Automated system for relative sound velocity and ultrasonic attenuation measurements

J. Toulouse and C. Launay[a)]

Lehigh University, Bethlehem, Pennsylvania 18015

(Received 13 August 1987; accepted for publication 30 November 1987)

In order to facilitate measurements of ultrasonic velocity and attenuation, we have developed an automated version of the phase sensitive technique with quadrature detection. The technique is outlined and the precision of relative transit time or velocity measurements that can be obtained from it is analyzed. We describe the computer-controlled experimental setup and report relative velocity and attenuation results obtained on a single crystal of pure $KZnF_3$. The temperature dependence of the elastic constants C_{11} and C_{44} calculated from these results agree very well with that obtained from echo overlap measurements performed on the same crystal. The relative error on the present velocity measurements is $\Delta v/v \sim 10^{-5}$.

INTRODUCTION

The most widely used technique to measure the velocity of sound in solids is the echo overlap, which was developed by May[1] and extended by Papadakis.[2] The method consists in superposing successive rf echoes, cycle for cycle, using a frequency controlled delay circuit. Provided the echoes are properly matched, relative precision of order 0.0002% (2×10^{-5}) can be obtained.[2] These measurements are time consuming, however, and, by nature, cannot be automated. The phase velocity can also be measured by a phase-sensitive technique which was developed by Williamson[3] and separately by Penvushin and Filippov[4]: The latter method can provide absolute and relative sound velocity measurements with a precision comparable to the former method. The method for absolute measurements has been described by Petersen *et al.*[5] In the present work we describe an automated system for relative sound velocity measurements using the phase-sensitive technique with quadrature detection and present experimental results.

I. THEORY

The phase-sensitive technique used here is based on the measurement of the phase of a given echo relative to the phase of a reference which is drawn from the same signal source as that driving the transducer. The phase of the mth echo, i.e., after m round trips through the crystal, is lagging behind the phase of the reference by an amount

$$\phi_m = 2\pi f t_m, \quad (1)$$

where t_m represents the travel time through the crystal as well as through the bond, the transducer, and the electronics and f is the frequency of the rf signal. A general expression for the travel time can be written as

$$t_m = m t_{rt} - (m-1)\gamma/2\pi f + t_e, \quad (2)$$

where t_{rt} represents the round trip time through the crystal, γ the phase change caused by the reflection at the transducer–bond interface, and t_e the transit time through the electronics. An approximate expression for γ has been derived by McSkimin.[6]

$$\gamma = (-4\pi\rho_B l_B/Z_s)f - 2\pi(Z_t/Z_s)[(f-f_r)/f_r], \quad (3)$$

with ρ_B and l_B the density and thickness of the bond, respectively, f_r the resonant frequency of the transducer, and Z_t and Z_s the acoustic impedance of the transducer and of the sample, respectively. From Eq. (2), the change in round trip time Δt_{rt} with temperature can be seen to be approximately equal to

$$\Delta t_{rt} = (1/m)\Delta t_m,$$

where we neglect the temperature dependence of γ. Making use of Eq. (1), Δt_{rt} can in turn be obtained from a measurement of the change in phase $\Delta\phi$ by using Eq. (1)

$$\Delta t_{rt} = (1/m)(\Delta\phi_m/2\pi f). \quad (4)$$

The phase of an echo m can be measured by decomposing the output signal into its in-phase component A_{1m}, and its quadrature component A_{2m} with respect to the rf reference (see Fig. 1). The phase angle is then given by

$$\phi_m = \arctan(A_{2m}/A_{1m}) \quad (5)$$

and the phase change $\Delta\phi_m$ can be calculated from the measured amplitudes A_{1m} and A_{2m}. We note that the change in phase can be decomposed into an integer number of half-periods, p, and a fraction of a half-period, n, so that it can be written as

$$\Delta\phi = (p+n)\pi. \quad (6)$$

We shall see that because of the arctan expression, Eq. (6) represents a central point in the evaluation of the change in round trip time. We also note that $\Delta\phi$ does not, in general,

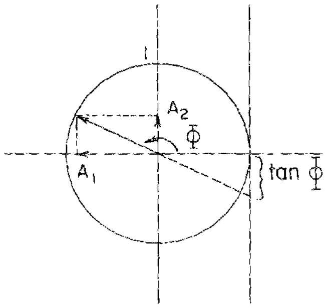

FIG. 1. In-phase and quadrature components and phase change ϕ of the propagating sound wave.

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depend on m and can be obtained from each echo independently. Comparing phase or round-trip time measurements from several echoes thus provides a check of accuracy. The precision of the measurement is limited by the precision with which the phase separation can be performed and is discussed below.

II. EXPERIMENTAL SETUP

The ultrasonic setup is schematically represented in Fig. 2. It consists of a rf signal source fed through a gated rf amplifier (Matec 310) to obtain pulses of variable widths ($>0.5\,\mu$s). These pulses are directed to the transducer and sample through one branch of a T circuit. The reflected signals (successive echoes) are phase analyzed through a superheterodyne detection stage composed of two mixers (ZAY-3), a 90° hybrid transformer (ZSCQ-2), and two phase detectors (ZAY-3) (all five components from Minicircuits). The gain curve of the IF amplifier is centered at 60 MHz; this is, therefore, also the frequency at which the detector operates (Matec 625). The outputs of the two phase detectors are fed into low-pass filters and then directed to the two inputs of a computer-controlled switch box (Siliconix FET-DG187 BP, not shown in Fig. 2). In this way each of the two phase detectors can be connected successively to the input of a PAR single-channel boxcar integrator. The output of the boxcar is digitized and read into a Zenith 158 PC via an interfacing board (Metrabyte, DASH 8).

The major steps of the program are listed as follows:

(i) Choice of experimental and graphic parameters.

(ii) Positioning of the integration window on selected echoes.

(iii) Amplitude measurements of echoes, in-phase components, and quadrature components.

(iv) Calculation of phase variation, change in transit time, and attenuation.

(v) Data storage and graphics.

A flow chart of the computer program is given in the Appendix.

Steps (ii) and (iii) are performed by the computer without any external intervention. Step (ii) is a particularly important one and calls for a few remarks. The time positions of the integration window must be selected initially to coincide with the position of the various echoes to be measured, as well as with a background position. Once selected, these positions are recorded by the computer, which then reproduces the positioning sequence automatically. In the course of the experiment, the sound velocity changes and so does the time delay between successive echoes. An automatic tracking procedure is, therefore, built into the program so that the window position always coincides with the position of the initially selected echoes. At each position, the boxcar must measure the amplitudes of both phase components, A_1 and A_2, and switching between them is controlled by the computer at the boxcar input. The integration time for each component is determined by the choice of a time constant on the boxcar.

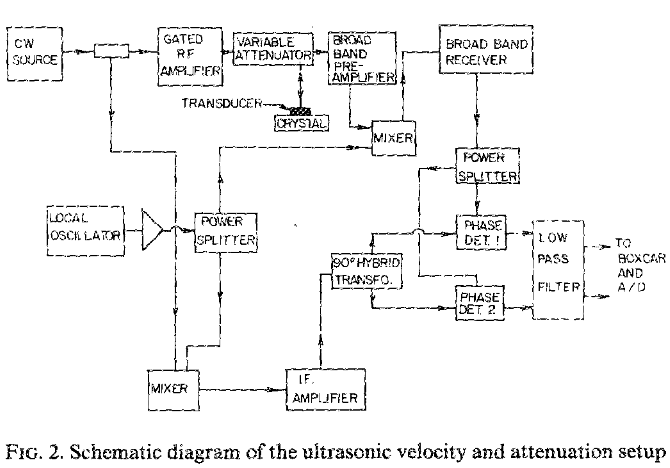

FIG. 2. Schematic diagram of the ultrasonic velocity and attenuation setup with quadrature phase sensitive detection.

As we indicated in Eq. (5), the phase angle is obtained as an arctan function and can only be determined to within π. (See Fig. 1.) To eliminate this uncertainty it is necessary to determine the quadrant in which the vector amplitude lies. This can be done by following the sign of ϕ_m as well as that of either the in-phase component A_{1m} or the quadrature component A_{2m}. The principal limitation of this technique is, of course, that the phase must be monitored continuously and the signal frequency cannot be changed.

A. Sources of error

Experimentally, the primary source of error in the measurement is due to the phase separation of the two components of the signal, which is done through the 90° hybrid transformer and the two phase detectors following it. According to the manufacturer, the transformer can introduce a deviation of $\pm 3°$ relative to exact quadrature. Also, one must take into account possible differences in the respective amplitude response of the two detectors. Assuming a maximum 10% difference in amplitude response, the two components can be written as

$$A_{1m} = A_m \cos\phi' \times 1,$$

$$A_{2m} = A_m \sin(\phi' + 3) \times 1.1.$$

Comparing with the exact measurement obtained from $A_{2m}/A_{1m} = \tan\phi$, we can find $(\phi' - \phi)$ as a function of the phase angle ϕ. We note that this difference is periodic with period 2π. With the present figures, the maximum difference is 0.08 rad, and using Eq. (4) this corresponds, at 45 MHz, to an error on the round trip time of approximately 3×10^{-15} on the tenth echo. A good measure of the actual experimental error is given by comparing the transit time obtained from different echoes since they correspond to different phase angles. Such an error is usually found to be about 5×10^{-11} s or a relative error $\Delta t/t \sim 1\times 10^{-5}$. Because the phase of any given echo changes with temperature this error, due to the phase separation of the hybrid transformer, must be taken into account.

On the basis of Eq. (2), the other source of relative error in the measurement of the transit time can be seen to be associated with γ given in Eq. (3). This error is temperature dependent since the acoustic properties of the bond and transducer are themselves temperature dependent. When operating the transducer near its resonant frequency, the second term in Eq. (3) is zero. The temperature dependence of the first term can be estimated if the coefficient of thermal expansions of the bond and the sample are known. For respective values of 10^{-4} and 10^{-5} we find $d(\rho_B l_B / Z_s)/dT \sim 10^{-14}$, 10^{-15}, which leads to an error on the transit time of the same magnitude, i.e., much smaller than the error discussed above.

In summary, the major source of error is found to lie in the phase separation of the signal and the relative error on the round trip time $\Delta t / t$ is approximately 1×10^{-5}, i.e., comparable to the relative error in the echo overlap method.

The sample holder used in the present setup is represented schematically in Fig. 3. This particular design of the spring-loaded contact was chosen to approximate, as much as possible, an uninterrupted coaxial line to the transducer. The transducer used was a 15-MHz lithium niobate operated on its third harmonic. A calibrated silicon diode sensor (Lake Shore Cryotronics DT-500) was silvered directly onto the sample for precise temperature measurements.

III. RESULTS

We now present experimental results obtained on $KZnF_3$ with the phase-sensitive technique and compare them with results obtained previously with the echo overlap technique.[7] The same $KZnF_3$ sample was used for both sets of measurements. Figure 4 illustrates the accuracy of the present method by showing the change in round trip time Δt obtained for longitudinal waves from each of 13 echoes as a function of temperature. The curves are staggered arbitrarily to make the display clearer; if not staggered, all curves would superpose on the top curve, which corresponds to the first echo. We then take the average of Δt_n at each temperature. In order to calculate the velocity and elastic constant,

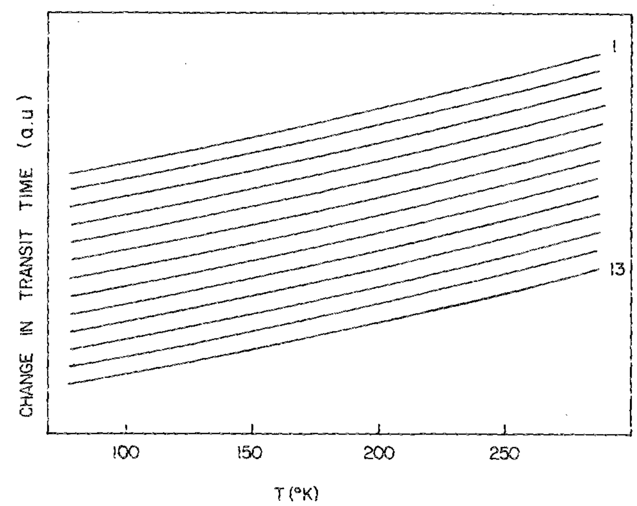

FIG. 4. Change in the transit time for 13 successive reflections (echoes) of an initial pulse into pure $KZnF_3$.

an absolute measurement of the round trip time is needed. This absolute measurement was obtained by a phase sensitive technique described previously.[5] The velocity was corrected for thermal expansion using the same lattice parameter data as those used in the echo overlap study.[7]

The final curves for C_{11} and C_{44} are shown in Fig. 5 along with the previous echo overlap results.[8] The accuracy of our relative measurements are exemplified by the near parallelism of the two C_{11} and C_{44} curves, respectively. The error bars on the present curves are only due to the absolute velocity measurement.

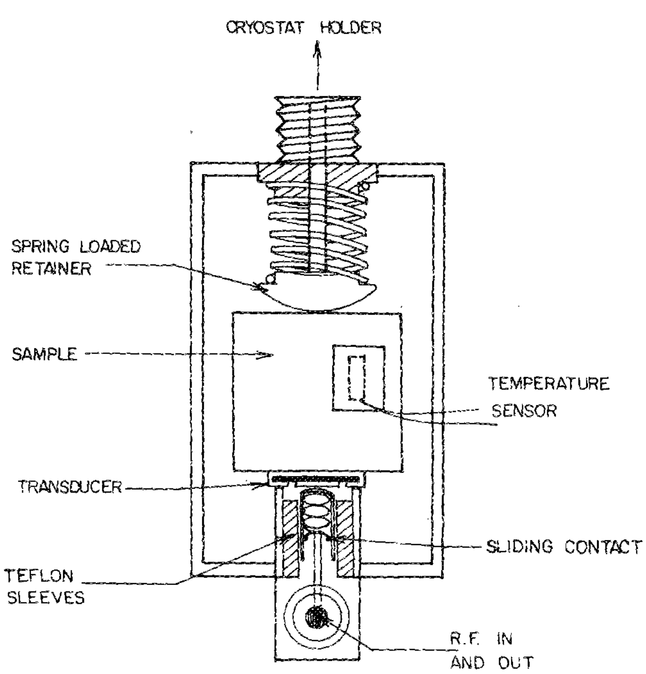

FIG. 3. Sample holder.

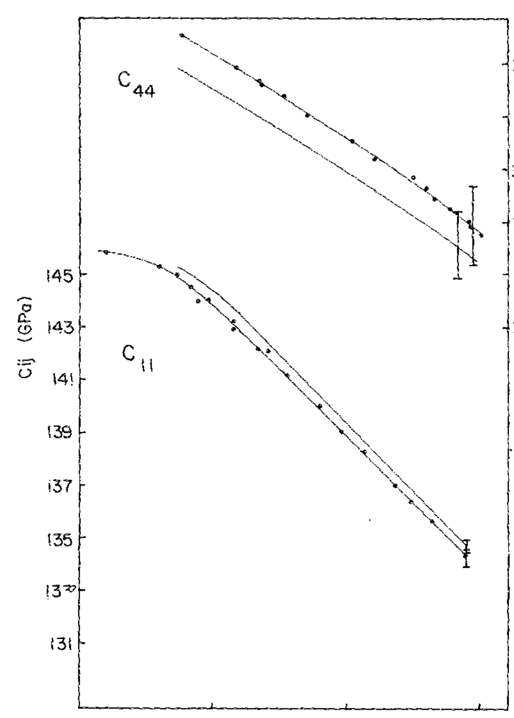

FIG. 5. Elastic constants of pure $KZnF_3$, C_{11}, and C_{44}, vs temperature; the continuous line represents the results of the present study and the dotted line with the discrete data points are echo overlap results from Ref. 7. The error bars in the present work refer to the absolute velocity measurement.

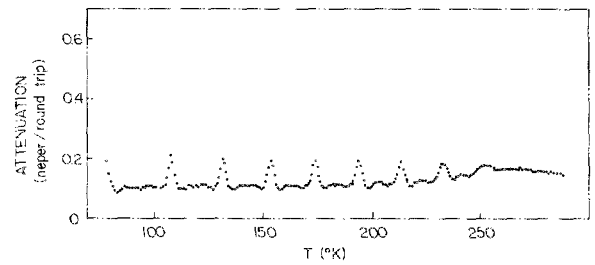

FIG. 6. Attenuation as a function of temperature in pure $KZnF_3$ (0.2 neper/round trip≡0.32 dB/cm).

It is important to notice that the curves obtained in the present work have been drawn continuous and do not show any specific data points. This is due to the fact that data points can here be obtained at arbitrarily close temperatures provided the temperature is drifted slowly enough. Drift rates of 0.06 K/mn are most often used; given the fast measurement technique employed, the temperature drift over the time required for the measurement of ten echoes is less than 0.002 K. The sensitivity (or resolution) of the present velocity measurements exceed 10^{-8} s, i.e., more than an order of magnitude greater than the sensitivity of echo overlap measurements.

Finally, in Fig. 6 we present the attenuation results obtained from the automated measurements for longitudinal waves. The ordinate represents the attenuation measured in neper/round trip. For the particular sample measured, 0.1 corresponds to 0.32 dB/cm. The oscillations in the attenuation are probably due to a small departure from parallelism of the transducer–crystal interface or mosaicity of the crystal, which result in a modulation of the echo train and artificial attenuation periodic with temperature. Further examination of this point is under way. This particular set of attenuation data was nevertheless chosen because it corresponds to the elastic constant data reported here and because the excellent resolution of the periodic peaks constitutes a good test of sensitivity. The present data show that attenuation can be measured with a resolution superior to 0.05 dB/cm. Attenuation curves with little modulation have also been obtained on other crystals using the present technique.

In summary, the phase-sensitive detection technique presented here allows automated measurements of the relative sound velocity and ultrasonic attenuation. These measurements can be performed, in a much reduced time, with greater sensitivity and an accuracy comparable to those made with the manual echo overlap technique.

ACKNOWLEDGMENTS

This work was supported by the US Department of Energy under Grant No. DE-FG02-86ER45258. We particularly wish to thank B. Chick and G. L. Petersen for sharing their expertise in the development of the present system. We also very much thank Professor J. Nouet of Le Mans for making the present collaboration possible and lending us the single crystal of $KZnF_3$.

APPENDIX: FLOW CHART OF COMPUTER PROGRAM FOR AUTOMATED ULTRASONIC MEASUREMENTS

INITIALIZATIONS OF INTERFACES
. IEEE 488 MULTIMETER INTERFACE (temperature measurements)
. DASCONI A/D COMPUTER INTERFACE (ultrasonic data acquisition)
LOAD OPERATIONS
. IE 488 AND DASCONI BINARY CODES
. CALIBRATION CURVE OF TEMPERATURE SENSOR
POSITIONING OF BOXCAR INTEGRATION WINDOW ON SELECTED ECHOES
CHOICE OF PARAMETERS
. EXPERIMENTAL: MINIMUM TRANSIT TIME AND TEMPERATURE STEPS
. GRAPHIC: UPPER AND LOWER LIMITS OF GRAPHIC DISPLAY OF DATA AUTOMATIC MEASUREMENT OF THE FIRST FOUR ECHOES (to allow manual adjustment of maximum echo amplitude)
DATA ACQUISITION
. MULTIMETER VOLTAGE (temperature)
. AMPLITUDE OF QUADRATURE AND IN-PHASE COMPONENTS OF EACH PREVIOUSLY SELECTED ECHO
CALCULATION OF AVERAGE TRANSIT TIME VARIATION
IF TEMPERATURE OR TRANSIT TIME CHANGE LESS THAN CHOSEN MINIMUM, GO TO DATA ACQUISITION
CALCULATION OF ATTENUATION [least-square linear fit over log (echo amplitude)] CALCULATION OF TEMPERATURE RATE OF CHANGE (K/mn)
DISK STORAGE AND GRAPH PLOTTING OF DATA POINTS
ADJUSTMENT OF POSITIONS OF BOXCAR INTEGRATION WINDOW
GO TO DATA ACQUISITION OR STOP
END

[a] Permanent address: Laboratoire de Physique de l'Etat Condense, Universite du Maine, 70217 Le Mans, France.

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