NONLINEAR ACOUSTIC BEHAVIOR IN A NICKEL-BASE SUPERALLOY

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NON-LINEAR ACOUSTIC BEHAVIOR IN A NICKEL-BASE SUPERALLOY

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ABSTRACT

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Several studies of nonlinear acoustic properties in metallic materials in recent years have shown promise as a new nondestructive evaluation technique for early detection of fatigue damage. Majority of these studies has been conducted on laboratory specimens with optically smooth surfaces using a capacitive detector with air as the dielectric medium. A recent study has shown on Ti-6Al-4V, has shown that the nonlinear acoustic parameter increases by hundreds of percent before the material fails due to accumulated fatigue damage. In order to implement the technique on components and materials that are not optically smooth several modifications have to be developed and implemented to the existing capacitive detector methodology. The aim of the thesis is to develop methodology to improve the sensitivity of the capacitive detector and to utilize it to investigate the accumulated fatigue damage due to mechanical and thermal loading in nickel-base superalloy samples with rough surfaces.

Also included in this study is the new development of new methodology for using capacitive detectors to detect higher order harmonic signals. Using liquid dielectrics to aide in the catching of acoustic signals yielded new levels of detector sensitivity. Using these methods produced data on the third harmonic signal. This method only requires slight modifications to almost any capacitive detector. All necessary equipment, equations and procedures are included in this thesis. Developing nondestructive damage evaluation techniques is very important to the continuing improvement of materials and material evaluation techniques. As fatigue damage accumulates in metal the increasing formation of these harmonic frequencies can be detected. The formation of these harmonic signals forms a dimensionless parameter labeled β . β has a direct connection with the amount of internal damage observed in laboratory samples. Combine with the new methodologies of using liquid dielectrics to provide increased sensitivity to capacitive detectors, using nonlinear acoustics, as a fatigue damage evaluation technique is becoming a more viable method for damage evaluation.

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LIST OF SYMBOLS AND RELEVANT EQUATIONS

A	Acoustic Wave Amplitude
F	Test Frequency
Т	Time of flight (1 st acoustic pulse to 3 rd acoustic pulse ms)
V _L	Longitudinal Wave Velocity
ε ₀	Permittivity of Free Space = $(8.845*10^{-12} \text{ C}^2/\text{Nm}^2)$
α	Detector Area
Vs	Substitute Voltage
Vs _{real}	Real Voltage Without Amplification
V _{out}	Voltage Amplitude out of Amplifier
V _b	Bias Voltage Applied to Detector
Cd	Detector Capacitance
β	Nonlinear Acoustic Parameter
k	Wave Number
a	Wave Propagation Distance
A ₂	Fundamental Wave Amplitude
A ₁	Harmonic Wave Amplitude
K _{test}	Dielectric Constant For the test ($K_{test} = 1$ when using air in
	capacitive gap)

$$\beta = \frac{8}{k^2 a} \left(\frac{A_2}{A_1^2} \right)$$
 (Eq. 1)

$$A_1 = \frac{K_{\text{test}} \varepsilon_0 \alpha \text{ Vs}}{2 \text{ V}_b \text{ Cd}}$$
(Eq. 2)

$$A_2 = \frac{K_{\text{test}} \epsilon_0 \alpha \, \text{Vs}_{\text{real}}}{2 \, \text{V}_{\text{b}} \, \text{Cd}}$$
(Eq. 3)

$$k = \frac{2 \pi F}{V_L}$$
(Eq. 4)

CHAPTER I

INTRODUCTION

One of the most important aspects in the development of materials is the determination of their properties. Accumulation of fatigue damage due to thermal and mechanical loading is a common cause of catastrophic failure of materials and components. Developing methods to monitor the amount of accumulated fatigue damage is critical to further the understanding of failure of materials and help in designing to avoid catastrophic failures. The most promising experimental techniques and methodologies to monitor fatigue damage accumulation should cause little or no damage to the current state of the material.

Nondestructive evaluation (NDE) techniques hold promise to determine the amount of damage in materials without altering the current state of the material. At present many of the NDE methods play a dominant role in the evaluation of structural integrity of components. Current NDE methods have been very successful in detecting cracks initiated due to accumulated fatigue damage. Although these techniques have provided a basis for damage tolerant design of components, there is a growing need to detect early signs of accumulation of fatigue to avoid catastrophic failures. One of the NDE methods that has shown a significant promise in detecting fatigue damage at early stages is the nonlinear acoustic method. Recently, Frouin et al. [1] have utilized this methodology to evaluate the fatigue damage in Ti-6Al-4V samples. In nonlinear acoustic measurements, a short burst of monochromatic ultrasonic wave is propagated through the sample and the harmonic frequency signals generated by the material during the propagation are monitored. In majority of the experimental measurements, the second harmonic frequency signal amplitude has been measured. The amplitude of the second harmonic signal has been observed to increase with the number of fatigue cycles. The nondimensional, nonlinear acoustic parameter, β , associated with the amplitude of the second harmonic signal is given by

$$\beta = \frac{8}{k^2 a} \left(\frac{A_2}{A_1^2} \right)$$
 Eq. 1

where k is the wavenumber, a is the propagation distance, and A_1 and A_2 are the wave amplitudes of the first and second harmonics, respectively. The increase in nonlinear acoustic parameter has been directly linked to the accumulation of fatigue damage.

Frouin [1] used non-linear acoustic analysis to determine fatigue damage in Ti-6Al-4V samples, using interrupted fatigue measurements and in-situ testing in a fatigue machine. Frouin has shown that the nonlinear acoustic parameter (β) changes almost by 200% as a result of fatigue damage accumulation before failure. He utilized two different measurement techniques to measure the nonlinear acoustic parameter. In in-situ tests two transducers one to propagate a fundamental frequency signal and another to detect the second harmonic signal was used. This method provided a relative measure of the nonlinear acoustic parameter. In the other measurements a capacitive detector was used to measure the absolute nonlinear acoustic parameter.

The capacitive detector is an excellent tool to measure very small displacements generated by ultrasonic waves. The instrument is very sensitive and requires tremendous care in the measurements. The distance between the sample and the detector button is of the order of 5-7 μ m. Because of this restriction the surface of the sample should have an optically smooth surface finish. Some times a polyethylene film is used to cover the detector button to improve the dielectric constant of the gap. This has been found to enhance the sensitivity. This film is usually very thin and breaks easily. If any voids and defects are present in the film it will lead to arcing and dielectric breakdown. Whenever arcing occurs entire detector has to be dissembled and the detector button has to be replaced. In spite of the shortcomings nonlinear acoustic measurements performed using capacitive detector are considered the golden standard.

This thesis investigates the use of nonlinear acoustic parameter measurements to evaluate the fatigue damage accumulated in a nickel-base superalloy through mechanical loading at elevated temperature. The samples have an as-machined surface or electropolished surface. Both of these surfaces are rougher than smooth surfaces required for capacitive detector measurements. In order to perform measurements with these types of samples a new method of using a liquid dielectric medium in the gap between the sample and the detector button of the capacitive detector has been explored. Different types of liquid dielectric materials have been used and interesting results have been obtained. Capacitive detector with liquid dielectric medium has been used to measure nonlinear acoustic parameter in mechanically fatigued and thermally fatigued samples.

Localized nonlinear acoustic parameter measurements across a fatigued sample clearly shows a significant increase in the nonlinear acoustic parameter in the middle of the gauge section.

Some of the important results presented in the thesis are related to the

- a) improvement of capacitive detector for nonlinear acoustic measurements on machined and electro-polished surfaces,
- b) application to mechanical fatigued samples of nickel-based superalloy under elevated temperature isothermal exposure, and
- c) measurement of third harmonic signals with improved capacitive detector.

Chapter II provides a description of the equipment and experimental setup and of the application of liquid dielectrics in a capacitive detector to improve sensitivity. The procedure for measurement of the nonlinear acoustic parameter, β , can be found in Chapter III. Chapter IV contains a description of the specimens used in the measurements, test matrices for calibration samples and nickel-base superalloy fatigue specimens, and experimental results from all samples. Chapter V provides a discussion of the nonlinear acoustic measurements on calibration samples and fatigue specimens. Conclusions and recommendations for further improvements of the measurement methodology are presented in Chapter VI.

CHAPTER II

EXPERIMENTAL METHODOLOGY

Experimental setup used during this study was constructed using references from previous studies. Many of the samples that were used as standards in the determination of β values also were obtained from Frouin's [1] study. A listing of all necessary equipment and electronic schematics necessary to perform nonlinear acoustic measurements are also included in this chapter.

A. Description of Equipment and Electronic Schematics

Setting up and using a nonlinear acoustic system can be complicated. Using the descriptions laid out in this section should simplify the process.

Included in this chapter is a block diagram for the setup used to acquire the acoustic measurements Fig 2.1. As well as a listing of all the equipment used in the block diagram Table 2.1. Also in this chapter is a description of the detector button setup.

A fair amount of equipment needs to be gathered in order to produce and catch acoustic signals in materials. In Table 2.1 is a listing of all the hardware that was used in this set of experiments. An important feature that is not listed in this table is the necessity of the waveform generator capable of producing tone bursts. Also it takes





Manufacture	Model Number	Description
LeCroy	Wave Runner LT 342 500 MHz	Digital Oscilloscope
Hewlett-Packard	6253A	Dual DC power supply ± 15 Volts DC
Tenma	72-2075	Lab DC Power Supply Current Controlled
Hewlett-Packard	4275A	Multi-Frequency LCR meter
Agilent	6634B	System DC Power Supply
Agilent	33250A	80 MHz Function/Arbitrary wave form generator
Amplifier Research	150A1008	150 Watt 10Khz-100Mhz Broad Band Amplifier
Miteq	VGC-8-30/6	30 MHz 80 Db Frequency Amplifier
Miteq	VGC-8-20/4	20 MHz 80 Db Frequency Amplifier

Table 2.1 Equipment Listing

two power supplies to run one Miteq amplifier. Miteq amplifiers are capable of 80 Db of frequency specific amplification and are used to look at specific harmonic signals. Miteq amplifiers require a duel voltage supply that can produce \pm 15 Volts DC. As well as a current controlled power supply to control the amount of amplification. In general these amplifiers were run at the maximum amplification possible.

In Fig 2.1 is the block diagram of the necessary connections. Notice that in the block diagram dashed lines are used to represent the signal path to measure harmonic signals.

Most important to using this type of detection method is the ground ring and the capacitive gap. A simple diagram is illustrated of the detector assemble is included in Fig 2.2. In Fig 2.2 it is important to notice that a small gap exists between the bottom of the sample and the detector button. This is the capacitive gap. Filling this gap with the dielectric material is what yielded the boosts in sensitivity.

Also in Fig 2.3 is the schematic for the signal separation box listed. Constructing this box is not very difficult it simply consists of a resistor and a capacitor. Although this box is simple is very important to the proper function of the capacitive detector. Using

Fig 2.2 Slice View of Capacitive Detector



Fig 2.3 Electronic Schematic for Signal Separation Box



Fig 2.4 Schematic for Signal Substitution Box



this type of box allows the user to connect the DC bias voltage to the capacitive detector and connect the capacitive detector to the oscilloscope.

The final piece of hardware that will need to be assembled is the signal substitution box. During the final stage of the nonlinear acoustic testing procedure this box will be necessary to calibrate the capacitive detector. Two values will need to be entered into this signal substitution box. These values are C_d and C_s . They stand for the capacitance of the detector gap and the stray capacitance of the system. Acquisition of the values will be covered in the proceeding chapter.

Entering these values into the signal substitution box is not very difficult but needs to be done accurately to ensure that the displacement amplitude is properly calculated. The electrical schematic of this signal substitution box is given in Fig. 2.4.

B. Sample Surface Conditions

The main alloy that was analyzed during this study was a Waspaloy of unknown heat treatment. Flat Dog bone samples were made of this material and electro-polished to provide an acceptable surface finish.

Electro-polishing does not provide ideal surface conditions for nonlinear acoustic testing. In most cases nonlinear acoustic samples are prepared in the standard manner described in Appendix A. Electro-polishing does not yield optically flat surfaces such as those studied in previous nonlinear acoustic tests [1]. Using capacitive detection to catch the acoustic waves makes this a major factor.

Capacitive detectors work by creating a capacitor using a surface of the sample as one plate of the capacitor and a detector button as the other. This method is highly sensitive to very small amounts of displacement but only at very short distances. Typically a distance of five to ten microns is required to provide an acceptable level of sensitivity. At a working distance of five microns even dust on the sample surface can cause false readings. Working within these types of tolerance can be very difficult.

Electro-polishing samples does not yield the optically flat surfaces that are normally required. Samples used in this study had surface conditions far from optically flat. When placed in a profilometer deviations of up to seventy-five microns can be detected. Since the detector button is only five microns away from the sample surface it becomes almost impossible to maintain the required distance constraints. It is easy to see that the worst-case scenario acoustic measurements are being taken at approximately eighty microns away.

These Waspaloy samples were also exposed to elevated testing temperatures causing a rough oxide formation on the surface. Changing surface conditions of the samples also caused problems to the ability of the capacitive detector to properly detect the acoustic waves. Formation of this rough oxide layer changes the surface condition of the sample. Oxidation of the surface of the sample also makes electrical grounding difficult. Producing feedthrough signals during testing. Formation of this feedthrough signal can also yield false β values. Most cases in this study showed a rise in β value due to this change in surface condition.

Further obstacles stemmed from the use of thin dog bone samples, requiring use of a smaller detector button. Decreasing the detection area also decreases the sensitivity of the capacitive detector. Testing of these samples required the almost exclusive uses of a detector button 1/4 inch in diameter. Although this type of testing had been preformed before it had not been attempted under these types of surface conditions.

C. Using Liquid Dielectrics

Nonlinear acoustics is a tremendously useful tool. Using nonlinear acoustic methods allows engineers to form a non dimensional value known as the beta (β) parameter. Part of the usefulness of this β parameter is its non dimensionality because it can be used to compare materials of varying sizes and compositions. Forming this β parameter provides engineers a means of looking at the internal damage of samples without causing additional damage. This NDE (Nondestructive Evaluation) technique is invaluable in the determination of underlying reasons for material failures. Causing very

little or no damage to the current state of samples allows for pinpointing of specific regions of interest without further disruption to test samples.

Evaluating the β parameter is the main focus of this thesis. Included in this study are data on Waspaloy a, nickel-base superalloys having varying levels of fatigue. This data provides a basis from which other studies may stem. Also covered in this thesis are the effect and procedures of using liquid dielectrics to aide in the measurement of acoustic waves.

Initial failure in the formation of quality data prompted the interest in improving the detection capabilities of the capacitive detector. Using air as the dielectric in the capacitive gap yields a myriad of problems. Some of which include electrical arcing between the surface of the sample and the detector button. When this happens the surface condition of the detector button is compromised and reading can be affected. Previously, this problem had been solved by applying a thin layer of plastic over the surface of the detector button. Applying this thin plastic layer is difficult. Creating a plastic layer that is uniform and thin enough to fit in the 5-micron capacitive gap is very difficult. Also if any air gaps form between the detector button and the thin plastic layer false readings are obtained. Many times it is very difficult to see these air gaps since the plastic layer is transparent.

Interest in the improvement of the capacitive detectors reliability dictated that this plastics layer be replaced. Researching how capacitors are made yielded another possible solution. Using a liquid dielectric layer in the capacitive gap was the most feasible idea. When the initial tests were conducted false readings were also obtained. But signal clarity was so good that further testing was conducted to see if this method of using liquid

dielectrics was viable. Locating the error in these initial reads was as fairly simple, since the only adjustments needed was a correction to the equations used to calculate the amplitude of the displacement Eq 2,3 located at the beginning of this thesis.

The current equations used the assumption that air was the dielectric in the capacitive gap. After filling the capacitive gap with a dielectric the permittivity of space in the gap has changed. So the simple correction that was made to the current equations was to multiply the permittivity of free space ε_0 by the dielectric constant K_{test} that is determined during the testing phase. In performing this modification the amplitude measuring capabilities are adjusted for the permittivity of space for every individual test. Another benefit of this method is the dielectric constant itself is also a non dimensional parameter thus it will not affect the non-dimensionality of β . Once this error was corrected, proper β readings were obtained using testing procedure in Chapter III.

Sensitivity of the capacitive detector is greatly increased by using dielectric materials. The main dielectric used in this study was oil commonly used to cool high frequency fatigue testing systems. Shell Oil Company produces this oil and it is known as Diala Ax. Diala Ax has a fairly low dielectric constant of K=2.2-2.3 at room temperature. This oil was chosen because of it's attainably and is high resistivity properties.

The resistivity property of any dielectric used in this kind of testing is critical. Tests conducted using materials with lower resistive properties were unsuccessful. Unfortunately there is a direct relationship between materials with high dielectric constants and materials with low electrical resistivity. The best example of this is water. Water has a dielectric constant of approximately eighty which would greatly increase the

sensitivity of the capacitive detector. But any contamination of the water makes it highly conductive.

Tests run with a medical gel named hydro-gel, which has medium resistive property (5 MOhm) and very high dielectric properties tests (K~10) were also unsuccessful. These tests confirmed the fact that highly resistive dielectrics must be used in order for this method to be effective. However there are ways of using these dielectrics without fear of losing the required resistive properties. A preliminary method for conducting this type of testing was used to produce acoustic signals using glycerol. Data on these tests will also be presented in Chapter IV with the test results. This methodology will also be included in the discussion of recommendations in Chapter VII.

Nonlinear acoustics and liquid capacitive detection can be very powerful techniques. By using liquid dielectric in the capacitive detector a door is opened to new levels of detection sensitivity. I would like to point out once again that the majority of these tests were conducted using oil with a fairly low dielectric constant. Capacitive detection equations dictate that the higher the dielectric constant the higher the microphone sensitivity. Since the relationship is a direct multiplication. The potential for continuing sensitivity improvements to explore further more minute signals exists. Important to note here is the highest dielectric constant I could find was K=11,000 so using something on this order of magnitude would yield a capacitive microphone (11,000/2.2)= 5000 times more sensitive than the one used in these tests.

Without this new level of sensitivity I am doubtful that this study would have been successful. By using these liquid dielectrics both the physical geometry and the surface roughness of the sample were conquered. Yielding some very useful data in the

exploration of the use of nonlinear acoustics. Using these methodologies is also opening the door to acquiring higher order harmonic signals.

CHAPTER III

PROCEDURE FOR DETERMINATION OF β

When acquiring nonlinear acoustic values, attention must be given to every detail. Dealing with the acquisition of acoustic waves can be difficult because the signal can often be difficult to detect. When working under these very sensitive conditions, cleanliness becomes very important. It is recommended that users wear plastic gloves at all times. Another good suggestion is to use compressed air to clean any surfaces suspected of being contaminated. Using dielectrics to aide the catching of acoustic waves will help to eliminate many of these factors but a little prevention can't hurt.

Determination of the nonlinear acoustic value (β) will be covered in this section. Description of the acquisition of proper data will also be covered. I will describe the lessons that I have learned over the past year so that other nonlinear acoustic experiments may go smoothly.

To begin the process of obtaining a β value you will need to perform some basic procedures that are laid out in Appendices A and B. These include the sample preparation and transducer bonding. Implementing these procedures is imperative prior to nonlinear acoustic testing.

A. Determination of C_s

Once you have successfully bonded a transducer to the sample testing can begin. For proper bonding procedures refer to Appendix B.

Place the proper size detector button into the base unit and make sure that the ground ring is in contact with the base unit. Improper grounding can occur due to poor contact between the ground ring and the base unit. To enhance grounding place aluminum foil spacers between the ground ring and the base unit to complete the circuit. Zero the capacitance meter and measure the capacitance of the test setup with no sample present. The capacitance that is displayed on the capacitance meter is the stray capacitance of the system labeled C_s . These values usually range from 10 - 20 pF depending on the diameter of the detector button. Record this value and label it C_s .

B. Determination of Test Dielectric Constant

If the dielectric constant of the material that is to be used is well known it is sufficient to skip this step and go to section C.

But if the dielectric constant is not well known or testing conditions are questionable, determination of the dielectric constant for this test becomes necessary. Take extra precautions here since damaging the transducer or the bond that is holding the transducer to the test sample is very easy.

Determination the dielectric constant is not a very difficult process. First place the sample over the detector button and center the transducer over the detector button Fig 2.2.

Carefully place the upper plate onto the setup and screw the lugs down until they are loosely snug and even. Record the total capacitance of the system with the capacitance meter. The capacitance meter will read the total capacitance of the system with air as the dielectric, record this number. The

value recorded is labeled C_{tair}.

Remove the upper plate and sample. Using the wooded end of a clean swab place a droplet of dielectric oil on the surface of the detector button. Using the same end of the swab make sure that the entire surface of the detector button is well



Figure 3.1 Dielectric Oil Application

covered. Oils will generally wet the surface of the button and form a half sphere Fig. 3.1. Make sure that the oil droplet will is large enough to ensure contact with the bottom of the sample when it is in place. Finally inspect the oil droplet to ensure that no air bubbles have formed inside the oil droplet. If any air bubbles have formed inside the dielectric oil then the detector button needs to be cleaned and the process needs to be repeated.

If no air bubbles have formed in the dielectric material then place the sample back on the test setup and center the transducer button over the detector button again. Now place the upper plate back on top of the test setup and tighten the thumbscrews down evenly as before. Now record the total capacitance of the system. The value that is displayed by the capacitance meter is the capacitance of the system with oil as the dielectric material. Record this value and label this value $C_{tDielectric}$. The dielectric constant for this test is the total capacitance of the system with oil as the dielectric divided by the total capacitance of system with air as the dielectric $(C_{tdielectric}/C_{tair})$ and is labeled K_{test}.

C. Testing Procedure

Connect the waveform generator to the microwave amplifier. Then connect the microwave amplifier to the upper plate of the test setup. Connect the signal separation box to the lower portion of the test setup labeled signal out. Connecting the signal separation box connects both the DC voltage for the capacitive detector and channel 1 of the oscilloscope. Refer to Fig. 2.1 for the schematic of full system when set up.

Before turning the DC voltage on make sure to look for feedthrough in the system Fig. 3.2. Some specific features make this feedthrough signal easy to recognize. One feature of a feedthrough signal is that it will occur instantaneously i.e. time marked zero on the oscilloscope. Most feedthrough is due to improper grounding. Left over voltages feed to the oscilloscope and create a false signal. Ideally no signal should be viewable on the oscilloscope at this point but some feedthrough is usual identifiable.

Another way to identify feedthrough is to turn on the DC voltage and view the acoustic signal that should appear. When the DC voltage is activated and the acoustic signal appears on the scope, any signal on the scope previous to the activation of the DC voltage is feedthrough signal. If the feedthrough exceeds ~1mV then some adjustments

20

-



need to be made. One way to eliminate feedthrough is to make sure that all of the lugs on the test apparatus are all tightened evenly. Another method of eliminating feedthrough is to place aluminum foil spacers between the ground ring and the base unit as previously discussed in this paper.

Eliminating as much of the feedthrough as possible is desirable since these signals will also feedthrough with the harmonic wave and be more pronounced due to amplification.

Once the feedthrough has been eliminated to an acceptable degree. Disconnect the signal separation box and check capacitance of the system. If the capacitance of the system has changed a new $C_{tdielectric}$ must be recorded. The new capacitance on the capacitance meter is the new $C_{tdielectric}$ use this value to replace the previous $C_{tdielectric}$.

Now it is ok to turn on the DC voltage to the capacitive detector. A good idea is to start with low voltage, such as half the indented testing voltage. Make sure that the acoustic signal appears on the scope then proceed to the intended testing voltage.

The acoustic wave will appear at some point after the initial triggering of the oscilloscope and appear in an exponentially decreasing echo train Fig 3.2.

The first measurement to be acquired is the speed of sound in the sample. The amount to time that passes from the detection of the first acoustic pulse to the third acoustic pulse needs to be determined. The reason that the third acoustic wave is used is because sometimes an inversion occurs from the first acoustic wave to the second acoustic wave. By aligning these two portions of the wave on the oscilloscope, the time that passes can be determined using the measurement tools in the oscilloscope. This value can then be used to determine the speed of sound in the sample.

Recording the amplitude of the voltages formed by the capacitive detector is what forms the β parameter. First step to recording these amplitudes is to center the first acoustic pulse in the center of the oscilloscope screen. Zoom in on this first transmission as much as possible to ensure the most accurate readings. Using the amplitude measuring capabilities of the oscilloscope measure the amplitude of the first acoustic pulse. Enter this value into the data sheet next to the input voltage that was used. Change the input voltage from the function generator to the next interval and measure the amplitude of the first acoustic pulse again. Repeat this process six times filling out the data sheet after each measurement.

Measuring the amplitude of the first acoustic wave takes some practice but an easy way to think of taking these measurements is to take the average of the signal. Some points may be over and some portions of the wave may fall under but it is important to try to have as many portions over as under thus obtaining the average readout of the signal. These voltage measurements of the first acoustic wave are labeled A1.

Reading the harmonic signal off of the oscilloscope is generally more difficult but requires the same processes as reading first acoustic wave. Placing a filter in between the test apparatus and the amplifier filters out all signals except that of the harmonic frequency Fig 2.1. I found it easiest to use the second channel on the oscilloscope since fewer adjustments needed to be made from test to test. Many times when looking at the harmonic signal it will only be viewable when the math functions on the oscilloscope are used to average the signal over a thousand sweeps.
Some difficulty may occur when trying to find the second harmonic wave. The best way to locate this signal is to look at a time slightly after the were the first acoustic pulse occurred. This small delay is due to the use of the filters and the amplifier.

If you are in doubt whether you are viewing the first pulse of the second harmonic wave, a couple of things that can be done. First, turn off the DC voltage to the detector button. The acoustic signal will disappear. This may provide conformation that the correct signal is being viewed because the signal that needs to measured will be the first pulse of the signal that disappeared. There is one other method can be used to view the harmonic signal. Once again turn off the DC voltage to the detector button so that the only signal that is being viewed on the scope is the feedthrough noise of the system. Using the memory inside the oscilloscope store the averaged noise signal over a thousand sweeps and then turn the DC voltage to the detector back on. Using the math functions on the oscilloscope take the difference between the new averaged signal of the second harmonic wave and the stored image of the noise wave. Using this method should provide a clear image of the second harmonic wave only. Unfortunately, no possibility exists to take measurements this way since the amount of feedthrough noise changes.

Once the harmonic signal is located place the first pulse of the second harmonic signal in the center of the scope and zoom in. Use the same input voltages on the function generator and record these values in the data sheet next to the corresponding input voltage; these values are labeled A2.

Once enough reading are acquired to provide a clear view of the acoustic signals it is now necessary to determine the amplitudes of the motion of the sample. Understanding that what is read on the scope is not the amplitude of the displacements of

the surface of the sample is very important. What is read out on the scope is only the change in voltage produced by the capacitive detector. Therefore, to calculate the amplitude of the motion of the sample it is necessary to convert these voltages in to displacement amplitudes.

To do this a substitution box is used. This box consists of a modeled circuit that is included in Fig. 2.4. The C_s , which was measured at the beginning of the test, is the same as the C_s that is used here. Also the C_d that is used in the model circuit is

$C_d = (C_{tdielectric} - C_s).$

Setting these values is the next step. Using a BNC shorting connector short the OUT BNC PLUG on the box and attach the capacitance meter to the side labeled IN BNC PLUG on the substitution box. Adjust the C_d capacitor until the proper capacitance is read. To set the C_s value simply the places the BNC shorting plug on the UPPER PLUG side of the substitution box and connect the capacitance meter to the side labeled OUT BNC PLUG on the substitution box. Adjust the C_s capacitor to the proper value.

Now using a BNC cable connect the function generator directly to the IN BNC PLUG connection. Connect the oscilloscope to the OUT BNC PLUG side of the substitution box and place a BNC shorting plug on the UPPER PLUG completing the circuit. Make sure that the output on the function generator is well below any voltage that will damage the oscilloscope and set the frequency to the test frequency. Turn on the output on the function generator and view the output on the oscilloscope.

To generate the true amplitude A_s1 , set the amplitude cursors on the oscilloscope to the amplitude of the measured voltage for the initial wave A1. Now adjust the voltage output of the function generator until reads the amplitude of A1 on the oscilloscope. The output voltage on the function generator is the substitute voltage A_s1 . Record the value of the amplitude of the voltage of the function generator in the data sheet. Repeat this process for all of the voltage readings in the initial wave data set.

Generating the second harmonic substitute voltage is much the same as generating the substitute voltages of the initial signal. Attach the OUT BNC PLUG side of the substitution box to the attenuator and attach the other side of the attenuator to the signal filter that is attached to the amplifier. Leave the IN BNC PLUG side of the substitution box attached to the function generator and keep the BNC shorting plug on the UPPER PLUG connection. Change the frequency out on the function generator to twice the testing frequency. Now repeat the process of finding the substituted voltage used in the previous section and enter these values into the data sheet.

The last step to calculating the amplitude of the surface movements is to calibrate the attenuator. Connect the function generator to the attenuator and attach the attenuator to the to the oscilloscope. Using the twice the testing frequency and selecting a voltage that is high enough to read the signal on the oscilloscope, average the signal. Enter these values into the data sheet and the calculation of the beta parameter should be complete.

Chapter IV

EXPERIMENTAL RESULTS

Using new liquid dielectric techniques yields some very useful data. Concerning both nonlinear acoustics and the new levels of sensitivity produced by using liquid dielectrics. In addition this is the first study done on Waspaloy of this pedigree.

A. Calibration and Application of Capacitive Detector Using Various Dielectrics

Using liquid dielectrics to detect nonlinear acoustic values is very helpful in detecting the displacements on the surface of the samples to produce β values. Formation of this idea came from my physics book. Attempting to learn more about how capacitors function I read about dielectrics and their constants. This held promise since the main problems at the time were arcing and minute signal. By simply assuming that the only variable changing was permittivity of space in the capacitor gap calibration began. After an initial correction to the amplitude calculation equations, this method became a viability method of acquiring acoustic values.

Beginning with the three standard cylinder samples of Ti 6-4, aluminum and silicon prepared in the method described in Appendix A that had known β values. Also using a fourth and fifth sample of Waspaloy produced using the standard method, but β

was unknown in these new Waspaloy samples and needed to be determined. The first value to be recorded were β values using air as the dielectric material to perform a standard test and then two test with oil in the capacitive gap produces Fig 4.1 and Table 4.1. In Fig 4.1 the β values obtained for nickel, aluminum, Ti-6-4 and the standard Waspaloy samples are displayed. Also in Table 4.1 the values of β for both liquid dielectrics and air are listed. I am very confident that this data clearly proves that using liquid dielectrics I a viable method of viewing acoustic signals.

i. Calibration Samples and Testing Matrix

Using dielectric oils to aide in the acquisition of acoustic waves is an unproven method. It is so new that the University of Dayton Research Institute is currently seeking patents on this methodology. So the first testing matrix that had to be put together with what was known about the materials that we had in house in order to prove that this method works

In order to prove that using dielectric oils is a viable method of catching acoustic wave it is important to first prove the methodology. Using the five standard samples described in a previous section is were we began.

The first sample type is a standard cylinder sample. These cylinders range from $\frac{1}{2}$ - 2 inches in length and from 3/4 - 2 inches in diameter. Standard samples are made from a single type of material. Three of the standard samples were acquired from a previous study [1]. These samples were made from Aluminum, Titanium 6-4 and Silicon, all had known β values. A fourth and fifth sample were cut from a Waspaloy block. These

								H X -1	Silicon	∓ 2.54 ₹ 2.42	I 2.37	
Wasplov	10.80	¥ 10.46 ▼ 10.08 ⊥ 10.02			Aluminum	Tic 1 ± 6.16	5.23 ± 5.72	4.63 4.57				
Beta Silicon Air G	 Beta Silicon Dielectric G 	Beta Silicon Dielectric G	× Beta Ti-6-4 Air Gap	* Deta 11-0-4 Dielectric ca	• Ti-6-4 Dielectric Gap #2	+ Beta Aluminum Air Gap	- Beta Aluminum Dielectric Gan	- Beta Aluminum Dielectric Gap #2	 Wasploy Sample #1 Air G 	Wasploy Sample #2 Air G	Wasploy Sample #1	× Wasploy Sample #1

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Materials

Waspaloy samples had no known β value. All standard samples were prepared in the manner covered in Appendix A. Standard samples are designed to eliminate as many unknowns as possible. By using the tolerances of optical flatness and a high degree of parallelism these samples should perform the same under any circumstances. Thus providing a solid base from which to calibrate any non-linear acoustic system.

Three of the standard samples were acquired from the previous study [1] this included the Aluminum, Ti 6-4 and silicon samples. For these samples one test using air as the dielectric was run first to ensure that both the sample and the capacitive detector were working properly. Once this test provided assurance that all systems were functioning properly two tests were conducted using the dielectric oil in the capacitive gap.

Two standard Waspaloy samples were made but had unknown β values. But the same procedure was used to test both these samples and the validity of using dielectrics. First the two samples had tests conducted with air as the dielectric and then another using the oil.

Sample	Beta Air Gap	Beta Dielectric Gap	Beta Dielectric Gap #2
Silicon	2.37	2.42	2.54
Ti-6-4	4.57	4.63	5.23
Aluminum	6.72	5.72	6.16
Wasploy Sample #1	10.80	10.02	
Wasploy Sample #2	10.48	10.08	

Table 4.1 Dielectric Calibration Matrix

Wave Amplitude Comparison Using Various Dielectrics ii.

One of the most important aspects of this study is the use of dielectric to improve the sensitivity of capacitive detectors. In order to illustrate the gains that are possible using dielectric, a series of tests were preformed on just the wave data in order to show the sensitivity gains. These measurements were all taken under the same conditions. A capacitive gap distance of approximately 20 microns was used. Other similar settings include 35% gain on the Amplifier Research microwave amplifier, a capacitive detector bias of 100 V and the Miteuq amplifiers were set to maximum amplification.

These settings by no means represent to the best conditions for acquiring acoustic values. The reason these settings were used to avoid amplifier saturation when looking at the second harmonic using glycerin. The signals that are plotted for comparison are the first pulse of the initial signal, second and third harmonic signal represented by A1, A2 and A3 respectively. All these test were preformed on an aluminum sample that was prepared in the standard manner using a .5-inch diameter detector button and a testing frequency of 10 MHz and a tone burst of 20 cycles.

Table 4.2 Dielectric Comparison

Standard Aluminum Sample .5" Detector Button 10 Mhz Testing Frequancy						
Dielectric Used	Air	Diala Ax K = 2.2-2.3	Glycerine K ~ 10			
Amplitude Test A1	X	X	X			
Amplitude Test A2	Х	X	X			
Amplitude Test A3	X	Х	Х			

Using liquid dielectrics give capacitive detectors a must needed sensitivity boost while at the same time preventing many of the unwanted side effects of applying high voltages across air gaps such as arching. Included in this section is a set of representative graphs to illustrate the how the sensitivity varies with the use of different dielectrics. In these graphs, of the echo train there are a few things to notice. The only portion of wave plotted is the initial pulse of the echo train. Doing this makes it easier to see the difference between dielectrics without crowding the graph but these initial pulses are representative of the rest of the echo trains. Referring to Fig 3.2 you can easily see which portion of the wave is plotted in these graphs.

Secondly the initial pulse is plotted twice once with the voltage amplitude for glycerin plotted on the secondary y-axis and then again with all wave data plotted on the same y-axis, Fig 4.2 and Fig 4.3 respectively. This same format is again followed for the second harmonic were glycerin values are initially plotted on the secondary y-axis and then again on the same y-axis, Fig 4.4 and Fig 4.5 respectively. In the final graph of the third harmonic wave Fig 4.6 all values are plotted on the same axis. Also notice that the x-axis is labeled adjusted time. These pulses occur at the same time relative to the initial signal introduction in order to plot them across the x-axis it was necessary to adjust the time to a relative time. However, no adjustments were made to the wave data other than this provision which made the data appear staggered on the same axis.



Fig 4.2 Initial Wave Burst (Glycerin Plotted on Secondary Axis)















iii. Third Harmonic Waves

Using liquid dielectrics makes it possible to see the third harmonic wave using a .5" diameter detector button. A graph of the third harmonic wave taken under more ideal conditions in aluminum is included here in Fig.4.7. Very few studies exist on this subject since viewing the third harmonic signal has been too difficult using previous methods. Using this new methodology of capacitive detection capability could make it possible too explore the use of the third harmonic to detect internal damage in materials. Even though this signal is minute, it is clearly visible.

The last piece of third harmonic data that needs to be represented the linear variation of the third harmonic with a variation in the input voltage. A plot of A_3 Vs. A_1^3 in aluminum is represented in Fig. 4.8.

Note: Second harmonic data was taken with this test to ensure accuracy and yielded a suitable β value for aluminum.



Fig 4.7 Third Harmonic Wave in Aluminum Using Diala Ax

Fig 4.8 A₃ versus. A₁³ In Aluminum







B. Nonlinear Acoustic Value in Waspaloy Fatigue Samples

i. Fatigue Sample Descriptions and Testing Matrix

Also used in this study were two sets of dog bone samples made from Waspaloy.

These Waspaloy samples had a heat treatment **Fig 4.9 Waspaloy Dog Bone** that was unknown to the author because of proprietary information. Dog bone samples were designed to have a uniform gauge section of approximately 20 mm long by 10 mm wide an overall length of 125 mm and a thickness of 2 mm Fig. 4.9.

Electro-polishing these samples gave the surface a fairly uniform appearance but may not have been the best option as it sometimes limits the ability to measurements nonlinear acoustic values. Surface conditions of the sample were highly polished but not optically flat as

specified in Appendix A. Electro-polishing also forms a lip at the base of the sample, which will later hinder the gathering mapping data.

Testing in the dog bone samples was a little more complex than that of calibrating the liquid dielectric capacitance method. The testing matrix that governed the Waspaloy samples was created to generate sets of samples that had varying amounts of damage. Specifically this matrix was designed to generate samples with damage levels based on a fatigue failure at 40,000 cycles with the samples at 650°C. Tests were conducted in a standard servo-hydraulic testing machine until fatigue conditions of 100%, 75%, 50%, and of life were researched 25%. Also β values were recorded at one mechanical cycle at



elevated temperature and pretest conditions of just electropolishing. The maximum stress was 931 MPa with a stress ratio of 0.05 at 1/3 Hz.

By recording the as received condition of these samples gives two key pieces of information that are critical to successful β comparison measurements. First this sets the β value for this pedigree of Waspaloy for this sample. Also β changes with the surface condition so it is important to make sure that the surface deformities do not affect the β reading to an unacceptable degree.

By following these guidelines the test matrix for the Waspaloy dog bone samples was formed Table 4.3. Two sets of Waspaloy specimens were required in the study. In the first set of specimens (03-467 – 03-476) two problems occurred. First, the griping of the sample was nonuniform and unpredictable failures. This griping problem also affected β readings throughout testing. Second, using dielectrics to aide in the acquisition of catching acoustic wave had not been established yet so only the air gap capacitance method was used. These two factors combined to produce unacceptable data scatter that will be discussed in Chapter V.

Sample set number two did not experience the same problems as sample set one. This set of samples is made from the exact same material as the first set of samples. This set of samples also exclusively used dielectric materials in the determination of β values. Predictable testing conditions, along with improved methods for determining β produce a much smoother set of data that will also be covered in Chapter V.

Table 4.3 Waspaloy Test Matrix

	As Received	1 Cycl	25%	50%	75%	100%
03-467	X					
03-468	X	X				Х
03-469	X					Х
03-470	X	Х	X	X		
03-471	X	Х	X	X		
03-472	X	X	X			X
03-473	X	Х	X	X	X	X
03-474	X	Х	X	X	X	Х
03-475	X	X				
03-476	X					
Second S	Set of Tests					
03-775			X			
03-776		X				
03-779	X	Х		X		Х
03-780	X	Х	X	X		
03-781	X	Х	X	X	X	
03-782	X					Х

First Set of Tests

ii. First Set of Waspaloy Samples (03-467 - 03-476)

Nonuniform griping produced failures as low as 6000 cycles in the first set of samples. Also due to the time allotted to measure β only one chance to obtain measurements was available. Using air gap capacitance method produced a β scatter that is very large. In Fig. 4.10 it is clear to see that some errors can occur when multiple tests cannot be performed using air gap capacitive methods. Some of the β values on this graph are well above the β values obtained in this study. These values are tabulated in Table 4.4 in section iii of this chapter. Due to the unknown variability in loading conditions a correlation between fatigue cycle and β values is impossible to asses.

Fig 4.10 β First Set only Samples 03-467 - 03-476





iii. Second Set of Waspaloy Samples (03-775 - 03-782)

The second sample set did not experience nearly the same amount of technical difficulty as the first set. Referring to Table 4.3 you can see all the intervals that β values were recorded at in this sample set. This produced the set of samples that was originally planned on. Nonlinear acoustic β values were obtained at all conditions of the testing matrix are shown in Fig. 4.11. This graph illustrates the rise in β as well as the continuation of β to approximately 16 at failure. Data scatter on this graph is reduced since the signal to noise ratio is much lower creating a much more repeatable means of acquiring β values.

In Table 4.4 all recorded values of β can be found. In Fig.4.12 is an exploded plot of the nonlinear acoustic value from the as received state through the first mechanical cycle. In Fig. 4.12 the initial jump that most samples experience is easily viewable. In Fig. 4.13 sample 03-779 after the initial rise in due to only the thermal exposure for the first cycle, some stress reliving affects can be seen.



Fig 4.11 B Second Set of Samples (03-775 - 03-781)

Fig 4.12 B As Received & One Thermo-Mechanic Cycle at 1200°F





03-467 8.84 6.46 6.46 6.6 7 12 13.84 12.87 3.61 13.62 3.649 13.62 3.649 10.43 2.1 16 7 16 7 16 7 16 7 16 16 7 16 16 16 7 16 16 16 16 16 16 16 16 16 16 16 16 16 16 16 16 16 16		As Received	1 Cycl	25%	50%	75%	100%
03-468 21.12 5.46 6 6 03-469 7.82 13.84 12.87 51.04 9.32 14 03-470 13.84 12.87 51.04 9.32 14 03-472 13.84 12.87 51.04 9.32 14 03-473 12.13 3.61 13.62 19.65 16 03-474 12.13 3.61 13.62 19.65 16 03-475 178.98 30.75 4.19 8.09 10.43 2 03-475 19.52 4.72 4.19 8.09 10.43 2 03-475 19.52 4.72 4.19 8.09 10.43 2 03-475 3.47 10.38 10.29 10.43 2 2 03-776 6.49 10.38 10.67 10.29 10.43 2 03-776 6.49 10.88 10.37 12.63 14.62 1 03-778 3.75 9.46	03-467	8.84					
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Averaged Data Second Set 7.82 10.17 10.81 12.68 14.62	Averaged Data First Set	8.62	6.94	8.91	12.28	13.25	11.27
	Averaged Data Second Set	7.82	10.17	10.81	12.68	14.62	15.72
Averaged Data (all) 8.22 8.55 9.86 12.48 13.94	Averaged Data (all)	8.22	8.55	9.86	12.48	13.94	13.50

Table 4.4 B Test Values

C. Mapping β on Waspaloy Samples

Values of β are represented in this section for sample 03-781. This sample was fatigued to approximately 75% of its fatigue life. β measurements were taken at approximately the center of the sample throughout the load history indicated by Table 4.4. In the interest of viewing the spatial distribution of β along the center axis of the sample, a β map was created. This sample was chosen because it had the most cycles and it would produce large spatial change with respect to β . Measurements were taken at 2.5 mm intervals along the entire length of the sample. Due to both the size of the ground ring and the lips produced by the electro-polishing only a single value of β could be obtained in the grip section in each of the griping tabs. Graphing these values produces Fig. 4.8, these values are also listed in Table 4.4. Clearly seen is a spike in β value as the middle of the gauge section is approached.





Postion (mm)		Beta
(0.00	
22	2.50	7.78
55	5.00	10.30
57	7.50	10.28
60	0.00	11.88
62	2.50	15.01
65	5.00	11.99
67	7.50	10.78
70	0.00	10.52
102	2.50	8.22
125	5.00	

Table 4.5 β Mapping Values Sample 03-781

Chapter V

Interpretations of Nonlinear Acoustic Values

Interpretations of the nonlinear acoustic values are the very important. Making sense of the data collected is the final step for the engineer. Validation of the use of liquid dielectrics to improve the sensitivity of the capacitive detector will be included in this chapter. As well as a discussion of the β parameter ability to detect internal damage incurred in Waspaloy samples due to fatigue testing at elevated temperatures.

A. Using Dielectric Materials to Evaluate β

Using liquid dielectric materials can provide a tremendous boost to help catch acoustic waves after they propagate though samples. Using these liquids with high dielectric strength will help improve the signal to noise ratio as well as prevention of some negative affects such as arcing.

In the initial study after the development of the possibility of using this liquid dielectric technique became a reality, I decided that a calibration needed to be done using all the available standard samples. Using a multi-test calibration a list of all the tests conducted values produced can be found in Table 4.1 and also in Fig. 4.1 both standard tests using air as the dielectric and tests using Diala Ax as the dielectric were

conducted. These values fall in a very tight grouping around the accepted values for these materials. Conducting these tests makes it very clear that using liquid dielectrics is a viable means of catching acoustic waves and using them to form the β parameter.

Although these values fall on top of each other there is one thing to notice. After viewing Fig. 4.1 it is easy to notice that for aluminum and Waspaloyploy the β values fall under the air gap values. Also to notice is that for Ti-6-4 and Silicon the β dielectric values fall above the air gap values.

These values fall within an acceptable degree but it is still important to notice these variations. Using liquid dielectrics is a new methodology and there maybe a few unseen effects that are occurring here that are important to notice. By using liquids in the capacitive gap there may be some damping of the motion of the sample due to cohesive bonding. I do not feel this damping will cause many problems because the harmonic waves are formed by the propagation of the initial wave (A₁) thus any damping will be proportional and will therefore be eliminated in the equations for β by (A₂/A₁²).

Another factor is the elimination of feedthrough. The elimination of feedthrough signal will produce values that are slightly lower because feedthrough signal generally produces false signals that are slightly larger. Limiting the amount of feedthrough voltage is very desirable and thus producing more accurate readings.

Another major factor in reducing the amount of data scatter that will be produced when using liquid dielectric is the signal to noise ratio. It is very clear after viewing Fig 4.2 - 4.6 that using liquid dielectrics gives the capacitive detector a major signal boost. More accurate reading are obtained by strengthening this making it easier to obtain the correct value is for a given signal. With any set of experimental data some scatter is expected. In this set of data, the difference of the β values may just be some data scatter. The consistency of these calibration values with earlier air gap data provided suitable proof that this method works. Thus, the entire second set of samples were tested using dielectric materials in the capacitive gap.

B. Comments on Using Liquid Dielectrics to Acquire Acoustic Waves

Using dielectric materials has greatly increased the sensitivity of capacitive detectors. One of the best features of this new method is that it requires almost no redesign of parts or specialized materials. All that is needed is a liquid with high electrical resistance. By using materials with higher dielectric constants, levels of sensitivity will continue to increase. Using this method will open the door to unexplored signals that may lead to more robust NDE techniques.

Another affect of using liquid dielectric is the wetting of the surfaces of both the sample surface and the button surface. By doing this many flaws in the surface condition can be ignored. By wetting the surface ensures that a capacitor is formed. Rough surfaces have a tendency to cause electrical arcing between the sample surface and the detector button. Arcing is a major concern because it damages both the sample and detector button surfaces. Using liquid dielectric it is physically impossible for arching to occur. In the worst case user can experience dielectric breakdown. Dielectric breakdown occurs when a voltage that is too high is applied across to short a distance. When this occurs materials

that were previously non-conducting will begin to conduct. But this will not damage the detector or the sample.

Using liquid dielectrics is a good way to improve the sensitivity of almost any capacitive detector. Requiring only a small change to the equations governing the amplitude calculation, making this a very powerful technique that can be applied to almost any capacitive detector.

C. Third Harmonic Wave Data

A discussion about the third harmonic data recorded is included as well as the comparison of using various dielectrics. The most important factor here is just the recording of the data. Very few studies exist on the third harmonic due to the inability to record third harmonic values. Using liquid dielectric allows for capacitive microphones that are sensitive enough to view this signal and possibly higher harmonics.

Wave data were recorded directly from the LeCroy oscilloscope and plotted in Microsoft Excel Fig. 4.2-4.6. As previously discussed these are graphs of the initial pulse of the initial, second and third harmonic wave. These wave train were produced under similar condition to illustrate the change in sensitivity as the dielectric constant changes. Although these conditions were not the optimal condition for acquiring acoustic waves, parameters had to be set so that all waves could be viewed without adjustments to the peripheral equipment.

Plotting the data in this way one can easily see the change in signal amplitude as the dielectric constant changes. All measurements were made on the same sample at the
same signal input level and yet only one plot Fig. 4.6 produces a discernable third harmonic pulse. As previously stated these were not optimal conditions but rather conditions that prevented amplifier saturation while viewing the second harmonic using glycerin.

When optimum conditions are used to acquire third harmonic signals in aluminum Fig. 4.6 is produced. Clearly displayed here, well above the noise band and feedthrough signal is the third harmonic wave. This wave was produced using Diala Ax which has a dielectric constant K=2.2-2.3 at room temp. Graphing this data clearly shows the exponententialy decreasing echo train. Also when amplitude values are recorded at varying levels of input voltage and plotted in as V_3 versus V_1^3 Fig 4.8 a linear pattern develops that when extrapolated will approach zero. Included with this fact is that there is almost no visible feedthrough voltage associated are all marks of a good third harmonic signal. Since no real studies exist on this third harmonic signal it is only sufficient to mention that recording these signals is now possible.

Diala Ax by no means posses the highest attainable dielectric constant. Rather it had the highest electrical resistance of the dielectrics used. Much more optimization and use of dielectric materials to catch higher order harmonics will soon be possible. It is noteworthy to mention the glycerin used to produce this data was highly contaminated with water, which lowered its dielectric constant to approximately ten. This contamination also significantly lowers its resistive properties. So some of the uncalibrated methods that will be discussed in Chapter VI had to be used to produce this data. The particular method used to produce the data with glycerin involved the use of a

resistive barrier between the glycerin and the detector button in order to provide the necessary restive properties to make the capacitive detector usable.

Using dielectric materials increases the noise to signal ratio giving the user conformation that the data are correct. Dielectric is also opening the door to discovering higher order harmonic signals.

D. Accumulation of Fatigue Damage in Waspaloy

This study includes two sets of data on the unknown Waspaloy dog bone samples. The test matrix that was laid out for this study was to create a set of samples with varying fatigue states Table 4.2. Samples were to be exposed to 25,50,75 and 100 % of their expected fatigue life. Samples were also exposed to 1200°F testing temperature that causes the formation of oxides on the sample surface.

Initial study of the β values had some errors. Some of these errors occurred due to a malfunction in the griping system on the testing system discussed in previous chapters. This caused a random bending moment during the fatigue testing and caused samples to break at random intervals. Thus, obtaining a set of samples that had 25,50,75 and 100% of their life expectancies was impossible. Other errors are also apparent in the first set of data. Many of these errors occurred as a result of electronic feedthrough. Data in the first set used exclusively air in the dielectric gap. Using air, as a dielectric does not yield enough sensitivity to acquire reads with a high degree of certainty. Therefore, the data scatter on this first set of data is very large. This first set of β ranges from 3 – 179. Some

obvious problems occurred with a data scatter whose largest value is over 9 times larger than the theoretical limit.

However some trends that can be observed in this first set of data. A definite rise in β was experienced by all samples during the testing procedure. Also initial β values of this group excluding errant data yield an initial reading of approximately β =8 for this Waspaloyploy. After one mechanical cycle and one thermal cycle had been applied to the samples β were no trend can be seen. Data points in this region of the graph show the reason that when using air gap capacitors it is important to have highly polished surfaces. Some failure criteria can also by extracted from this first set of data. Almost all failure β values were above $\beta \ge 16$. Many of these samples experienced very rapid failure. Causing the location that the β reading was taken at to become a critical factor. In these failures it is clear that a portion of the sample failed due to fatigue loading. Also obvious is that part of these samples experienced rapid failure. In order to find the correct β values measurements needed to be taken as close to the fatigue failure as possible. Other sections of the fracture surface may not yield the correct values for this β .

As stated before there are two sets of data include in this study. After resolving the problem with both the grips and discovering how to use liquid dielectrics the second set of data was produced. As predicted earlier this data set is much more accurate.

Some similarities exist between data set #2 and data set #1. First the initial value of approximately $\beta=8$ is confirmed in this second set of tests. An average jump of 2.52 also occurs due to the initial change in surface condition by the initial heating and mechanical cycle. After the initial heating and mechanical cycle a rise of only .46 on the average is observed between this initial cycle and the first 25% damage. The following

suggestive damage levels of 50, 75 and 100% yield an almost constant average increase of 1.65. The highest β value that was obtained during this second set of tests was 15.72. This value is close to the highest β value of β =16 that was observed in the initial set of testing. A value β =16 in this study seams to be the β the highest readable value for these sets of data.

Although there are some major errors in the initial study of β , the second set of data yields a much better look at what the real values for β may be.

Chapter VI

Conclusions and Recommendations

The interpretations of the results from this study are not ground breaking. Only conformation that nonlinear acoustics and the β parameter are useful tools in the detection of internal damage in materials. Using dielectrics has only given the capacitive detectors a much-needed boost.

A. Final Conclusions

Using nonlinear acoustics will become a very powerful methodology in the determination of internal damage. Using these types of methods will appeal to almost anyone who works with damage tolerance and determination. Development of these types of tools is critical to the development of other types of equipment. Giving engineers to ability to determine pre-failure conditions make it possible to design better equipment and determine how long it can safely be used.

Using β and monitoring the second harmonic are not new ideas. In fact people have been conducting this type of testing for many years. Finally the technology is catching up with the theory. Most studies to this point have been just to prove that

harmonic signals are sensitive to internal damage. In this study as in others using β values provided a picture of a localized damage site in the sample. So in this aspect nothing new is presented in this study.

Using dielectrics to increase the signal to noise ratio and to aid in dealing with rough surfaces has direct field applications. Many times it is easy to use methodologies in the lab that are not practical for use in the field. Using these new methods allows for the conditions necessary to perform this type of testing to be more realistic.

Using liquid dielectric and coatings is a significantly new approach to this problem. By using these techniques new signals will be discovered and new and better damage tolerance methods will be developed.

B. Recommendations for Future Work

It seems to very clear to this author that nonlinear acoustics needs to be continually be developed. Many attributes make using the β parameter very appealing. One of these major attributes is its non-dimensionality. Providing a means of looking at materials on a non-dimensional level. Using a non-dimensional value provides the ability to compare samples of different sizes shapes and compositions. More studies need to be conducted to determine more about the usefulness of the non-dimensional β parameter. Further work also needs to be conducted using various other dielectrics to discover the limit of the sensitivity of the capacitive detector. Using dielectrics will yield continually more sensitive capacitive detectors. I feel that this will open the door to explore signals that have been previously undetectable as well as providing a clearer view of the acoustic signals that are currently used.

More work will be necessary to determine how to use various dielectrics. I have been able to produce signal at distances up to 1 mm away for the lower sample surface. Using a plastic coating and the medical gel previously discuss hydro-gel was one method of producing these signals. But due to the brevity of this study I was not able to complete these tests and no data is included in this study. Using non-conductive coatings on the detector button will provide a means of using almost any liquid dielectric. By using coatings the capacitive detector will be able to hold the charge necessary to produce the electrical signal necessary to view acoustic signals. Using coatings and liquid dielectrics has the potential to become another very powerful off shoot from the method laid out in this thesis. Many acoustic signals still exist that have never been explored. I believe that using these coatings and liquid dielectrics the sky will be the limit and some very powerful tools will be produced using this method. Another important note is that almost no modification to the any current existing capacitive detector is needed.

Signal exploration is very interesting and needs to have more studies done on it. Were nonlinear acoustics will be the most powerful is in automated scanning β method.

NDE techniques need to be able to pinpoint locations of damage without further causing damage to the sample. Having the ability to look at an entire sample or part at one time and be able to determine were the β parameter is the highest will yield some very useful damage detecting equipment. Only now possible since the current equipment is becoming sensitive enough to detect vibration on this level. In the future using interferometers this may be very possible.

It seems to this author that nonlinear acoustics now has the option to go in two directions both of which hold untold promise. Third harmonic signals are theoretically more sensitive to dislocation motion than the second harmonic and may yield some very useful information on the undying reasons for material failure. While developing methods to automatically scan sample and map their β values also holds tremendous promise in the development of the nonlinear acoustic field and NDE techniques.

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Appendix A

Sample Preparation

Sample preparation method used to prepare both the standard samples and the detector buttons is the same. The first and most important pieces of equipment that need to be purchase are an optical flat and a light source. This light source should produce light of the longer wavelength of green line in the mercury spectrum. Also purchase at least three or more 12×12 glass plates that are $\frac{1}{4}$ " or more thick. Clean these plates with soap and water and let them dry. Once they are completely dry use the optical flat to check that your plate are optically flat. Most plate glass is optically flat and this should not be a problem. You will also need to collect silicon carbide grit paper 320 - 600 grit as well as diamond paste 15μ m - 1μ m.

The first step is to select a grit paper that is commensurate with the current surface condition of the sample. If the surface condition of the sample is good enough to skip grinding with the grit paper skip this step. Place the grit paper on a flat surface and wet it with distilled water. Begin to polish the sample by moving the sample in a figure eight pattern being careful to apply even pressure to all surfaces of the sample and rotating the sample forty-five degrees after every stroke. Holding the sample flat here prevents ruining the parallelism of the surfaces of your sample. The larger the sample the more attention to holding the sample flat is required and not to tip the sample while grinding.

Once you have reached the 600 grit level check your sample for parallelism. To check the surface parallelism, clean the sample and place it on a clean glass plate. Setup a surface to reflect to laser beam onto, this surface should be as close to parallel to the glass plate as possible. Next aim the laser beam at the top surface of the sample so that it reflects onto the opposing surface and rotate the sample. The laser beam will deviate, measure the deviation over 360 degrees of rotation. A desirable surface parallelism is 12 arc seconds. Once this surface parallelism is obtained move to the polishing.

Polishing of the surface of the sample or the detector button is a long and arduous process but take your time because it is critical to proper non-linear acoustic measurements. Cleanliness is absolutely essential to this success. A recommendations to wear plastic gloves, also if any contamination is suspected clean the sample and the glass plate you are working with and start over. Starting with the 15µm diamond paste dab it over a clean glass plate and spread the paste using a clean tongue depressor. Applying even pressure, move the sample in a figure eight pattern around the glass plate turning the sample 45 degrees after each rotation. Normally a minimum 100 strokes is needed to complete one side of the sample. When one side of the sample is completed flip the sample over and begin the process on the other side. After both sides are completed wash the sample with soap and water and dry with compressed air. Place the sample in an ultrasonic cleaner with isopropanol and run for the appropriate amount of time to provide adequate cleaning. Remove the sample and once again dry with compressed air. Repeat this process until the 3-micron level is completed.

The last step is to remove any lingering damage on the surface of the sample or detector button is to using a lapping cloth. Place one-micron paste on the lapping cloth and move the sample in a circular motion for about 50 strokes. Be careful not to over polish because lapping can affect the surface flatness that was established with the rougher grits. Once again clean the sample and check for surface flatness using the optical flat and the green light.

Appendix B

Transducer Bonding

Once a desirable surface condition is reached. Bond a piezoelectric transducer to the sample. Three different cuts of lithium niobate transducers that come in a variety of frequencies. Three different types of piezoelectric transducers are available and there are labeled as x, y and z cuts. A z-cut transducer produces a pure longitudinal wave. An x-cut transducer produces a shear wave. The third type of cut and the one that was used in this study is a 36° Y-cut lithium niobate crystal transducer. A 36° Y-cut lithium niobate transducer expands almost entirely in the z direction,

although some shear component to the wave production it is negligible. This transducer was selected over a pure longitudinal wave because it is the most efficient type of transducer.

The simplest method for applying the transducers to samples is to construct a setup in (Fig. 2). Using a



pair of fine point tweezers to get a pinch of phenyl salicylate and place it on top of the

sample. Collect this pinch into a small pile on top of the sample. Using a hot air gun to heat the phenyl salicylate until it is completely liquefied. Apply some heat to the surfaces of the transducer heating it slightly. Be careful not to apply too much heat, as it will damage the transducer. About ten seconds or less will suffice. Then place the transducer onto spot with the melted phenyl salicylate. Using the setup in (Fig. 2) center the hole in the stand over the transducer. Placing the ball bearing between the tip of the load table and the transducer. Be very careful at this point, it is very easy to slip and drop the ball bearing or the load table onto the transducer cracking it and you will have to start over.

Once everything is in placed check to make sure that everything is centered over the lithium niobate transducer. If everything is centered then proceed to place an appropriate amount of weight on top of the load table. Never exceed two pounds of weight as you risk damage to the transducer. Most of the time a one-pound weight will be enough weight to squeeze the excess phenyl salicylate from under the transducer. Phenyl salicylate is a non-conductive substrate so it is important for proper grounding that as thin a layer as possible is laid down. Leave the sample to cool for ten minutes. Place some phenyl salicylate should form a layer of super-saturated liquid on the sample. Apply a few crystals of phenyl salicylate to the super saturated liquid layer that has formed. This will seed the crystal and let the setup stand for 30 minutes.

When you return to the setup a crystal should have formed around and under the transducer button. If extra phenyl salicylate gets on top the transducer it will need to be

removed. To clean off this extra phenyl salicylate use a sterile cotton swab and wet the tip with isopropanol and swab the surface clean. Pay careful attention to make sure that you do not touch the bonded surface with and isopropanol since it will dissolve the bond and you will have to re-bond the transducer.