Cohesive Strength Prediction of Adhesive Joints

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Cohesive Strength Prediction of Adhesive Joints

Abstract
My part of this program was to investigate a nondestructive method for measuring the cohesive strength properties of adhesively bonded joints.

I started this problem by viewing a reasonably fundamental study in modeling the adhesive as a layer between two infinite aluminum adherends. You can set up wave potentials in these different regions in terms of reflected and transmitted waves, as is seen in Fig. 1. If you set up your wave number in a very classical manner, where everything is elastic and there is no damping, you just get a regular Rayleigh-type solution. If you desire to include the damping of the adhesive layer, then you must have a complex wave number. The appropriate relationship was derived by Brekhovshikh. We took his relationships and did parametric studies on the bond line in terms of the acoustic properties of the adhesive.

Keywords
nondestructive testing, nondestructive evaluation

Disciplines
Materials Science and Engineering | Structures and Materials
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Figure 1. Model of Three-Region Laminate Treated in Analytical Calculation

Figure 2 shows the type of theoretical spectrum you get when reflected intensity is plotted against frequency in megahertz. The top curve is for the case where you don't have any attenuation in the adhesive layer. It is a simple Rayleigh solution. Then we have successive plots of bond lines with attenuation coefficients of 10, 20, and 30 Neper/cm. You will notice that as the attenuation coefficient is increased, the resonances tend to become both broader and shallower for this .0254 cm thick bondline.

We set up our code so it would scan the information that it produced and give the magnitudes of the depths of the resonances, the separations, and the quality factors of the resonances.

Figure 2. Theoretical Spectra for a 0.0254 on Adhesive Layer Between Aluminum Adherends

One of the terms that can be measured using Fourier transform spectroscopy is the resonance quality, or Q, which is defined as the resonance frequency divided by the half-power bandwidth. It's an easily measured term to characterize the shape of the resonance. However, in our parametric study we saw that with reasonable attenuations in the bond line, such as you get at the higher frequencies where the resonances occur, you end up really flattening out these curves as seen in Fig. 3. So, you don't really have very much sensitivity.

Figure 3. Theoretical Relationship Found Between Resonance Quality and Acoustic Impedance as a Function of Attenuation
In order to look at other types of measurables you can get from an adhesive bond line, we looked at the returned signal from the top aluminum-adhesive interface, seen in Fig. 4 as \( R_2 \), which is inverted because of the impedance mismatch, and the return signal from the bottom of the adhesive layer, \( R_3 \). We took these amplitudes in the time base signal and extracted one measurable, \( R_3/R_2 \), which will be referred to as the Amplitude Ratio of the bond-line.

From very simple energy partitioning relationships with a superimposed attenuation term, you can make a plot of how amplitude ratio varies with acoustic impedance and then plot this for different amounts of attenuation in the bond line, as seen in Fig. 5. Again, the zero attenuation case returns the highest signals and increasing attenuation drives the ratio to lower values.

Tennison Smith made some specimens for us that were one eighth of an inch thick aluminum adherends with a one-inch overlap. These specimens were made with Chemlok 304 adhesive, and the surfaces were properly prepared. The only variation between the specimens was the mixture of the two components of the Chemlok 304 adhesive. Mixing was done over the range of 2 parts A to 3 parts B, to 3 parts A to 1 part B. These were cured under the proper cure cycle to give different cohesive properties.

We investigated these from an ultrasonic standpoint with a pulse that is seen in Fig. 6. This was a 15 MHz highly damped pulse. We examined the laminates with a normal incidence compressional wave in the pulse echo made. We had the capability of digitizing the signal that was returned from the bond line, transforming it to get its bond line spectrum, Fig. 7. These resonances correspond to standing waves in the adhesive layer, with the resonance separation given by the velocity of sound divided by twice the thickness. On these curves, you can look at the resonance quality, the resonance separation and the resonance depth.
Figure 7. Reflected Spectrum of Adhesive Bondline

You notice that as you increase in frequency you have a higher and higher damping which gives you less intense resonances and also broadens them. So, in order to compensate for this factor, I used the tangents to the maxima in measuring the resonance depths.

Using the resonance separation, we were able to extract the velocity of sound by measuring the thickness of the adhesive layer. Figure 8 shows how the amplitude ratio varied with the velocity of sound. The first set of specimens all fell inside the dark lines. These received a cure at 93°C for an hour. The second set of specimens received another cure, 121°C for 20 minutes, a higher temperature cure for a shorter amount of time. They fell above the data of the first set. This separation in data according to cure will follow on through the rest of the correlations, but will be rectified at the end.

From the amplitude ratio data we were also able to extract the attenuation coefficient of the bond line at the frequency of the transducer, which was 15 MHz. The calculated attenuation coefficients were reasonably high, in the range of about 10 nepers per centimeter to about 30 nepers per centimeter. For those of you who aren't used to looking at things in terms of nepers, a neper is about 8.6 db. So, these adhesives are relatively highly attenuating polymers.

When the attenuation coefficient was plotted against sound velocity, Fig. 9, we had a very nice relationship among the first set of specimens, but the specimens cured at the higher temperature fell below the trend.

Figure 8. Experimental Relationship Between Amplitude Ratio and Adhesive Sound Velocity for Chemlok 304 Adhesive Specimens

Figure 9. Experimental Relationship between attenuation coefficient and sound velocity for Chemlok 304 Adhesive

When we finished nondestructively inspecting these specimens, we strength tested them, and we got a very nice correlation between ultrasonic amplitude ratio and the maximum load sustained by the specimens before failure as seen in Fig. 10. You notice by comparing Figs. 10 and 11 that the ultrasonic amplitude ratio was largely determined by the attenuation coefficient. The alternately cured specimens did not deviate from the correlation that was set up by the rest of the specimens.

We also saw a correlation between attenuation coefficient and strength, where the two sets of points in the upper left are the alternately cured material which had much lower attenuation than the initial material.
Figure 10. Experimental Relationship Between Ultrasonic Amplitude Ratio and Bond Strength in Chemlok 304 Adhesive

In Fig. 12, we see the relationship that was found between the strength of the adhesive joints and the velocity of sound that was measured, and here the alternate cure ones do fall a little bit above the spread of the other data.

Considering the strength correlations seen in the time domain, we tried to look at the quality of the resonances that we measured experimentally. We did not see a correlation, and that was as originally thought from the type of relationship we had found between the velocity of sound and the attenuation coefficient.

Figure 11. Experimental Relationship Between Attenuation Coefficient and Bond Strength for Chemlok 304 Adhesive

To search for a meaningful parameter in the frequency domain, I took my analytically prepared code and substituted into it the experimentally derived acoustic parameters of the different adhesive mixtures. The results of this study are seen in Fig. 13. You notice that the widths of the resonances don't vary appreciably. The only thing that really is very striking is the depth of the resonances. The top curve in each box is the total amount of energy emanating from the adhesive layer, the center curve is the reflected spectrum and the bottom curve is the transmitted spectrum. The upper left box is the 2 to 3 mixture which was very soft and compliant and highly attenuating. The lower right box is the 2 to 1 mixture which was hard and had a low attenuation coefficient.

Figure 12. Experimental Relationship Between Adhesive Velocity of Sound and Bond Strength for Chemlok 304 Adhesive

In Fig. 13, we see the theoretical spectra obtained using experimentally determined adhesive properties.

Seeing this relationship in the analytical work, we went back and took a closer look at some of the spectra we had experimentally generated on
these specimens. I measured the resonance depth and it correlated very nicely with the strengths of the specimens. Figure 14 shows the specimen strength plotted against the depth of the first standing wave resonance in the bondlines. Some of this scatter is due to the fact that the thickness of the specimens varied a little and the velocity of sound varied a little, so you end up having the resonances at different frequencies. And the attenuation, which is largely responsible for the resonance depth, varies quite strongly with frequency. We also looked at the depth of the second resonance, and we got a pronounced knee in the curve, as seen in Fig. 15.

In order to rectify the disparity seen between the strength and velocity of sound, we went back and looked at the stress/strain curves (actually, load/displacement curves) of the adhesively bonded joints, as seen in Fig. 16. We looked at the slope of the curves as being an indication of the stiffness of the adhesive material. Because you can draw a valid relationship between velocity of sound and stiffness, we made the correlation shown in Fig. 17. In this case, the alternately cured specimens did not vary from the scatter band set up by the initial specimen set. And if you plot the joint strength versus joint stiffness, Fig. 18, you see that the alternately cured specimen did separate out very nicely under those terms.
Figure 17. Experimental Relationship Between Joint Stiffness and Adhesive Sound Velocity for Chemlok 304 Adhesive Specimens

Figure 18. Experimental Relationship Between Joint Strength and Stiffness for Chemlok 304 Adhesive Specimens

I also looked at the failure surfaces generating by these specimens. The initial specimen set is represented by the three fracture surfaces on the left, and the higher temperature-shorter time cure specimens are represented by the two on the right in Fig. 19. The fracture surfaces are arranged, one side above the other, to show both sides of the fracture surface.

Figure 19. Fracture Surfaces of Single Overlap Specimens

The 2/3:1 mixture was a very soft, compliant, highly attenuating adhesive, and had a definite interfacial type of failure, even though the surfaces were properly prepared. We had an interfacial failure just because the adhesive was not mixed properly and thus did not have the right chemistry to enable it to cure properly and set up the right interfacial strength. The intermediate specimens, 1 1/3:1 and 1/1 in each cure set showed almost a purely cohesive failure. The adhesive ended up stuck to both of the adherends and the fracture path was directly down the center. In the higher strength materials they were not much stiffer but were stronger and had lower attenuation coefficients. The fracture started at the leading edge of the overlap in a cohesive manner as indicated by the light color, and the final fracture event was an adhesive failure as marked by the dark area in the center for both these specimens.

So, in conclusion, I would have to say that the basic finding was that the strength was most directly correlated to the attenuation coefficient of the adhesive layer, and the stiffness was most directly related to the velocity of sound.

And finally, the only thing that really keeps this analysis from practical application is the necessity of measuring the bondline thickness to extract the velocity of sound and the attenuation coefficient. This is because the sound velocity and attenuation coefficient are paired with the bondline thickness. This is true even in the analytical analysis, as was seen in Fig. 7. One hope lies in the very nice correlation seen between the attenuation coefficient and the velocity of sound in this adhesive material. If you can fully characterize this relationship and use it in your analysis, then that provides you with the extra known that relieves you from the necessity of measuring the bondline thickness in order to be able to extract the material properties of the adhesive layer and predict the strength of the adhesive bond.

Thank you.
REFERENCES


DISCUSSION

PROF. MAX WILLIAMS (Pittsburgh University): Dr. Flynn has succeeded in finishing before the bell.

DR. TENNISON SMITH (Science Center): I just wondered if in your analysis you take into consideration roughness effects?

DR. PAUL FLYNN (General Dynamics): No, I don't. You mean roughness of the---

DR. SMITH: No, I don't. I modeled the interfaces between the adhesive and the aluminum in a classical sense in that the pressures matched their displacements matched. They're much the same as most of the routine wave mechanics analysis.

MR. DAVE KAELBLE (Science Center): Paul, in your model studies, do you take into account the fact that the attenuation actually varies with frequency?

DR. FLYNN: No, I didn't. I can, but I didn't in this case.

MR. KAELBLE: It's not difficult to do.

DR. FLYNN: No, it's not at all. The way those curves were generated, you set up your reflection coefficient in terms of frequency and then iterate the frequency to plot out a spectrum, and while you're iterating, you can also change the attenuation coefficient with frequency. It plots out and draws an envelope depending on the relationship between the frequency and the attenuation coefficient just like the experimental analysis I did.

DR. BILL BASCOM (Naval Research Laboratory): Since Tennison Smith's lap shear joints have become a standard for this conference, I wonder if he could tell us what the general composition of the adhesive is which might explain something about the heavy damping that you saw.

DR. FLYNN: I can answer part of that question. The adhesive used was the same adhesive that was used in the other part of the program. It's Chemlok 304 adhesive. Do you know what the generic makeup of that is, Tennison?

DR. SMITH: I'm going to turn to Dave.

MR. KAELBLE: I think it's an epoxy polyamylene.

DR. SMITH: We tried to pick something that didn't have any glass scrim in it, didn't have carriers and fillers and things in it, and we put three little wires in between to keep a constant bond line thickness which came out fairly close to 10 mils, plus or minus half a mil.

DR. BASCOM: It does not have dispersed rubber?

DR. SMITH: No, I don't think so.

DR. FLYNN: What happens to all of this if there is a scrim cloth?

DR. FLYNN: I can't give you any answer on the basis of the work that was done here, but in some other work that is going on in our department at General Dynamics on commercial aircraft adhesive we always have nylon or dacron scrim. You don't see the scrim in AF-147 or the filler in FM-400 for example. I'm not sure what the definite size of that glass would be, but I think the fiber would be pretty small; probably smaller than the wave length you are working with. You would have to go to pretty high wave frequencies to see it.

PROF. WILLIAMS: I want to thank Dr. Flynn for his presentation.