Magnetophonon and Shubnikov–de Haas oscillations in n-type PbTe epitaxial films

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We have observed Shubnikov–de Haas (SdH) and magnetophonon (MP) oscillations in the magnetoresistance of single crystal, vapor-phase, epitaxial films of n-PbTe on BaF_2 substrates. For a film with $n = 6\times10^{16}\text{cm}^{-3}$, the SdH oscillations show that all electrons are in a single $\langle 111\rangle$ valley at 4.2 K. This result is a consequence of a thermally induced shear strain which pulls the valleys toward the bismuth structure. Both the cyclotron mass and cross-section anisotropy of the corresponding ellipsoid were obtained. Several series of MP oscillations were detected. Use of the measured cyclotron masses allows each series to be identified with intravalley scattering by long-wavelength LO phonons with energies of about 14 meV.

I. INTRODUCTION

The relative contributions of the possible types of phonon scattering in the lead salts has been open to question for many years. For example, extensive studies of the temperature dependence of the mobilites of PbTe and SnTe by Allgaier *et al.*[1] suggest that long-wavelength acoustical phonons are the only lattice vibration modes that need to be considered. In contrast, analyses of a variety of transport data by Ravich *et al.*[2,3] show the need for a significant contribution from polar optical-phonon scattering.

A sensitive probe of the optical-phonon–electron interaction is the measurement of the temperature dependence of the magnetoresistance in the quantum regime. In this regime the interaction will manifest itself as a resonance between the energy of nondispersive optical phonons and the energy separation between Landau states. In a parabolic band, the result is a periodic oscillation in the magnetoresistance. The period of these oscillations is directly related to the energy of the phonon involved. When there are interactions with more than one phonon, several periods may be present simultaneously. The conduction band of PbTe is a multivalley structure consisting of four $\langle 111\rangle$ ellipsoids of revolution located at the L points of the Brillouin zone of the fcc lattice. Thus, for an arbitrary orientation of the magnetic field, intervalley scattering by a single phonon may produce four periods. Intervalley scattering between ellipsoids, typically by zone edge phonons, will lead to additional periods. Experiments on Si,[4] and Ge,[5,6] provide excellent examples of these types of phenomena.

The condition for intravalley magnetophonon MP resonance in a parabolic band can be written

$$\hbar\omega_p = N\hbar\omega_c = N\hbar eB/m^{\text{cyc}}c\ , \tag{1}$$

where ω_p is the angular phonon frequency, ω_c is the angular cyclotron frequency, N is the quantum number difference of the initial and final Landau states, m^{cyc} is the cyclotron effective mass, and B is the magnetic field intensity in gauss. These resonances occur periodically in $1/B$, and, as shown in detail by the theories of Gurevich and Firsov[7] and Efros,[8] lead to corresponding oscillations in the magnetoresistance. Using Eq. (1), the frequency of the oscillations (defined as the reciprocal of the period) can be expressed in G by

$$F = (c/\hbar e)m^{\text{cyc}}\hbar\omega_p\ . \tag{2}$$

Thus, the energy of the phonon involved can be determined from the frequency of the oscillation and an independent measurement of the cyclotron effective mass. On the other hand, if the energy of the phonon is known, the effective mass may be obtained. This approach is often very useful for determinations of effective mass at high temperatures where Shubnikov–de Haas (SdH) or de Haas–van Alphen studies are not feasible.

II. EXPERIMENTAL

Figure 1 is a schematic diagram of the experimental apparatus. The sample current was provided by a Princeton Applied Research Model No. TC-602CR constant current supply. The resistivity voltage was amplified by a model 149 Keithley dc amplifier. The output of this amplifier was filtered to eliminate internally generated modulation noise which became very large when differentiated. When the magnetoresistance was to be measured, the output of the filter was coupled directly to the y axis of the x-y recorder. Observation of MP oscillations in our films required at least one differentiation of the magnetoresistance signal and, in general, the second derivative produced the best MP signal relative to the magnetoresistance background.

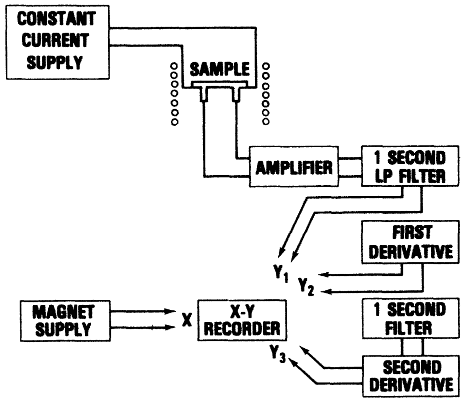

FIG. 1. Schematic diagram of experimental apparatus.

The magnetic field was provided by a $1\frac{1}{4}$-in.-bore NiTi superconducting magnet which generates fields up to 85 kG at 4.2 K. The field is linear with respect to current so that the voltage from a resistor in series with an Intermagnetics General 150-V power supply determines the field and drives the x axis of the x-y recorder. Temperatures between 4.2 K and 130 K were maintained to within 0.1 degree or better using helium gas flow in a Janis supervaritemp Dewar, and a GaAs diode as a temperature sensor for a Lakeshore Cryogenics controller.

The samples we measured were thick films grown on BaF_2 substrates by the hot wall technique.[9] Film thicknesses were on the order of 2 μm, mobilities at 77 K were about 25 000 cm^2/V sec, and carrier densities ranged from 5×10^{16} to 1×10^{18} cm^{-3}. The films were grown on a $\{111\}$ surface of BaF_2 and were single crystals with a $\langle 111\rangle$ direction normal to the growth surface. BaF_2 is a useful subtrate for the growth of high quality films of the IV-VI compounds because the thermal coefficients of expansion of these materials are closely matched at room temperature and above. Below about 300 K, however, the difference between the coefficients becomes appreciable.[10] The resulting strain, which destroys the cubic symmetry, influences the band structure and transport properties of PbTe, as we will show. Films are very useful for measurement of the small signals described here because large voltages are achieved with small currents. Using the system shown in Fig. 1, oscillations with amplitudes as small as 1×10^{-4} of the background magnetoresistance can be detected.

III. RESULTS

A. Shubnikov-de Haas oscillations

As indicated by Eq. (2), a determination of the energies of the phonons responsible for the MP oscillations requires a knowledge of the cyclotron effective masses of the carriers involved. Therefore, we first consider the SdH oscillations from which these masses are derived.

The geometry of the Fermi surface of PbTe is shown in Fig. 2. For the data reported here, the sample current was parallel to the $[1\bar{1}0]$ axis, and the transverse magnetoresistance was measured for $\vec{B}\parallel[111]$ and $\vec{B}\parallel[11\bar{2}]$. For ellipsoids of revolution, the frequencies of the corresponding SdH oscillations can be written[11]

$$F_k=(3.15\times10^5)(n_k/10^{18})^{2/3}K^{1/6}\times[1+(K-1)\cos^2\theta]^{-1/2}\,, \qquad (3)$$

where n_k is the number of carriers per cm^3 enclosed by the k^{th} ellipsoid, θ is the angle between the magnetic field direction and the ellipsoidal major axis, and K is the square of the extremal cross-section anisotropy. Figures 3 and 4 show the SdH oscillations for the two orientations of $\vec{B}$. For $\vec{B}\parallel[11\bar{2}]$, three series of oscillations, corresponding to the three different extremal cross sections of ellipsoids 1, 2, and 3, should be observed. Instead, only the fundamental frequency $F=(7.0\pm0.1)\times10^4$ G (and its second harmonic) from the largest extremal cross section of ellipsoid 3 are detected. For $\vec{B}\parallel[111]$, instead of two series of oscillations from ellipsoids 2 and 3 (1 and 2 are now equivalent), only the fundamental $F=(2.35\pm0.05)\times10^4$ G (and its second harmonic) from the smallest extremal cross section of ellipsoid 3 are observed. Using these values of F in Eq. (3), we obtain

$$K=[F(11\bar{2})/F(111)]^2=9.0\pm0.6$$

and (4)

$$n_3=6.0\times10^{16}\ cm^{-3}\,.$$

Comparison of n_3 with the total electron density, n obtained from the high-field Hall coefficient, indicates that all the electrons are in ellipsoid 3. This accident, which will be explained in Sec. III A 1, has made it possible for the first time to make a determination of K in n-type PbTe from a direct measurement of the large extremal cross section. Because of the low carrier density, the value obtained is essentially a band-edge value and is in agreement with values previously reported by Fujimoto[12] and Cuff *et al.*[13]

1. Strain effects

An explanation for the depletion of electrons from all but ellipsoid 3 can be seen in the effect of strain on the multivalley band structure of PbTe. Magneto-optical measurements by Palik *et al.*[14] first showed that the low-temperature band gaps of $\{100\}$ epitaxial films of the lead salts on

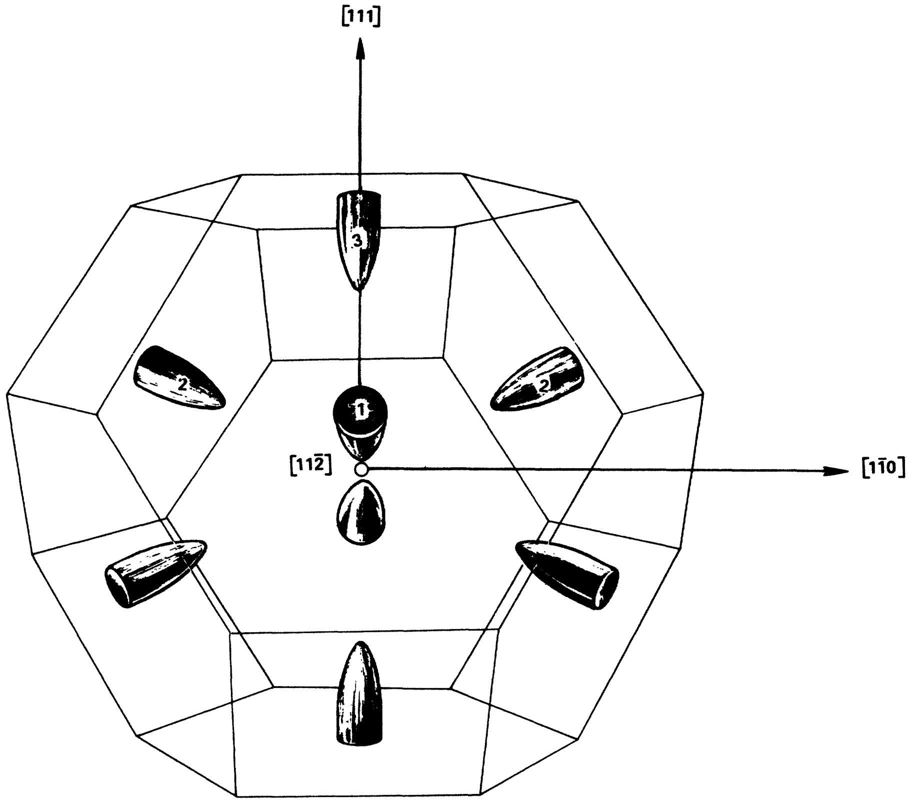

FIG. 2. Brillouin zone of the fcc lattice and the Fermi surface of n PbTe. In the experiments described here, the sample current was parallel to [1$\bar{1}$0] and the magnetic field was parallel to [111] or [11$\bar{2}$].

{100} substrates of NaCl are smaller than those in bulk material. Their result was explained by a calculation of the compressional strain in the films which should occur as a consequence of the difference between the thermal coefficients of expansion of the lead salts and NaCl. For {111} films of the lead salts on {111} substrates of BaF_2, there is the additional effect that the shear com-

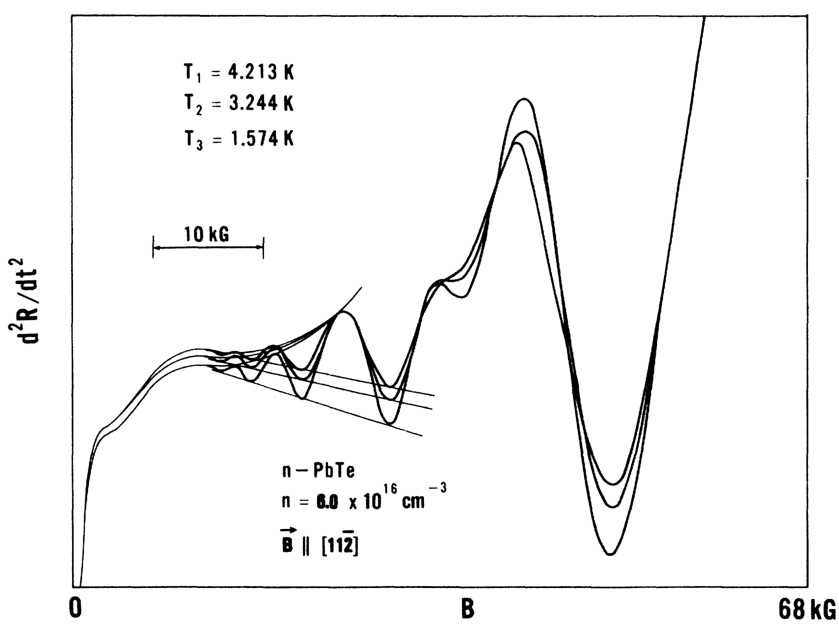

FIG. 3. Second derivative of the Shubnikov–de Haas oscillations for $\vec{B}$∥ [11$\bar{2}$]. The oscillations are generated by ellipsoid 3 (Fig. 2).

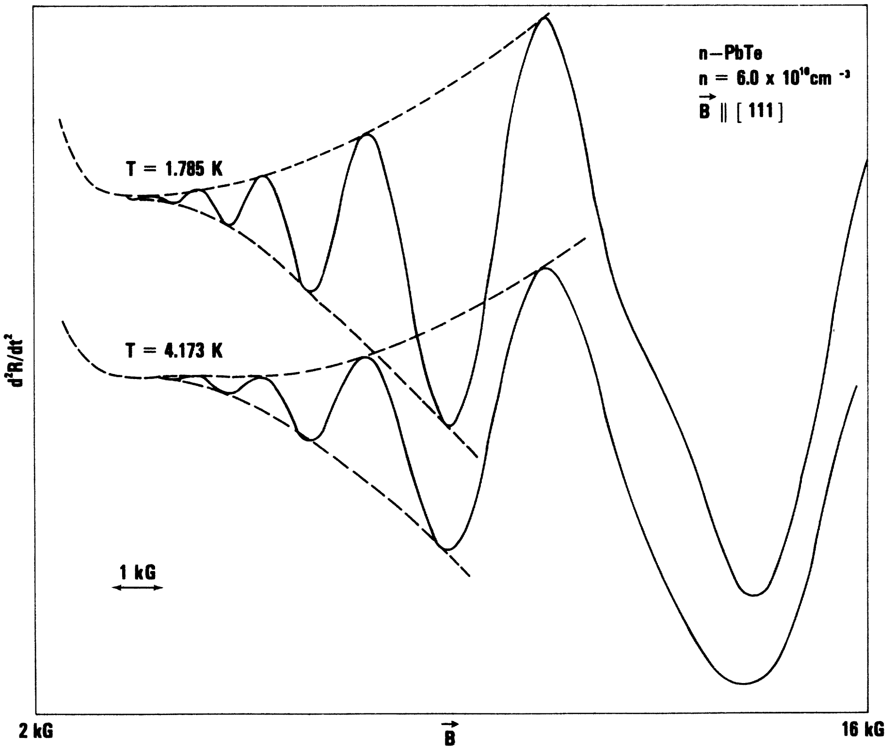

FIG. 4. Second derivative of the Shubnikov–de Haas oscillations for $\vec{B}$ || [111]. The oscillations are generated by ellipsoid 3 (Fig. 2).

ponent of the tension removes the degeneracy of the valleys.[15] The energy shift of the kth valley can be written

$$\delta E^{(k)} = \sum_{ij} D_{ij}^{(k)} \epsilon_{ij} , \qquad (5)$$

where ϵ_{ij} is the strain tensor and $D_{ij}^{(k)}$ is the deformation-potential tensor which, in the cubic axis system, can be written

$$D_{ij}^{(k)} = D_d \delta_{ij} + D_u u_i u_j . \qquad (6)$$

Here D_u and D_d are, respectively, the uniaxial and dilatational deformation-potential constants in the Herring and Vogt[16] notation, and the u's are the direction cosines of the angles between the major axis of the kth ellipsoid and the cubic axes.

We estimate the strain by integration of the difference between the expansion coefficients of PbTe and BaF_2 between 300 and 4.2 K.[10] Assuming, as a number of experiments have shown,[17] that there is no strain at 300 K, and also that there is no strain relief at lower temperatures, the (111) plane of the film is under a tension of 2×10^{-3} at 4.2 K. Using the elastic constant tensor,[18] one then computes that there is a compression of about this magnitude in the [111] direction. Transforming these strains into the cubic axis system, and using the conduction band values $D_u^c = 8.3$ eV and $D_d^c = -4.4$ eV calculated by Ferrara,[19] from Eqs. (5) and (6) we find that ellipsoid 3 (the [111] ellipsoid) is 0.030 eV lower in energy than the other three ellipsoids which remain degenerate. This shift is more than six times what the Fermi level would be if our film was unstrained. Thus, the transfer of all electrons into ellipsoid 3 at 4.2 K is easily accounted for. Using the valence-band deformation potentials $D_u^v = 10.5$ eV and $D_d^v = -8.9$ eV to calculate the shift of the valence-band extrema,[19] we obtain the band structure shown in Fig. 5. The separation between valleys 3 has increased 13 meV relative to the

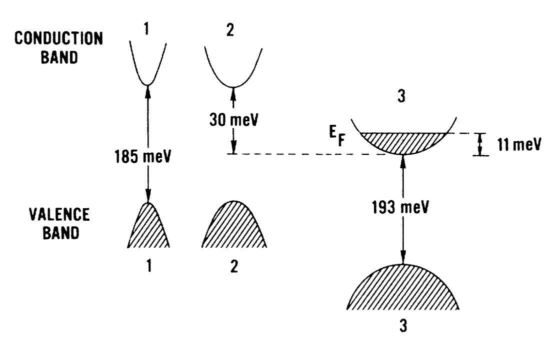

FIG. 5. Proposed band structure of the 2.8-μm epitaxial film of n-type PbTe on BaF_2 at 4.2 K. Fermi energy is for an electron density of 6.0×10^{16} cm^{-3}. Curvatures shown are for $\vec{B}$ || [11$\bar{2}$].

180-meV energy gap in the unstrained crystal, while that between valleys 1 and valleys 2 has increased by 5 meV. This structure approaches that of bismuth, and, at low temperatures and electron densities, is the first example of a single valley ellipsoidal band.

n-PbTe films on BaF_2 have also been grown by the hot-wall technique by Lopez-Otero at the Univeristy of Linz in Austria.[20] These films have much higher mobilities than previously reported and our SdH data on a film 16 μm thick suggest that all ellipsoids are populated. Far infrared cyclotron resonance measurements by Bauer[21] indicate that the ellipsoid population is independent of film thickness. This is in contrast to our recent SdH measurements on the White Oak material which shows that the ellipsoid population and thus the strain is a function of film thickness. Taken together, these results demonstrate that the properties of PbTe films on BaF_2, in particular the degree of strain, are significantly affected by the choice of growth parameters in the hot-wall technique. A study of the thickness dependence of the SdH and MP effects, using both White Oak and Linz films, will be described in a future publication.

2. *Cyclotron masses*

The amplitudes of SdH oscillations at a given $\vec{B}$ for two temperatures T_1 and T_2 are related by[11,22]

$$\frac{A(T_1)}{A(T_2)}=\frac{T_1}{T_2}\,\frac{\sinh(x)}{\sinh[(T_1/T_2)x]}\,, \tag{7}$$

where $x=2\pi^2kT_2/\hbar\omega_c$, $\omega_c=eB/m^{cyc}c$, and T_1 is the higher temperature. Equation (7) is solved graphically for x, for several values of $\vec{B}$. The results for $\vec{B}\parallel[11\bar{2}]$ and $\vec{B}\parallel[111]$, corresponding to Figs. 3 and 4 are shown in Fig. 6. The cyclotron masses are obtained from the slopes of these curves. This is a useful procedure because extrapolation through the origin indicates that systematic errors in the construction of amplitude envelopes, T_1/T_2, and $\vec{B}$ have been eliminated. For $\vec{B}\parallel[11\bar{2}]$, $m_h^{cyc}=(0.0667\pm0.0007)m_0$, and for $\vec{B}\parallel[111]$, $m_l^{cyc}=(0.0230\pm0.0003)m_0$, where the quoted uncertainties are derived from the uncertainties in slope determination. The cyclotron mass anisotropy is, therefore,

$$m_h^{cyc}/m_l^{cyc}=2.9\pm0.1$$

or (8)

$$(m_h^{cyc}/m_l^{cyc})^2=m_h/m_l=8.4\pm0.6\,,$$

where m_h and m_l are the principal ellipsoidal masses. m_l^{cyc} is in good agreement with that reported by Cuff *et al.*[13] for the conduction-band edge in bulk material. m_h^{cyc} has not been measured previously.

Comparison of Eqs. (4) and (8) shows that within experimental error the values of K and of

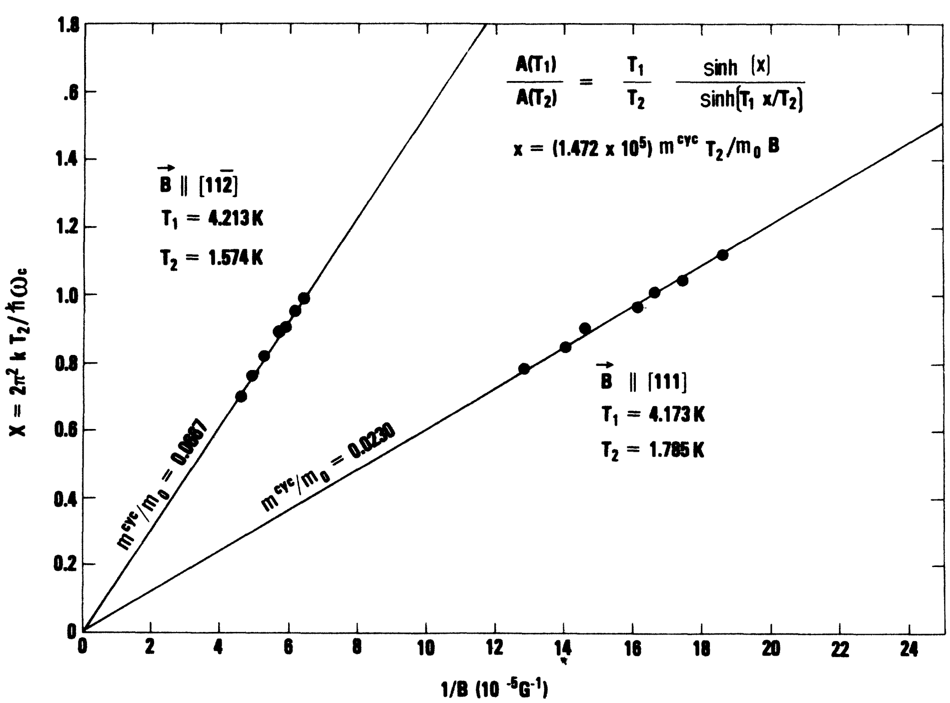

FIG. 6. Graphical solutions (x) of the equation shown in the figure for corresponding combinations of $A(T_1)/A(T_2)$ and $1/B$, vs $1/B$. The slopes of these curves determine m^{cyc}/m_0.

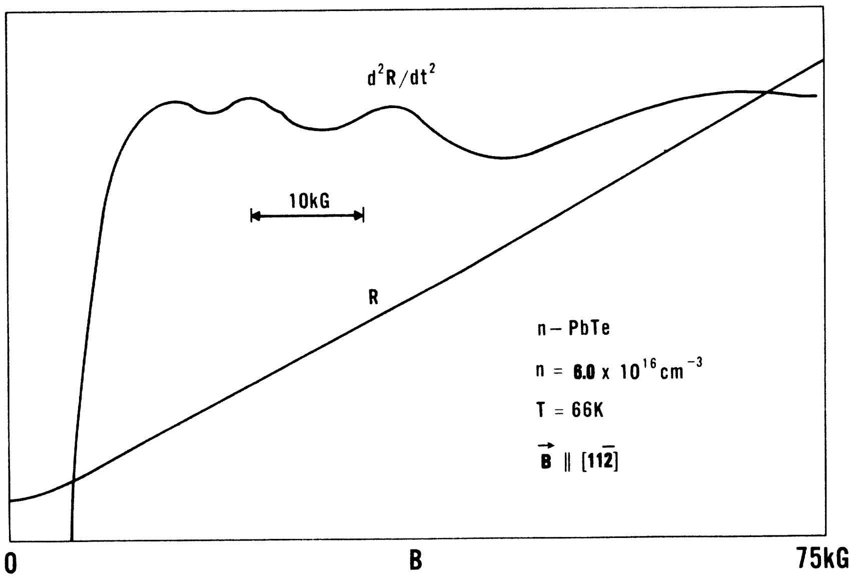

FIG. 7. Magnetoresistance R and its second derivative d^2R/dt^2 at 66 K for $\vec{B}$ ∥ [11$\bar{2}$]. Magnetophonon oscillatirons are seen in d^2R/dt^2.

$(m_h^{cyc}/m_l^{cyc})^2$ are equal. This result follows from a PbTe band model, previously proposed by Burke *et al.*[11] in which both principal masses have the same energy dependence. For this model K is a constant and, for all carrier densities,

$$K = (m_h^{cyc}/m_l^{cyc})^2 . \qquad (9)$$

The model for the valence band was confirmed by SdH measurements which showed that within experimental error, K is constant at least up to a hole density of 1×10^{19} cm^{-3}.[23,24] A recent analysis of a variety of PbTe data by Foley and Langenberg[25] indicates that a "best" statistical fit is obtained when K for the valence band increases slightly, and K for the conduction band decreases slightly, with increasing carrier density.

B. Magnetophonon oscillations

The magnetoresistance R and its second derivative d^2R/dt^2 at 66 K for $\vec{B}$ ∥ [11$\bar{2}$] are shown in Fig. 7. By calibrating the differentiating circuits, it is found that the undifferentiated amplitudes of the oscillations are about 8×10^{-4} of the zero-field resistivity. The small size of this signal can be visualized by recognizing that the large slope of d^2R/dt^2 near $\vec{B}=0$ is due to the slight curvature of R in this region.

The temperature dependence of the oscillations

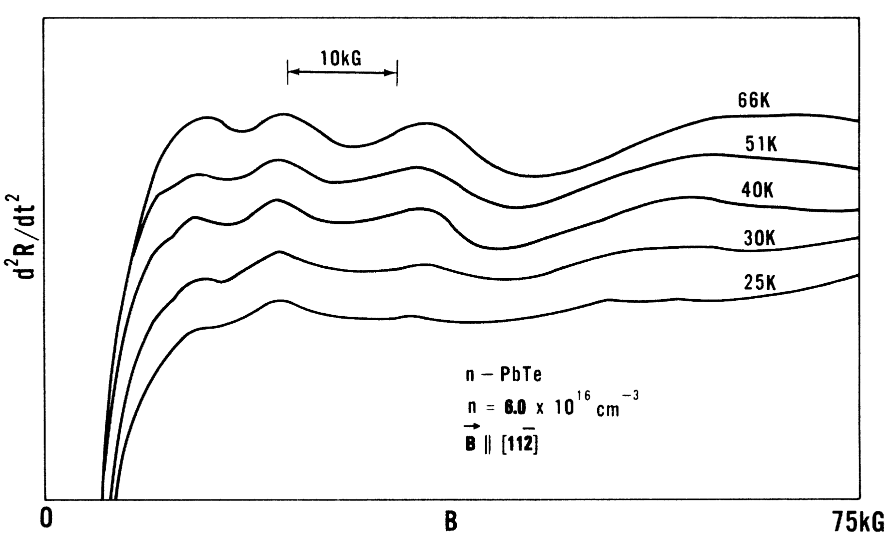

FIG. 8. Temperature dependence of the magnetophonon oscillations for $\vec{B}$ ∥ [11$\bar{2}$].

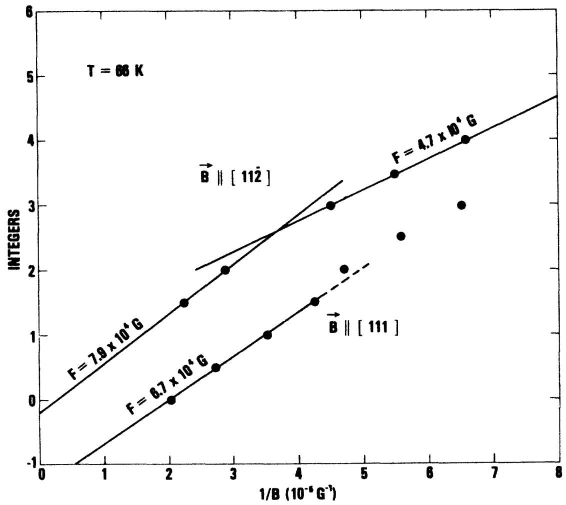

FIG. 9. $1/B$ values of the magnetophonon extrema at 66 K or $\vec{B} \parallel [11\bar{2}]$ and $[111]$. Slopes of these curves give the frequencies F [Eq. (1)]. The data indicate the presence of more than one frequency for each orientation of $\vec{B}$.

for $\vec{B} \parallel [11\bar{2}]$ is shown in Fig. 8. The decrease of the amplitudes with decreasing temperature is the expected behavior for MP oscillations when the decrease in the number of phonons is dominant over the accompanying increase in the relaxation time of the carriers.[26] In contrast, the amplitude of SdH oscillations always increases with decreasing temperatures. For a number of n-type PbTe samples, we have found that the amplitude of the MP oscillations has a broad maximum between 50 and 70 K.

Equation (1) shows that we may deduce the frequency of the oscillations from the slope of a plot of integers versus the $1/B$ values at which the extrema occur. Figure 9 is such a plot for T = 66 K for the two orientations of $\vec{B}$ studied. The oscillations for $\vec{B} \parallel [111]$ are given in Fig. 10. If only one frequency or series of oscillations were present for each orientation of $\vec{B}$, the points would lie on straight lines. For $\vec{B} \parallel [11\bar{2}]$, the data can be interpreted as a change over from $F = 7.9 \times 10^4$ G at high fields to $F = 4.7 \times 10^4$ G at low fields. The behavior of the data for $\vec{B} \parallel [111]$ is not as obvious, but it is clear both from the oscillatory pattern in Fig. 10 and the break in the data at about $1/B = 4.5 \times 10^{-5}$ G^{-1} that in addition to $F = 6.7 \times 10^4$ G another frequency is also present.

The results from Fig. 9 and from the SdH and cyclotron-mass measurements are summarized in Table I. The cyclotron-mass values in parentheses were calculated from the formula at the bottom of the table, using $m_h^{cyc}/m_0 = 0.0667$, $m_l^{cyc}/m_0 = 0.0230$ and the appropriate value of θ, the angle between the magnetic field direction and the major axis of the ellipsoid. The MP frequencies scale approximately as the cyclotron masses of the ellipsoids showing that the same phonon is responsible for each frequency. This result also shows that in contrast to the situation at 4.2 K, all the ellipsoids are at least partially populated at 66 K by a combination of thermal excitation and a reduction in the thermally induced strain. From the MP frequencies, the 4.2-K values of m^{cyc}/m_0, and Eq. (2), the average value of $\hbar\omega_p$ is 14 meV. Because of the temperature and strain dependence of the band gap between 4.2 and 66 K, the exact values of m^{cyc}/m_0 to be used in Eq. (2) for the computation of $\hbar\omega_p$ at 66 K are difficult to obtain. However, comparison of the 14-meV result with neutron diffraction data[27-29] shows that each of the observed MP frequencies derive from intravalley scattering by long-wavelength LO phonons. Using the neutron diffraction result for $\hbar\omega_p$, values for m^{cyc}/m_0 at 66 K could be cal-

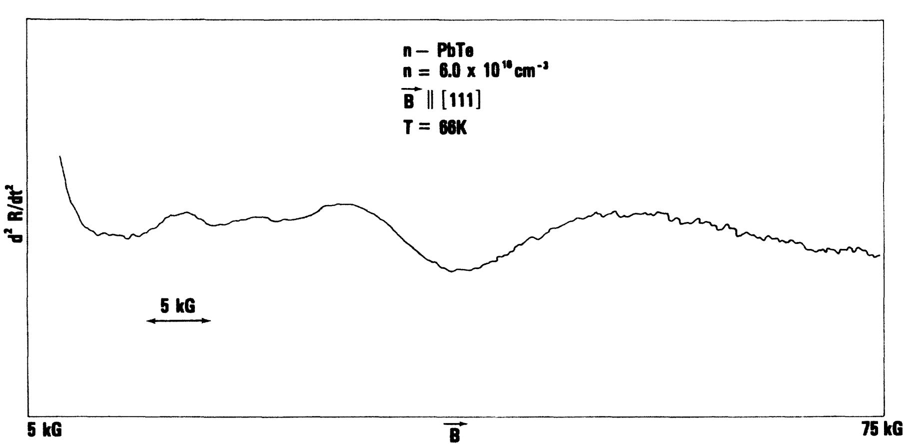

FIG. 10. Magnetophonon oscillations at 66 K for $\vec{B} \parallel [111]$.

TABLE I. Summary of Shubnikov–de Haas (SdH) and magnetophonon (MP) data from an n-PbTe epitaxial film on BaF_2. $n = 6.0 \times 10^{16}$ cm^{-3} and film thickness is 2.8 μm.

	$\vec{B} \parallel [111]$			$\vec{B} \parallel [11\bar{2}]$		
Ellipsoid	F(SdH) (10^4 G) 4.2 K	F(MP) (10^4 G) 66 K	m^{cyc}/m_0 4.2 K	F(SdH) (10^4 G) 4.2 K	F(MP) (10^4 G) 66 K	m^{cyc}/m_0 4.2 K
1		6.7	(0.0494)[a]			(0.0242)
2		6.7	(0.0494)		4.7	(0.0410)
3	2.35		0.0230 ± 0.0003	7.00	7.9	0.0667 ± 0.0007

[a]Numbers in parentheses calculated from $m^{cyc} = m_h^{cyc}\{1 + [(m_h^{cyc}/m_l^{cyc})^2 - 1]\cos^2\theta\}^{-1/2}$.

culated from Eq. (2). We have not presented these values in Table I because it is difficult to extract sufficiently reliable frequencies for their calculation. This is a consequence of not being able to identify a magnetic field range in which one series of oscillations can be clearly separated from the other. This may be possible when the MP spectra are Fourier analyzed.

Tsui *et al.*[30] have observed MP oscillations in bulk n-type PbTe samples over a range of electron densities. Their data is also explained by intravalley long-wavelength LO phonon scattering. However, the amplitude of the oscillations in our films are typically 20 times smaller than those observed by Tsui *et al.*[30] even though the electron mobilities are comparable. A study, presently in progress, shows that as the White Oak films are made thicker, the MP amplitudes approach bulk values while the mobility remains constant. These results are surprising because typically the amplitudes of oscillatory phenomena only increase when the mobility increases and collision broadening of the Laudau states is reduced.

No evidence for intervalley scattering by zone-boundary x phonons was observed either in our experiment or in those on bulk material.[30] The associated electron-phonon coupling is the proposed mechaniam[31,32] for the existence of the superconducting state in SnTe,[33] and $Pb_{1-x}Sn_xTe$,[34] semiconductors with multivalley band structures approximating that of PbTe.

Tsui *et al.*[30] identify the MP oscillations with electron scattering by unscreened LO phonons on the basis of the calculations by Cowley and Dolling[27] which show that small $\vec{q}$ screened modes have much lower energies than that derived from the MP oscillations. The experimental LO dispersion curves however show a much weaker $\vec{q}$ dependence. Thus, within experimental error, we do not feel that the interaction with unscreened modes has been demonstrated.

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