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Dependence of the indirect energy gap of GaP on quasi-hydrostatic pressure and phase transition

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Abstract. We have measured the dependence of the absorption coefficient on quasi hydrostatic pressure in GaP up to 23.3 GPa in the region of the indirect gap energy (E_g) at room temperature. We have found a linear behaviour of E_g with pressure up to 17 GPa with a pressure coefficient of -14.9 ± 0.5 meV GPa $^{-1}$. Transmission measurements, electronic diffraction, x-ray diffraction, Raman scattering and photographs under microscope have been used to characterise the phase transition. It begins between 21 and 22 GPa. The transitioned phase, after depressurisation from 32 GPa, contains a mixture of polycrystalline and amorphous GaP.

1. Introduction

The study of the dependence on hydrostatic pressure of energy gaps in semiconductors was the object of considerable interest some 25 years ago. With the advent of the diamond anvil cell to generate pressure, and of the ruby luminescence scale to measure it, it became possible to perform transmission experiments up to very high pressure under nearly hydrostatic conditions. The whole situation is summed up in a recent paper and references therein (Ves *et al* 1985). Gallium phosphide is a III–V semiconductor with the zincblende structure. It has been shown to undergo a transition to a centred tetragonal phase around 22 GPa and the transition has been claimed to be reversible (Hu *et al* 1984, Ves *et al* 1985).

We measured the pressure dependence of the absorption coefficient at room pressure under nearly hydrostatic pressure up to 23.3 GPa. In order to characterise the phase transition and its possible reversibility, GaP was studied as a function of pressure by Raman scattering, optical transmission, microphotographic technique. The phase obtained after a rapid decrease of pressure was studied by electron diffraction, x-ray diffraction and optical transmission.

In a first part, we present the absorption measurements and the analysis of the behaviour of the indirect band gap. The second part is devoted to the analysis of the phase transition.

2. Pressure dependence of the indirect gap (transmission measurements)

2.1. Experimental procedure

Monocrystalline samples, were polished and chemically etched. The thickness of the

samples varied between 7 and 30 μm . We used a diamond anvil cell of Block and Piermarini type with ethanol–methanol as pressure transmitting medium. The pressure was obtained from the wavenumber shift $\Delta\sigma = \sigma(P) - \sigma(0)$ of the ruby R_1 fluorescence peak taking $\Delta\sigma/\Delta P = -7.53 \text{ cm}^{-1} \text{ GPa}^{-1}$. The ethanol–methanol mixture is fluid up to 10.5 GPa, so the pressure is truly hydrostatic up to that pressure. Above, we had to increase the pressure rapidly from low pressure to keep nearly hydrostatic conditions (Polian *et al* 1980). This has been possible up to the phase transition. After each pressure variation, we waited a sufficiently long time for the pressure to stabilise in the experimental volume ($t \geq 30 \text{ mn}$). At 17 GPa, we have measured the pressure from several ruby chips dispersed within the experimental volume. The largest pressure difference measured was 0.5 GPa. Then, the broadening of the ruby luminescence peak corresponds to about 0.6 GPa or 0.8 GPa between 17 and 27 GPa and 1.3 GPa at 30 GPa.

Transmission experiments were performed on a CODERG triple monochromator. The diameter of the experimental chamber (the hole drilled in the inox gasket) was 200 μm and the initial thickness 60 μm . Typical dimensions of the samples were $90 \times 90 \times 20 \mu\text{m}$. The sample was illuminated through a microscope with a field-limiting aperture which always allowed us to select the same region of the sample (about 20 μm) at all pressures to measure the transmitted intensity I . The intensity I_0 of the reference beam was measured on the side of the sample.

2.2. Absorption coefficient

The absorption coefficient α was obtained from the experimental transmission using the expression

$$\alpha = (1/d) \ln \{ [2kT_{\text{exp}} R^2 / \{ -(1-R)^2 + [(1-R)^4 + 4k^2 T_{\text{exp}}^2 R^2]^{1/2} \}] \} \quad (1)$$

where d is the sample thickness, R the reflection coefficient $(n^* - 1)^2 / (n^* + 1)^2$ with $n^* = n_{\text{GaP}} / n_{\text{ethanol-methanol}}$ the refractive index of GaP divided by that of the ethanol–methanol mixture (Welber 1977). We have also taken into account the pressure variation of n^* (Strössner *et al* 1985). T_{exp} is the experimental transmittance and k a corrective coefficient to take into account the defects of the crystals. It was calculated assuming $\alpha = 0$, when the transmittance becomes constant at low energies.

At high pressures, when one could not reach a constant transmittance, we have used the correction obtained from the zero pressure transmittance of the sample which has been subjected to a pressure of 32.5 GPa before quenching down to room pressure. On the high energy side, the stray light ($\approx 10^{-2}$) was subtracted from the transmittance.

At room pressure, the experimental absorption coefficient obtained from (1) has been compared with the absorption coefficient calculated from the formula of Elliott (1957) to discover if our results could be interpreted from it and in this case to have a starting point for the interpretation of high pressure results:

$$\begin{aligned} \alpha = & \sum_{i=1} \frac{a_i}{h\nu(\exp(\hbar\omega_i/kT) - 1)} \{ (h\nu - E_{\text{gx}} + \hbar\omega_i)^{1/2} + \rho_i \exp(\hbar\omega_i/kT) \\ & \times (h\nu - E_{\text{gx}} - \hbar\omega_i)^{1/2} \} + \sum_{i=1}^n \frac{b_i}{h\nu(\exp(\hbar\omega_i/kT) - 1)} \\ & \times \{ (h\nu - E_{\text{g}} + \hbar\omega_i)^2 + \rho_i \exp(\hbar\omega_i/kT)(h\nu - E_{\text{g}} - \hbar\omega_i)^2 \}. \end{aligned}$$

Here, $h\nu$ is the incident photon energy and $\hbar\omega_i$ the energy of the phonons, a_i is a weakly temperature-dependent parameter containing the density-of-states effective masses of electrons and holes and $b_i = Ca_i$ with $C = 43 \text{ eV}^{-3/2}$ (Lorentz *et al* 1968).

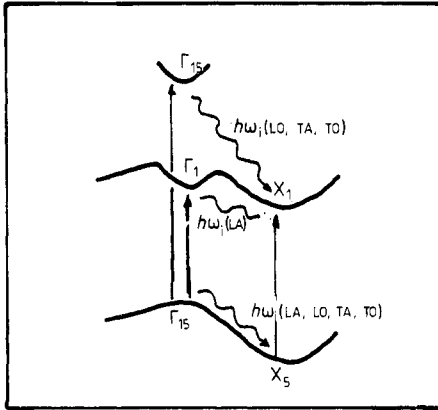


Figure 1. Band structure of GaP (Auvergne *et al* 1975). The energy differences of the various transitions are: $\Gamma_{15}^V \rightarrow \Gamma_1^c \equiv E_0 = 2.76$ eV (Ves *et al* 1985); $\Gamma_{15}^V \rightarrow X_1^c \equiv E_g = 2.261$ eV (Lorentz *et al* 1968); $X_5^V \rightarrow X_1^c \equiv E_2 = 5.3$ eV (Zallen and Paul 1964); $\Gamma_{15}^V \rightarrow \Gamma_{15}^c \equiv E'_0 = 3.71$ eV (Zallen and Paul 1964).

Table 1. Values used to calculate the absorption coefficient at room pressure from Elliott's formula.

Phonons	$\hbar\omega_i$ (meV)	ρ_i	a_i ($\text{cm}^{-1} \text{eV}^{1/2}$)	b_i ($\text{cm}^{-1} \text{eV}^{-1}$)
TA	12.8 ^a	1.0359	39.9 ^a	1715.7
LA	31.3 ^a	1.2850	151.0 ^a	6943.0
TO	46.5 ^a	1.0663	59.5 ^a	2558.5
TA + LO	64.3 ^a	1.1942	11.3 ^a	485.9

^a Dean and Thomas (1966).

E_g is the indirect gap energy which corresponds to the phonon-assisted transition $\Gamma_{15}^V \rightarrow X_1^c$ (figure 1), E_{gx} is equal to E_g minus the binding energy of the free exciton, which is taken to be 0.010 eV (Zallen 1964) and $\rho_i = (\Delta E + \hbar\omega_i)^2 / (\Delta E - \hbar\omega_i)^2$ with ΔE being the energy difference between the principal virtual intermediate state and the final state.

To evaluate ρ_i we supposed that the edge-of-zone longitudinal acoustical LA(X) phonon has the dominant contribution. The band structure (figure 1) indicates two processes in which it can be involved: emission or absorption from Γ_{15}^V via Γ_1^c or Γ_{15}^V via X_5^V . To simplify, we assumed the intermediate state to be represented by the lowest-lying conduction band extremum compatible with selection rules. So we have chosen $\Delta E = E_0 - E_g = 0.50$ eV for the LA phonon. The other phonons are involved in the transition from Γ_{15}^V via Γ_{15}^c or via X_5^V . In that case the smallest ΔE corresponds to $E'_0 - E_g = 1.45$ eV. We used the values listed in table 1 for the evaluation of α from Elliott's formula (α_{cal}).

2.2.1. Room pressure results. In the evaluation of α_{cal} the phonon-assisted excitonic effects have the same importance at room temperature (even if excitonic structures are not observed at this temperature) as the band-to-band processes assisted by phonon absorption or emission and we have to take into account the contribution of all the phonons to find an absorption coefficient in agreement with the experimental values.

The function $(\alpha_{\text{cal}}\hbar\nu)^{1/2}$ versus $h\nu$ has a quasi linear behaviour in the considered energy range, although it is not obvious in view of the mathematical form of Elliott's (1957) formula. Figure 2 and table 2 show this for α between 70–850 cm^{-1} (the range over which the precision of the DAC measurements is good), a comparison between our

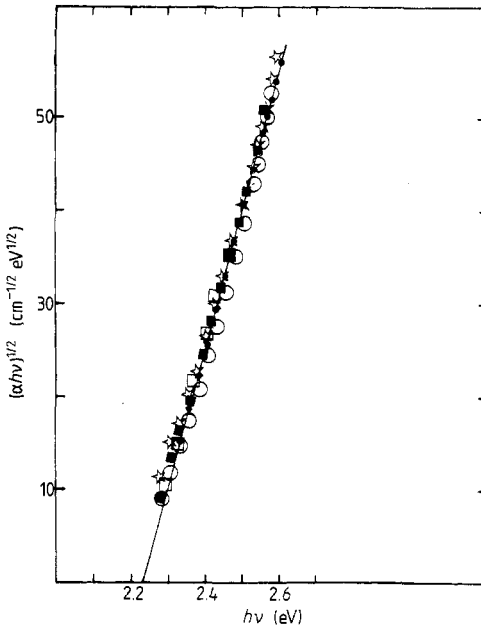


Figure 2. $(\alpha h\nu)^{1/2}$ versus $h\nu$ at room pressure. \circ , sample 1 ($22.7\ \mu\text{m}$); \triangle , sample 2 ($23.1\ \mu\text{m}$); \blacksquare , (sample 3 ($30.6\ \mu\text{m}$)).

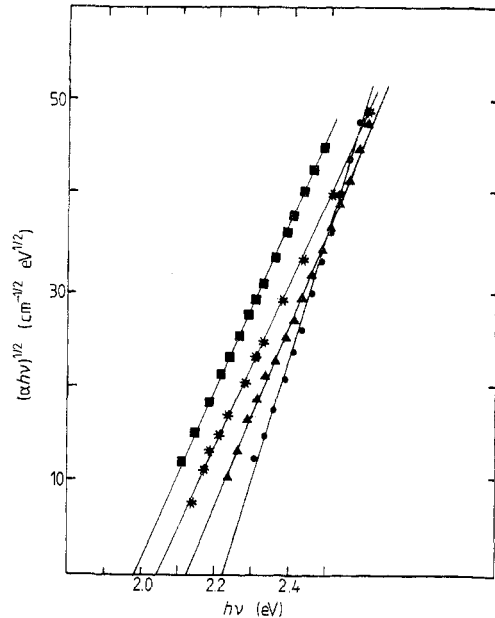


Figure 3. $(\alpha h\nu)^{1/2}$ versus $h\nu$ \bullet , 0.72 GPa; \blacktriangle , 7.44 GPa; $*$, 12.63 GPa; \blacksquare , 16.75 GPa.

Table 2. $(\alpha h\nu)^{1/2} = f(h\nu)$ at room pressure. Comparison between experimental and calculated values.

$h\nu$ (eV)	$(\alpha h\nu)^{1/2}$ ($\text{cm}^{-1/2}\ \text{eV}^{1/2}$)				
	Elliott's formula	Sample 1 ^(a)	Sample 2 ^(b)	Sample 3 ^(c)	Lorentz (1968)
2.331	15.4	14.8	17.1	16.3	16
2.356	18.8	17.5	20.2	19.6	19.8
2.380	22.1	20.8	22.8	23	23.6
2.405	25.7	24.5	26.5	26.6	27
2.43	29.4	27.7	30.0	30.0	31
2.455	33.1	31.3	33.1	33.6	34.6
2.48	36.9	35.1	36.7	37.2	
2.504	40.6	38.8	40.7	40.6	
2.529	44.4	42.8	44.8	44.4	
2.542	46.4	45.1	46.9	46.4	

^a Out of the diamond anvil cell (thickness $22.7\ \mu\text{m}$).

^b In the diamond anvil cell at room pressure (thickness $23.1\ \mu\text{m}$).

^c Out of the diamond anvil cell (thickness $30.6\ \mu\text{m}$).

experimental data, those of Lorentz *et al* (1968) and the values calculated from the complete formula of Elliott.

The extrapolation to $\alpha = 0$ of the straight line $(\alpha h\nu)^{1/2} = f(h\nu)$ corresponds to $E'_g = E_g - \hbar\omega_{\text{LA}} \approx 2.230\ \text{eV}$.

These two last remarks have been used as a starting point for the interpretation of our results under pressure.

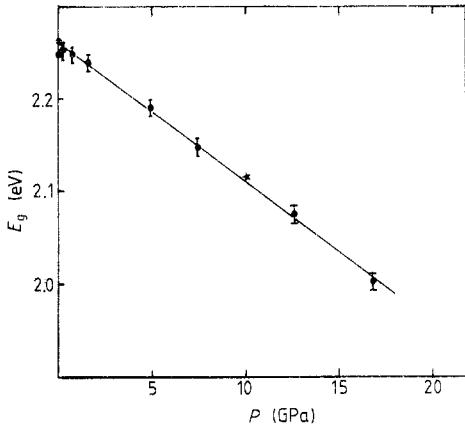


Figure 4. Indirect band gap E_g versus pressure. The star denotes a value calculated from Elliott's formula.

Table 3. Comparison of dE_g/dP values.

dE_g/dP (meV GPa ⁻¹)	Method	Reference
-20.2 ± 2.5	Experiment	Ves <i>et al</i> (1985)
-26 ± 5	Experiment	Mathieu <i>et al</i> (1979)
-18	Calculation	Gorczyca (1984)
-17	Calculation	Tsay <i>et al</i> (1974)
-11 ± 1	Experiment	Zallen and Paul (1964)
-14.6 ± 0.7	Experiment	Gil <i>et al</i> (1984)
-14.9 ± 0.5	Experiment	Present work

2.2.2. High pressure results. To determine the pressure coefficient we have plotted $(\alpha h\nu)^{1/2}$ versus $h\nu$ for each pressure and calculated the best straight line by using a least-squares method for $13 \leq (\alpha h\nu)^{1/2} \leq 42 \text{ cm}^{-1/2} \text{ eV}^{1/2}$ (figure 3). This energy range has been chosen to be narrow enough for it to be used over the whole pressure range considered. Indeed, transmission measurements in a DAC are precise only for $\alpha \geq 70 \text{ cm}^{-1}$; on the other hand, at low pressure the proximity of the direct gap perturbs the high absorption range ($\alpha > 700 \text{ cm}^{-1}$). Then, we have extrapolated the straight line $(\alpha h\nu)^{1/2} = f(h\nu)$ to $\alpha = 0$ to determine $E'_g = E_g - \hbar\omega_{\text{LA}} = f(P)$.

The pressure dependence of the LA mode is deduced from the equation

$$(\hbar\omega_{\text{LA}})_P = [(\hbar\omega_{\text{LA}})_{P=0}/B_0^{\gamma/B'_0}](B_0 + B'_0 P)^{\gamma/B'_0}$$

where the bulk modulus $B_0 = 88.2 \text{ GPa}$, its derivative $B'_0 = 4.79$ (Yogurtçu *et al* 1981) and the LA mode Grüneisen parameter $\gamma = 1.0$ (Weinstein 1984) and then E_g is evaluated by $E_g \approx E'_g + \hbar\omega_{\text{LA}}$. The variation of $\hbar\omega_{\text{LA}}$ with pressure is two orders of magnitude smaller than the pressure variation of E_g in the pressure range considered. Then we take $dE'_g/dP \approx dE_g/dP$.

We have found a linear behaviour of E_g with pressure up to 17 GPa (figure 4) within the experimental precision. The error bars in the figure 4 are obtained from the dispersion of the various extrapolations to $\alpha = 0$ of the different lines $(\alpha h\nu)^{1/2} = f(h\nu)$ which can fit the experimental points. The validity of the determination of $E_g(P)$ by the above method has been checked at 10 GPa by computing $(\alpha h\nu)^{1/2}$ with the full Elliott formula and deducing E_g from $(\alpha h\nu)^{1/2}$. The calculated and experimental E_g -values agree very well.

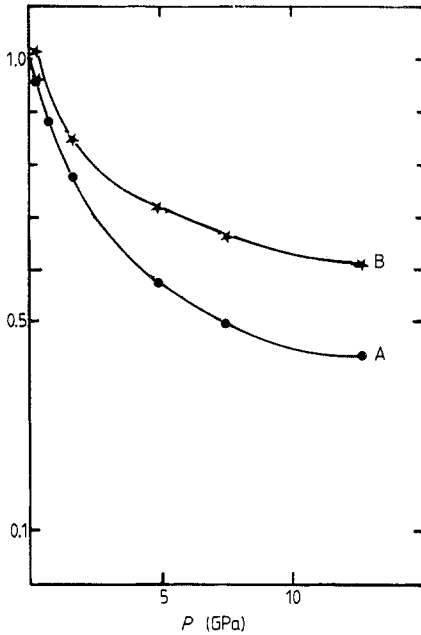


Figure 5. Variation of the slope of $(\alpha h\nu)^{1/2} = f(h\nu)$ with pressure. Comparison between theoretical evaluation and experimental values: curve A, $(E_0^{3/4}/\Delta E)_P/(E_0^{3/4}/\Delta E)_{P=0}$ (theoretical); curve B, S/S_0 (experimental).

Table 3 gives a comparison between the various published values (Mathieu *et al* 1979, Gorczyca 1984, Tsay *et al* 1974, Gil *et al* 1984) of dE_g/dP and our result. It is worth noting that the result of Gil *et al* (1984) obtained up to 0.8 GPa remains valid up to 17 GPa. The data of Ves *et al* (1985) and Mathieu *et al* (1979) are obtained indirectly from uniaxial pressure experiments. This is probably the reason for the discrepancy with our result.

We now consider variation of the slope of $(\alpha h\nu)^{1/2} = f(h\nu)$. If we take the contribution of the LA phonon as being preponderant and if we neglect the excitonic part in the absorption coefficient, then the slope S of $(\alpha h\nu)^{1/2} = f(h\nu)$ is roughly proportional to $(b_i)^{1/2}$ since we can consider $\rho_i \approx 1$ when $\Delta E = E_0 - E_g$ increases. $(b_i)^{1/2}$ is proportional to $(m_c^* m_v^*)^{3/4}/\Delta E$ where m_c^* and m_v^* are respectively the effective masses of the electron and the hole (Bassani 1975). The curvature of the valence band is less than that of the conduction band so we can assume that m_c^* will vary much more than m_v^* and take m_v^* as a constant. Therefore, the slope must roughly vary with pressure as $(m_c^*)^{3/4}/\Delta E$ which is proportional to $E_0^{3/4}/\Delta E$. We have for P in GPa

$$E_0 \approx 2.76 + 9.7 \times 10^{-2} P - 35 \times 10^{-4} P^2 \quad (\text{Ves } et al \text{ 1985})$$

$$E_g \approx 2.261 - 1.49 \times 10^{-2} P$$

$$\Delta E \approx 0.5 + 11.2 \times 10^{-2} P - 35 \times 10^{-4} P^2$$

up to about 12 GPa. Figure 5 shows a similar behaviour of the slope S and $\Delta E_0^{3/4}/\Delta E$ with pressure. The difference between the two curves comes from the terms that we have neglected.

3. Phase transition

Transmission experiments, electron diffraction, x-ray diffraction, Raman scattering and

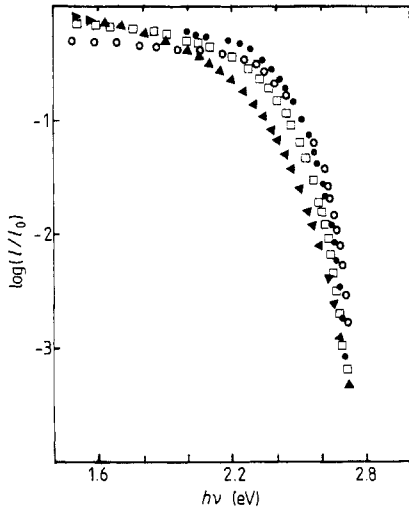


Figure 6. Transmission at low pressure versus the energy after several runs. ●, at 0.22 GPa; ○, at 0.53 GPa down from 21.16 GPa; □, at 0.52 GPa down from 22.3 GPa; ▲, at 0.84 GPa down from 23.8 GPa.

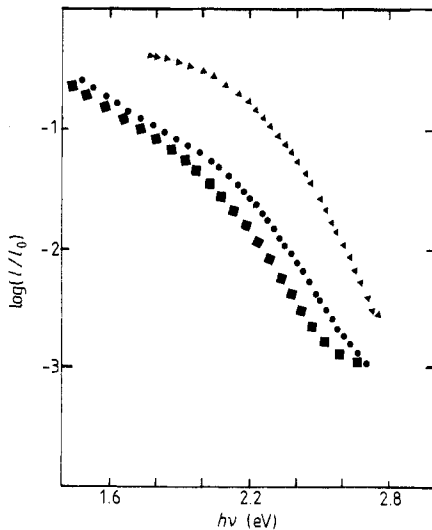


Figure 7. Comparison between the transmission when the pressure increases and the transmission when the pressure decreases after a run up to 21 GPa. ▼, transmission at 15.2 GPa (P increasing); ●, transmission at 15.1 GPa (P decreasing); ◆, transmission at 21 GPa.

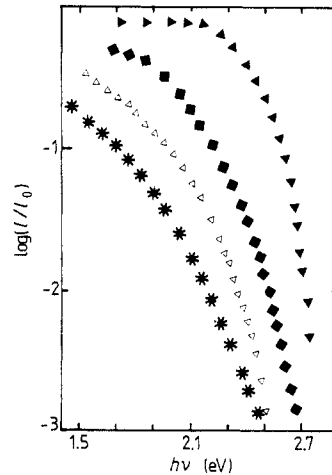


Figure 8. As for figure 7, but at the following pressures: ▼, transmission at 4.87 GPa; ◆, transmission at 22 GPa; ▽, transmission at 4.58 GPa down from 22 GPa; *, transmission at 23.3 GPa.

photographs under microscope have given results which are in agreement with each other.

The sample begins to blacken at about 22 GPa but we could measure the optical transmission up to 23.3 GPa. Figure 6 shows the irreversibility of the phenomenon by comparing low-pressure spectra before and after pressurisation. We also note that the transition begins between 21 and 22 GPa. Figures 7 and 8 illustrate the irreversibility of the transition. From 21 GPa, when the pressure is decreased, the absorption edge shifts towards high energies as we can expect if no transition occurs (figure 7) but it does not return to the initial position. On the other hand, when the pressure is decreased from

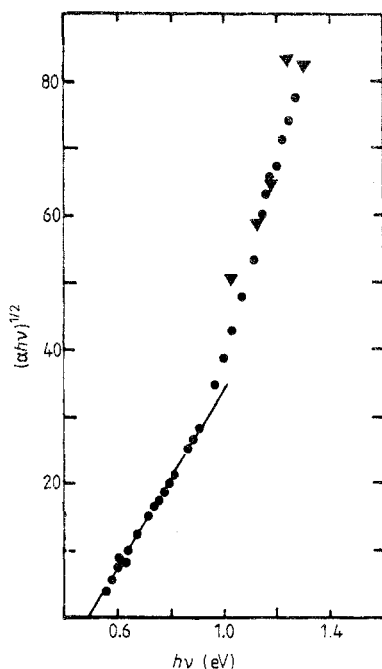


Figure 9. $(\alpha h\nu)^{1/2}$ versus $h\nu$. Comparison between a sample of GaP at room pressure after a run up to 32.5 GPa (●) and annealed amorphous GaP (▼) (Gheorghiu 1982).

22 GPa, then the absorption edge continues to shift towards low energies (figure 8). Then the sample was transitioned enough to have the absorption edge shifted as if we had been increasing the pressure.

After a run up to 32.5 GPa, the sample was depressurised to room pressure. The transmission spectrum was measured in near infrared region (figure 9). We find, between 0.4 and 0.8 eV, a linear behaviour of $(\alpha h\nu)^{1/2}$ with $h\nu$ which extrapolates to 0.49 eV for $\alpha = 0$. This value agrees well with that of Connell and Paul (1972) for the absorption edge of amorphous GaP (0.42 eV). In the region between 0.8 and 1.3 eV we find results which are in good agreement with those on annealed amorphous GaP (Gheorghiu 1982).

Electron diffraction and x-ray diffraction confirm the presence of a mixture of polycrystalline and amorphous GaP when the sample is depressurised from high pressure. After a run up to 28.4 GPa, electron diffraction at room pressure shows polycrystals with various sizes (between 4 and 400 nm) and a region of amorphous GaP, but few regions are diffracting. As for x-ray diffraction at room pressure, the films show polycrystalline GaP after a run up to 21 GPa and broadened and very weak diffraction rings after a run up to 32 GPa compatible with a mixture of polycrystalline and amorphous GaP.

We can follow the LO and TO modes up to 22 GPa by Raman scattering on samples oriented along (111). Our results are similar to those of Pinceaux *et al* (1979), and we have also confirmed the irreversibility of the transition: when the samples are depressurised the LO and TO modes do not reappear.

We have also measured by a microphotographic method, in the 0–10 GPa range, the sample dimension l for four various directions. The four series of measurements gave us 52 values of $(l - l_0)/l_0$ which have been fitted by the function $CP^2 + DP$. Then, from the determination of the coefficient C and D , and using Murnaghan's equation of state we could determine the volumic compressibility χ_0 and the first pressure derivative of the bulk modulus. The comparison between our results and the values published in the

Table 4. Comparison of χ_0 and B'_0 values.

χ_0 (10^{-2} GPa $^{-1}$)	B'_0	Reference
1.10 ± 0.05	5.1 ± 0.5	Present work
1.134	4.79	Yogurtçu <i>et al</i> (1981)
1.114	—	Boyle (1975)
1.121	—	Pesin <i>et al</i> (1976)
1.171	5.75	Gerlich and Wolf (1979)
—	5.0	Rimai and Sladek (1979)

literature are summarised in table 4. The high pressure deformation of the sample is not isotropic and when it is quenched to room pressure the surface of the sample is larger (about 7%) than before pressurisation. After several months at months at room pressure, the samples are still black. This result seems to be in contradiction with observations of Ves *et al* (1985).

Photographs show the coexistence of two phases between 21 and 24 GPa. Hu *et al* (1984) indicate the presence of two phases at 25 ± 0.5 GPa from x-ray experiments in polycrystalline samples.

4. Conclusion

We have shown that the pressure dependence of E_g up to 17 GPa is well reproduced by a linear law and located the beginning of the transition between 21 and 22 GPa. After lowering pressure down to ambient, the samples become partly polycrystalline with an absorption coefficient similar to that of annealed amorphous GaP. The transition is irreversible. We are now performing high pressure EXAFS experiments to confirm our results.

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