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Transport measurements in InSe under high pressure and high temperature: shallow-to-deep donor transformation of Sn related donor impurities

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Abstract
We have investigated the temperature dependence of the transport parameters of Sn-doped InSe at different pressures, up to 2.5 GPa. A noticeable change in the temperature dependence of all the transport parameters has been observed above 1.2 GPa. This fact is explained by assuming the transformation of Sn shallow donors into deep donors at a hydrostatic pressure of 1.1 GPa, and by taking into account the transfer of electrons from the absolute minimum to higher energy minima in the conduction band. At ambient pressure, the position of the Sn deep level is estimated to lie 75 ± 20 meV above the absolute conduction-band minimum.

1. Introduction

Room temperature (RT) Hall effect measurements in n-type indium selenide (InSe) doped with tin (Sn) showed that the electron concentration abruptly decreases as the pressure (P) increases above 1 GPa, this decrease being more pronounced for samples with higher Sn concentrations [1, 2]. The observed behaviour is reversible with pressure, so it cannot be attributed to permanent structural defects created by the high pressure. On Sn-doped GaAs a qualitatively similar behaviour has been observed and explained through the existence of a deep donor level, which traps electrons as the band structure is modified under compression [3, 4]. Such a highly localized state is also found in many n-type semiconductor compounds [3–7]. In GaAs, this deep level lies above the Γ minimum of the conduction-band and roughly moves with the L minima under pressure. In this way, above a critical pressure, it is pushed below the Γ minimum and therefore the trapping of free electrons increases with increasing pressure.

In spite of the crystalline-layered structure of InSe, its band structure has some features in common with that of GaAs. In the most usual polytype of InSe (γ-polytype) the triply-degenerated B conduction-band minima are located about 325 meV above the lowest one (Z minimum) at ambient pressure [8–11]. Upon the application of hydrostatic pressure, the energy difference between the subsidiary minima and the absolute minimum decreases [8, 11, 12] as happens in GaAs [13]. Then, the decrease of the electron concentration under compression observed in Sn-doped InSe is consistent with the assumption that there exists a deep donor level which shifts towards lower energies, thus entering the band gap and trapping electrons as it approaches the Fermi level. In fact, the RT pressure dependence of the electron concentration has been qualitatively reproduced by assuming this hypothesis in [1].

In this work we report on the temperature (T) dependence of the transport parameters of Sn-doped InSe at different pressures, up to 2.5 GPa. The present results provide supplementary evidence that Sn donors in InSe

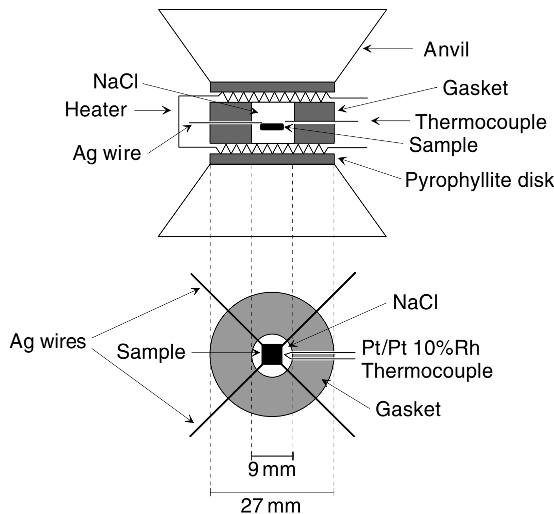

Figure 1. Schematic drawing of the pressure cell used to carry out the Hall measurements.

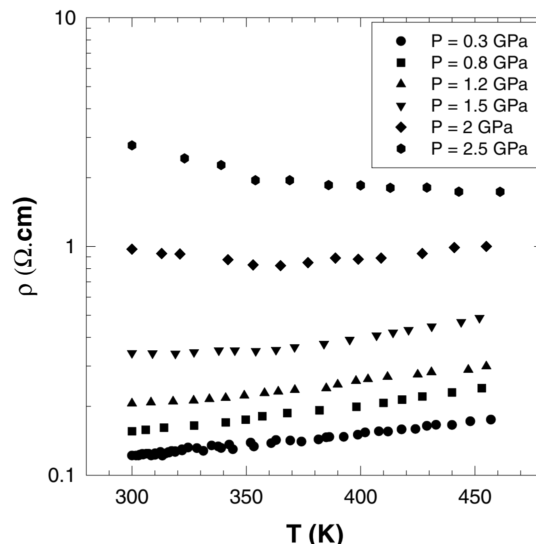

Figure 2. Resistivity as a function of temperature at different pressures.

present simultaneously at least one shallow and one deep state, suggesting that the shallow-to-deep donor level transformation might be a common phenomenon also in III–VI semiconductors.

2. Experimental details

2.1. Material and measurements

The present Hall effect measurements under variable pressure and temperature conditions were carried out using a Bridgman cell. To perform the study, square samples, with faces perpendicular to *c* axis, were cleaved from InSe monocrystals, which were grown by the Bridgman method [14]. Sn impurities were introduced as SnSe in the polycrystalline melt. Due to the high impurity segregation effect in InSe [14], starting concentrations up to 10% of Sn in the polycrystalline melt are needed in order to get free carrier concentrations above 10^{17} cm^{-3} in the Bridgman grown monocrystalline ingot. Samples were typically 50 μm thick and 4×4 mm^2 in size. Indium contacts were vacuum evaporated on the corners of the samples in the van der Paw configuration [15] (see figure 1) taking care that the contact size was always much smaller than the distance between the contacts. Silver (Ag) wires of 100 μm in diameter were used as electrical leads, being soldered with high-purity indium to the evaporated indium electrodes. To avoid inaccuracy problems in the determination of the Hall coefficient (R_H) coming from offset voltages, we acquired two sets of Hall measurements, one for positive and one for negative magnetic field (0.6 T) directions. The linearity of the ohmic voltages on the injected current was checked out at different pressures and temperatures.

2.2. High-pressure cell and heating arrangements

In our device, a 150 ton oil press is used to apply a load in two opposing non-magnetic tungsten carbide (WC) anvils. This system facilitates a large volume sample and allows for the application of an external magnetic field, which makes it quite convenient to perform Hall effect studies. The high-pressure cell is placed between the anvil flats. It consists of two pyrophyllite gaskets of optimized thickness (0.5 mm each), in split gasket geometry [16, 17]. The initial internal diameter of the gaskets was 9 mm and the anvil diameter 27 mm. They were thermally treated to get suitable mechanical properties and avoid breakings in the first stages of the compression [18]. The sample was inserted inside the gasket between two sodium chloride (NaCl) discs used as a pressure-transmitting medium. The diameter of these discs was closely adjusted to the internal gasket diameter and their thickness was about three quarters of that of the gaskets to ensure the best stability of the gasket. The sample pressure was determined by the calibration of the load applied to the anvils against known fixed points [17, 18].

The pressure cell was externally heated by including a heater between the anvil flats and the gasket. The heater consisted of two resistances of nichrome glued over two phyrophyllite discs. Due to its low electrical and thermal conductivities, phyrophyllite acted as thermal and electrical isolator between the nichrome wire and the anvils. As the sample is located just between the two discs a good thermal efficiency is ensured. A 200 μm diameter thick Pt/Pt–10%Rh thermocouple (type S) was used to measure the temperature. Since the absolute effect of pressure in the emf of this thermocouple introduced a correction smaller than 3% [19], we have neglected it. The details of the whole set-up are schematically shown in figure 1.

3. Results

Figure 2 shows the temperature dependence of the resistivity (ρ) at several different pressures. One can clearly observe that ρ increases with T up to 1.5 GPa. In contrast, at higher pressures, the temperature behaviour of ρ changes. At $P = 2$ GPa, ρ decreases up to 360 K, but for higher temperatures it increases, and at $P = 2.5$ GPa, ρ decreases with T in all the studied range of temperatures. These

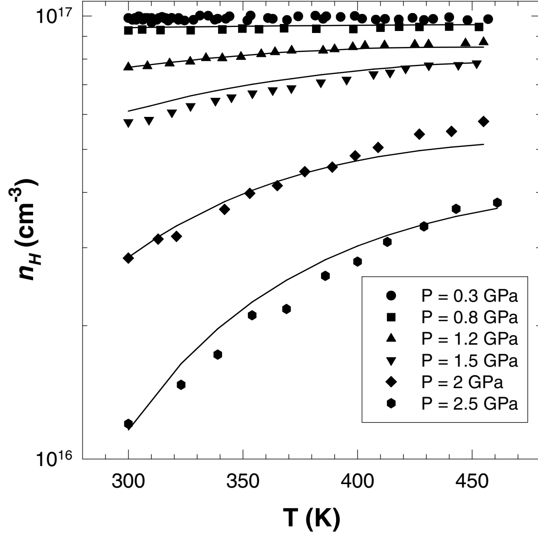

Figure 3. Hall concentration as a function of temperature at different pressures. Symbols correspond to the experimental results and solids lines to the fits with equation (7).

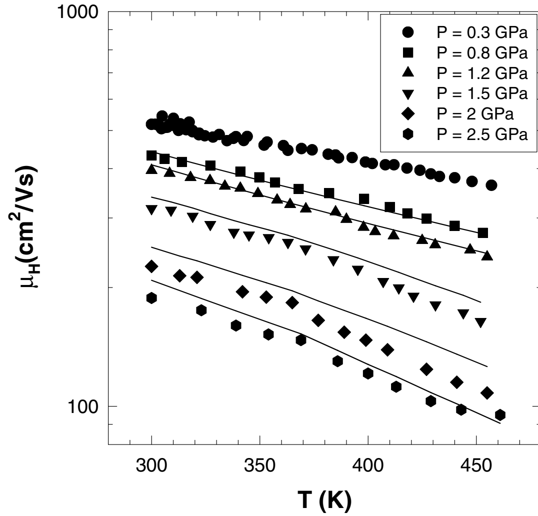

Figure 4. Hall mobility as a function of temperature at different pressures. Symbols correspond to the experimental results and solids lines to the fits with equation (8).

different behaviours of ρ are basically related to the different temperature dependences of the Hall electron concentration ($n_H = 1/eR_H$, where e is the electron charge). Figure 3 illustrates the temperature dependence of this parameter. In this figure, it can be seen that at low pressures n_H is nearly constant. However, for $P > 0.8$ GPa, the apparent electron concentration increases with temperature, this increase being higher at higher pressures.

Regarding the Hall mobility (μ_H), figure 4 shows that this parameter is a decreasing function of T at all the pressures studied. In this way, for $P < 1.2$ GPa, when n_H is constant or increases slowly, the decrease of μ_H produces the observed increase of ρ. In contrast, for $P > 1.2$ GPa, n_H increases rapidly, compensating first the decrease of μ_H, and determining then the observed decrease of ρ at $P = 2.5$ GPa.

4. Discussion

It is well known that a donor impurity, besides giving rise to a hydrogen-like level, associated with the normal site configuration (e.g. Sn in In site in the present case), can also form a deep donor level, which in some cases arises from a lattice distortion near the donor [13]. On the other hand, it is also clear at present that a deep level can be due to an isolated substitutional donor, which undergoes a shallow-to-deep transition under certain conditions of hydrostatic pressure [20, 21]. Based on this, we have analysed the temperature dependence on n_H, shown in figure 3, by assuming that any Sn impurity introduces a shallow level, which under pressure could change its character. In order to do this, we have postulated that the charge state of the localized donor level is neutral. In addition, as the conduction-band minima at the B point of the Brillouin zone are closer in energy to the minimum at Z than the subsidiary minima at A to the minimum at Z [8], and shift to lower energies with a larger pressure coefficient than those at A [8], we have considered that redistribution of electrons occurs only between Z and B minima. Under these hypotheses the following electroneutrality equation must be satisfied:

$$n_Z + n_B + N_a = N_d + \frac{N_T}{1 + 2\exp[(E_F - E_T)/K_B T]} \qquad (1)$$

where N_d is the shallow-donor concentration, N_a is the compensation acceptor concentration, N_T is the deep impurity level concentration, K_B is the Boltzmann constant and E_F and E_T are the Fermi level and deep impurity level energies, respectively. In equation (1) n_Z and n_B are given by

$$n_Z = N_{CZ}\exp[(E_F - E_Z)/K_B T] \qquad (2)$$

and

$$n_B = 3\left(\frac{m_B^*}{m_Z^*}\right)^{3/2} n_Z \exp[(E_Z - E_B)/K_B T] \qquad (3)$$

respectively, where $n_{Z(B)}$ and $m^*_{Z(B)}$ are the electron concentrations and effective masses of the density of states in the Z and B minima of the conduction band, respectively, and $N_{CZ} = 4.89 \times 10^{15} T^{3/2}(m_Z^*/m_0)^{3/2}$ cm^{-3} is the density of states of the conduction band, m_0 being the free electron mass.

From equations (1)–(3) one readily obtains

$$n_Z = \frac{N_{CZ}\alpha}{4}\left(1 - 2\frac{N_d - N_a}{N_C\alpha}\right) \times \left[\sqrt{1 + 8\frac{N_T + N_d - N_a}{N_C\alpha}\left(1 - 2\frac{N_d - N_a}{N_C\alpha}\right)^{-2}} - 1\right] \qquad (4)$$

with

$$N_C = N_{CZ}\left[1 + 3\left(\frac{m_B^*}{m_Z^*}\right)^{3/2}\exp[(E_Z - E_B)/K_B T]\right] \qquad (5)$$

and

$$\alpha = \exp[(E_T - E_Z)/K_B T]. \qquad (6)$$

Equation (4) describes the pressure and temperature dependences of the electron concentration on the Z minimum. Once $n_Z(P, T)$ is known, it is straightforward to know

Table 1. Calculated values for μ_Z at RT as a function of pressure, estimated effective mass of the density of states at the *Z* point as a function of pressure and best fitting parameters at different pressures.

P (GPa)	μ_Z (cm^2 V^{-1} s^{-1})	m_Z^*/m_0	m_B^*/m_0	N_d/N_T	E_T–E_Z (meV)
0.8	460	0.1165	0.85	1.5	5
1.2	440	0.1171	0.85	1	−25
1.5	415	0.1175	0.85	0.66	−50
2.0	360	0.1185	0.85	0.43	−95
2.5	290	0.1196	0.85	0.25	−140

$n_B(P, T)$. Then, for a full analysis of the present results, it is reasonable to consider that the electrons located in both minima (*Z* and *B*) of the conduction band are involved in the charge transport process. According to this assumption, the Hall electron concentration and the Hall mobility are given by [22, 23]:

$$n_H = \frac{(n_B\mu_B + n_Z\mu_Z)^2}{n_B\mu_B^2 + n_Z\mu_Z^2} \tag{7}$$

and

$$\mu_H = \frac{n_B\mu_B^2 + n_Z\mu_Z^2}{n_B\mu_B + n_Z\mu_Z} \tag{8}$$

where μ_Z and μ_B represent the mobility of the electrons located in the *Z* and *B* minima, respectively.

The temperature and pressure dependences of μ_Z can be accounted for, by including LO polar phonon, homopolar phonon and ionized impurity scattering mechanisms [1, 24]. On the other hand, from our measurements it can be deduced that at low pressures (where as we will show later $\mu_H \approx \mu_Z$), μ_Z can be expressed as $\mu_Z \propto T^{-\gamma}$ where $\gamma = 1.15$. This dependence is very similar to that observed at ambient pressure in previous works [24] and indicates that the contribution of the different scattering mechanisms does not change substantially with pressure. Therefore, it can be assumed that the temperature dependence of the mobility of the electrons located at the *Z* minimum is not drastically modified upon compression within the pressure range of this study. Hence, we have fitted the experimental results shown in figures 3 and 4 with equations (7) and (8), assuming the following phenomenological expression for μ_Z:

$$\mu_Z(P, T) = \mu_Z(P)\left(\frac{T}{300\,\mathrm{K}}\right)^{-1.15} \tag{9}$$

where $\mu_Z(P)$ gives the pressure dependence of the mobility at 300 K and it has been calculated following [1]. The values obtained for it at different pressures are given in table 1.

The calculation of $\mu_B(P, T)$ is not an easy task because in addition to the usually considered scattering mechanisms in InSe [1, 24] other mechanisms, as intervalley scattering, should be considered [25, 26]. However, intervalley scattering in III–V semiconductors usually leads to a temperature and pressure dependence for the mobility in the subsidiary minima which is not significantly different from that of the absolute minimum [27–29]. Presumably, similar results can be obtained for the temperature and pressure dependences of the mobility of the electrons located at the *B* minima in InSe. Then, in order to reduce the number of unknown parameters in the present model, it looks reasonable to assume that the mobility ratio (μ_B/μ_Z) is essentially independent of the temperature and the pressure within the range of the present study (300 K < *T* < 460 K and *P* < 2.5 GPa). It should be stressed that this assumption is an oversimplification, but as we will see later it leads to results consistent with it. The value of μ_B at ambient conditions can be estimated from the observed value of μ_H at pressures where the majority of the electrons occupy the *B* minima (i.e. $\mu_H \approx \mu_B$ following equation (8)). As will be clear afterwards at 4 GPa most of the electrons are transferred from the *Z* minimum to the *B* minima and therefore, we estimated from the results of [1] that $\mu_B = 150$ cm^2 V^{-1} s^{-1} at ambient conditions. Thus, according to our approximation, $\mu_B/\mu_Z \approx 1/3$, which is similar to the value of μ_L/μ_Γ in III–V semiconductors [30–32].

In order to fit the *P*–*T* behaviour of n_H and μ_H, the value and the pressure dependences of most of the parameters involved in the different equations (4–9) can be obtained from the literature [1, 8, 10, 11, 33–35] with the exception of m_B^*, N_d/N_T and $E_T - E_Z$, which were considered as fitting parameters. In particular, we calculated the pressure dependence of m_Z^* within the framework of the $\mathbf{k}\cdot\mathbf{p}$ theory [36] by considering that its pressure variation is proportional to that of the direct band gap. The values of m_Z^* obtained at different pressures following this approximation are given in table 1. The fits obtained by considering $E_B - E_Z = 325\,\mathrm{meV} - 76P$ (meV GPa^{-1}) [8], $N_a/N_d = 0.6$ and $N_d + N_T = 1.8 \times 10^{17}$ cm^{-3} are shown in figures 3 and 4. It can be seen that the present model reproduces well the experimental results at every pressure for very reasonable values of the fitting parameters (see table 1) in spite of its simplicity.

From the analysis of the obtained fitting parameters at each pressure reported in table 1, it can be deduced that, up to 1.2 GPa, most of the Sn impurities have associated shallow levels. However, when the sample is subjected to a hydrostatic pressure of 1.2 GPa or above, most of the Sn impurities form deep donor states, which trap electrons and produce the marked decrease of the electron concentration previously observed [1]. Note that this is just the pressure at which the deep level is expected to be located below the *Z* minimum. On the other hand, a least-squares fit to $E_T - E_Z$ with a linear function gives

$$E_T - E_Z = 75(20)\,\mathrm{meV} - 85(15)P(\mathrm{meV\ GPa^{-1}}) \tag{10}$$

where *P* is in GPa. From this result, it can be estimated that the deep level is located about 250 ± 35 meV below the *B* minima at ambient pressure. Furthermore, it can be also deduced that the deep level enters into the band gap at 0.9 ± 0.35 GPa, which confirms our previous estimates [1]. Based upon this fact together with the observed change of N_d/N_T, and knowing that the ionization energy of the shallow donor is 17.4 meV [37], we have established that the configuration of the donor states changes at about 1.1 GPa. Nevertheless, some of the donors seem to remain in their shallow donor configuration up to higher pressures, as inferred from table 1. We think that the present disagreement probably indicates that there is a range of pressures in which both donor configurations coexist.

On the other hand, the obtained pressure coefficient of the energy difference between the *Z* minimum of the conduction band and the deep level ($\partial(E_T - E_Z)/\partial P = -85 \pm 15$ meV GPa^{-1}) is very similar to that of the energy difference between the *Z* minimum and the *B* minima

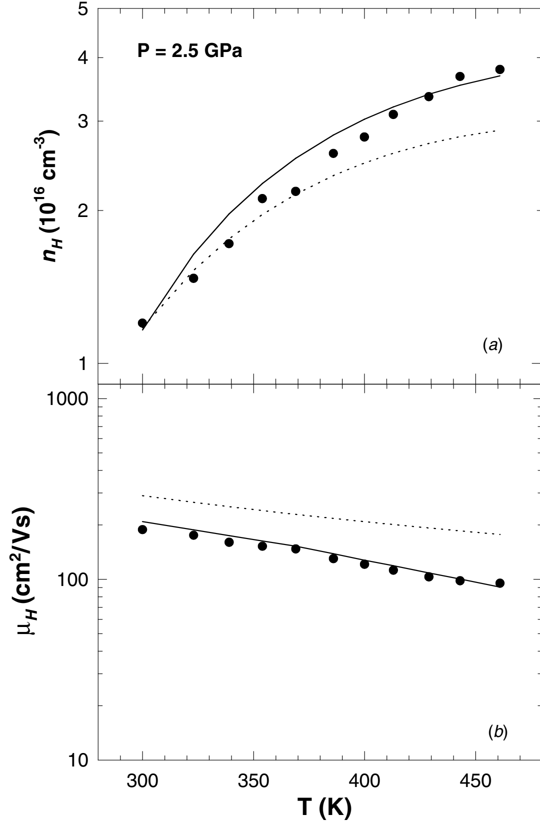

Figure 5. (*a*) Calculated temperature dependence of the Hall concentration at $P = 2.5$ GPa. (*b*) Calculated temperature dependence of the Hall mobility at $P = 2.5$ GPa. The solid lines draw the results obtained following the present model, the dashed lines draw the results obtained considering N_d/N_T as pressure independent and equal to 1 as in [1] and the circles represent our experimental results.

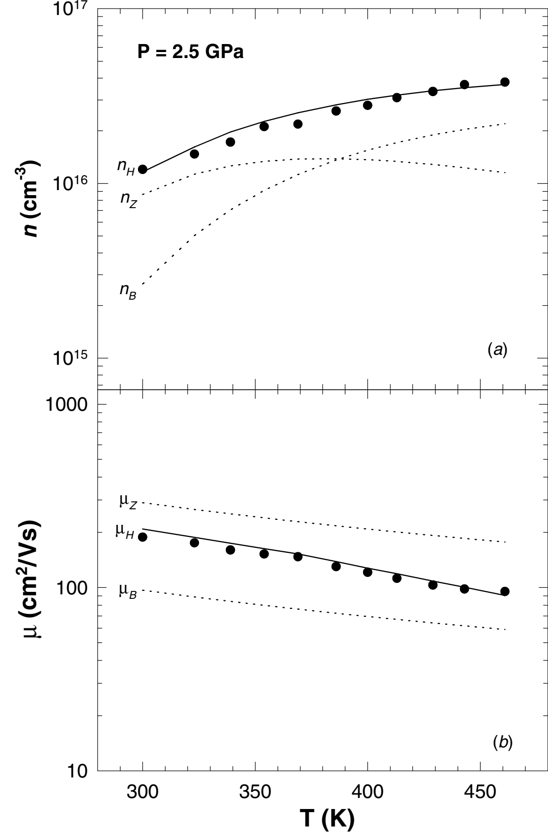

Figure 6. (*a*) Points: experimental behaviour of n_H as a function of temperature at $P = 2.5$ GPa. Solid line: fitted behaviour of n_H with equation (7). Dashed lines: calculated n_Z and n_B using equations (3) and (4). (*b*) Points: experimental behaviour of μ_H as a function of temperature at $P = 2.5$ GPa. Solid line: fitted behaviour of μ_H using equation (8). Dashed lines: calculated μ_Z using equation (9) and calculated μ_B assuming $\mu_B/\mu_Z \approx 1/3$.

($\partial(E_B - E_Z)/\partial P = -76 \pm 10$ meV GPa^{-1}) [7]. This result was expected since the pressure dependence of a deep donor level is mainly determined by the pressure dependence of the nearest subsidiary minima at the conduction band. For example, in GaAs the deep level moves together with the L minima, which are the closest subsidiary minima. From the room temperature studies of [1] the pressure coefficient of the deep donor level was estimated to be $\partial(E_T - E_Z)/\partial P = -98 \pm 20$ meV GPa^{-1}, but in that work [1] it was considered that only the electrons in the Z minimum were involved in the charge transport process. That way, the pressure coefficient of the deep donor lever could be overestimated in order to explain the decrease of the apparent electronic concentration under pressure.

For comparison, the results obtained at $P = 2.5$ GPa for n_H and μ_H excluding the possibility of the electron transfer from the Z minimum to the B minima and considering $N_d = N_T$ as in [1] are shown in figure 5 together with the results obtained according to the present model. This figure shows that even when the model of deep states proposed in [1] allows us to fairly estimate the pressure behaviour of the deep level energy at RT, it does not allow us to reproduce accurately the temperature dependence of n_H and gives a high value for μ_H. These facts show the necessity of including a new fitting parameter, N_d/N_T, that accounts for the possibility of a shallow-to-deep transformation and of considering the effects of the electron transfer from the absolute minimum to the subsidiary minima of the conduction band in order to explain the present experimental results.

Regarding the effective mass of the density of states at the B point, the value obtained for m_B^* ($0.85m_0$) is around 7 times the value of m_Z^* and does not change with pressure within the accuracy provided by our model. This value, which should be considered as a rough estimation of m_B^*, fairly agrees with the value deduced from photoluminescence studies ($m_B^* = (0.75 \pm 0.05)m_0$) [38]. The present estimates of m_B^* are also consistent with previous band-structure calculations [8] that show that the Z minimum is sharper than the secondary minima. On the other hand, the obtained m_B^* values imply that the electrons transferred to the B minima of the conduction band are heavier than those located at the Z minimum and, therefore, are expected to be less mobile, in agreement with our present assumption ($\mu_B/\mu_Z \approx 1/3$). This fact explains the decrease observed in the Hall mobility at high pressures in [1]. At ambient pressure all the electrons are in the Z minimum and thus μ_Z represents the observed mobility. However, at pressures much higher than the direct–indirect crossover pressure, the pressure-induced electron transfer from the Z minimum to the B minima makes the Hall mobility to approach

μ_B, which is reflected in the drop by a factor of 4 in the Hall mobility between 1 GPa and 4 GPa at RT [2].

This pressure-induced change of the electron population of the Z and B minima is also reflected in the temperature dependence observed in our measurements in those samples compressed to 1.5 GPa and beyond. At these pressures, when heating, the trapped electrons can be excited to both minima, since they are close in energy. This, added to the smaller mobility of the electrons excited to the subsidiary minima, induces a change in the temperature dependence of the Hall mobility (γ changes from 1.15 to 1.95). This phenomenon can be clearly understood from figure 6. Figure 6(*a*) shows the variation with temperature of n_H, n_Z and n_B at $P = 2.5$ GPa illustrating the fact that at low temperatures the electrons tend to mostly populate the Z minimum but when the temperature increases they are transferred to the B minima. Figure 6(*b*) shows the temperature dependence of μ_H, μ_Z and μ_B at the same pressure. There, it can be seen that above 380 K the increase of the charge redistribution produces a change in the slope of μ_H. A similar change has also been observed in $Al_xGa_{1-x}As$ compounds after the band crossover [29], which is also related to the smaller mobility of the electrons transferred to the subsidiary minima.

5. Conclusions

In summary, we have observed evidence of the existence of a pressure-induced deep donor level in InSe. The pressure at which the new level becomes more stable than the shallow one is 1.1 GPa. The fact that deep donor levels could be formed in InSe through the application of pressure suggests that the existence of these states may be common not only to many n-type tetrahedrally coordinated semiconductors [3–7], but also to n-type III–VI semiconductors when the subsidiary conduction-band minima lie close to the bottom of the conduction band. Complementary experiments, such as deep levels transient spectroscopy, photocapacity and photoconductivity are needed in order to determine whether the observed localized state has DX-like properties or not.

Acknowledgments

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