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Piezoelectric stiffening and attenuation of ultrasonic waves in *n*-type GaSb doped with sulfur*

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Measurements of the velocity and attenuation of [110] [001] ultrasonic waves and of the velocity of [100] [001] waves have been made on single-crystal samples of sulfur-doped *n*-type GaSb down to liquid-helium temperatures. (The first and second set of digits in brackets give the crystallographic indices of the propagation and polarization directions of the waves, respectively.) It is found that the [110] [001] waves exhibit an attenuation maximum and an additional increase in velocity as the temperature is lowered sufficiently. The additional velocity increase begins at a temperature which depends on the sample involved and the measuring frequency, but the total increase is not dependent on these factors. Both the additional velocity increase and the attenuation maximum are understandable in terms of the decreased screening of piezoelectric fields as electrons freeze out of the conduction band into impurity levels. (Evidence for such freeze out is provided by resistivity and Hall-effect measurements we have made at a few temperatures.) Theoretical formulas of Hutson and White are found to fit our most accurate velocity and attenuation versus temperature data almost exactly when a semiempirical expression is used for the resistivity which suggests that the conductivity at low temperatures is controlled by levels closer to the conduction band than is the sulfur level whose ionization dominates the electrical behavior above 77 K. Comparison of the theoretical formulas with the velocity enhancement observed at lowest temperatures and the amount by which the maximum attenuation exceeds a small background value yields values of 0.170 ± 0.005 and 0.160 ± 0.016 C/m^2, respectively, for the magnitude of the piezoelectric constant. Both of these values are larger than that obtained previously by others using a different method. However only the 0.170-C/m^2 value exceeds the previous value by more than the sum of the quoted experimental errors. Possible reasons for the discrepancy are discussed, but no definitive one is identified.

I. INTRODUCTION

The piezoelectricity of a material may cause the velocity of those ultrasonic waves which have longitudinal piezoelectric fields associated with them to be greater than that of non-piezoelectrically-active waves which involve the same elastic constant(s).[1] In a piezoelectric semiconductor the attenuation, as well as the velocity, of such ultrasonic waves may be affected when there is an appropriate concentration of mobile electrons or holes. The effects on both the velocity and the attenuation have been represented mathematically[1] by means of a complex elastic constant. Thus,

$$\tilde{c} = c\left(1 + \frac{e_p^2}{2c\epsilon}\,\frac{1+(\omega_c/\omega_D)+(\omega/\omega_D)^2 - i(\omega_c/\omega)}{1+2(\omega_c/\omega_D)+(\omega/\omega_D)^2+(\omega_c/\omega)^2}\right), \tag{1}$$

where $\tilde{c}$ is the complex elastic constant, c is the real part of the elastic constant, e_p is the effective piezoelectric constant, and ω is the angular ultrasonic frequency pertinent to the (piezoelectric) wave being propagated. In addition, ϵ is the dielectric permittivity of the material; ω_c is the conductivity relaxation frequency and equals $1/\rho\epsilon$, where ρ is the electrical resistivity; and ω_D is the carrier diffusion frequency given by $ev^2/\mu f_0 kT$, where e is the charge and μ is the mobility of the carriers, f_0 is the fraction of the acoustically produced space charge which is mobile, v is the velocity of the ultrasonic waves and equals $\mathrm{Re}(\tilde{c})^{1/2}/\sqrt{\rho_m}$, where ρ_m is the mass density, k is Boltzmann's constant, and T is the absolute temperature.

A variety of work has been done on CdS (Refs. 2–16), which can be interpreted either by means of Eq. (1) or by generalizations of it which includes effects due to carrier trapping and an applied electric field. There has been ultrasonic work on *n*-type InSb,[17,18] using a magnetic field to modulate the conductivity in the range $\omega \ll \omega_c$. Hickernell[19] measured the attenuation in GaAs as a function of temperature in a range where ω_c changed enough to produce most of the maximum predictable from Eq. (1). Other measurements[20] of attenuation versus temperature have revealed a complete maximum in *n*-type GaAs. Measurements of velocity versus temperature in the same sample and in an oxygen-doped *n*-GaAs sample[20] have shown the velocity increases expected from Eq. (1). Both the attenuation and velocity data have yielded very accurate values of the piezoelectric constant of GaAs. These values agree very well with each other and with that determined previously by another method.[21]

Attenuation maxima have also been found[22,23] in n-GaP and p-InSb and in n-InP.[24] They can be understood qualitatively in terms of the attenuation implied by Eq. (1) although a quantitative fit of the whole attenuation maximum in some cases required the use of a somewhat different value of resistivity than that obtained from dc measurements. A value for the piezoelectric constant of each material was obtained. The value for InSb agreed with that measured previously by others.[21] Values for e_{14} had not been determined previously for GaP and InP although an approximate value had been given for GaP.[25]

In this paper we shall report ultrasonic velocity and attenuation measurements on n-type GaSb doped with sulfur. These experiments reveal effects due to piezoelectricity and the temperature dependence of the concentration of conduction-band electrons. Analysis of our data is based on the theory of Hutson and White[1] and the expressions for the velocity and attenuation of [110][001] ultrasonic waves which are derivable from Eq. (1) when $\omega_D \gg \omega$. (Here and henceforth the first set of crystallographic indices gives the direction of propagation of the waves and the second set of indices gives the direction of polarization of the waves.) The expressions are

$$v = v_0 \left(1 + \frac{e_{14}^2}{2c_{44}\epsilon} \, \frac{1}{1+(\omega_c/\omega)^2} \right) \tag{2}$$

and

$$\alpha = \frac{e_{14}^2}{2c_{44}\epsilon} \, \frac{\omega}{v_0} \left(\frac{\omega/\omega_c}{1+(\omega/\omega_c)^2} \right), \tag{3}$$

where $v_0 = (c_{44}/\rho_m)^{1/2}$, e_{14} is the piezoelectric constant, and the other quantities are defined after Eq. (1).

II. EXPERIMENTAL DETAILS

Ultrasonic velocities were determined from the transit times of pulses of ultrasonic waves by means of the pulse-echo-overlap method.[26,27] The attenuation of pulses of 31-MHz [110][001] ultrasonic shear waves was measured using an echo-voltage-ratio attenuation measuring system.[28] The velocity and attenuation measuring systems and technical details have been described elsewhere[22] as have the transducers and bonds which were employed. For our initial measurements (shown in Figs. 2 and 3) the temperature was allowed to drift slowly upward from 4.2 K (1 K every few minutes) and our final measurements (shown in Figs. 1, 4, and 5) were made at temperatures held constant to 0.1 K or better.

Various characteristics of the samples are listed in Table I. The resistivity and Hall effect were determined by means of conventional dc potentiometric measurements or with a very high input impedance digital voltmeter. A 4-in. Varian electromagnet was used in making Hall-effect measurements.

Sulfur-doped n-type samples, rather than the more common tellurium-doped ones, were used. This choice was made to ensure the presence of enough electrons in the conduction band at high temperatures so that the piezoelectric fields associated with appropriate ultrasonic waves would be screened out but so few electrons remaining in the conduction band at low temperatures that the effect of piezoelectricity would be manifest in the velocity and attenuation of appropriate waves. Such extremely strong freeze out of conduction electrons has been observed previously[29,30] in S-doped n-GaSb between about 373 and 100 K. From our electrical data in Table I it can be seen that for sample 75A there is strong freeze out down to 77 K like that expected for the deionization of sulfur impurity levels which are located about 0.07 eV below the conduction band edge. However for sample 75A both the dc resistivity at 4.2 K and the resistivity deducible from our ultrasonic velocity and attenuation measurements (see Sec. IV) imply that for some range of temperature below 77 K, deionization of shallower impurity levels (i.e., levels which are closer to the conduction band) must be most important for determining the temperature dependence of the resistivity. We do not know what impurities the shallower levels are due to. In view of previous work[30] they might be due to sulfur and/or selenium.

From Table I it can be seen that samples 1B and 1T also show a large amount of carrier freeze out by 77 K, which, however, is an order of magnitude smaller than that exhibited by sample 75A. This may be due to there being less sulfur in samples 1B and 1T relative to some other kind of impurity with a smaller ionization energy (selenium perhaps[30]). We infer this from the fact that samples 1B and 1T have somewhat larger values of $|R_H|$ at 296 K than does sample 75A but have values of $|R_H|$ at 77 K which are orders of magnitude smaller than that of sample 75A.

At 77 K we observed that the resistivity and the magnitude of the Hall coefficient increased appreciably with time in a manner consistent with the long electronic equilibration times observed by other workers[31,32] for sulfur-doped GaSb. This effect made it difficult to obtain resistivity data accurate enough to account for the velocity rise and attenuation maximum to be discussed in this paper. Therefore, as will be seen in Sec. IV, we contented ourselves with being able to account for

TABLE I. Characteristics of our sulfur-doped n-type GaSb samples.

Designation	T (K)	ρ [a] (Ω cm)	$-R_H$ [a,b] (cm^3/C)	Ultrasonic samples Length (cm)	Orient.
75A	296	0.066	156	1.1567	[110]
				1.1574	[100]
	77	273	2.6×10^5	1.1554 [c]	[110]
				1.1561 [c]	[100]
	4.2	4.3×10^5	···	1.1554	[110]
				1.1561	[100]
1B	296	0.117	230	0.8933	[110]
				1.2542	[100]
	77	24.3	1.45×10^4	0.8922 [c]	[110]
				1.2527 [c]	[100]
1T	296	0.067	195	0.8422	[110]
	77	7.7	9.8×10^3	0.8413 [c]	[110]

[a] Measured on a sample cut from the ingot adjacent to region which provided the ultrasonic sample.

[b] Measurements of the Hall coefficient were made using magnetic inductions between 1000 and 5000 G.

[c] Obtained by correction for thermal contraction using results of S. I. Novikova and N. Kh. Abrikosov {Fiz. Tverd. Tela 5, 2138 (1963) [Sov. Phys.-Solid State 5, 1558 (1964)]}.

both the velocity and attenuation phenomena with the same resistivity rather than trying to do so employing measured resistivity values.

III. RESULTS

Figure 1 shows the velocity and attenuation of shear ultrasonic waves as a function of temperature for our sample 75A. These data were obtained at a number of accurately controlled temperatures (see Sec. II). From Fig. 1 it can be seen that there is an extra increase in velocity of waves propagating in the [110] direction and polarized in the [001] direction at low temperatures which does not occur for [100][001] waves. In addition there is a maximum in the attenuation of the [110][001] waves which is correlated with the extra velocity increase of these waves. No attenuation maximum was observed for [100][001] waves.

Since the [110][001] modes are known to be piezoelectrically active in zinc-blende compound semiconductors, while the [100][001] modes are not, we attribute the low-temperature behavior of the [110][001] modes shown in Fig. 1 (and in Figs. 2–5 also) to the reduction in the amount of screening of the piezoelectric interaction as enough electrons freeze out of the conduction band onto impurities. Our electrical data presented in Sec. II provide direct evidence that electrons do freeze out sufficiently in sample 75A at low temperatures.

A detailed quantitative analysis of the extra velocity increase and attenuation maximum will be made after we present data to show how the velocity increase depends on sample characteristics and frequency.

Shear wave velocity versus temperature data are shown for three sulfur-doped n-GaSb samples in Fig. 2. Velocity results for two different frequencies are given in Fig. 3 for sample 75A. Since the data in Figs. 2 and 3 were obtained early in the course of our investigation when

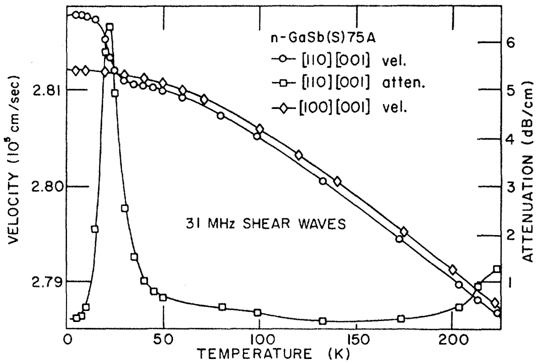

FIG. 1. Velocity and attenuation of 31-MHz shear ultrasonic waves in sulfur-doped n-type GaSb as a function of temperature. The data points were obtained at accurately controlled temperatures (see Sec. II of the text). The directions of propagation and polarization of the waves are given by the first and second set of crystallographic indices, respectively.

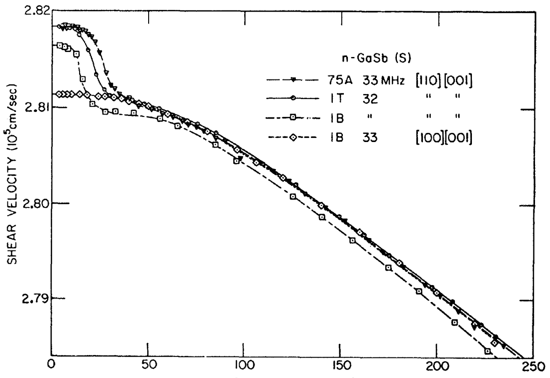

FIG. 2. Velocity vs temperature for three different sulfur-doped *n*-type GaSb samples. The measurements were made with the temperature drifting upward slowly (see Sec. II of the text).

measurements were made with the temperature drift method (see Sec. II), they are not as accurate as the velocity data in Fig.1 and we shall give only a qualitative analysis of them.

From Figure 2 it can be seen that the additional increase in velocity of all three samples is approximately the same. This is to be expected from Eqs. (1) and (2) if the resistivity of each sample is such that $\omega_c \gg \omega$ at high temperatures and $\omega_c \ll \omega$ at low enough temperatures.

The extra increase in velocity begins at a higher temperature for sample 75*A* than for samples 1*B* and 1*T*. This is to be expected from Eq. (2) if the resistivity of 75*A* remains higher than that of 1*B* and 1*T* at low temperatures as is implied by the fact that it is much higher at 77 K. Because the resistivity of 1*B* is larger than that of 1*T* at 77 K it is not clear why the additional velocity increases of these two samples begin where they do relative to each other.

From Fig.2 it can be seen also that above 50 K all velocity data show the same type of temperature dependence. This dependence can be explained in terms of the anharmonicity of bonding forces and has been discussed elsewhere.[33] Three of the velocity curves are almost coincident above about 30 K and one of the [110][001] curves for sample 1*B* is about 1% lower than the comparison curves. We do not believe that this difference represents an actual difference in velocity. Rather it is due to inaccuracy in the absolute value of the velocity. This belief is supported by the fact that for sample 1*B* the [100][001] velocity curve lies somewhat above the [110][001] curve for this sample at all but the lowest temperatures.

From Fig. 3 it can be seen that the additional increase in velocity with decreasing temperature begins at a higher temperature for 33-MHz than for 11-MHz [110][001] waves and that the final velocity reached at the lowest temperature is the same for both frequencies. The former occurrence is to be expected from Eqs. (1) and (2), since ω_c/ω is smaller at a given temperature the higher the value of ω. The equality of the 11- and 33-MHz velocities at low temperatures is also understandable in terms of Eq. (2) since for both frequencies $\omega_D \gg \omega \gg \omega_c$. That these conditions are true can be seen by calculating values for ω_D and ω_c using their definitions given after Eq. (1). We find $\omega_D \approx 9.1\times10^{11}/T$ rad/sec and $\omega_c = 1.67\times10^6$ rad/sec at 4.2 K, whereas $\omega = 6.9\times10^7$ rad/sec at 11 MHz and $\omega = 2.1\times10^8$ rad/sec at 33 MHz. In calculating ω_D we used values of 0.1 m^2/V sec for the mobility and 1 for f_0. The mobility value was deduced from our resistivity and Hall data at 77 K. It should be noted that the highest electron mobility reported for GaSb at 77 K seems to be 1.0 m^2/V sec,[34] and that the mobility of conduction band electrons at 4.2 K is about 0.25 m^2/V sec for samples with the lowest concentration of conduction electrons (10^{17} cm^{-3}) reported on at that temperature.[35] The value of ϵ was obtained from the literature.[36] Our poor knowledge of the mobility and of f_0 at low temperatures cause uncertainty in the value calculated for ω_D. Of course since $0 \leq f_0 < 1$, ω_D might even be larger than that calculated above. Other evidence that $\omega_D \gg \omega$ is provided later by the fact that the attenuation maximum shown in Fig. 1 can be accounted for quantitatively by means of Eq. (3).

Now we shall make a quantitative analysis of the extra rise in velocity and of the attenuation maximum which occurred for [110][001] waves

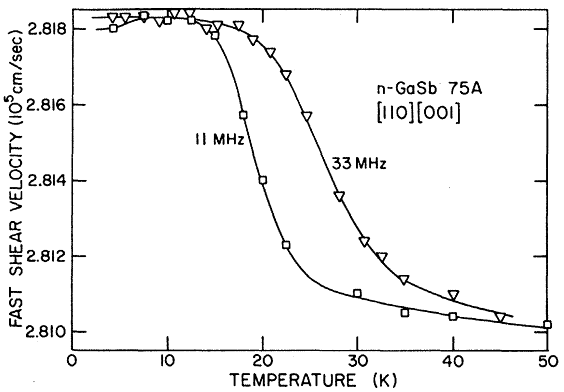

FIG. 3. Measurements showing how the velocity vs temperature curves for [110] [001] waves differed for different ultrasonic frequencies. The data were obtained on sulfur-doped *n*-type GaSb sample 75*A* as the temperature drifted upward slowly.

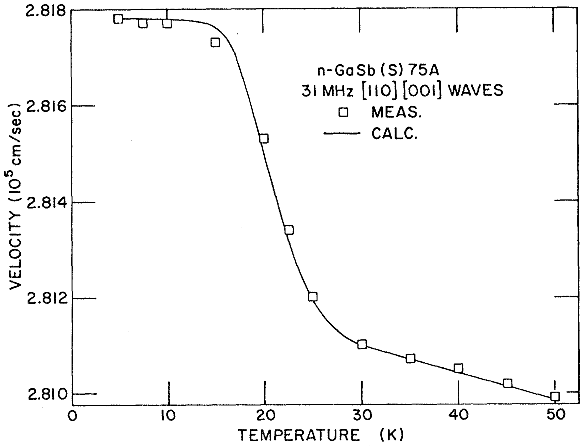

FIG. 4. Velocity of 31-MHz [110] [001] ultrasonic waves in sulfur-doped n-type GaSb between 5 and 50 K. The squares repeat the pertinent measured values from Fig. 1. The curve shows the velocity calculated by means of Eq. (2) of the text using the resistivity given by Eq. (4) of the text. Values of v_0 were obtained from the velocity measured for [100] [001] waves as indicated in the text. The difference between the measured [110] [001] wave velocity and v_0 at 4.2 K yields a value of 0.170 C/m^2 for the magnitude of the piezoelectric constant.

in sample 75A. In order to do so, we have prepared Figs. 4 and 5 which show large scale plots of the data for smaller temperature ranges than in Fig. 1 along with curves calculated using Eqs. (2) and (3). Both calculated curves were obtained by choosing the conductivity to be

$$1/\rho_{\Omega\,\mathrm{cm}} = (19.3 e^{110.7/T})^{-1} + (4.832\, e^{9.18/T})^{-1}. \qquad (4)$$

Choice of this *form* for the conductivity was guided by the behavior of other semiconductors at low temperatures as first exemplified by Ge.[37] The *numerical quantities* were chosen so that Eq. (4) gave the observed dc resistivity at 4.2 K and yielded resistivity values at higher temperatures which allowed Eqs. (2) and (3) to fit the extra velocity increase and attenuation maximum observed at low temperatures.

The first term in Eq. (4) is probably due to the freezing out of electrons from the conduction band into impurity levels located about 0.0095 eV below the conduction band edge. No levels at this location have been reported previously although it has been suggested[30] that there might be both selenium and sulfur levels associated with and somewhat below the bottom of the Γ_1 primary conduction band miminum. The deeper sulfur level, located about 0.07 eV below the Γ_1 minimum and associated with the X_1 minimum by some authors[29] and with the L_1 minimum by others,[30] which is mainly responsible for the temperature dependence of the resistivity (and Hall effect) above 77 K, is not represented in Eq. (4). This is because such a term is negligible at temperatures where the extra velocity increase and attenuation maximum occur. The origin of the second term is unknown. It might be due to a level only about 0.0008 eV below the conduction band edge, due to Te perhaps, or alternatively, could be due to conduction by carriers in the impurity states having the 0.0095-eV ionization energy.

In using Eq. (2) to fit the extra velocity rise, we used a value for v_0 deduced from the velocity versus temperature data curve for [100][001] waves by moving this data curve so that the velocity of these waves agreed with that observed for [110][001] waves at high temperatures where $\omega_c \gg \omega$ and the velocities of both kinds of waves must be equal.

In calculating the curve shown in Fig. 4 we took $e_{14}^2/2c_{44}\epsilon$ to be equal to the experimental value of $(v - v_0)/v_0$ at 4.2 K, where v is observed for [110][001] waves and v_0 the velocity given by the shifted [100][001] data curve. Because of the excellent fit of the calculated velocity curve to the data, as shown in Fig. 4, we feel confident to proceed to calculate a value for the magnitude of the piezoelectric constant e_{14} from $\Delta v/v_0$ at 4.2 K. Specifically, we find

$$|e_{14}|_{\Delta v} = [2c_{44}\epsilon(\Delta v/v_0)]^{1/2}_{4.2\,\mathrm{K}} \simeq 0.170 \pm 0.005 \ \mathrm{C/m^2}$$

by using $c_{44} = 4.44 \times 10^{10}$ N/m^2,[33] and $\epsilon = 15.7\epsilon_0$,[36] where ϵ_0 is the permittivity of free space.

In order to compare our attenuation data with that calculable from Eq. (3) we have plotted in Fig. 5 experimental points which give the difference between the total observed attenuation and a small background value obtained by interpolating between the attenuation values measured at temperatures outside the region of the maximum. The calculated curve in Fig. 5 was obtained by making the attenuation calculated from Eq. (3) for $\omega = \omega_c$ equal to the maximum experimental attenuation. Then the whole attenuation-versus-temperature curve was calculated by using Eq. (3) and the resistivity given by Eq. (4). From Fig. 5 it can be seen that the experimental points are fitted extremely well by Eq. (3). Therefore, we feel justified in deducing a value for the magnitude of the piezoelectric constant from our attenuation data. Specifically, we find

$$|e_{14}|_{\Delta\alpha} = (4c_{44}\epsilon v_0 \alpha_m/\omega)^{1/2} = 0.160 \pm 0.016 \ \mathrm{C/m^2},$$

where the maximum "experimental" attenuation α_m is 6.1 dB/cm. Values for c_{44} and $v_0 = (c_{44}/\rho_m)^{1/2}$ were taken from Ref. 33 and the same value of

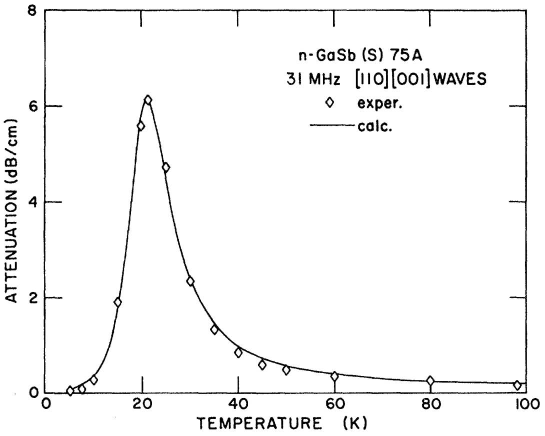

FIG. 5. Attenuation of 31-MHz [110] [001] ultrasonic waves in sulfur-doped *n*-type GaSb vs temperature. The experimental points show $\Delta\alpha$, the difference between the measured attenuation shown in Fig. 1, and a background value deduced from attenuation values measured outside the region of the maximum. The curve shows the attenuation calculated by means of Eq. (3) of the text using the resistivity given by Eq. (4) of the text and a value of 0.160 C/m^2 for $|e_{14}|$ deduced by equating the maximum value of $\Delta\alpha$ to that given by Eq. (3) when $\omega_c = \omega$.

ϵ was employed as previously. The fact that Eq. (3) can be made to fit the observed attenuation peak with a value for $|e_{14}|$ within experimental error of that which we deduced from our velocity data is excellent evidence that $\omega_D \gg \omega$, since as shown in Ref. 1 if this condition were not true, the attenuation peak would be smaller and wider than that calculable from Eq. (3) (and would not occur at $\omega = \omega_c$).

It is now time to compare our values of $|e_{14}|$ with that obtained from previous work. Arlt and Quadflieg[21] (henceforth referred to as AQ) obtained a value of $e_{14} = -0.126 \pm 0.025$ C/m^2 using what might be called an ultrasonic Hall-effect method which requires knowing the value of the applied magnetic induction and the ultrasonic strain. Note that both our values of $|e_{14}|$ are larger than that of AQ. Our value of $|e_{14}|$ deduced from the attenuation is within the sum of the quoted experimental errors of the AQ value but our value of $|e_{14}|$ from the velocity is not. Since we believe the latter value is our most accurate one it is necessary to consider the origin of the discrepancy between it and the AQ value.

We believe the discrepancy is not due to some defect in our analysis since we have previously obtained values of $|e_{14}|$ for[20,22] GaAs and[22,23] InSb which agree very well with those determined by AQ. A possible reason why AQ's value for GaSb is lower than ours is that their sample had a much larger density of dislocations. Some workers[38] have reported that in CdS a dislocation density of 10^6/cm^2 can cause a reduction in the piezoelectric constant. Such a large dislocation density seems to be unlikely for GaSb in view of previous investigations.[39] (In our sample the highest dislocation density which we obtained by counting etch pits was a few thousand per cm^2.) A low value of $|e_{14}|$ might also be attributable to resistivity inhomogeneities and stacking faults since these imperfections have been invoked to explain low values of the electromechanical coupling constant $e_p^2/2c\epsilon$ in CdS.[13,15]

Since in deducing values of $|e_{14}|$ from our ultrasonic data we employed a literature value for ϵ, one might wonder just how appropriate such a value of ϵ is. Previous work[40] on Ge has shown that ϵ might be appreciably greater in a sample with a relatively large concentration of isolated neutral impurities than in a purer sample. Other investigators[41] have found that in Si and Ge hopping of electrons from neutral to ionized impurities may also enhance ϵ.

Unfortunately, we do not know enough about the types and concentrations of impurities in our samples nor in the material which was used to determine ϵ to be certain that we have used an appropriate value for ϵ. It might be surmised that due to the rather low Hall mobilities exhibited by our samples they might have enough impurities to cause ϵ/ϵ_0 to have a value greater than 15.7. If so, even higher values of $|e_{14}|$ would result from our ultrasonic data than those which we have deduced above, thereby increasing the discrepancy between our values and that of AQ. Since we have no physical reason to expect ϵ/ϵ_0 to be less than 15.7 in our samples, we must conclude that our use of it is appropriate and our values of $|e_{14}|$ are not flawed because of uncertainty in the value of ϵ.

IV. CONCLUSION

An enhancement of the velocity and a maximum in the attenuation of ultrasonic waves propagating in the [110] direction and polarized in the [001] direction have been observed in S-doped, *n*-type GaSb at low temperatures. They can be accounted for using theoretical formulas of Hutson and White for piezoelectric semiconductors in which there is negligible diffusion of the electronic space charge produced by the ultrasonic waves. Fitting the theoretical formulas to our experimental results indicate that the electrical conductivity is comprised of two exponential terms controlled by impurity levels which are closer to the conduction band edge than are the sulfur levels which control the concentration of conduction electrons above 77 K. Values for the magnitude of the piezoelectric constant $|e_{14}|$ deduced from our velocity and attenuation data are within experimental error of each other. Both our values are larger, and we believe more accurate, than the previous value obtained by others using an "ultrasonic-Hall-effect" method. Our smaller value of $|e_{14}|$ is within experimental error of this previous value but our larger more-accurate value is not. The reason for this disagreement is unknown.

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