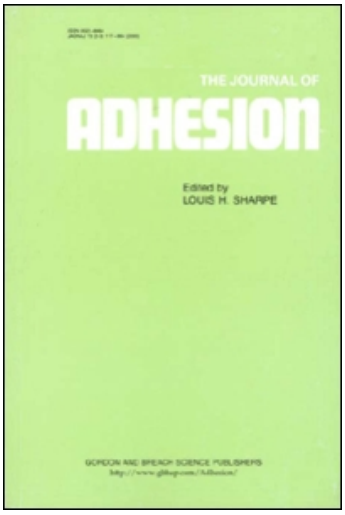

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Ultrasonic Determination of Bond Strength Due to Surface Preparation Variations in an Aluminum—To—Aluminum Adhesive Bond System

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This study demonstrates the application and use of analytical models in the experimental ultrasonic evaluation of interface conditions in an Aluminum–FM-47–Aluminum adhesive bond system. The results of the study show that a variation in bond strength due to surface preparation can be detected ultrasonically through careful inspection and signal processing analysis.

INTRODUCTION

The use of ultrasonics in the evaluation of adhesive bonds is currently limited to the detection of voids or debond areas within the bondline. Rose and Meyer[1] mention that even those bonds free of voids, have been known to fail at relatively low loads due to such bondline "flaws" as improper adhesive cure, inadequate surface preparation or various environmental conditions. Research by Zurbrick[2, 3] to correlate certain pre-bond parameters with ultimate bond strength proved inconclusive due to possible variations during the bonding process. Rose and Meyer[1] mention that hopefully the ultimate strength of an adhesive bond could be related to some ultrasonic NDT parameter determined during inspection of the completed bond. In a recent paper by Meyer and Rose,[4] physical bond models were developed, and results of the ultrasonic echo computations indicated that

small changes in the ultrasonic reflections may occur with small variations in the characteristics of the adhesive properties or interfacial characteristics. It was shown, however, that these differences may not be noticed unless careful comparison techniques are employed.

The purpose of this paper is to demonstrate one application of the analytical model results to the experimental evaluation of a particular adhesive bond system. The results of this study have shown that a variation in bond strength due to surface preparation can be detected using currently available ultrasonics equipment and careful experimental inspection and evaluation procedures.

SPECIMEN FABRICATION

Fifty step-lap test specimens, shown in Figure 1, were manufactured from 2024 aluminum flat stock. Substrates were machined, individually measured and paired so that a predetermined joint gap could be obtained. The bondline thickness of the individual finished substrate pairs was checked by placing the finished specimens on a flat surface and measuring the gap with a thickness gauge. The thickness of each substrate at the bonding surface was also measured at this time. After the specimens were bonded, these values and the overall thickness of the specimen at the bond would give an indication of the

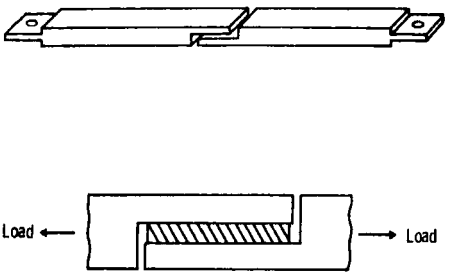

FIGURE 1 Step-lap joint test specimen.

bondline thickness. The bond surface of the substrates was milled to produce a smooth flat bonding surface having an area of 1 square inch. All specimens were manufactured to produce 0.010 inch bondlines and were to be used to study the detectability of the substrate surface preparation in completed bonds using ultrasonics.

For the purpose of this paper, it was decided to vary the substrate surface preparation, in an attempt to produce a variation in the interfacial bond strength which could be detected ultrasonically as indicated by the models

presented by Rose and Meyer.[4] Therefore, prior to the actual bonding operation, the bonding surface of each substrate was subjected to one of two prescribed cleaning operations depending whether or not the specimen was to represent adequate surface preparation. Half of the specimen substrates were treated using the substrate cleaning procedure that is recommended by the adhesive manufacturer and is listed below.

1) First wipe the bonding surface with a clean cloth soaked in acetone to remove any remaining oil from the machining operation.

2) Rinse the specimens in water at 145°F for a period of 10 minutes.

3) Spray rinse the bond surface checking the cleanliness by the "water break free test". If the surface is not yet sufficiently clean, the water will not spread on the surface but will bead as on wax paper. If the surface is clean, the water will spread over a large area.

4) Within 30 minutes, immerse the substrate bond surface in a chromic sulphuric acid solution at 145°F for a period of 10–13 minutes.

5) Repeat step 3.

6) Rinse in cold deionized water for 4–6 minutes.

7) Within 30 minutes, force dry the substrate in a vented oven at 145°F.

The remaining specimen substrates were treated using a second cleaning procedure which is used when it is desired to simulate a bond system having inadequate surface preparation. The procedure is similar to that described above except that the chromic-sulphuric acid etch is eliminated.

After the substrates are chemically cleaned, the bond surface is then coated with a primer to minimize contamination and oxidation of the surface prior to bonding. The primer is air dried at room temperature for 2 hours. This procedure is repeated until a primer thickness of 0.001 to 0.003 inches is obtained. A piece of the film adhesive is then cut to size and placed on the primed surface of one of the substrates which is then placed in an oven at approximately 225°F to allow attachment of the film to the primer and also allow partial curing of the primer. At this point, the substrates are then stored until the bonding operation.

The actual bonding procedure used for assembly of the specimens was that recommended by the adhesive manufacturer as is discussed below.

1) A vacuum is drawn in the sealed bag containing the specimens.

2) While maintaining the vacuum the specimens are heated until the minimum temperature in the adhesive is 280°F and the maximum temperature has not been above 300°F for more than 10 minutes.

3) At this time, nitrogen is added to the autoclave to bring the ambient pressure to 85 psi producing a total effective bonding pressure of 100 psi.

4) The specimens are heated until the minimum temperature in the adhesive is 300°F.

5) The 300°F, 100 psi condition is maintained for a period of 30 minutes.

6) While maintaining 100 psi, the temperature is reduced to 210°F.

7) The pressure is released and the specimens are allowed to cool uniformly.

In order to minimize effects in the completed bonds due to cure conditions, specimens using both types of surface preparation were included in each group to be bonded.

ULTRASONIC INSPECTION OF TEST SPECIMENS

The equipment used for the inspection of the test specimens is shown schematically in Figure 2. An Aerotech UTA–2 pulser-receiver-gate is used to apply the electrical pulse to the transducer. The test was initiated with a 1/4 inch 10 MHz broadband focused transducer but this was replaced after the purchase of a 1/4 inch 20 MHz broadband focused transducer which was used for inspecting the remaining specimens in the test sequence. Reflections from the bond are received by the transducer and amplified by the receiver and the gated portion of the reflection is displayed on both a Hewlett–Packard oscilloscope and a Hewlett–Packard Spectrum Analyzer. Both the oscilloscope

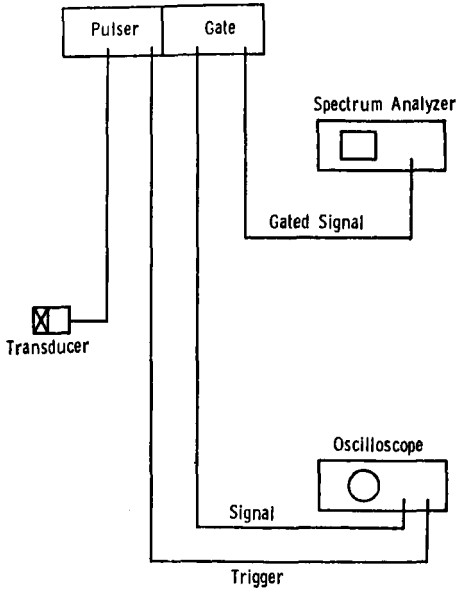

FIGURE 2 Block diagram of test equipment.

and the spectrum analyzer have screen mounted Polaroid cameras so that permanent records of each specimen's reflection characteristics can be maintained.

The specimens were inspected using an immersion tank to minimize coupling problems. The transducer was placed 1 inch above the surface of the specimen. The transducer was first adjusted for beam perpendicularity with the substrate-adhesive interface by adjusting for maximum echo amplitude of the upper substrate-adhesive interface echo. It was found that a slightly more sensitive method of determining beam perpendicularity was to observe the spectral depressions on the spectrum analyzer. These appear to be most distinct when the beam is normal to the bond interface. The transducer was then moved over the bond to determine if the echo amplitude varied with position. Nonuniform bond specimens would not be suitable for this test sequence due to the necessity of weighing each amplitude according to its location. Once these conditions were met, the amplitude-time and amplitude-frequency profiles were photographically recorded for analysis. The gate had been adjusted to encompass the entire echo train reflected from the bondline. The first thirty-three specimens were ultrasonically inspected only once. The remaining seventeen specimens were reinspected several times prior to destructive testing to obtain an indication of the consistency of the results. This reinspection is discussed in more detail later in the paper.

DESTRUCTIVE TEST PROCEDURE

The bond specimens were loaded to failure on an Instron tensile testing machine. A constant displacement rate of 0.01 in./min was used. The total load was recorded as a function of time on a strip chart recorder. Those specimens subjected to the recommended chemical surface preparation usually failed cohesively at the middle of the bondline leaving adhesive and portions of the scrim cloth over the entire bond area of each substrate. Those specimens given the inferior chemical preparation failed adhesively showing a clearly defined separation between the adhesive and one or both substrates.

RESULTS

The results of the analysis by Rose and Meyer[4] indicate that a variation in the interfacial condition of the bond is most noticeable ultrasonically by an amplitude change of the interfacial echo. As the quality of the interfacial bonding decreases, the amplitude of the reflection from the adhesive-substrate

interface should increase. The interfacial characteristics will also affect the Fourier spectrum of the bondline echo train. However, since the spectrum additionally indicates internal variations in the bondline, analysis in the time domain was chosen. The experimental bond specimens, however, were manufactured and ultrasonically inspected in groups and it was found that the interfacial echo amplitude range varied considerably from group to group although the variation within each group was relatively small. The ultrasonic inspection systems which are presently available are manufactured for the purpose of flaw detection but not necessarily flaw characterization. Most systems contain uncalibrated amplifiers, which, in addition, may be non-linear. It is very difficult, therefore, to use such a system at different times without the results varying to some degree. It is for these reasons that only half of the specimens in each group received the recommended surface preparation. To account for this variation between groups, the data groups were scaled so that the center of each group had the same value. This would allow comparison of all the data without changing the relative variation within each group. As the quality of interfacial bonding decreases, the amplitude of the reflection from the adhesive-substrate interface should increase. Figures 3a and 3b show the failure load of the specimens as a function of the average peak-to-peak echo amplitude from the adhesive-substrate interfaces. The data from the individual groups were scaled by a constant to account for the variation in the inspection system. As predicted by the Surface Preparation bond model discussed by Meyer and Rose[4] specimens with the inferior surface treatment produced a larger interfacial echo from the interface than those subjected to the recommended process. The specimens also failed at a load substantially below those properly prepared and the failure was totally adhesive in nature. It can also be seen that the use of the newly acquired 20 MHz transducer did, in most cases, enhance the predicted amplitude difference between the weak and strong specimens.

Certain specimens were inspected ultrasonically several times over a period of a few weeks in an attempt to notice time effects in the inspection process. In analyzing these data, the first data set usually produced the most pronounced indication of a variation in surface preparation. Thereafter, each data set appeared to be less indicative of the type of specimen being inspected. One possible reason for this phenomenon is the absorption of moisture by the adhesive during the inspection process. It is hypothesized that those specimens having the poorer surface preparation contain microscopic unbond points uniformly distributed over the adhesive substrate interface. The magnitude or extent of these interfacial flaws would decrease the wave coupling between the substrate and the adhesive as well as reduce the overall strength of the bond. If, however, moisture penetrated the adhesive during the first inspection

of the specimens, filling some of the very small voids, the wave coupling would be increased without the strength being substantially affected. The moisture absorption theory is corroborated to some extent by Walton and Cowling[5] who show that moisture migration through the adhesive to the sub-

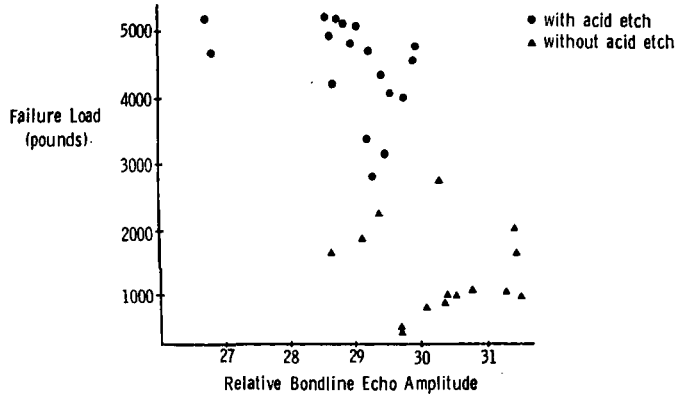

FIGURE 3a Correlation of bond failure load with bondline echo amplitude (for specimens inspected with a 10 MHz transducer).

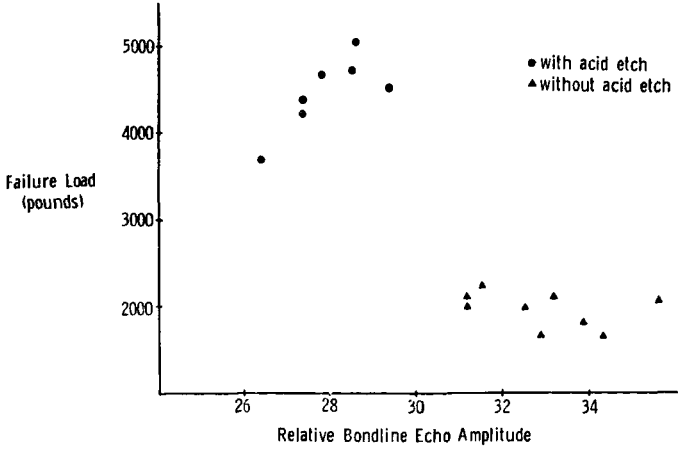

FIGURE 3b Correlation of bond failure load with bondline echo amplitude (for specimens inspected with a 20 MHz transducer).

strate interface is one of the major causes of bond failure after environmental exposure. It has also been shown by Cagle[6] that inferior bonds tend to degrade more rapidly than good quality bonds. The hypothesized porosity at the

interface would allow the rapid penetration of moisture to the interface with the subsequent formation of a weak hydrate layer causing the ultimate failure of the bond.

CONCLUDING REMARKS

The work conducted in this study demonstrates the value of the analytical models developed in Ref. 4 as a guide in experimental work. It has been shown that bond strength due to a particular variation in surface preparation can, for a particular adhesive system, be detected by a careful signal amplitude measurement. Within each of the two groups, however—the properly prepared specimens and the "poor quality" specimens—there is a high degree of data dispersion. Several factors did restrict the results to a qualitative nature. In this study, the experimental inspection of the bondline was limited to an area approximately 1/2 inch square in the center of the joint due to reflections from the substrate edges. A scan of this area was used to determine the uniformity of the bond. However, an analysis of the stress distribution at the bondline by Erdogan and Ratwani,[7] indicates that this is the region of the lowest stress concentration. In a uniform bond, failure would initiate at the ends and propagate towards the center. Any variations in the interfacial characteristics of the bond near the ends of the joint, however, could vary this distribution and severely alter the ultimate strength without being detected in this study. In addition, the form of the stress distribution is dependent on the mechanical properties of the adhesive. Therefore, possible internal adhesive anomalies such as inadequate cure could vary the ultimate load even in the case of an interfacial failure.

Efforts should also be made to manufacture ultrasonic inspection equipment to specifications suitable for research requirements. This, in itself, could eliminate the need to restrict data comparison to those results obtained during one data acquisition session.

Future work should also be directed toward the development of high speed data acquisition and analysis systems which will allow the inspection of the bond at several points, especially near the ends. The strength evaluation at each point should be weighed in accordance with its position and the expected stress distribution for the bond.

A mini-computer data analysis system could be very useful in automating an ultrasonic inspection. For instance, by evaluating the frequency content of the system output pulse prior to inspection initiation, a compensation routine could be used to determine the reflection of a "white noise" input.

With these advancements in the state-of-the-art, ultrasonic inspection can become a reliable quantitative nondestructive test technique for the determination of bond strength in the not too distant future.

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