



## TEMPERATURE VARIATION OF LATTICE PARAMETER AND OPTICAL ENERGY GAP VALUES OF THE COMPOUNDS $\text{CdIn}_2\text{Te}_4$ AND $\text{MnIn}_2\text{Te}_4$

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**Abstract**—X-Ray powder diffraction measurements, in the range from 300 K to 700 K, and optical absorption measurements, in the range 20–300 K, were made on polycrystalline samples of  $\text{CdIn}_2\text{Te}_4$  and  $\text{MnIn}_2\text{Te}_4$  compounds. In each case, from the analysis of the X-ray diffraction lines, accurate lattice parameter values were determined as a function of temperature, thus the thermal expansion coefficients of the materials are obtained. The absorption measurements were used to determine values of the optical energy gap  $E_g$  as a function of temperature. The resulting curves of  $E_g$  vs  $T$  were fitted to a simplified Manooogian–Leclerc equation and the fitted coefficients used to give values of  $(dE_g/dT)_1$  and  $(dE_g/dT)_2$  due to lattice dilation and electron–phonon contributions respectively. Hence, the deformation potentials of the valence and conduction bands were estimated for both compounds.

**Keywords:** A. semiconductors, C. X-ray diffraction, D. optical properties.

### INTRODUCTION

semiconductor materials containing manganese have received attention because of the manner in which the magnetic behaviour associated with the manganese atoms can modify and complement the semiconductor properties [1, 2]. The materials that have been the most studied are the alloys based on standard tetrahedrally coordinated semiconductor compounds, such as the I–VI compounds, and the corresponding Mn–VI materials, e.g.  $\text{Cd}_{1-x}\text{Mn}_x\text{Te}$ , etc. [1]. These studies have been extended to the investigation of the tetrahedrally coordinated I–III–VI<sub>2</sub> chalcopyrite base alloys, e.g.  $(\text{CuIn})_{1-x}\text{Mn}_x\text{Te}_2$ , etc. [3]. Another group of compounds which show similar tetrahedrally coordinated form are some of the II–III<sub>2</sub>–VI<sub>4</sub> compounds, and these with II = Mn can be produced easily. Most of these compounds have a defect tetragonal structure with one in four cation sites being vacant, and it has been shown [4] that the  $\text{CdIn}_2\text{Te}_4$  and  $\text{MnIn}_2\text{Te}_4$  compounds have this form. In recent works [5, 6], the phase diagrams of the  $\text{Cd}_{1-x}\text{Mn}_x\text{In}_2\text{Te}_4$  and  $\text{Mn}_{3-2x}\text{In}_{2(1-x)}\text{Te}_3$  systems were investigated, it was found that the compounds  $\text{CdIn}_2\text{Te}_4$  and  $\text{MnIn}_2\text{Te}_4$  undergo an order–disorder transformation at the

temperatures of about 720 and 650°C, respectively. Above 720°C the  $\text{CdIn}_2\text{Te}_4$  represents a partially disordered tetragonal structure, while below this temperature, the ordered tetragonal I4 structure occurs. In the case of  $\text{MnIn}_2\text{Te}_4$ , above 650°C, the structure is a disordered defect zinc-blende, and below this temperature, the ordered tetragonal I42m structure is presented. In earlier work carried out on this compound, Doll *et al.* [7] have determined lattice parameter values, in the temperature range 90–300 K, and Medvedkin *et al.* [8] have measured energy gap values for some temperatures in the range 77–370 K on samples of  $\text{MnIn}_2\text{Te}_4$  before and after heat treatment. Also, in recent work [9] the DTA data for this compound was re-investigated using more sensitive equipment (Perkin Elmer DTA 7) than used in the previous work [6] and it was shown that a previously unobserved transition occurs at 420°C [9]. Magnetic susceptibility measurements made on quenched and slowly cooled to room temperature samples showed that the phase  $\gamma_2$  above 420°C is a little more disordered than the tetragonal I42m phase  $\gamma_1$  which is present below this temperature [9].

In the present work, measurements of X-ray powder diffraction and optical absorption have been

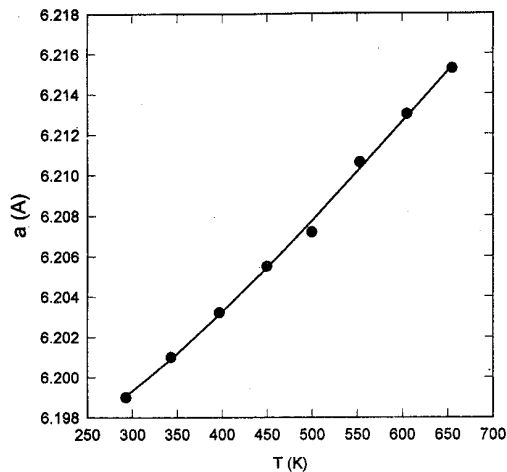


Fig. 1. Variation of the lattice parameter  $a$  with temperature  $T$  for  $\text{CdIn}_2\text{Te}_4$ . Full circles: experimental data; full curve: fitted curve.

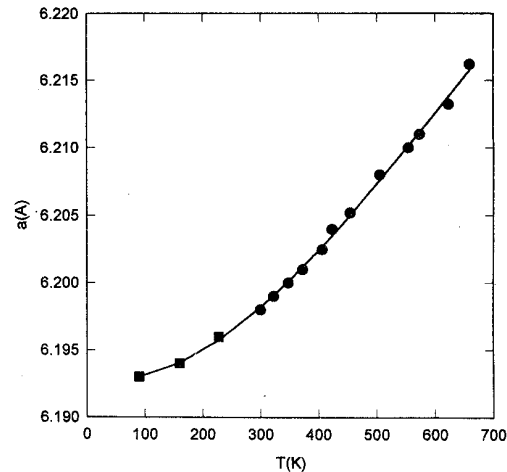


Fig. 2. Variation of the lattice parameter  $a$  with temperature  $T$  for  $\text{MnIn}_2\text{Te}_4$ . Full circles: experimental data; full squares: data from Ref. [7]; full curve: fitted curve.

carried out on the  $\text{CdIn}_2\text{Te}_4$  and  $\text{MnIn}_2\text{Te}_4$  compounds to determine values of the crystallography parameters in the temperature range between 300 K and 700 K, a range where  $\text{MnIn}_2\text{Te}_4$  showed the  $\gamma_1$  phase, and had values of fundamental optical energy gap  $E_g$  in the range 20–300 K for these compounds.

#### EXPERIMENTAL DETAILS

The samples used were prepared by the melt and anneal technique, the components of a 1.5 g sample were sealed under vacuum in a small quartz ampoule. In the case of the  $\text{MnIn}_2\text{Te}_4$  compound, the ampoule was previously carbonized to prevent interaction of the  $\text{Mn}^{2+}$  ions with the quartz. The components were melted together at 1150°C for about an hour, annealed to equilibrium at 500°C, then cooled to room temperature by leaving the ampoule in the switched-off furnace. Previous experience indicates that for this type of material, this procedure usually gives samples showing conditions corresponding to equilibrium near room temperature.

The X-ray powder photographs were obtained, for both compounds, in the temperature range 300 K–700 K, using a Guinier–Lernne high temperature camera with a speed of 40 h (2 mm/h) and the radiation was  $\text{CuK}\alpha$  ( $\lambda_{\text{Cu}}^* = 1.5418 \text{ \AA}$ ). The Bragg angles were corrected by using KCl powder as standard material. The lattice parameter values at different temperatures were calculated by using the usual least squares method together with all of the reflections. The accuracy in the determination of the lattice parameters was estimated to be  $\pm 0.001 \text{ \AA}$  in all cases.

Optical energy gap values were determined using measurements of optical absorption. Slices cut from

the sample ingot were polished down to give specimens suitable for standard transmission measurements. Values of  $\ln(I_0/I_t)$ , where  $I_0$  is the incident intensity and  $I_t$  the transmitted intensity, were determined as a function of photon energy  $h\nu$  at a number of temperatures in the range 20–300 K. These values were corrected by subtracting a background value to give values of the optical absorption coefficient  $\alpha$ . As has been shown, these materials have a direct energy gap [5, 8]. For each temperature a graph of  $(\alpha h\nu)^2$  was plotted against  $h\nu$  and the linear region was extrapolated to  $(\alpha h\nu)^2 = 0$  to give a value of the optical energy gap  $E_g$ .

Magnetic susceptibility measurements in the range 20–300 K were made using a Quantum Design SQUID magnetometer on the  $\text{MnIn}_2\text{Te}_4$  sample and it was found that the value of the Curie–Weiss temperature  $\theta$  corresponds to the  $\gamma_1$  phase.

#### LATTICE PARAMETER RESULTS AND ANALYSIS

The X-ray powder photographs showed that the samples have the expected defect tetragonal structure with space groups I4 and I42m for  $\text{CdIn}_2\text{Te}_4$  and  $\text{MnIn}_2\text{Te}_4$  respectively. It was found that after approximately 700 K, the line quality became too diffuse to make any accurate measurement of the Bragg angle of these lines, so that the line measurements were made up to about 700 K. In every case, no splitting of the (220/204), (312/116), (400/008), etc. structure lines were observed in the X-ray diffraction patterns, indicating that for both compounds the ratio  $c/a$  is very close to 2. The values of the lattice parameter  $a$  were determined at each temperature as

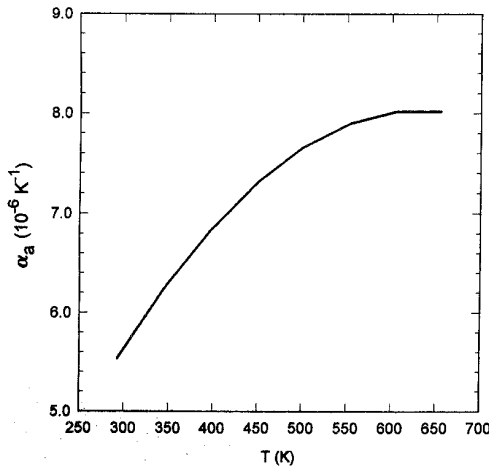


Fig. 3. Variation of the thermal coefficients  $\alpha_a$  with temperature  $T$  for CdIn<sub>2</sub>Te<sub>4</sub>.

indicated above, and at 293 K (for CdIn<sub>2</sub>Te<sub>4</sub>) and 320 K (for MnIn<sub>2</sub>Te<sub>4</sub>) these values were  $a = 6.199$  and  $6.198 \text{ \AA}$ , and a standard deviation of the experimental points of  $\sigma = \pm 0.001 \text{ \AA}$  were obtained for both materials. These results are in very good agreement with those reported in earlier works [5]. The variations of the crystallographic parameter  $a$  with temperature, for CdIn<sub>2</sub>Te<sub>4</sub> and MnIn<sub>2</sub>Te<sub>4</sub>, are plotted in Figs 1 and 2, respectively. In Fig. 2, the values reported by Doll *et al.* [7] in the temperature range between 90 K and 300 K, are also shown as solid circles, and it is observed that these values matched very well with the present results. It is seen from Figs 1 and 2 that the values of  $a$  increase nonlinearly with the temperature. It is found that, within the limits of experimental errors, in each case the variation of  $a$  can be very well fitted to a relation of the form  $p = A + BT + CT^2 + DT^3$ , and the equations obtained are:

$$a(T) = 6.1941 - 4.0290 \times 10^{-6}T + 8.5193 \times 10^{-8}T^2 - 4.4941 \times 10^{-11}T^3 (\text{\AA}) \quad (1)$$

$$a(T) = 6.1929 - 7.8782 \times 10^{-6}T + 1.0176 \times 10^{-7}T^2 - 5.6667 \times 10^{-11}T^3 (\text{\AA}), \quad (2)$$

the standard deviations of the fitted points being  $\pm 0.0007 \text{ \AA}$  and  $\pm 0.001 \text{ \AA}$  for CdIn<sub>2</sub>Te<sub>4</sub> and MnIn<sub>2</sub>Te<sub>4</sub> respectively, the resulting curves are shown in Figs 1 and 2 as solid lines.

The thermal expansion coefficient  $\alpha_p$  of a given crystalline parameter  $p$  can be determined by using the relation given by:

$$\alpha_p(T) = (1/p_0) dp/dT \quad (3)$$

where  $p_0$  is the value of the parameter  $p$  at a given temperature. Then, the expressions for the parameter

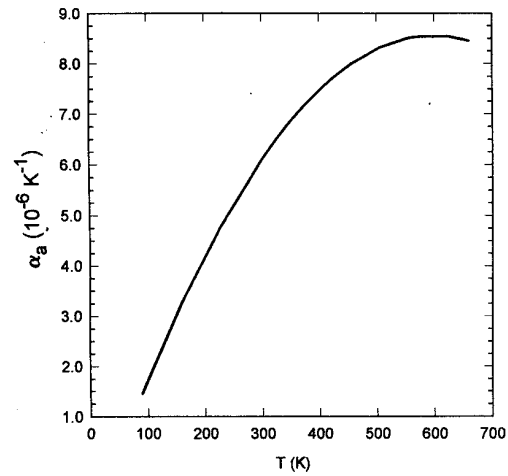


Fig. 4. Variation of the thermal coefficients  $\alpha_a$  with temperature  $T$  for MnIn<sub>2</sub>Te<sub>4</sub>.

$a$ , obtained by using relation (3) with eqns (1) and (2) and by taking  $p_0$  as the values of the lattice parameter  $a$  at 293 and 90 K for CdIn<sub>2</sub>Te<sub>4</sub> and MnIn<sub>2</sub>Te<sub>4</sub> respectively, are:

$$\alpha_a(T) = -6.4993 \times 10^{-7} + 2.7486 \times 10^{-8}T - 2.1749 \times 10^{-11}T^2 (\text{K}^{-1}) \quad (5)$$

$$\alpha_a(T) = -1.2721 \times 10^{-6} + 3.2865 \times 10^{-8}T - 2.7451 \times 10^{-11}T^2 (\text{K}^{-1}) \quad (6)$$

for CdIn<sub>2</sub>Te<sub>4</sub> and MnIn<sub>2</sub>Te<sub>4</sub> respectively. The corresponding results are shown plotted in Figs 3 and 4. It is observed from these figures that, in each case, the values of  $\alpha_a$  increase with temperature until about 550 K, where these values level out. This behavior of  $\alpha_a$  is to be expected because the anharmonic effects, which are considered to be responsible for the thermal expansion of the material, should increase as the temperature is increased.

#### OPTICAL ABSORPTION RESULTS AND ANALYSIS

Optical absorption measurements in the range 20–300 K were made, on each compound, as indicated above. Typical sets of  $(\alpha h\nu)^2$  vs  $h\nu$  curves at various temperatures are shown in Figs 5 and 6 for CdIn<sub>2</sub>Te<sub>4</sub> and MnIn<sub>2</sub>Te<sub>4</sub>, respectively. The values of  $E_g$ , obtained from the extrapolations of  $(\alpha h\nu)^2 = 0$ , as a function of temperature  $T$  for each of the compounds are shown in Figs 7 and 8. The values of  $E_g(T)$  for the MnIn<sub>2</sub>Te<sub>4</sub> are in good agreement with those obtained by Medvedkin *et al.* [8]. It can be seen in Fig. 8, i.e. for MnIn<sub>2</sub>Te<sub>4</sub>, that no magnetic effects are expected to be important near to a magnetic transition of a material,

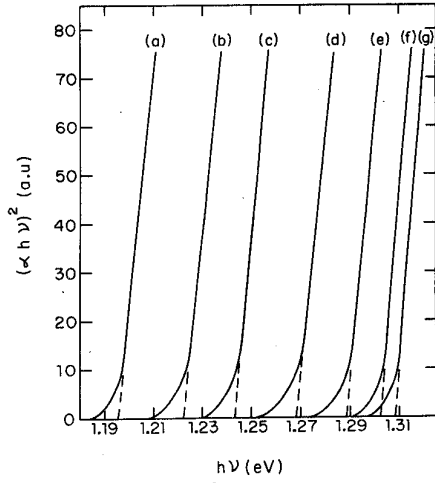


Fig. 5. Variation of  $(\alpha h\nu)^2$  with photon energy  $h\nu$  for  $\text{CdIn}_2\text{Te}_4$ , at the temperatures (a) 300 K, (b) 250 K, (c) 200 K, (d) 150 K, (e) 100 K, (f) 50 K and (g) 20 K.

on the energy gap  $E_g$  [10] are observed at low temperature, although these are expected to be important near to the magnetic transition of a material. Values of  $E_g(T)$  for the compound  $\text{CdIn}_2\text{Te}_4$  were not found in the literature.

It has been shown for a range of semiconductor compounds and alloys that the  $E_g$  vs  $T$  curves can be well fitted by a simple Manooogian–Leclerc equation of the form [11, 12]

$$E_g(0) - E_g(T) = UT^x + V\theta(\coth \theta/2T - 1) \quad (7)$$

where  $E_g(0)$  is the energy gap at  $T = 0$  K, and the parameters  $U$ ,  $V$ ,  $\theta$  and  $x$  are constant and independent of temperature. The first term in eqn (7) represents the effect of the lattice dilation and the second term represents the electron–phonon interaction.  $\theta$

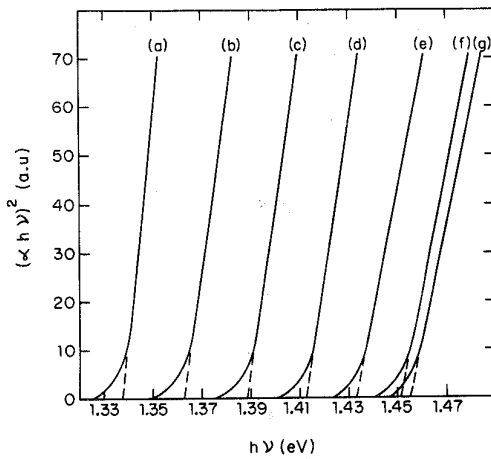


Fig. 6. Variation of  $(\alpha h\nu)^2$  with photon energy  $h\nu$  for  $\text{MnIn}_2\text{Te}_4$ , at the temperatures (a) 300 K, (b) 250 K, (c) 200 K, (d) 150 K, (e) 100 K, (f) 50 K and (g) 20 K.

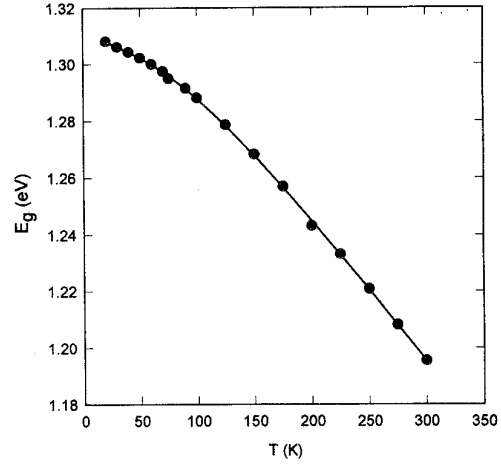


Fig. 7. Variation of optical energy gap  $E_g$  with temperature  $T$  for  $\text{CdIn}_2\text{Te}_4$ . Full circles: experimental data; full curve: curve fitted to Manooogian–Leclerc equation.

correspond to the mean frequency of the entire phonon spectrum, and its value is of the order of the Debye temperature  $\theta_D$  [11].

When fitting eqn (7) to the  $E_g$  vs  $T$  experimental data, in previous work (e.g. Refs [12] and [13]) it was assumed that  $x = 2/3$ . However, in the analysis of the optical energy gap data for  $\text{CuInTe}_2$  [14], it was found that a value of  $x = 1$  was necessary to use in order to obtain agreement between the values obtained from the variation of  $E_g$  vs  $T$  and  $E_g$  vs pressure. Hence, in the present analysis values of  $x = 1$  and  $2/3$  have been assumed, and then the parameters  $U$ ,  $V$ ,  $E_g(0)$  and  $\theta$  treated, in each case, as unknown parameters to be determined from the fitting procedure, the final criterion for the overall best fit being the minimum standard deviation. When this was done, it was found that the value of  $x = 1$  gave the best fit to the experimental data, and when  $x = 2/3$  is used negative values of  $U$  are obtained. The final values for the various parameters obtained from fitting to the data in Figs 7 and 8 are listed in Table 1 and the resulting fitted curves for  $x = 1$  are shown in Figs 7 and 8.

Another point of interest here is to estimate values for the deformation potentials of the valence and conduction bands,  $C_h$  and  $C_e$ , respectively. These parameters can be related to the thermal coefficients of the energy gap. Thus, for the lattice dilation contribution [15],

$$(dE_g/dT)_1 = 2\alpha_L(C_e + C_h) \quad (8)$$

while for the electron–phonon interaction [16],

$$(dE_g/dT)_2 = - (8/9\pi)(3/4\pi)^{1/3} (K_B\Omega^{2/3}/\hbar^2 Mv^2) \times (m_e C_e^2 + m_h C_h^2) \quad (9)$$

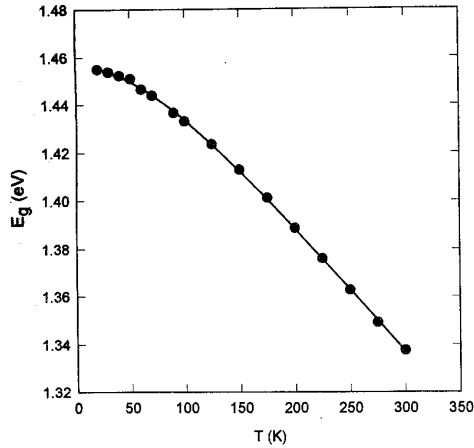


Fig. 8. Variation of optical energy gap  $E_g$  with temperature  $T$  for MnIn<sub>2</sub>Te<sub>4</sub>. Full circles: experimental data; full curve: curve fitted to Manoogian–Leclerc equation.

where  $\alpha_L$  is the average thermal expansion coefficient of the material.  $M$  and  $\Omega$  are, respectively, the mass and volume of the unit cell,  $v$  is the sound velocity in the material,  $m_e$  and  $m_h$  are respectively the electron and hole effective masses. In previous work [12], eqns (8) and (9) have been used to make estimates of the deformation potentials of the CuGa(S<sub>z</sub>Se<sub>1-z</sub>)<sub>2</sub> alloys. Here, it is proposed that a similar analysis can be made with the present  $E_g$  vs  $T$  data using the U and V of the Manoogian–Leclerc relation to give values for the temperature coefficients of  $E_g$ .

As indicated above, in eqn (8) the term in U is the dilation contribution, while the term in V is the electron–phonon contribution. Thus, the dilation term can be used to give  $(dE_g/dT)_1$ , and the electron–phonon term to give  $(dE_g/dT)_2$ ,

$$(dE_g/dT)_1 = -U \quad (10)$$

$$(dE_g/dT)_2 = -V\theta^2/2T^2 \operatorname{cosec}^2(\theta/2T). \quad (11)$$

It is seen from the above equations that  $(dE_g/dT)_2$  varies to some extent with  $T$ , while  $(dE_g/dT)_1$  is independent of  $T$ . Taking the range 150–300 K over which the variation of  $E_g$  vs  $T$  is, within the limits of experimental errors, linear and using the values of  $\theta$ , U and V for  $x = 1$  given in Table 1, the following mean

value for  $(dE_g/dT)_2$  of  $-1.118 \times 10^{-4}$  eV/K and  $-1.0 \times 10^{-4}$  eV/K for, respectively, CdIn<sub>2</sub>Te<sub>4</sub> and MnIn<sub>2</sub>Te<sub>4</sub> are obtained.

In order to use these data to calculate  $C_e$  and  $C_h$ , it is necessary to know values of  $\alpha_L$ ,  $v$ ,  $m_e$  and  $m_h$  in eqns (8) and (9). In the present compounds the ratio  $c/a$  was found to be very close to 2, and this gives  $\alpha_a = \alpha_c$ . Hence  $\alpha_L$ , which for a tetragonal crystal can be taken as  $(2\alpha_a + \alpha_c)/3$ , also gives  $\alpha_L = \alpha_a$ . Thus, in the present work, the average value of  $\alpha_L$  between 300 and 500 K of, respectively,  $6.60 \times 10^{-6}$  K<sup>-1</sup> and  $7.21 \times 10^{-6}$  K<sup>-1</sup> for CdIn<sub>2</sub>Te<sub>4</sub> and MnIn<sub>2</sub>Te<sub>4</sub>, given by eqns (3) and (4), are used. The values of  $v$  can be estimated from the Debye temperature  $\theta_D$  by using the well-known relation  $v = (K_B\theta_D/\hbar)\Omega^{1/3}/6\pi^2$ , where  $\Omega$  is the mean volume per atom. As values of  $\theta_D$  are not given in the literature for the compounds, the values of  $\theta$  obtained by the Manoogian–Leclerc equation, which are of the order of  $\theta_D$ , are used to estimate values of the second velocity  $v$  in the compounds. No values are available for  $m_e$  and  $m_h$  in the literature. Thus, these values are assumed to be the same ones as for the CuInTe<sub>2</sub> [14, 17] ( $m_e/m_h = 0.078$  and  $m_h/m_o = 0.78$ ), for both compounds, which is a reasonable approximation.

Using the above values, eqns (8) and (9) can be solved for  $C_e$  and  $C_h$ . When this is done, two solutions are possible for each compound. For CdIn<sub>2</sub>Te<sub>4</sub> either  $C_e = -25.89$  and  $C_h = 17.42$  or  $C_e = 10.49$  and  $C_h = -18.96$ , while for MnIn<sub>2</sub>Te<sub>4</sub>  $C_e = -22.01$  and  $C_h = 15.10$  or  $C_e = 9.45$  and  $C_h = -16.36$  (all in units of eV/unit dilation) are found. In all cases,  $C_e$  and  $C_h$  have opposite sign, indicating that under any given stress the conduction band minimum and the valence band maximum move in the same direction on compression. In previous work [12], it has been indicated that  $|C_e| > |C_h|$ , and so the first solution is taken here, i.e.,  $C_e = -25.89$  eV/unit dilation and  $C_h = 17.42$  eV/unit dilation for CdIn<sub>2</sub>Te<sub>4</sub>, and  $C_e = -22.01$  eV/unit dilation and  $C_h = 15.10$  eV/unit dilation for MnIn<sub>2</sub>Te<sub>4</sub>. The deformation potential of the energy gap,  $a_g$ , of a material is given by  $C_e + C_h$ . The resulting values of  $a_g$ , of 8.47 and 6.90 eV/unit dilation for the CdIn<sub>2</sub>Te<sub>4</sub> and MnIn<sub>2</sub>Te<sub>4</sub> respectively, are in the range of the values obtained for the I–III–VI<sub>2</sub> [14, 18–20] and II–IV–V<sub>2</sub> [21] chalcopyrite compounds and alloys.

Table 1. The fitted values of  $x$ ,  $\theta$ ,  $E_g(0)$ , U and V.  $\sigma$  is the standard deviation of the experimental points

Compound	$x$	$\theta$ (K)	$E_g(0)$ (eV)	U ( $\times 10^{-4}$ eV/K)	V ( $\times 10^{-4}$ eV/K)	$\sigma$ ( $\times 10^{-3}$ eV)
CdIn <sub>2</sub> Te <sub>4</sub>	1.0	310.9	1.312	1.892	1.746	2.08
	2/3	100.0	1.297	-12.12	3.057	5.15
MnIn <sub>2</sub> Te <sub>4</sub>	1.0	274.7	1.459	1.872	1.805	2.46
	2/3	100.0	1.445	-11.72	3.158	4.05

## CONCLUSIONS

The results showed that the values of the lattice parameter  $a$  increase nonlinearly with temperature and that the variation of  $a$ , for each compound, can be very well fitted to a relation of the form  $p = A + BT + CT^2 + DT^3$ . The behaviour of the thermal coefficient  $\alpha_a$  with  $T$ , for both compounds, is found to be typical of a semiconducting material. It is shown that a good fit of  $E_g$  against  $T$  data can be obtained using the Manoogian–Leclerc equation with the parameter  $x$  taken as unity. The resulting fitted parameters can be used to determine values of the thermal coefficients  $(dE_g/dT)_1$  and  $(dE_g/dT)_2$ , and hence values of  $C_e$ ,  $C_h$  and  $a_g$  estimated. In addition, when  $x$  is taken as unity in the above analysis, the value of  $(dE_g/dT)_1$  is found to be independent of temperature, which is to be expected in the range where  $E_g$  varies linearly with  $T$ . The estimated values of the deformation potential of the energy gap,  $a_g$ , for the compounds, are found to lie in the range of the reported values for the I–III–VI<sub>2</sub> and II–IV–V<sub>2</sub> chalcopyrite materials. It is to be pointed out that using values of  $\alpha_L$ , taken in the temperature range of the present measurements, the values of  $C_e$  and  $a_g$  change by only 0.014% eV/unit dilation, while the value of  $C_h$  changes by 0.002% eV/unit dilation. Hence, the main source of error in the values of  $C_e$  and  $C_h$  is probably due to uncertainty in the values of  $v$ ,  $m_e$  and  $m_h$ .

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