2.1 Ultrasonic measurement techniques

2.1.1 Survey of ultrasonic measurement techniques

For the measurement of ultrasonic velocity and attenuation a number of techniques can be used. These techniques can be broadly classified into three categories, which are,

(a) Pulse methods,
(b) Continuous wave methods,
(c) Low frequency methods.

(a) Pulse methods

In these techniques, a short pulse of sound waves is generated using a piezoelectric transducer (usually quartz) which is bonded to the crystal under investigation. The crystal for the experiment should be cut and polished to have a pair of end faces plane and parallel in the desired direction. The sound waves excited in this direction will get multiply reflected from the end faces and will produce an electric signal each time it hits the transducer. These electric echo pulses are amplified and displayed on an oscilloscope or processed otherwise. From the length of the sample, the transit time of the pulse in the sample, and the decay rate of the pulse amplitudes of the successive echoes, the velocity and attenuation can be estimated. The absolute accuracy of such methods are generally about 1%.

Using Phase sensitive methods the absolute accuracy can be increased in certain cases of pulse techniques and very high precision of $10^{-6}$ can be obtained in the measurement of changes in velocity. There are various kinds of phase sensitive methods: pulse superposition [2.1,2.2], phase
comparison [2.3], sing-around method [2.4,2.5], and pulse echo overlap method [2.6]. More details of these methods are available in specialized review articles [2.6-2.10].

b) Continuous wave methods

Standing waves or Continuous wave (CW) methods have also been successfully applied in various problems in physical acoustics. Similar to a Fabry-Perot interferometer one excites standing wave resonances generally also with quartz transducers. For a sample length $L$ the number of excited resonances of frequency $f$ is,

$$n = \frac{2Lf}{v} \quad (2.01)$$

whereby the sound velocity $v$ can be determined. For 10 MHz $n$ is of the order of $10^2$. Using frequency modulation techniques one can measure changes in velocity and attenuation with high precision. A detailed discussion about this method is given in review articles [2.11,2.12].

(c) Low frequency methods

The lower limit of frequency is given by the sample dimensions. Here the number $n$ in (2.01) is of order unity. In this case the elastic compliances (Youngs modulus $Y$ and Shear modulus $G$) are determined by a CW resonance method or by measuring flexural and torsional oscillations. These techniques are described by Read et al. [2.13]. These methods are particularly suitable for piezoelectric materials which can be excited into mechanical resonances by an electric field directly without transducers [2.14]. Other nonpiezoelectric crystals can be measured in a similar way with additional dc bias field using the electrostrictive effect [2.15]. The low frequency dynamic resonance methods are also described by Schreiber et al. [2.16].

In ultrasonic experiments the frequency is usually in the range of 10 to 100 MHz. The upper limit in frequency is given
by the precision to which planeness and parallelism of the two reflecting end faces can be achieved. For coherent detection this precision has to be about 1/10 of the acoustic wavelength. For high acoustic quality materials one can go to microwave frequencies. But at GHz frequencies the Brillouin light scattering is a better technique for the measurement of elastic properties.

A serious problem with ultrasonic propagation near structural phase transitions is the bonding of transducers to the sample. Because of thermal expansion and the occurrence of spontaneous strains in the low symmetry phase the transducer sample bond may crack under such conditions [2.17].

2.1.2 The method of Pulse Echo Overlap

The Pulse Echo Overlap (PEO) method was first invented in 1958 by John E. May [2.18] and modified to essentially its present form in 1964 by E. P. Papadakis [2.6]. This modification, to a large extent, has depended on the development of McSkimin's Pulse Superposition Method [2.1,2.2]. The necessary factor in McSkimin's method, which was borrowed for the PEO method, is McSkimin's calculation of the correct cycle-for-cycle superposition (overlap in PEO method) of the rf cycles in echoes from long pulsed rf wave forms. The correct overlap, obtained by using McSkimin's calculation, permits travel time measurements with high absolute accuracy. The PEO method is still the most widely used technique to measure the velocity of sound in solids [2.8].

The fundamentals of PEO measurement can be understood by examining the block diagram in Fig. 2.1. The CW oscillator which is the basic clock of the system supplies the frequency which is to be set at the reciprocal of the delay time between the echoes to be measured. The CW signal goes to three places; (1) to the counter which measures the exact frequency of the
Figure 2.1
Block diagram of the Pulse Echo Overlap Method for measuring the travel time of the waves.
CW oscillator, (2) to the x-axis of the CRO to display the overlapped echoes when the CRO is in the x-y mode, (3) to the frequency divider to provide synchronous triggers for the pulsed rf oscillator and for the delay circuits. The delay generators provide the two synchronized intensifying pulses of adjustable width and delay to permit the observation of two selected echoes by intensifying the CRO display at the two echoes of interest. The rf generator must be a pulsed oscillator (not gated type) so that the phase of the rf is synchronous with the divided trigger generated from the CW oscillator. The rf pulse energizes the piezoelectric transducer, which sends the ultrasonic signal and receives its echoes. The echoes go to the y-axis of the CRO after amplification. The diode pulse limiter protects the amplifier input from the high power rf pulses.

When the CRO is in linear sweep mode the x-axis sweep is triggered by the sync input signal which is the same as the one triggering the rf pulsed oscillator. In this mode if the time base is properly set then all the echoes, with exponentially decaying amplitudes, along with the first rf pulse can be seen on the screen. The two echoes of interest between which the time delay is going to be measured can now be selected by positioning the intensifying pulses on them by adjusting the delay and width of the intensifying pulses. The approximate time interval between the echoes can now be noted from the CRO to enable an initial frequency setting for the CW oscillator. When the CRO is switched to the x-y mode the x-axis sweep is produced by the cw oscillator and a sweep is there corresponding to every echo. The echoes appear on the screen one after the other on successive sweeps. Due to the persistence of vision the echoes appear as if one is overlapped on the other. By adjusting the intensifying pulse amplitude the two echoes of interest alone can be made visible in the overlapped condition. The overlap is exact if the CW frequency is equal to the reciprocal of the time interval
between the echoes. The echoes appear on the screen in an expanded form with the individual rf cycles in the echo visibly resolved and an rf cycle to cycle overlap can be achieved by fine tuning of the CW oscillator. The frequency of the CW signal can now be obtained from the frequency counter, the reciprocal of which gives the round trip travel time of the echoes in the sample. By knowing the length of the sample the velocity can be computed. In PEO method the next rf pulse is applied only after all the echoes in the sample have died out. This is ensured by switching the frequency divider to 10, 100 or 1000 division mode.

In contrast with PEO method, in pulse superposition method the rf pulses are applied at a rate which corresponds to simple multiple of echo interval, thus producing actual interference or superposition of waves in the sample. The cycle to cycle overlap is achieved in pulse superposition technique from the amplitude variations, resulting from the constructive and destructive interference of the waves as the CW frequency is varied.

Due to attenuation and other pulse distortion effects the number of rf cycles in the two selected echoes will be different and hence there is no easy way to find which cycle of the first echo should be overlapped with which cycle of the second echo. In Section 2.3 we discuss how Mc Skimin’s calculation can be used to find the correct overlap along with our contributions to the basic technique.

2.1.3 Measurement of attenuation

Ultrasonic attenuation is defined by the solution

\[ A = A_0 e^{-\alpha x} \cos(kx - \omega t) \]  

(2.02)

for the ultrasonic wave propagating in the x-direction with a propagation constant \( k = 2\pi/\lambda = 2\pi f/v \), a radian frequency \( \omega = 2\pi f \), and an attenuation coefficient \( \alpha \). In these
definitions \( \lambda \) is the wavelength, \( f \) the frequency and \( v \) the phase velocity. The ultrasonic attenuation as defined in Eq. (2.02) must be measured as the logarithm of the ratio of the amplitude of the ultrasonic wave at two distances along its propagation path. Then \( \alpha \) is given as

\[
\alpha = \frac{\ln(A_1/A_2)}{(x_2 - x_1)} \quad (2.03)
\]

in units of nepers per unit length or

\[
\alpha = \frac{20 \log_{10}(A_1/A_2)}{(x_2 - x_1)} \quad (2.04)
\]

in decibels per unit length for amplitudes \( A_1 \) and \( A_2 \) sensed at positions \( x_1 \) and \( x_2 \).

Various schemes have been used in pulse techniques to find the amplitudes of echoes. Comparison pulses run through an attenuation box and a delay line have been applied on alternate or chopped oscilloscope sweeps to find amplitude directly in decibels [2.19]. An ingenious arrangement has been devised by Chick et al [2.20] displaying an electrically generated decaying exponential function and the echoes on alternate sweeps of an oscilloscope. The decaying exponential is calibrated in decibels per microsecond as a matter of convenience from the electronic standpoint, although nepers per centimeter is a more natural unit in theoretical derivations. The slope of the decaying exponential can be varied by a ten-turn dial, so that the attenuation between any pair of echoes can be measured.

The attenuation can be most conveniently measured by an automatic procedure for which commercial equipment is available (Matec. Inc. (USA) Model 2470). In the automatic system, two gates with variable delay are set on the two echoes of interest to sample them. The amplitude of the first echo is held constant by AVC circuitry, and the amplitude of the second echo is sampled at its peak. A calibrated logarithmic amplifier converts the sampled amplitude to
decibels relative to the constant amplitude of the first echo. The decibel level is recorded on a built-in strip chart that has several calibrated scales and a variable baseline, so that small changes in attenuation can be measured at various total loss levels. In this equipment the attenuation can also be noted from a panel meter calibrated in dB.

2.2 The experimental setup

2.2.1 The basic experimental setup

The experimental setup used for making the ultrasonic measurements consists of mainly the PEO system, the temperature measuring and control system, the cryostat for low temperature measurements and the oven for high temperature measurements.

The PEO system was setup mostly by using equipments from MATEC. Inc. (USA). These equipments include Matec Model 7700 pulse modulator and receiver together with model 760 V rf plug-in, Model 110 high resolution frequency source, Model 122 B decade divider and dual delay generator, Model 2470 B attenuation recorder, Model 70 impedance matching network etc. The frequency counter used was HIL (India) Model 2722 and the oscilloscope was a 100 MHz one with z-axis input (HIL Model 5022). For temperature measurement and control Lakeshore Cryogenics (USA) Model DR 82C temperature controller was used. The cryostat used was specially designed and fabricated for the ultrasonic measurements at low temperatures using liquid nitrogen as the cryogen. The details of this cryostat will be discussed separately in the next section (sec. 2.2.2). A high temperature oven for Ultrasonic measurements from System Dimension (India) was used for high temperature measurements.

The block diagram of the experimental setup used is shown in Fig. 2.2. The tunable cw source (model 110) has a highly stable internal high frequency oscillator (12-50 MHz) from
Figure 2.2
Block diagram of the Experimental setup
which the required low frequency cw signal for PEO is generated by selectable frequency division. This signal is available at terminal 2, while the high frequency is available at terminal 1 for accurate counting by the frequency counter (model 2722). The dual delay and divider unit (model 122B) has dividers selectable as 10, 100 or 1000. The division factor 100 is quite acceptable for most measurements, which means that the next rf pulse is sent to the sample only after a time interval corresponding to 100 number of echoes. The terminal 2 of this unit gives trigger pulses for the CRO. The CRO is always operated in the external sweep trigger mode. By using switch SW2 in 122B the trigger at terminal 2 can be made direct trigger or divided trigger for observing the overlapped echoes or the full echo pattern respectively. The dual delay generators in 122B can be adjusted for delay and width of the intensifying pulses for selecting the two echoes of interest for overlap. These pulses are available at terminal 3 and are connected to the z-input of the CRO through the selector switch SW1.

The divided trigger from 122B goes to the pulse modulator & receiver unit (model 7700 with rf-plug-in model 760 V). This is the most important unit in the setup. An rf pulse packet of peak power 1 KW is obtained at the output when the unit is triggered at the input. The rf frequency of the triggered power oscillator can be adjusted in the range 10 to 90 MHz. The width and amplitude of the pulse are adjustable. For good pulse shape the unit is usually operated at full amplitude and the amplitude reduction is achieved by using an rf attenuator (Alan Attenuator) at the output as shown in Fig.2.2. The unit has a sensitive tunable superhetrodyne receiver with a maximum gain of 110 dB for amplifying the echoes. The amplified echoes are available through the receiver out terminal which is connected to the CRO channel 1 input. The amplified echoes are also detected and the detected output (envelope of the echoes) is available at the video out terminal which is connected to
the attenuation recorder and CRO channel 2 input. For optimum signal to noise ratio in the amplification of weak echoes, an impedance matching network (model 70) is connected before the quartz transducer.

The working of the attenuation recorder (model 2470B) is as discussed in Section 2.1.3. The switch SW1 can be toggled to pole 2 for connecting the intensifying pulse output from the attenuation recorder to the Z-input of the CRO. By this way the two echoes of interest can be selected for attenuation measurement. One important advantage of the PEO technique is the capability to measure the velocity and attenuation at the same time.

A timing diagram of the various signals in the measurement system is given in Figure 2.3. The first and second echoes are shown as selected. In a typical setting one divided sync pulse will be produced for every 100 direct sync pulses. The diagram shown is for an overlapped situation.

2.2.2 The fabricated cryostat

For the low temperature ultrasonic measurements on crystals, we have designed and fabricated a cryostat with Liquid Nitrogen as the cryogen. The essential parts of the cryostat are shown in Figure 2.4. The metallic outer case is one meter long. The top lid is removable and is made vacuum tight by using a rubber "O" ring of 27 cm diameter. The 50 cm long liquid nitrogen inlet tube along with the liquid nitrogen reservoir are welded to the top lid of the cryostat. The liquid nitrogen reservoir has a thick copper bottom to which the sample chamber assembly is tightly bolted. The sample chamber assembly is a 24 cm long, thick copper hollow cylinder with 8.5 cm internal diameter and having a removable bottom cover. A 6 mm dia copper tube is wound on the outer side of the sample chamber assembly and ends of this tube is connected to the nitrogen reservoir such that liquid nitrogen flows
Figure 2.3
A timing diagram of the various system signals. The interval $t$ is the travel time in an overlapped situation.
through this tube under the action of gravity. Thus the sample chamber assembly works like an efficient cold finger due to the circulating liquid nitrogen. This arrangement also helps to create a uniform temperature in the sample chamber. The sample holder and heater assembly are introduced into the sample chamber after removing the bottom cover of the sample chamber assembly. The sample holder consists of four long threaded rods with plates fixed to these rods using nuts. The sample along with the ultrasonic transducer are held between the plates with the help of a spring loaded arm fixed to the top plate. The sample holder is surrounded by a 20 cm long copper hollow cylinder having removable lids on top and bottom. Thin insulated copper wire is closely wound on this copper cylinder to almost fully cover its outer surface. This copper wire winding acts as the heater element for temperature regulation. Due to the extended nature of this heater winding the sample holder is uniformly heated and the temperature gradients are minimized.

The special features of this cryostat can be summarized as follows.

(1) It has a large sample chamber and sample holder with minimum temperature gradients to accommodate large crystal samples used in ultrasonic investigations.

(2) Large thermal mass provides slow cooling and heating and prevents sudden heating and cooling which would easily crack large crystal samples.

(3) A rope and multi-pulley arrangement with good mechanical advantage is provided to easily lift and lower the lid assembly. This makes the loading and unloading of the sample easy.

(4) It has a cold finger with circulating liquid nitrogen to ensure the uniform cooling of the sample chamber.

(5) A distributed heater coil with copper wire for uniform heating and temperature regulation.

(6) An optical window to perform experiments involving light
Figure 2.4

The cryostat for low-temperature measurements
interaction with ultrasonic waves. (This facility was not used for the present work but was included in the design as an optional facility for future applications of this cryostat). A photograph of the whole experimental setup including the cryostat is shown in Plate 2.1.

2.3 Bond correction and overlap identification

2.3.1 Basic theory

The following analysis [2.1, 2.2, 2.6-2.8, 2.21] pertains to a burst of ultrasonic waves of principally one frequency echoing back and forth within a specimen. The specimen is bare on one end but has a transducer bonded to the other end. The transducer is half a wavelength thick at its resonant frequency, and the bond has a finite thickness. The transducer is unbacked. We will consider the effect of the bond and transducer upon the phase of the reflected wave at the bonded end of the specimen in order to derive a method of choosing the correct cycle-for-cycle matching of one echo with any subsequent echo.

Consider the sketch of the specimen, bond, and transducer in Fig. 2.5. The phase angle \( \gamma \) relating the reflected wave phase to the incident wave phase is defined in that figure, as are the impedances of the specimen, bond, and transducer. The specific acoustic impedances are \( Z_s \) for the specimen, \( Z_1 \) for the bond, and \( Z_2 \) for the transducer. The impedance looking into the termination (bond and transducer) from the specimen is \( Z_d \). Since the transducer is unbacked the impedance \( Z_a \) seen by it at its back is that of air, which is approximately zero.

Assuming negligible attenuation in bond and transducer, the theory of lossless transmission line [2.22] can be used to obtain an expression for \( Z_d \) as
Plate 2.1
The photograph of the experimental setup
The phase angle $\gamma$ generated at each reflection of the echoes from the specimen/bond/transducer interface is shown here diagrammatically.
\[
Z_d = jZ_e = jZ_1 \left[ \frac{(Z_1/Z_2) \tan \beta_1 l_1 + \tan \beta_2 l_2}{(Z_1/Z_2) - \tan \beta_1 l_1 \tan \beta_2 l_2} \right]
\] (2.05)

where \( \beta_1 \) and \( \beta_2 \) are the propagation constants in the bond and transducer, and \( l_1 \) and \( l_2 \) are the thicknesses of the bond and transducer, respectively. The propagation constants \( \beta_1 \) and \( \beta_2 \) are related to the ultrasonic frequency \( f \) impressed upon the transducer by the RF pulse generator and also to the velocities \( v_1 \) and \( v_2 \) of the wave in bond and transducer respectively. The relations are

\[
\beta_1 = \frac{2\pi f}{v_1} \quad \text{and} \quad \beta_2 = \frac{2\pi f}{v_2}
\] (2.06)

\( Z_d \) can now be used to define the ratio of reflected to incident pressure as

\[
\frac{E_b}{E_i} = \frac{Z_d - Z_s}{Z_d + Z_s}
\] (2.07)

With \( Z_d \) imaginary \( (Z_d = jZ_e) \), the real and imaginary parts of Eq. (2.07) can be separated as

\[
\frac{E_b}{E_i} = \frac{Z_s^2 - Z_e^2}{Z_e^2 + Z_s^2} - \frac{j2Z_e Z_s}{Z_e^2 + Z_s^2}
\] (2.08)

from which the phase angle \( \gamma \) on reflection is given by

\[
\tan \gamma = \frac{-2Z_e Z_s}{(Z_s^2 - Z_e^2)}
\] (2.09)

for the vectors sketched in Fig. 2.5 (c). The impedance \( Z_e \) is as defined in Eq. (2.05).

In these formulae there is one unknown parameter, the bond thickness \( l_1 \), and one running variable, the ultrasonic frequency \( f \). By varying \( f \) one can change \( \beta_1 \) and \( \beta_2 \) using the
relations (2.06).

The phase angle $\gamma$ is the relevant measure of the effect of the transducer and bond upon the reflected wave. Mc Skimin has shown that the measured travel time $t_\text{H}$ is made up of the true round-trip travel time $t$ plus some increments:

$$t_\text{H} = pt - \left( \frac{p\gamma}{2\pi f} \right) + \left( \frac{n}{f} \right) \tag{2.10}$$

Here $p$ is the number of round trips in the measurement. The phase angle $\gamma$ per reflection yields a fraction $\gamma/2\pi$ of a period of the RF, so a time increment $\gamma/2\pi f$ is generated per round trip. Also a mismatch of $n$ cycles will yield a time error $n/f$. The object of the mathematical analysis is to develop a method to find the overlap case $n = 0$, and to minimize and estimate the residual $\gamma$.

It is clear from the above equations that both $\gamma$ and $t_\text{H}$ are functions of the frequency $f$. It is possible to utilize this dependence of $t_\text{H}$ on frequency to eliminate $n$, the mismatch. To do this consider the possibility of making the measurement of $t_\text{H}$ at a high frequency $f_\text{H}$ and at a low frequency $f_\text{L}$ (for example, at the resonant frequency $f_\text{H} = f_\text{r}$ and at $f_\text{L} = 0.9f_\text{r}$). Then $t_\text{H}$ is measured time at $f_\text{H}$ and $t_\text{L}$ is measured time at $f_\text{L}$. These times are

$$t_\text{H} = pt - \left( \frac{p\gamma_\text{H}}{2\pi f_\text{H}} \right) + \left( \frac{n}{f_\text{H}} \right) \tag{2.11}$$

$$t_\text{L} = pt - \left( \frac{p\gamma_\text{L}}{2\pi f_\text{L}} \right) + \left( \frac{n}{f_\text{L}} \right)$$

where the same overlap condition (same $n$) has been maintained by shifting the repetition frequency slightly as the RF frequency is changed. Subtraction eliminates $t$, the true travel time, as
Equation (2.12) expresses Mc Skimin's Δt criterion for finding n = 0 case. Stated verbally, Eq. (2.12) indicates that if \( f_L, f_H, t_L, \) and \( t_H \) are measured, and if \( \gamma_L \) and \( \gamma_H \) are computed from \( l_1, l_2, v_1, v_2, Z_1, Z_2, \) and \( Z_s \) by Equations (2.05), (2.06) and (2.09), then there is only one possible value for \( \Delta t_M \) when \( n = 0 \). Conversely, if \( n = 0 \) is set in the measurement, the measured value of \( \Delta t_M \) will agree with the value calculated theoretically.

2.3.2 The correction and identification technique

To apply the Mc Skimin Δt criterion to PEO technique, one follows the following procedure:

1. With the oscilloscope on triggered sweep with divided sync, find the approximate time between echoes of interest from the graticule of the CRO, and set the cw oscillator frequency to have that time as its period.

2. Switch to the direct sync sweep trigger mode and adjust the cw oscillator frequency to bring about a plausible overlap with the leading edges of the echoes nearly aligned and every cycle of the later echo smaller than the corresponding cycle of the earlier echo because of attenuation.

3. Measure \( t_L \) and \( t_H \) of Eq. (2.11) at proper frequencies \( f_L \) and \( f_H \), usually \( 0.9f_r \) and \( f_r \).

4. Repeat step 3 for several possible adjacent overlaps, e.g., three toward lower cw repetition frequencies and as many towards higher.

5. Compute \( \Delta t_M \) of Eq. (2.12) for each of these sets of measurements.

6. Compare \( \Delta t_M \) found experimentally with theory, and
choose the correct cycle- for-cycle match. Then $t_M$ measured at $f^n = f_r$ will be $t_M$ in Eq. (2.10) with $n = 0$. This time $t_M$ is the correct expression for the measured time before correction for bond phase shift.

The step no. 6 given above for choosing the correct overlap is not a straightforward procedure. This is because of the unknown bond thickness and consequent difficulty for computing a theoretical value of $\Delta t_M$ for comparison. To overcome this difficulty a graphical technique is often used. For this it is assumed that the bond is very thin, that is less than a quarter of the acoustic wavelength (this condition is usually satisfied in experimental situation). This means that the bond phase $\beta_1$ expressed in degrees can be in the range 0 to 90 degrees. Using the equations presented above, the value of $\Delta t_M$ can be computed (for $n = 0$ case) for increasing values of bond angle from 0 to 90 degrees. A plot of $\Delta t$ vs bond angle is then made. This gives a range of possible $\Delta t$ values for the given transducer, bond, and sample combination for $n = 0$ case. It can then be examined, which overlap-case in the measured values falls in this range and that overlap-case can be taken as the $n = 0$ case. Thus the correct overlap can be identified. Next step is to find the correction factor. In Eq. (2.10) we see that the correction can be calculated if the phase angle $\gamma$ is known. All parameters except the bond thickness (or bond angle), are known for the computation of $\gamma$. The bond angle can now be found out from the plot as the angle corresponding to the measured value of $\Delta t$ which has fallen in the possible range. Thus the correction factor in Eq. (2.10) can be estimated and the true round-trip time $t$ in the sample can be obtained. It may be noted that a graph has to be plotted for every measurement.
2.3.3 The computer program developed for bond correction and overlap identification

The usually used manual computation and graphical procedure for finding the correct overlap and bond correction which have been discussed in Section 2.3.2 is very laborious especially if a number of measurements are to be made. We have developed a numerical procedure and a computer program which can process the experimental data and output the corrected velocity and elastic constant. This program is extremely useful for experimenters who are making ultrasonic measurements using pulse echo overlap technique or pulse superposition technique. The special features of our numerical procedure includes a linear regression technique for eliminating random errors in experimental observation, automatic computation of RF pulse frequency, auto finding of the correct overlap and a manual mode for selecting an overlap-case in the event when no overlap-case is found in the possible range of \( \Delta t \) values.

The importance of the linear regression technique which we have introduced as an improvement of the basic technique can be understood as follows.

Consider the equations (2.11). They define the measured values of travel time \( t_L \) and \( t_H \) corresponding to frequencies \( f_L \) and \( f_H \) respectively. An examination of these equations reveal that they are equations of straight lines of the form

\[
t_H = A_H + nB_H \\
t_L = A_L + nB_L
\]

(2.13)

where,

\[
A_H = pt - \left( p\gamma_H / 2\pi f_H \right) \\
A_L = pt - \left( p\gamma_L / 2\pi f_L \right)
\]

(2.14)

and
\[ B_H = 1/f_H \quad (2.15) \]
\[ B_L = 1/f_L \]

In an actual measurement the correct value of \( n \) is initially unknown and Eq.(2.13) can be written as

\[ t_H = A_H + XB_H \]
\[ t_L = A_L + XB_L \quad (2.16) \]

where \( X \) is an arbitrary overlap number and it can be defined as \( X = (n + m) \), where \( m \) is an unknown integer whose value is known when the correct overlap is identified.

In usual technique the value of \( \Delta t \) for successive overlap number \( X \) is obtained as the difference \( (t_L - t_H) \) of the measured \( t_L \) and \( t_H \). Here \( t_L \) and \( t_H \) are large numbers when compared to their difference \( \Delta t \) and hence even very small errors in the measurement of \( t_L \) or \( t_H \) can greatly influence the value of \( \Delta t \). This can lead either to incorrect identification of the correct overlap or to a situation where none of the measured \( \Delta t \) values are in the calculated range.

In the method we have developed the \( \Delta t \) value is not directly obtained from the measured values of \( t_L \) and \( t_H \) for each case of overlap but it is obtained from the equation

\[ \Delta t = (A_L - A_H) + X(B_L - B_H) \quad (2.17) \]

The coefficients \( A_L, A_H, B_L \) and \( B_H \) are obtained from linear regression analysis with least squares fitting of measured \( t_L \) and \( t_H \) on successive values of overlap \( X \). Eq. (2.16) shows that the points of \( t_L \) plotted against the overlap no. \( X \) should ideally lie on a straight line. This is also the case with \( t_H \). In regression analysis \( t_L \) or \( t_H \) are fitted to two different straight lines against \( X \) values. Because of the fitting process the random errors in the measurement of \( t_L \) and \( t_H \) cancel out to some extent and the overall accuracy is
increased. The correlation coefficient is also computed which gives a measure of how well the measured data fits into a straight line. Further, the deviation of each of the measured values of \( t_L \) and \( t_H \) from the fitted straight line is also computed to clearly indicate the errors involved.

Another useful feature of our straight line fitting technique is the estimation of the frequency of the RF pulse which excite the ultrasonic waves. This frequency is usually difficult to measure. A frequency counter cannot be used because of the small duty cycle plus the pulsed nature of this waveform. Eq. (2.15) shows that the required frequencies \( f_L \) and \( f_H \) can be estimated as the reciprocal of the fitting coefficients \( B_L \) and \( B_H \) respectively. Hence a separate measurement of these frequencies is not necessary in the present technique.

A description of the program developed for correct overlap identification and to apply bond correction is given below. The program was written in BASIC language and the source code of the program is listed in Appendix 2.1. The program can run on an IBM PC or compatible with GWBASIC interpreter or with compilers like Turbo Basic or Quick Basic.

The program opens with a menu of three choices: 1) To create a data file, 2) To process a data file, 3) To quit the program. If the first option, for creating data file, is selected then the program requests various parameters like sample name, bond material name, direction of propagation, length of sample in cm, whether wave is longitudinal or transverse, and if transverse the direction of polarization, number of data values, value of \( p \), repetition rates in MHz for high and low RF frequencies for different cases of overlap, bond impedance, sample density, and any other information. These experimental data are then written on to a data file whose name can be specified. The program then returns to the opening menu where the next option to process the data can be selected.
If the process data file option is selected then on entry of the data file name the file is read from the disk and processing begins. The linear regression process is first performed to find the straight line fitting coefficients and the correlation coefficients for $t_L$ and $t_H$. The intersection of the straight lines corresponds to the zero $\Delta t$ value. It can be shown that for the case of correct overlap the value of $\Delta t$ is a small negative value which is usually one or two overlap cases away from the overlap case near to the zero $\Delta t$ value. From the fitted lines the experimental $\Delta t$ values are estimated for different cases of overlaps, 4 no. of $+\Delta t$ cases and 4 no. of $-\Delta t$ cases from the overlap case near the zero $\Delta t$ value, and are tabulated. The low and high RF frequencies are then estimated from the slope of the lines. It is assumed in the processing that the estimated high RF frequency corresponds to the resonant frequency of the transducer. The acoustic impedance of the sample is also estimated as the product of sample density and approximate velocity which is estimated from sample length and $t_H$ corresponding to the overlap case near the zero $\Delta t$ value.

All parameters necessary to calculate the $\Delta t$ value corresponding to any bond angle is now ready. It is assumed that for thin bonds the bond angle is in the range from 0° to 60°. The $\Delta t$ values corresponding to 0° and 60° are now estimated and one of this $\Delta t$ values is the minimum $\Delta t$ value in this range of bond angle. The curve of $\Delta t$ vs bond angle is like an approximate parabola and it usually has a maximum in between 0° and 60° bond angles. This maximum $\Delta t$ in this range of bond angle is now estimated using the Interval Halving numerical search procedure. The maximum is found in less than 20 iterations. The estimated minimum and maximum values of $\Delta t$ gives the range of possible $\Delta t$ values for $n = 0$ case or for the correct overlap. The experimental values from the straight line fitting is now searched for the case which fall in the possible range of $\Delta t$. Thus the correct overlap case is
identified. To apply the bond correction the bond angle should be known. For this Equation (2.12) is solved numerically by using Bisection Method to find the root (bond angle) corresponding to the observed $\Delta t$ value in the possible range. The correct velocity is now calculated and the corresponding elastic constant is also found as the product of the square of velocity and the density of the sample. In case, due to some experimental problem, no experimental $\Delta t$ values are falling in the possible range then there is an option to manually select an overlap case and calculate the velocity and elastic constant without bond correction. After printing or displaying the results the program goes back to the opening menu.

2.4 Sample preparation techniques

In this section details of sample preparation technique and the instrumentation developed for the same are described. The samples used were Lithium Hydrazinium Sulphate, Lithium Ammonium Sulphate, and Lithium Potassium Sulphate. These crystals are water soluble and hence they can be crystalized from their aqueous solutions.

2.4.1 Crystal growth from solution

Growth from solutions [2.23-2.28] is the most widely used method of growing crystals. It is always used for substances that melt incongruently, decompose below the melting point, or have several high-temperature polymorphic modifications. Even in the absence of above restrictions the crystal growth from solution is an efficient method. The equipment needed is relatively simple, the crystals exhibit a high degree of perfection, and the conditions of growth-temperature, composition of the medium and types of impurities can be widely varied. On the other hand, in contrast to other methods like growth from melt or vapor, in solution growth the
crystals are not grown in a one component system. The presence of other components (a solvent) materially affects the kinetics and mechanisms of growth. The migration of nutrient to the crystal faces is hindered, and thus diffusion plays an important part. The heterogeneous reactions at the crystal-solution interface are complicated by adsorption of the solvent on the growing surface and by the interaction between the particles of the crystallizing substance and the solvent (hydration in aqueous solutions). A theoretical analysis or description of the effects of the above factors on the mechanism of growth, the morphology, and the deficiencies of crystals is generally rather complicated. For this reason, empirical investigations on solution growth are to be given more weight. The skill of the crystal grower is of great importance in producing good quality crystals.

Controlled crystal growth is possible only from metastable solutions. The driving force of the process is the deviation of the system from equilibrium. This can be conveniently characterized either by the supersaturation $\Delta C$ or by the value of "supercooling", $\Delta T$, i.e., the difference between the temperature of saturation of the solution and that of growth. The supercooling $\Delta T$ is related to the supersaturation $\Delta C$ by the expression

$$\Delta C = (\delta C_0 / \delta T) \Delta T$$  \hspace{1cm} (2.18)

where $\delta C_0 / \delta T$ is the temperature coefficient of solubility, i.e., the change in solubility of the substance per 1°K change in the temperature of the solution.

The methods of growing crystals from solutions are classified into several groups according to the principle by which supersaturation is achieved.

1) Crystallization by changing the solution temperature. This includes methods in which the solution temperature differs in different parts of the crystallization vessel (temperature-difference methods), as well as isothermal crystallization, in which the entire volume of the solution is
cooled or heated.

2) Crystallization by changing the composition of the solution (solvent evaporation)

3) Crystallization by chemical reaction.

The choice of the method mainly depends on the solubility of the substance and the temperature solubility coefficient $\frac{\partial C_0}{\partial T}$. For many crystals both the slow cooling technique and the constant temperature solvent evaporation technique can be successfully used.

For a continuously growing crystal the substance has to be transported to the growing face from the solution. In a motionless solution the delivery of the substance is by a slow diffusion process. In a pure diffusion regime the supersaturation differs over different areas of the faces. To reduce this nonuniformity of the supersaturation and nutrition of different areas of the faces, and for faster mass transport for increased growth rate, motion of the crystal and solution relative to one another must be ensured. In low-temperature aqueous solutions, rotation of the crystal in solution or stirring is usually applied.

The samples necessary for our investigations have been grown by constant temperature solvent evaporation method with solution stirring as well as with crystal rotation.

2.4.2 The fabricated constant temperature bath

In this section the details of the bath which has been fabricated for crystal growth at constant temperature by solvent evaporation method are discussed. A special feature of this constant temperature bath is that it is protected against mains power failures by using an automatically recharged battery backup.

The bath is a glass tank on iron frame measuring about 40 cm in length, 28 cm in width and 25 cm in height. Figure 2.6 illustrate the essential details of the bath. The tank is
1 - Crystal rotator
2, 3 - Bath stirrers
4 - Thermocol granules
5 - Bath liquid
6 - Solution
7 - Growing crystal
8 - Temperature sensor
9, 10 - Bath heaters
11 - Outer puf lining

Figure 2.6
The constant temperature bath for crystal growth
given a 1 cm thick heat insulating outer lining made of polyurethine foam. This is to prevent unnecessary heat loss from the bath and hence to reduce the power required to keep the tank at a regulated temperature above room temperature. For the same reason, thermocole granules are also allowed to float on the bath-water surface to reduce evaporation and consequent heat loss. There is a window with a foam shutter in front of the tank made in the foam lining for observing the growing crystal. Heater coils which are sealed inside glass tubes are fitted at the bottom of the tank. This prevents any electrical contact between bath-water and heater coils. A Diode temperature sensor enclosed in an oil filled glass tube is kept in the bath-water for temperature sensing. This arrangement of heater and sensor permits a closed loop proportional temperature control of the bath. The details of the temperature controller are discussed in the next section (sec. 2.4.3). Two stirring motors with stirrers are fitted near left and right ends of the bath for uniform temperature distribution in the bath.

The solution for crystal growth may be taken in a beaker or a wide mouth conical flask of 500 ml capacity and can be kept dipped in the bath at a suitable depth by using a bench made of perspex. The seed crystal was attached to a thin perspex strip and was hung in the solution from a rotation mechanism. The rotation mechanism consists of a DC motor which can rotate in both directions and a reduction gear arrangement for slowing down the motor speed. The motor is driven by specially designed control circuit which can periodically reverse the rotation direction and having facility for presetting the motor speed. A description of this control circuit is given in section 2.4.4. The bidirectional rotation of the growing crystal is very essential for perfection in growth. The same control circuit can also be used for controlling a stirrer in the solution and this way the solution can be periodically stirred in opposite directions.
2.4.3 The designed temperature controller

A temperature controller for crystal growth by constant temperature solvent evaporation technique has to meet several requirements. It is necessary to have precise control of the temperature of the bath for durations as long as several weeks or for a few months when large single crystals of good quality are required. Any temperature fluctuation during this period is likely to introduce defects into the growing crystal. In a temperature controlled bath which is operating from AC mains, even a brief power failure can cause a damaging temperature fluctuation. One solution to this problem is to use a battery backup for the control power. But the available bath temperature control circuits [2.29,2.30] and general purpose temperature control circuits [e.g. 2.31] are not easily adaptable for battery backup.

We have designed and fabricated a proportional temperature controller that can be used ideally for crystal growth experiments of long duration. The control is protected against power failures by using a battery backup. The circuit diagram and other details of this controller have been published by us [see Ref. 2.32]. A description of the circuit and its operation is given below.

A block diagram of the temperature controller is shown in Fig. 2.7 and its circuit diagram is shown in Fig. 2.8. For sensing the temperature, a forward biased emitter-base junction of a silicon transistor (BC178) is used with its collector shorted to the base [2.33]. This forms a sensitive and linear temperature sensor as the forward voltage has a negative temperature coefficient of about -2 mV K\(^{-1}\). The junction is forward biased with a constant current of about 200 \(\mu\)A. Transistor TR1 acts as a constant current source. The three terminal voltage regulator IC 7805 provides a stable output voltage of 5V, from which the reference voltage for temperature setting is derived. A multi-turn potentiometer VR1
is used to set the temperature. An Instrumentation amplifier formed with two operational amplifiers A1 and A2 acts as an error amplifier. The gain of this stage is $R_6/R_9$, with $R_6 = R_7$ and $R_9 = R_8$, and is about 50 in this circuit. A triangle-wave generator with operational amplifier A3 provides a triangle wave of amplitude 0.5 V across C3 with a positive offset with respect to the midpoint voltage at M. The period of this waveform is

$$T \approx 0.2(R_{14})(C_3)$$

and is about 10 ms in this circuit.

An operational amplifier comparator A4 compares the error signal with the triangle wave and provides a square wave, the pulse width of which is proportional to the error signal. The output of A4 is amplified by the current amplifier configuration formed by TR2, TR3, and TR4 which in turn drives the heater load. Any decrease in temperature from the set value will increase the error voltage, causing the pulse width to increase, resulting in more average power to the load, which in turn corrects the decreasing temperature, thereby completing the control loop. This pulse width modulation technique of proportional power control was also successfully used by the author earlier in an high-temperature control circuit [2.34]. The switching type of power output has an advantage that there is no power loss in the output transistor and the system becomes highly power efficient, a factor which is highly relevant when battery backup is used.

The circuit diagram of the power supply for the temperature controller is shown in Fig. 2.9. Operational amplifier A5 along with transistors TR5 and TR6 produce a symmetrical dual supply voltage for the controller circuit. While the power is ON, TR7 acts as a series pass voltage regulator with the reference voltage taken from the battery which is being charged through R22 and D3. During the power ON period the relay is active and the normally open (NO) relay contact is closed. As a result, power is taken from the
Figure 2.7
Block diagram of the Temperature controller
Figure 2.8
Circuit diagram of the temperature controller
Figure 2.9
Circuit diagram of the temperature controller power supply
emitter terminal of TR7. During a power failure the normally closed (NC) relay contact is closed and power is taken from the battery. During the power ON period the battery is given only a trickle charging to avoid overcharging. However, the charging rate can be adjusted to the required value by selecting an appropriate value for the resistance R22. The gain of the controller may be adjusted by changing the gain of the error amplifier. The proportional band can be changed by adjusting the amplitude of the triangle wave, which is possible by changing the value of R11. The amplitude of the triangle wave in volts is approximately equal to $5.1(R_{11})/(R_{12}+R_{11})$.

The present controller uses a silicon junction sensor, which has a large signal output compared with thermocouples, and this avoids problems of noise and drift at microvolt level. Also its low non-linearity permits an easy temperature calibration, in contrast with the highly nonlinear thermistors. This controller is not provided with a temperature indicating display because a dial calibration is sufficient for the present purpose. A digital panel meter can be incorporated in the circuit in case temperature indication is also needed, as we have done for a general purpose PID temperature controller [2.35].

The performance of the controller has been monitored while controlling the temperature of the bath discussed in section 2.4.2 and it was found that the temperature of the bath was steady within ± 0.1 K at 312 K over several weeks, even with mains power off for several hours.

2.4.4 The designed crystal rotation controller

In this section, a motor control circuit which was designed for controlling the crystal rotation is described. The need for crystal rotation or solution stirring in solution growth of crystal was explained in section 2.4.1. The present
circuit is versatile and has some novel features. It has provision for adjusting the speed of rotation. The period of time between rotation reversals can be adjusted. Further, there is a dead time before a rotation reversal during which no power is given to the motor. This dead time is to allow the inertia of the rotating crystal to die out before a reversal. In the absence of the dead time, a jerky movement can be produced which would strain the crystal and the suspension system. The motor control circuit has a fully solid state design and electromechanical relays are not used.

The circuit diagram of the crystal rotation motor controller is shown in Figure 2.10. The circuit essentially has two parts. One is the oscillator, counter and logic section and the other is the complementary transistor bridge type power output stage. The oscillator and counter function is performed by the CMOS type IC CD4060 (IC1). This IC has a 14 bit binary counter and an internal oscillator, the frequency $f$ of which can be set using external components as

$$f = \frac{1}{(2.3 \times R \times C)} \quad (2.20)$$

where $R = (V_{R1} + R2)$ and $C = C1$ in the circuit. This frequency is divided by the internal binary counter and fixes the period between reversals of the motor. Hence the period is adjustable using VR1. The outputs of the counter from 7th to 10th stages ($Q_7$ to $Q_{10}$) are used for producing the control waveform. Examination of the 4 bit binary sequence shows that $Q_{10}$ produces a symmetrical square wave output. Further, when $Q_{10}$ goes to 0 or 1 state then all the three outputs $Q_7$ to $Q_9$ together goes to 0 state for a period equal to 1/16th of $Q_{10}$ output waveform period. This short period is decoded by using one section of a Dual 4 input CMOS NOR gate CD4002 (IC2) for producing the dead time (C). The other section of the NOR gate is used to invert the $Q_{10}$ output to produce the biphase output (A and B) for driving the power stage. It may be noted that the $Q_{10}$ output has a period which is $2^{10}$ times (i.e. 1024) the period of the internal oscillator. With VR1 wiper set at the
Figure 2.10
Circuit diagram of the Crystal rotation controller
midpoint the $Q_{10}$ output has a period of about 140 seconds.

The bridge type power stage is formed by two set of complementary pairs having high gain Darlington type power transistors (TR3 to TR6). With a biphase drive (A & B) from the logic section, the motor can be driven in forward and reverse directions. Diodes D1 to D4 protects the transistors from inductive surges produced by the motor. In an emitter follower mode, TR2 works as a speed regulator with speed setting possible with VR2. TR1 is driven by the dead time logic (C), and when C is High, the power to the motor is fully cutoff. D5 ensures effective switching of the power stage transistors.
Appendix 2.1

Program for overlap identification and bond correction
(See section 2.3.3 for a description of this program)

PEO.BAS

10 CLS:KEY OFF:SCREEN 0 :COLOR 15:LOCATE 25,5
20 PRINT"Program by L.Godfrey -For Ultrasonic measurements-
30 LOCATE 17,1:PRINT "1. Creates Ultrasonic Experiment Data files"
40 PRINT "2. Process data with bond correction"
50 PRINT "3. Quit":PRINT
60 INPUT "Select (1 - 3)";SEL
70 IF SEL<l OR SEL>3 THEN 30
80 ON SEL GOTO 2410,110,100
90 GOTO 10
100 COLOR 7:CLS:KEY ON:END
110 CLEAR:LOCATE 17,1
120 PRINT SPACE$(50):PRINT SPACE$(50):PRINT SPACE$(50):PRINT
130 PRINT SPACE$(50):LOCATE 1,1 :PRINT "files present are..."
140 FILES ".IN":PRINT
150 INPUT"enter the name of selected data file: [.IN]",DTA$
160 DEFDBL A-H,L-Z
170 OPEN "I",#2,DTA$+.IN"
180 INPUT #2,DTA$I$
190 INPUT #2,NA$,BM$,DR$:PRINT NA$,BM$,DR$
200 INPUT #2,W$,POL$:PRINT W$,POL$
210 INPUT #2,LS,N,P:PRINT LS,N,P:PRINT
220 DIM XD(N),YD(2,N)
230 FOR I=1 TO N
240 XD(I)=I
250 INPUT #2,YD(1,I),YD(2,I):PRINT I,YD(1,I),YD(2,I)
260 YD(1,I)=100/YD(1,I):YD(2,I)=100/YD(2,I)
270 NEXT I
280 INPUT #2,ZB,RHO,ZT :PRINT :PRINT ZB,RHO,ZT
290 INPUT #2,O TR$:PRINT O TR$
300 CLOSE
310 X1=0:X2=0:YSQ1=0
320 Y1=0:Y2=0:YSQ2=0
330 P1=0:P2=0
340 FOR I=1 TO N
350 X1=X1+XD(I)
360 Y1=Y1+YD(1,I):Y2=Y2+YD(2,I)
370 YSQ1=YSQ1+YD(1,I)*YD(1,I):YSQ2=YSQ2+YD(2,I)*YD(2,I)
380 X2=X2+XD(I)*XD(I)
390 P1=P1+XD(I)*YD(1,I)
400 P2=P2+XD(I)*YD(2,I)
410 NEXT I
420 D=N*X2-X1*X1
430 IF D<>0 THEN 460
440 PRINT "no solution"
450 STOP
460 B1=(N*P1-X1*Y1)/D:B2=(N*P2-X1*Y2)/D
470 A1=(Y1-B1*X1)/N:A2=(Y2-B2*X1)/N
480 PRINT:COR1=(N*P1-X1*Y1)/SQR((N*X2-X1*X1)*(N*YSQ1-Y1*Y1))
490 PRINT:COR2=(N*P2-X1*Y2)/SQR((N*X2-X1*X1)*(N*YSQ2-Y2*Y2))
500 PRINT
510 PRINT
520 PRINT " 1. SCREEN"
530 PRINT " 2. LPT1:"
540 PRINT " 3. " ;DTA$+.OUT"
550 PRINT " 4. Special"
560 INPUT "Enter output choice: ",CHO
570 PRINT:PRINT
580 IF CHO > 4 OR CHO<1 THEN 520
590 ON CHO GOTO 600,610,620,630
600 OF$="SCRN:":GOTO 640
610 OF$="LPT1:":GOTO 640
620 OF$=DTA$+.OUT":GOTO 640
630 INPUT "Output file/device name":OF$
640 OPEN "O",#1,OF$
650 IF CHO > 1 THEN PRINT "Dumping Output to ";OF$;
660 PRINT#1,"Sample name = ";NA$
670 PRINT#1,"Propagation direction = ";DR$
680 PRINT#1,"Wave Type = ";W$
690 PRINT#1,"Polarization direction= ";POL$
700 PRINT#1,"Sample dimension & p = ";
710 PRINT#1,USING "####.###":LS;:PRINT#1," cm , p = ";
720 PRINT#1,USING ":P
730 PRINT#1,"Linear fitting of the ";N," measurement data
740 pairs give:
750 F$="#.#######AAAA":F2$="#.###########"
760 PRINT#1,"TL= ";
770 PRINT#1,USING F$;A1;
780 PRINT#1," + ";
790 PRINT#1,USING F$;B1;
800 PRINT#1,"*X Cor.cof=";
810 PRINT#1,USING F2$;COR1
820 PRINT#1,"TH= ";
830 PRINT#1,USING F$;A2;
840 PRINT#1," + ";
850 PRINT#1,USING F$;B2;:PRINT#1,"*X Cor.cof=";
860 SG$=STRINGS$(62,45)
870 PRINT#1,SG$:FS0$="\n\":FS1$="\n"
880 PRINT#1,USING FS0$;" X";
890 PRINT#1,USING FS1$;" TL"," E(TL)"," TH"," E(TH)"," (TL-TH)"
900 PRINT#1,USING FS0$;" No"
910 PRINT#1,USING FS1$;" µs"," µs"," µs"," µs", ns"
920 PRINT#1,SG$
**ZXZ=INT((A2-A1)/(B1-B2))**

940 FOR I=(ZXZ-4) TO (ZXZ+4)

950 ETL=0;ETH=0


970 IF I>0 AND I<=N THEN ETL=YD(1,I)-TL;ETH=YD(2,I)-TH

980 PRINT#1,USING "### ";I;

990 PRINT#1,USING "###.##### ";TL,ETL,TH,ETH,(TL-TH)*1000

1000 NEXT I

1010 PRINT#1,SG$

1020 IF CHO=1 THEN INPUT "Strike ENTER to continue...",NUL$

1030 FL=1/B1;FH=1/B2;FR=FH

1040 PRINT#1, "Estimated Low RF frequency = ";

1050 PRINT#1,USING "###.##### MHz";FL

1060 PRINT#1, "Estimated High RF frequency= ";

1070 PRINT#1,USING "###.##### MHz";FH;

1080 PRINT#1,USING " LF/HF = ###";FL/FH

1090 PRINT#1, "Bond material used = ";BM$

1100 PRINT#1, "Bond impedance = ";

1110 PRINT#1,USING "###.#####";ZB

1120 PRINT#1, "Transducer impedance = ";

1130 PRINT#1,USING "###.#####";ZT

1140 PRINT#1, "sample density in g/cc = ";

1150 PRINT#1,USING "###.#####";RHO


1170 PRINT#1, "estimated sample impedance = ";

1180 PRINT #1,USING "###.#####";ZS;

1190 PRINT#1, "Other information = ";OTR$

1200 PRINT

1210 IF CHO=1 THEN INPUT "Strike enter to continue....",NUL$

1220 PI=3.141592654#:COLOR 31:LOCATE 25,55

1230 PRINT "COMPUTING"

1240 AL=0:X=AL:GOSUB 1650

1250 DTMIN=DT

1260 AH=60*PI/180:X=AH:GOSUB 1650:DTMIN2=DT

1270 X=(AL+AH)/2:GOSUB 1650:DTMAX=DT:XMAX=X

1280 AIN=(AH-AL)/4

1290 FOR I=1 TO 20

1300 X=XMAX-AIN:GOSUB 1650

1310 IF DT>DTMAX THEN DTMAX=DT:XMAX=X:CRMAX=CR

1320 X=XMAX+AIN:GOSUB 1650

1330 IF DT>DTMAX THEN DTMAX=DT:XMAX=X:CRMAX=CR

1340 AIN=AIN/2

1350 IF AIN<.000001 THEN 1370

1360 NEXT I

1370 COLOR 15

1380 PRINT #1,STRING$(16,45);"CALCULATION OF CORRECT OVERLAP"

1390 PRINT #1,STRING$(16,45)

1400 F3$="###.#####"

1410 PRINT #1,"estimated maximum value of delta t [ns] = ";

1420 PRINT #1,USING F3$;DTMAX

1430 PRINT #1,"bond angle corresponding to max delta t [deg]=
2400 RETURN
2410 PRINT
2420 CLEAR:LOCATE 17,1:PRINT SPACE$(50):PRINT SPACE$(50)
2430 PRINT SPACE$(50):PRINT:PRINT SPACE$(50):LOCATE 1,1
2440 PRINT "Files present are...":FILES ".IN":PRINT
2450 INPUT "enter the data file name to be created: [ .IN] ",DAT$
2460 DAT$=DAT$+.IN"
2470 DEFDBL Y
2480 INPUT "Name of sample";NA$
2490 INPUT "bond material used";BM$
2500 INPUT "direction of propagation";DR$
2510 INPUT "length in cm";LS
2520 INPUT "wave type L or T";W$
2530 IF W$="L" OR W$="l" THEN W$="Longitudinal":POL$=DR$:GOTO 2560
2540 IF W$="T" OR W$="t" THEN W$="Transverse":GOTO 2550 ELSE 2520
2550 INPUT "Polarization direction ";POL$
2560 INPUT "number of data values";N
2570 INPUT "value of p";P:PRINT
2580 DIM YL(N),YH(N)
2590 PRINT " yl is rep.rate in MHz x 100 for low
rf"
2600 PRINT " yh is rep.rate in MHz x 100 for high
rf"
2610 PRINT " m is a case of overlap"
2620 PRINT
2630 INPUT "Multiplication factor for yl&yh [1 for
default]";MULF
2640 PRINT
2650 FOR I=1 TO N
2660 PRINT "FOR m=";I;
2670 INPUT "; yl=";YL
2680 INPUT "; yh=";YH
2690 YL(I)=YL*MULF:YH(I)=YH*MULF
2700 NEXT I
2710 INPUT "Bond impedance in x1e6 kg/m².s";ZB
2720 INPUT "Sample density in g/cc ";RHO
2730 INPUT "transducer impedance x1e6 kg/m².s";ZT
2740 INPUT "any other information (null for nothing) ";OTR$
2750 PRINT "creating file ";DAT$
2760 OPEN "o",#1,DAT$
2770 WRITE #1,DAT$
2780 WRITE #1,NA$,BM$,DR$
2790 WRITE #1,W$,POL$
2800 WRITE #1,LS,N,P
2810 ON SGN(YH(1)-YH(N))+2 GOSUB 2890,2930,2950
2820 WRITE #1,ZB,RHO,ZT
2830 WRITE #1,OTR$
2840 CLOSE
2845 ERASE YL,YH
2850 PRINT " File ";DAT$; " is created"
2860 INPUT "More data file y/n ";SEL$
2870 IF SEL$="y". OR SEL$="Y" THEN 2450
2880 GOTO 10
2890 FOR I=N TO 1 STEP -1
2900   WRITE #1, YL(I),YH(I)
2910 NEXT I
2920 RETURN
2930 PRINT "data invalid"
2940 RETURN
2950 FOR I=1 TO N
2960   WRITE #1, YL(I),YH(I)
2970 NEXT I
2980 RETURN
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