure to determine the state of bonded joints and give greater confidence in the application of bonding technology to secondary and primary structure.

7. ACKNOWLEDGEMENTS

Analatom acknowledges SBIR funding provided by the US Air Force under SBIR Phase I contract number F33615-98-C-5204 and SBIR Phase II contract number F33615-99-C-5202.

8. REFERENCES

1. Arnott, D.R., Wilson, A.R., Rider, A.N., Lambrianidis, L.T. and Farr, N.G., "Studies of the Degradation of Metal-Adhesive Interfaces With Surface Analysis Techniques", App. Surface Science 70/71, (1993), 109-113.
2. Rider, A.N., Arnott, D.R., Wilson, A.R. and Vargas, O., "Influence of Simple Surface Treatments on the Durability of Bonded Aluminium Alloy Plates", Mats. Sci. Forum 189-190, (1995), 235-240.
3. Arnott, D.R., Rider, A.N., Olsson-Jacques, C.L., Lambrianidis, L.T., Wilson, A.R., Pearce, P.J., Chester, R.J., Baker, A.A. and Morris, C.E.M. "Bond Durability Performance – The Australian Silane Surface Treatment", 21st Congress of ICAS Conf. Proc., Melbourne, Australia, (1998), 13-18.
4. Rider, A.N., Olsson-Jacques, C.L. and Arnott, D.R., "Influence of Adherend Surface Preparation on Bond Durability", Surf. Interface Anal. 27, (1999), 1055-1063.
5. Pearce, P.j., Arnott, D.A., Camilleri, A., Kindermann, M.R., Mathys, G.I. and Wilson A.R. "Cause and Effect of Void Formation During Vacuum Bag Curing of Epoxy Film Adhesives", J. Adhesion Sci. Technol. 12, #6 (1998), 567-584.
6. Fred Rosebury, "Handbook of Electron Tube and Vacuum Techniques", Addision-Wesley, (1965), 25-26.
7. Tabata, O., Asahi, R., Funabashi, H., Shimaoka, K. and Sugiyama, S. "Anisotropic Etching of Silicon in TMAH Solutions", Sensors and Actuators A 34, (1992), 51-57.
8. Tabatam O. "pH-Controlled TMAH Etchants for Silicon Micromachining", Sensors and Actuators A 53, (1996), 335-339.
9. Wilson, A., Muscat, R. Jackson, I., Ollsson-Jacques, C. and Retchford, J. "Directly Electroplated Microstructure", SPIE Smart Materials and MEMS: Smart Materials, Melbourne, Australia, (2000).
10. Arnott, D.R. and Kindermann, M.R., "Durability Testing of Epoxy Adhesive Bonds", J. Adhesion 48, (1995), 101-109.
11. Alwitt, R.S. "Oxides and Oxide Films", Vol. 4, Diggle, J.W. ed, Marcel Dekker, NY, (1976), Chp. 3.