G. A. Alers Science Center, Rockwell International Thousand Oaks, California 91360

The most important aspect of measuring the strength of adhesion at an adhesive to metal interface is to understand the chemistry of the interface and to know what makes a good interface. The existence of a strong interface will make the joint less susceptible to fabrication defects that are going to be there no matter how carefully the joint is prepared.

It is also pretty obvious that we cannot use very sophisticated laboratory tools such as superconducting tunneling that we have just heard about on an airplane wing in the field. Our problem is to relate the kind of measurements made on the microscopic scale in the laboratory to the kind of measurements that can be made in the field. This paper is an attempt to bridge the gap between current ultrasonic techniques, which we have heard a little bit about already and some newer more sophisticated and sensitive ultrasonic techniques that may open up an avenue for bringing the microscopic chemistry of the interface out into a nondestructive test performed on macroscopic parts.

Our problem then is to find an ultrasonic test that is going to look at that tiny molecular dimension at the joint between the metal, its oxide, and the adhesive. It is obviously a difficult problem because we're working with a layer that is many times smaller than the wave length of our probing sound wave. Last year we tried some experiments directed at the interaction of very thin layers with sound waves, and the results were not too encouraging for the simple geometry of sending the sound waves perpendicular to the interface as many of the conventional experiments are done nowadays. That is probably to be expected because the sound wave does not have much time or distance in which to interact with the interface. Therefore, the idea in this year's program was to send the sound wave parallel to the interface so that it could run along for some distance and interact with the defects at the interface and accumulate an effect that could be measured. This immediately became a complicated problem because it involved mathematical analysis of the modes of propagation in a sandwich structure. It involved developing the experimental techniques to observe these modes and it also involved the preparation of specimens that would have variable adhesive strength at the joint between the adhesive and the metal while maintaining a constant cohesive strength.

In order to attack these many problems cooperative work with a lot of people was demanded. Dick Elsley and his computer helped immeasurably when it became apparent that we wanted to observe some special modes of vibration of the total panel and his computer was there waiting to go and answer those questions and to guide the experiments along a very easy path. Chris Fortunko, who has just recently joined our laboratory, provided electromagnetic non-contact transducers for exciting the surface waves of high purity. These new devices were already assembled and easy to use so they greatly simplified our experiments in exciting sound waves in the adhesive layer. Of course, Tennison Smith, working from the very beginning of the program, prepared some specimens that had poor adhesion between the adhesive and the metal and strong cohesive strength so we could test our modes to see if they would, indeed, tell the difference between high and low quality of adhesion.

Our basic experiment is to excite propagating modes of vibration that travel in the adhesive between the two pieces of metal being bonded together. Since the wave velocity is slow in the adhesive, one can imagine that when these trapped waves try to get out of the adhesive they are refracted back in by the higher wave speed in the metal. Thus, the energy is trapped in the adhesive layer. We shall seek to measure some property of the acoustic wave such as the velocity of propagation, although it would pay us to keep track of the attenuation of the wave also. It would appear to be a simple project to make some adhesive-metal sandwiches and then excite some trapped waves in the adhesive. However, there are a great many modes, and conventional transducers readily excite all of them. Therefore, our first task was to attack the problem of calculating the trapped modes and then to look for regions in the mode spectrum that might be particularly sensitive to the boundary conditions.

The first figure shows the experimental configuration we used. It is a simple lap shear specimen onto which we attached electromagnetic, noncontact surface wave transducers to excite and detect a surface wave on the metal that sticks out from the adhesive. By launching a surface wave into the adhesive from the metal tab, it was possible to excite one of the trapped modes in the adhesive layer which came out on the right-hand side where a receiver transducer detected it. It is a simple experiment also because the geometry of specimen allows one to test the strength of the bond in shear by pulling on the tabs. The electromagnetic transducer was important because conventional surface wave transducers are wedge type transducers which allow a lot of other modes to get excited, making a mess out of the signals that emerge from the bond line.

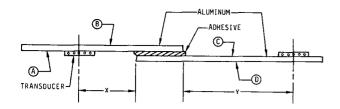


Figure 1. Typical lap shear specimen for mechanical testing of adhesive bond strength with electromagnetic transducers in place for exciting and detecting surface waves on surfaces A and C.

We were able to excite very nice clean surface waves at postion A and to pick them up with another transducer on the right side near the Letter D after going through the adhesive bond. Figure 2 shows the kind of signal we detected and is a photograph of the oscilloscope trace from the receiving transducer. That little triangular pulse labeled A is the signal that was excited as a surface wave on the left-hand side of our sample, went through the adhesive and came out the other side. By plotting the arrival time of this signal as a function of the separation between the two transducers, a straight line is obtained whose intercept corresponds to the time that the sound waves spent in the adhesive. From this amount of time and a knowledge of how long the adhesive is. it is easy to deduce the velocity of sound in the adhesive. Unfortunately, there are a lot of problems with this kind of measurement. First, it is not that accurate a technique and we had to use a different transducer every time we wanted to change the frequency. Thus, in order to measure velocity as a function of frequency and thereby examine some different modes, we had to use a series of specially built transducers. Furthermore, when we probed around the surface of the entire sample, we found acoustic energy all over the place. The adhesive bond not only converted the incident surface wave into a trapped mode, but it produced bulk modes in the aluminum that transferred energy to the other surfaces. It would be interesting to measure this mode conversion process since it might be a way of looking at the adhesive bond, but our objective was to look at the adhesive layer with trapped modes so we discontinued these experiments.

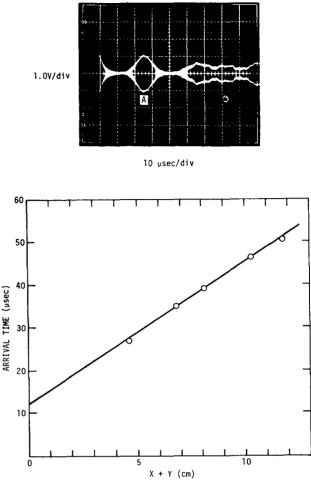


Figure 2. Oscilloscope trace photograph showing the surface wave signal A detected on surface C of the adhesively bonded structure shown in Fig. 1. The time of arrival of this signal varies as a function of transducer separation distance X + Y in Fig. 1.

Because so many modes are possible, it is important to consider the problem theoretically by asking which modes of motion can propagate along the adhesive layer parallel to the metal plates and which are most sensitive to the boundary conditions at the metal to adhesive interface. The method that we had available at the Science Center was a computer program that Bruce Thompson had prepared with the help of Dick Cohen for calculating the properties of acoustic waves in layered media. This program was modified by putting in different boundary conditions at the adhesive to metal interface and recalculating the propagation velocity of the modes of a simple three-layered sandwich consisting of two aluminum layers separated by an adhesive layer. Of course the conventional boundary condition of continuous stress and continuous displacement across the interface was used as a standard of comparison. There are a lot of dif-ferent kinds of boundary conditions one could choose. The one we chose was to put a step in displacement at the boundary whose magnitude was proportional to the local stress. You can rationalize this choice of boundary condition if you imagine that there is a very thin layer of very compliant material at the interface.

By inserting a value for the compliance of the thin layer and recalculating all the propagating modes, we were able to locate modes and frequencies where there were big effects in the velocity of propagation due to the boundary conditions. For the delight of the physicists present in the audience, Fig. 3 is the ωk diagram (the frequency versus wave number graph) for the modes of motion in which the energy is confined to the adhesive. As you can see, it is complicated and shows many possible modes. Thus, it is not surprising that our surface wave experiments showed the excitation of other modes all over our sample. I would like to call your attention to k=0 where a lot of those curves come in and hit the frequency axis at a finite value of frequency. These frequencies correspond to the standing wave modes in the thickness dimension of the total sandwich. They are not propagating modes parallel to the direction of the adhesive layer because they have infinite wave They actually correspond to the standing lengths. wave modes propagating normal to the surface and are the resonances that we saw in Scott's paper this morning and we will see more this afternoon. By recalculating this whole set of curves for a different set of boundary conditions, we looked to see where the curves were shifted the most; that is, frequencies and k vectors that were more sensitive to the boundaries.

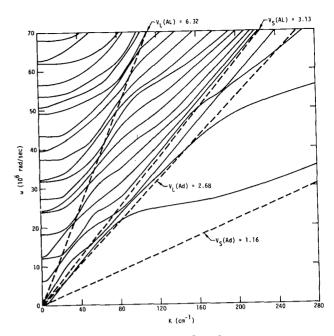


Figure 3. Curves of the angular frequency versus wave number for the acoustic wave modes that are trapped in the adhesive layer between aluminum plates.

Figure 4 is a graph of the percentage change in phase velocity of that first antisymmetric mode produced by a fixed change in boundary conditions. It shows that for a particular change in boundary conditions, there is a big effect in phase velocity right around 4 megacycles. By calculating the phase velocity versus frequency curve for this mode, as is shown in Fig. 5, along with some of the other modes, we found that the first antisymmetric mode starts at zero velocity at zero frequency, goes up over a peak and falls back down to a low velocity at high frequency.

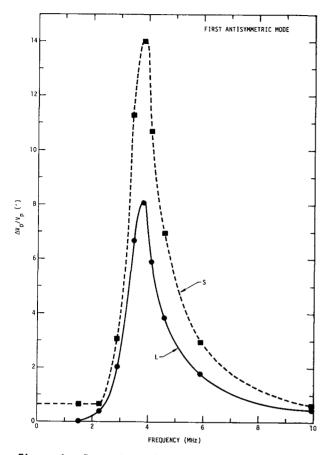


Figure 4. Percentage change in propagation velocity of the first antisymmetric trapped mode produced by insertion of a very thin layer of material at the interface having a small shear stiffness (curve S) or a small compressional stiffness (curve L).

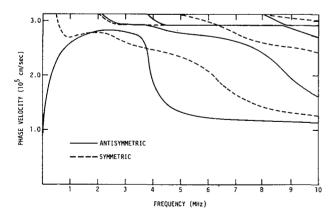


Figure 5. Curves of the phase velocity versus frequency for the trapped modes that propagate along the adhesive in an aluminum-adhesive aluminum sandwich.

At very low frequencies it is the flexure mode of the total sandwich. At intermediate frequencies where the wave length becomes comparable to the sandwich thickness, the velocity approaches the wave velocity of a surface wave on aluminum. Near 3 or 4 megacycles the velocity plunges downward to describe a shear wave confined to the adhesive and propagating with the shear wave velocity in the adhesive independent of the presence of the aluminum boundaries. Note that the transition from a wave that feels the effects of aluminum to one dominated by the adhesive occurs near 4 megacycles and that is where we have shown unusual sensitivity to the interface.

Based on these theoretical arguments, it is obvious that our experiment must be to try and excite this particular first antisymmetric mode where the curve is changing rapidly near 3 1/2 or 4 megahertz. This would require the design of special transducers for each frequency and probably imbedding them in the adhesive to avoid mode conversion at the edges of the adhesive layer when exciting the mode by external surface waves. At this point, Bruce Thompson suggested that by imbedding a single wire in the adhesive and by driving it with an alternating current in the presence of a strong magnetic field, we would have a broad band transducer imbedded in the adhesive and could excite it to any frequency we chose. Three wires were placed in the adhesive as shown in Fig. 6, and the whole thing was put in a magnetic field. When an alternating current was driven through the wire farthest on the left, it mechanically vibrated and excited mechanical waves in the adhesive. When these waves arrived at the wire farther down the sandwich, they moved it in the static magnetic field and thus generated an electrical signal which could be observed on an oscilloscope. Since only a single wire is involved, there is no particular frequency associated with it and one should be able to tune the frequency to any desired value. This concept worked just beautifully, as is shown in the oscilloscope signal photographs in Fig. 6. These pictures show the received, pulse signals at receiver 1 and at receiver 2. It can easily be seen that when the wave pulse is close to the transmitter, it is one burst, but after it has run a short distance the effects of dispersion and different modes appear, to split that burst up into three separate, broader signals. Thus it is clear that the broad band, single wire transducers have excited three modes in this experiment, and we could measure their velocities of propagation from a knowledge of the distance between the wires and the time delay. Unfortunately, the clean, large amplitude signals shown in Fig. 6 could only be obtained at one particular frequency. It turned out that our wire was acting as a little resonator consisting of the wire mass imbedded in a compliant adhesive with the adhesive acting as a spring. Good sensitivity was observed only when the vibrational resonance of that wire was excited. Thus when we tried to operate at the 4 megahertz that we wanted to use, all we saw was the tail end of the 2 megahertz resonant vibrations of the wires.

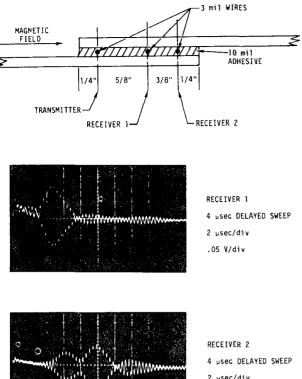




Figure 6. Geometrical configuration used to excite the trapped modes of the adhesive layer by a current carrying wire embedded in the adhesive in a magnetic field. The photographs show tone bursts picked up at the two receiver wires after launching a tone burst acoustic signal from the transmitter wire.

By adjusting the mass of the wires it should be possible to put the resonant frequency near 4 megahertz, but searching for the correct wire would be time consuming and could easily go beyond the scope of the current phase of the program. Instead, it was decided to return to the theory and look for other modes which would be sensitive to the boundary conditions. This examination of the theory showed that whenever the stress distribution of the waves within the adhesive put a maximum stress on the interface, then a maximum in sensitivity to the boundary conditions occurred. In particular, the thickness vibration of the whole sandwich (the lowest frequency k=0 intercept on Fig. 3) was found to be stressing the interface the most. This mode represents the two pieces of aluminum moving as rigid bodies with the adhesive acting as a spring, and occurs for usual bond geometries around 1/2 a megahertz to 1/4 of a megahertz. For the kind of specimens we were working with, it was actually down at 1/3 of a megahertz, which was somewhat lower than our available transducers. By making the aluminum sheets 1/16 inch thick instead of 1/8 inch, the standing wave frequency could be moved up to a half a megahertz where we had a transducer. Pulse-echo measurements of the reflectivity of the specimen

in a water bath with this transducer directing waves at normal incidence on the bonded sandwich showed a minimum in reflectivity at the frequency of this mode. Such measurements were easily performed by Dick Elsley and his computer who took the Fourier transform of the echo signal. Figure 7 shows the dip in reflectivity associated with the thickness resonance as it is observed superimposed on the response curve of the transducer.

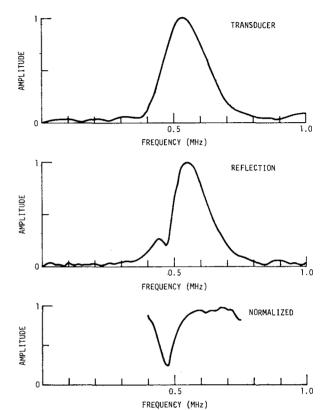


Figure 7. Fourier transform of the pulse signal reflected from a block of aluminum to define the transducer band pass response and from the adhesive bond sandwich to define its frequency of minimum reflectivity. The bottom curve is the ratio of the top two curves.

In order to observe a correlation with strength, eight lap shear specimens were prepared to have different adhesive strengths but a constant cohesive strength. These samples were constructed by Dr. Tennison Smith who used four different surface preparations on the aluminum to reduce the strength of adhesion. Wires were also imbedded in the adhesive so that data on the wave velocities of the propagating modes and the Q of the wire resonances could be recorded for possible correlation with the specimen strength.

Tables I and II show the results of the correlation obtained between the strengths of the eight lap shear specimens having different surface preparations, and the various measurable quantities that can be obtained from the standing wave resonances and the imbedded wire transducers. Table I shows the strengths and the measurements from the imbedded wires which are the velocities of propagation of the different waves as deduced from the time of arrival of the pulses; the frequencies of the wires vibrating inside the adhesive; and the Q of that resonance of the wire. The subscripts || and 1 denote the orientation of the external magnetic field relative to the adhesive bond plane. The resonant frequency of the thickness mode is shown in Table II along with the depth of the minimum in reflectivity. Since this resonant frequency depends upon the "spring constant" of the adhesive which in turn depends on the thickness of the bond, all frequencies have been

normalized to a uniform bond line thickness, and this is shown in the column labeled corrected. Examination of the observed strengths of the eight specimens showed that we succeeded in producing two distinct groups of different strength in spite of our attempt to achieve four different levels of strength. Four specimens exhibited strengths in the 2400 to 2500 psi range, and four of them were very weak having strengths in the 1500 to 1800 psi strengths. The weak samples showed a completely adhesive failure because they broke nicely right along the interface.

- Table I. Mechanical and ultrasonic properties of adhesive bond specimens deduced from embedded wires. The subscripts || and \perp refer to the direction of the magnetic field relative to the bond plane. V is the group velocity of a trapped mode pulse, f is the resonant frequency of the transmitter wire,

 - Q is the mechanical quality factor of the wire resonance.

Specimen	Surface Treatment	Bond Line Thickness mils	Failure Stress psi	V 10 ⁵ cm/sec	V ₁ 10 ⁵ cm/sec	f _H MHz	f }⊥ MHz	f _∥ /f⊥	Q _{II}	q_{\perp}
G-2	Degrease	10.1	1550	1.4	1.7	2.61	1.59	1.64	3.3	3.9
AR-1	As Rec'd.	10.1	1783	2.2	2.2	2.48	1.53	1.62	3.8	3.4
G-1	Degrease	10.8	1790	1.7	2.0	2.53	1.52	1.66	3.5	4.0
AR-2	As Rec'd.	9.8	1843	2.2	2.0	2.51	1.52	1.65	3.5	3.4
FM2	Monolayer	9.3	2440	2.0	2.3	2.5	1.69	1.50	3.4	3.7
FM1	Monolayer	9.5	2460	1.4	1.8	2.55	1.69	1.51	3.4	3.8
F2	FPL Etch	9.5	2476	1.9	3.0	2.46	1.75	1.40	3.7	4.5
Fl	FPL Etch	10.3	2533	3.0	2.4	2.50	1.58	1.58	3.8	4.4

Table II. Mechanical and ultrasonic properties of adhesive bond specimens deduced from fundamental thickness vibration mode.

Specimen	Surface Treatment	Bond Line Thickness mils	Failure Stress psi	Resonant Measured KHz	Frequency Corrected KHz	Depth of Minimum <u>%</u>
G-2	Degrease	10.1	1550	449	467	27
AR-1	As Rec'd.	10.1	1783	449	467	28
G-1	Degrease	10.8	1790	429	458	15
AR-2	As Rec'd.	9.8	1843	439	451	0.6
FM2	Monolayer	9.3	2440	469	469	17
FM1	Monolayer	9.5	2460	454	474	4
F2	FPL Etch	9.5	2476	454	472	27
Fl	FPL Etch	10.3	2533	468	474	13

The tables show that the resonant frequency of the thickness mode correlated with the strength in that for the weak bonds the resonant frequencies ran between 469 and 475. Thus there was a good 10 KHz difference between the groupings of the two resonant frequencies measured, and the resonant frequencies separated nicely according to whether it was a good bond or a bad bond. The resonant frequencies of the wires imbedded in the epoxy also correlated or fell into groups that corresponded to the actual strength measurements. Again, the differences were rather small, but there was a systematic trend in the data that was clear. The other quantities, the Q values and the wave velocities, did not seem to show any correlation. Our tentative conclusion, based on two sets of four specimens having different strengths, is that the low frequency resonance which vibrates the total sandwich seems to reflect the quality of the interface. This quantity is a property that could be easily measured with conventional ultrasonic techniques. We do not rule out the use of propagating waves, and we will proceed to try some more sophisticated methods of exciting these waves. Hopefully, next year we will be able to report some better statistical correlation between the propagating mode and the strength of the bond.

DISCUSSION

PROF. MAX WILLIAMS: There is not time for discussion, but the chair will entertain any questions of fact for Dr. Alers as far as the details or questions that were not clear. Are there any questions of fact?

DR. JOSEPH HEYMAN (NASA, Langley): What was the thickness of the adhesives in this?

DR. ALERS: 10 mils.