

Determination of the carrier concentration of doped ZnSe from infrared measurements

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(Received 18 September 1990; accepted 29 October 1990)

Determination of the carrier concentration of *n*-type ZnSe/GaAs heteroepitaxial films from absorption measurements is reported over the wave number range $30 < W < 600 \text{ cm}^{-1}$. Transmission and reflection curves are very sensitive to the doping level, *n* above 3×10^{16} and $3 \times 10^{17} \text{ cm}^{-3}$ levels, respectively. Absorption coefficients, α_f are derived from the transmitted intensities and vary between 500 and 14400 cm^{-1} in this wave number range. Around $W = 100 \text{ cm}^{-1}$, α_f varies as $n^{1/2}$. This allows the determination of *n* from α_f . The α_f variations versus wave number and doping are compared to available models for various absorption processes.

I. INTRODUCTION

ZnSe has a 2.7 eV direct band gap and is a good candidate for blue light emitting diodes or diode lasers.¹ Currently, however, the determination of carrier densities in ZnSe epilayers is rather difficult particularly in the case of *p*-type material due to the lack of suitable ohmic contact technology.² Optical measurements, which are contactless, are an alternative, simple, fast, non destructive method to characterize such thin doped films.

Optical techniques³ have been used to measure absorption coefficients associated with free carriers in *n*-type bulk ZnSe in the mid infrared range ($2 < \lambda < 11 \mu\text{m}$). However, the absorption coefficients reported ($1 < \alpha_f < 100 \text{ cm}^{-1}$) are not large enough to be measured in thin films. A theoretical model,⁴ however, predicts values of α_f from the free carrier absorption up to 10^3 cm^{-1} for higher doping levels (10^{19} cm^{-3}) near the wavelength (wave number) limit of the model ($\lambda < 11 \mu\text{m}$, i.e., wave number $W > 900 \text{ cm}^{-1}$).

In a previous short letter,⁵ we reported absorption coefficients in the range $10^3 < \alpha_f < 2 \times 10^4 \text{ cm}^{-1}$ for *n*-type ($9.4 \times 10^{16} < n < 8 \times 10^{18} \text{ cm}^{-3}$) ZnSe heteroepitaxial films in the wave number range, $30 < W < 600 \text{ cm}^{-1}$. These values were derived from transmission experiments alone using a simple approximation of the transmission from a single absorbing thin film and two stacked absorbing thin films deposited on a thick absorbing substrate.

In this work, we have extended the data to another set of samples with a larger range of *n*-type doping (from $3 \times 10^{16} \text{ cm}^{-3}$ to $3.82 \times 10^{18} \text{ cm}^{-3}$), and to both reflection (*R*) and transmission (*T*) measurements. We will show that both reflection and transmission values are sensitive to the doping level. Since the analytical expressions for *T* and *R* needed to calculate α_f in absorbing multilayer thin films on a thick

absorbing substrate are still unknown, the absorption coefficients are derived from the transmission measurements alone. However, we can now better verify the validity of this approach since the corresponding reflection values are now reported.

II. EXPERIMENTAL SETUP

The ZnSe films were grown by molecular-beam epitaxy (MBE) on undoped, semi-insulating ($\rho > 10^7 \Omega \text{ cm}$) (100) GaAs substrates under a fixed set of growth conditions, namely a substrate temperature of 275 C and a Zn to Se beam equivalent pressure ratio of 1:2. The ZnSe layers were doped *n*-type by substitutional incorporation of Cl atom during the MBE growth process using a ZnCl_2 effusion source. The doped layers were grown on a $\sim 0.5\text{-}\mu\text{m}$ thick undoped ZnSe buffer layer to avoid doping interaction with the GaAs substrate. The layers were grown in a custom designed MBE system previously used to grow ultra high purity homoepitaxial ZnSe layers.⁶ Room temperature carrier concentrations and electron mobilities in the ZnSe films were determined by Hall-effect measurements, the carrier concentration range being $3 \times 10^{16} < n < 3.82 \times 10^{18} \text{ cm}^{-3}$.

Transmission and reflection measurements were taken at room temperature using a Fourier transform infrared spectrometer (Bruker IFS 113 V) with four overlapping ranges, 35–100, 80–300, 200–600, and 450–5000 cm^{-1} . The resolution was 1 or 2 cm^{-1} in the *W* ranges 35–600 and 450–5000 cm^{-1} , respectively. The detector was a liquid helium cooled bolometer or a DTGS between 35–600 and 450–5000 cm^{-1} , respectively. The substrate and film thicknesses were determined from their interferences fringes in the far and mid infrared, respectively.

The characteristics of the samples are given in Table I.

TABLE I. Characteristics of the samples.

Samples No.	34	75	76	78
GaAs thickness (μm)	370	337	347	399
ZnSe thickness (μm) (undoped)	1.9	0.76	0.76	0.70
ZnSe thickness (μm) (doped)		1.52	1.52	1.41
n doping level (cm^{-3})		3×10^{16}	3×10^{17}	3.82×10^{18}

Since the back of the substrates had to be lapped and polished to remove the indium used to bond them onto the substrate holder, a slight variation of thickness among the various substrates was found (Table I).

III. RESULTS AND DISCUSSION

Examples of raw transmission data for lightly (no. 75, $n = 3 \times 10^{16} \text{ cm}^{-3}$) and heavily (no. 78, $n = 3.82 \times 10^{18} \text{ cm}^{-3}$) doped ZnSe are shown in Fig. 1. The raw transmission data (shifted by +0.3) of a bare GaAs substrate of similar thickness (372 μm) is also included in Fig. 1 for comparison. Most of the structure in the transmission curves of samples no. 75 and no. 78 originate from the GaAs substrate: (i) $310 < W < 550 \text{ cm}^{-1}$, multiphonon absorption; (ii) $160 < W < 200 \text{ cm}^{-1}$, plateau; (iii) $W < 150 \text{ cm}^{-1}$, interference fringes dependent on the exact GaAs thickness; and (iv) $240 < W < 320 \text{ cm}^{-1}$, zero transmission due to the reststrahlen band for GaAs. There is also an absorption band around 206 cm^{-1} from the reststrahlen band of ZnSe. The data in Fig. 1 show unambiguously that the doping has a tremendous effect on the transmission of the samples, over the entire range of $30 < W < 600 \text{ cm}^{-1}$. In the range $W < 200 \text{ cm}^{-1}$, substrate interference fringes were removed by sub-

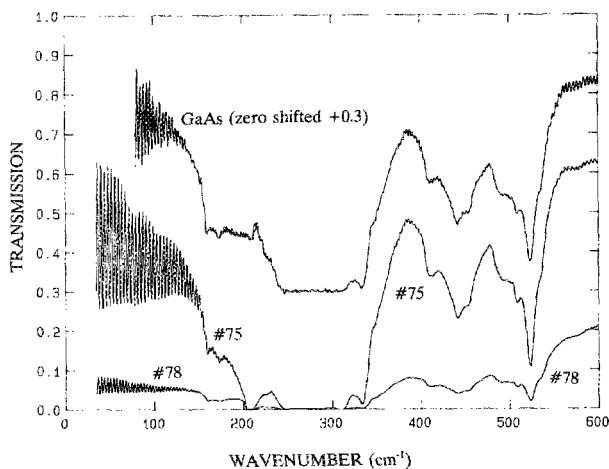


FIG. 1. Transmission vs wave number of the samples with lightly doped (no. 75, $3 \times 10^{16} \text{ cm}^{-3}$) and heavily doped (no. 78, $3.82 \times 10^{18} \text{ cm}^{-3}$) ZnSe films, and of a bare GaAs substrate (shifted by +0.3).

tracting the corresponding peak in the Fourier transform of both the transmission and reflection data. All remaining transmission and reflection data will be shown following the subtraction process.

Transmission curves from samples comprised of a GaAs substrate with a single ZnSe undoped film (no. 34), or with doped films of increasing doping level [no. 75 ($3 \times 10^{16} \text{ cm}^{-3}$), no. 76 ($3 \times 10^{17} \text{ cm}^{-3}$)], and no. 78 ($3.82 \times 10^{18} \text{ cm}^{-3}$) are given in Fig. 2.

Imperfect removal of the interference fringes in the range $35\text{--}100 \text{ cm}^{-1}$ introduces slight (but reproducible) structure and a slight mismatch in the continuity between neighboring ranges. The transmission curve of sample no. 75 between 300 and 600 cm^{-1} is not shown in Fig. 2, because it is nearly identical to that of sample no. 34. The absorption coefficient of GaAs decreases very rapidly with decreasing wave number below the reststrahlen.⁷ It is transparent ($\alpha < 10 \text{ cm}^{-1}$) for $W < 200 \text{ cm}^{-1}$, except for absorption ($\alpha \sim 40 \text{ cm}^{-1}$) between 160 and 200 cm^{-1} . Multiphonon bands with $20 < \alpha < 60 \text{ cm}^{-1}$ induce the sharp structures seen in Figs. 1 and 2 between $310 < W < 600 \text{ cm}^{-1}$. The absorption coefficient of undoped ZnSe⁸ has a negligible effect ($\alpha < 10^3 \text{ cm}^{-1}$) when W is outside the range of $180 < W < 300 \text{ cm}^{-1}$. Above and below the range $200 < W < 350 \text{ cm}^{-1}$, there is a large monotonic decrease in the sample transmission as the doping level of the ZnSe film increases from $n = 3 \times 10^{16}$ to $3.82 \times 10^{18} \text{ cm}^{-3}$, particularly for $W < 200 \text{ cm}^{-1}$. In this range, the transmission curve corresponding to $3 \times 10^{16} \text{ cm}^{-3}$ is significantly lower than that corresponding to the sample no. 34 with the undoped ZnSe film, indicating clearly that these doping level can easily be detected in $1.5\text{-}\mu\text{m}$ thick films.

Reflection data (after removing the GaAs interferences fringes) for the nos. 34, 76, and 78 are shown in Fig. 3. There is a striking difference between data from no. 78, whose reflection curve appears to be dominated by a slowly de-

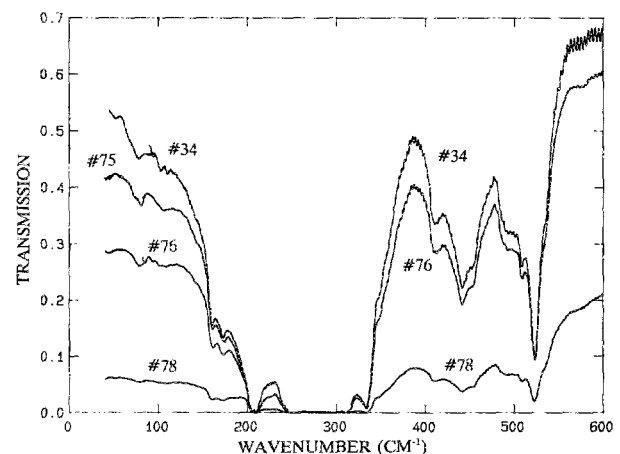


FIG. 2. Transmission vs wave number of samples with a single undoped ZnSe film (no. 34) and two stacked doped and undoped ZnSe films on a GaAs substrate with increasing doping [(no. 75) 3×10^{16} ($30\text{--}205 \text{ cm}^{-1}$), (no. 76) 3×10^{17} , (no. 78) $3.82 \times 10^{18} \text{ cm}^{-3}$] after removal of the interference fringes.

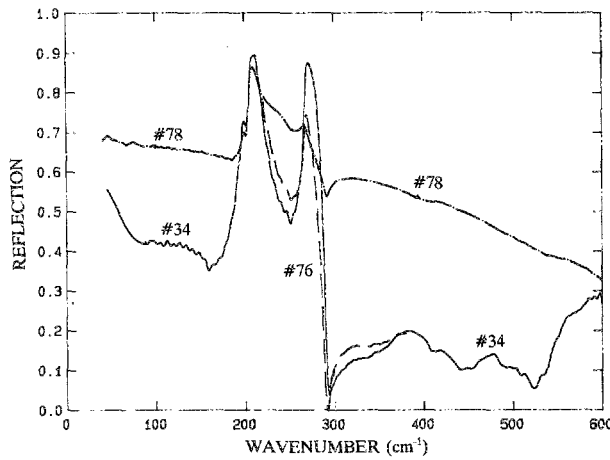


FIG. 3. Reflection vs wave number of undoped ZnSe film (no. 34, full line), ZnSe film doped to $3 \times 10^{17} \text{ cm}^{-1}$ (no. 76, 215–380 cm^{-1} , dashed line), $2.8 \times 10^{18} \text{ cm}^{-1}$ (no. 78, mixed line).

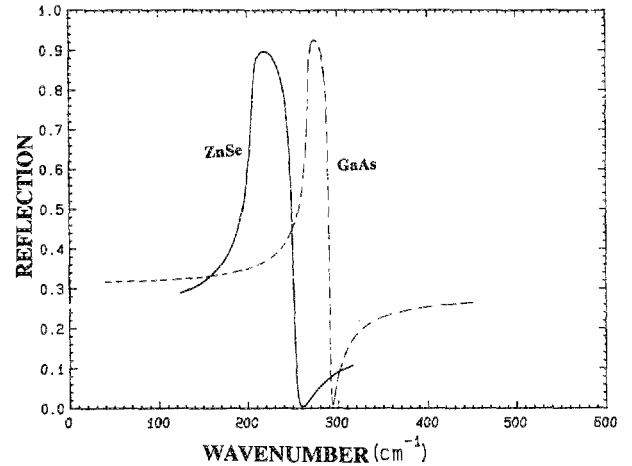


FIG. 4. Reflection vs wave number for bulk ZnSe (Ref. 8(b), solid line) and GaAs (Ref. 10, dotted line).

ing function of W , and no. 76 whose data are close to that of the undoped sample no. 34. For clarity the reflection curve of no. 76 has been drawn only between 215 and 380 cm^{-1} where it can be distinguished from that of no. 34. The reflection curve of no. 75 is nearly identical of that of the undoped sample and therefore is not shown in this figure. The reflection of bulk undoped ZnSe⁸ and GaAs¹⁰ (denoted by R_{13} and R_{41} , respectively, in the expressions of the transmission and reflection Eq. 1 to 4) from the literature have been plotted between 0–600 cm^{-1} in Fig. 4. By comparison with Fig. 3, it may be concluded that, (i) the peaks around $W = 206$ and 270 cm^{-1} originate from the interfaces with ZnSe and GaAs, respectively. However, their widths are more narrow than for bulk material, which indicates that our samples had some additional (complicated) interference effects resulting from the thickness of the thin films. (ii) In addition to the reflection at the air/ZnSe interface, there is, below 160 cm^{-1} , another component from multiple reflections. Only doping levels higher than $3 \times 10^{17} \text{ cm}^{-3}$ can be distinguished by reflection measurements as shown in Fig. 3. On the other hand, reflection data show that important effects from mul-

iple reflections are present in our samples.

Expressions for transmission and reflection of an absorbing thin film on a transparent substrate exist in the literature,⁹ but to our knowledge, expressions for transmission and reflection of one or two absorbing thin film on a thick absorbing substrate have not been published. These expressions would have to include the reflectivity R_{ij} and the phase shift ϕ_{ij} at the each interface (except for the “thick” GaAs/air), plus the absorption coefficient α_i and the phase shift, ϕ_i (except for the thick GaAs) for all media, where the subscripts ij designate the approach of light to the interface between i and j through media i . To simplify our designation, we will number the various media as follows, (1) air; (2) doped ZnSe film with α_f , ϕ_2 and thickness d ; (3) undoped ZnSe film with α_u , ϕ_3 , and thickness e ; and (4) GaAs substrate α_s and thickness D . From simple energy absorption balance and taking into account the occurrence of reflection at the interfaces, the reflection R_r and R and the transmission T_r and T of a single absorbing film and two stacked absorbing films on the thick absorbing GaAs substrate, respectively, would have the form

$$R_r = R_{13} + \frac{G' \{ [R_{13}, R_{34}, R_{41}, \phi_{13}, \phi_{34}, \phi_3, \exp(-\alpha_u e), \exp(-\alpha_s D)] \}}{H' \{ [R_{13}, R_{34}, R_{41}, \phi_{13}, \phi_{34}, \phi_3, \exp(-\alpha_u e), \exp(-\alpha_s D)] \}}, \quad (1)$$

$$R = R_{12} + \frac{G \{ [R_{12}, R_{23}, R_{34}, R_{41}, \phi_{12}, \phi_{23}, \phi_{34}, \phi_2, \phi_3, \exp(-\alpha_f d), \exp(-\alpha_u e), \exp(-\alpha_s D)] \}}{H \{ [R_{12}, R_{23}, R_{34}, R_{41}, \phi_{12}, \phi_{23}, \phi_{34}, \phi_2, \phi_3, \exp(-\alpha_f d), \exp(-\alpha_u e), \exp(-\alpha_s D)] \}}, \quad (2)$$

and

$$T_r = \frac{[(1 - R_{13})(1 - R_{34})(1 - R_{41}) \exp(-\alpha_u e - \alpha_s D)]}{1 - F' \{ [R_{13}, R_{34}, R_{41}, \phi_{13}, \phi_{34}, \phi_3, \exp(-\alpha_u e), \exp(-\alpha_s D)] \}}, \quad (3)$$

$$T = \frac{(1 - R_{12})(1 - R_{23})(1 - R_{34})(1 - R_{41}) \exp(-\alpha_f d - \alpha_u e - \alpha_s D)}{1 - F \{ [R_{12}, R_{23}, R_{34}, R_{41}, \phi_{12}, \phi_{23}, \phi_{34}, \phi_2, \phi_3, \exp(-\alpha_f d), \exp(-\alpha_u e), \exp(-\alpha_s D)] \}}, \quad (4)$$

where F , F' , G , G' , H , and H' are polynomial functions of multiples variables which account for reflections at the various interfaces and whose values remain < 1 .

Therefore,

$$\frac{T}{T_r} = \frac{(1 - R_{12})(1 - R_{23})(1 - F') \exp(-\alpha_f d)}{(1 - R_{13})(1 - F)} \quad (5)$$

Data in Fig. 3 show that for $n \leq 3 \times 10^{17} \text{ cm}^{-3}$ (samples 75

and 76), $R_r \sim R$, so the real parts n_2 and n_3 of the refractive index of doped and undoped ZnSe films are very close, and dominate R_{12} and R_{13} . Therefore,

$$R_{32} = \frac{(n_3 - n_2)^2}{(n_3 + n_2)^2} \sim 0,$$

and from Eq. (5)

$$\frac{T}{Tr} = \frac{(1 - F')}{(1 - F)} \exp(-\alpha_f d). \quad (6)$$

Assuming that the preexponential term is nearly constant or varies slowly in comparison of the exponential leads to

$$\frac{T}{Tr} \sim \exp(-\alpha_f d), \quad (7)$$

which allows a simple determination of α_f . For no. 78, the $(1 - R_{12})/(1 - R_{13})$ ratio can be guessed from Figs. 3 and 4. It is of the order of 0.35/0.75 which would yield a larger α_f by an additive factor of $5 \times 10^3 \text{ cm}^{-1}$. There will also be a larger yield from $R_{23} = 0$, but the effect of $(1 - F')/(1 - F)$ on α_f remains unknown.

This procedure for deriving α_f is limited to the W range where the transmission is not zero, i.e., outside the range 205–305 cm^{-1} . Actually, cancellations of the effects of the undoped layer and that of the substrate are not perfect. The undoped buffer layer are thinner than the single undoped layer on the GaAs substrate. The thicknesses of the GaAs substrates after grinding and polishing have a small dispersion (Table I). As discussed in our previous paper,⁵ all these spurious effects are negligible if the dead range for W is expanded to include 150–350 cm^{-1} . Only small and sharp structures from the multiphonon absorption bands, which can be easily identified on the α_f versus W curves, remain from differences in the GaAs substrate thicknesses. Variation of the absorption coefficient with wave number for the doped ZnSe films is given in Fig. 5. The larger "noise" in the transmission curve of sample no. 76 between 350 and 500 cm^{-1} originates from the interference fringes which cannot be removed in this range, as discussed previously. The wave number variation of the absorption coefficient of sample no.

78 in the range 550–1100 cm^{-1} , obtained by the same procedure, has been added in Fig. 6 for completeness.

The absorption coefficients increase monotonically as the doping level increases, i.e., from 9×10^2 (no. 75, $3 \times 10^{16} \text{ cm}^{-3}$) to $1.44 \times 10^4 \text{ cm}^{-1}$ (no. 78, $2.8 \times 10^{18} \text{ cm}^{-3}$) at 100 cm^{-1} at 100 cm^{-1} . For all samples, α_f decreases as W increases in the ranges 40–150 and 350–1100 cm^{-1} . The curves can be approximately fitted by $\alpha_f \propto W^{-p_1}$, over limited ranges for the far and mid infrared. In the mid infrared, $p_2 = 2.60 \pm 0.4$ for sample no. 76 ($3 \times 10^{17} \text{ cm}^{-3}$) and increases to 4.7 ± 0.3 at higher doping (sample no. 78, $3.82 \times 10^{18} \text{ cm}^{-3}$). In the far infrared, $p_1 < 1$ and decreases as the doping increases, $p_1 = 0.66 \pm 0.03$ for $3 \times 10^{16} \text{ cm}^{-3}$ (sample no. 75), 0.25 ± 0.02 for $3 \times 10^{17} \text{ cm}^{-3}$ (sample no. 76), and is nearly 0 for $3.82 \times 10^{18} \text{ cm}^{-3}$ (sample no. 78).

Compared to our previous data,⁵ a better polishing procedure and a liquid helium cooled bolometer which decreases the noise have allowed us to extend the α measurements down to $5 \times 10^3 \text{ cm}^{-1}$. The magnitude of α_f , the shape of the $\alpha_f(W)$ curves and the p values are similar and have similar variation with the doping to those reported earlier.⁵ The values of α_f , at 100 cm^{-1} (left scale) and those of p_1 (right scale) versus doping level are shown in Fig. 6, for this set of samples (\diamond and \times , respectively) and the previous one (\square and $+$, respectively). The figure shows that the agreement among the α_f and p_1 values derived between the two set of samples is very good. From Fig. 6, α_f varies as $n^{1/2}$, while p_1 is approximately proportional to $n^{-3/2}$. This figure allows the determination of the carrier concentration from the absorption coefficient of the doped film at $W = 100 \text{ cm}^{-1}$.

As in our previous work,⁵ the values of p_2 (mid infrared) are in good agreement with those predicted by Ruda⁴ when α_f is dominated by the absorption of the free carriers. The lower p_2 value for the lower doping correspond to the dominance of the scattering of electrons by optical phonons, while the higher values correspond to the dominance of ionized impurities in the scattering process. The absorption in the far infrared can be tentatively attributed to plasmon-phonon interactions.¹⁰ Work is in progress to clarify this point.

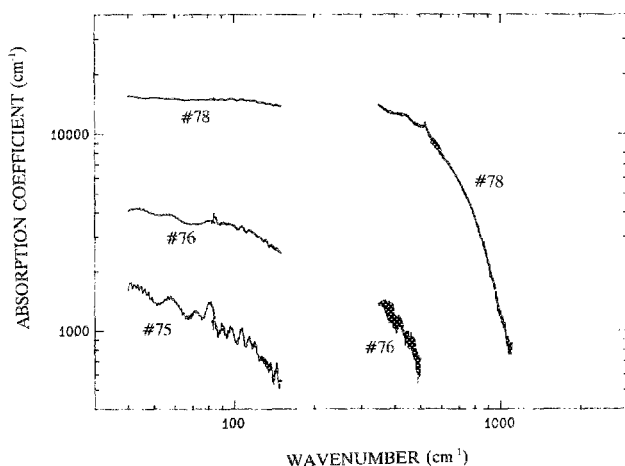


FIG. 5. Absorption coefficient vs wave number of the ZnSe doped films, (no. 75) $3 \times 10^{16} \text{ cm}^{-3}$, (no. 76) $3 \times 10^{17} \text{ cm}^{-3}$, (no. 78) $3.82 \times 10^{18} \text{ cm}^{-3}$.

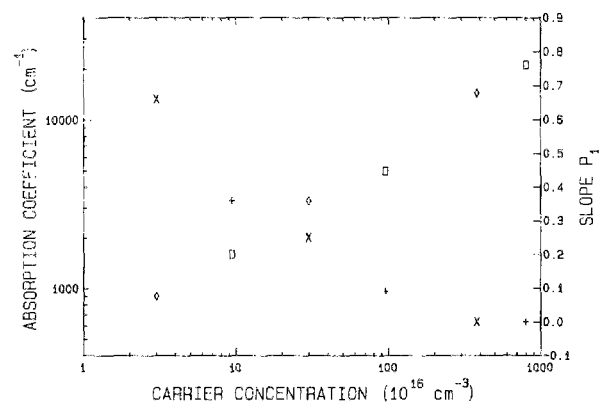


FIG. 6. Absorption coefficient (left scale) and slope p_1 (right scale) vs carrier concentration for this set of samples (\diamond and \times , respectively) and the previous set (\square and $+$, respectively).

IV. CONCLUSIONS

We have shown that over the wave number range 35–600 cm^{-1} , the transmission and reflection values of the doped ZnSe/undoped ZnSe/GaAs substrate are very sensitive to the doping level of the top film. The dopant concentration range studied was 3×10^{16} – $3.82 \times 10^{18} \text{ cm}^{-3}$.

The reflection values for $n \leq 3 \times 10^{17} \text{ cm}^{-3}$ are almost independent of the doping which justifies the use of a simple approximation of the transmission expressions to derive the absorption coefficient of the doped films by comparison of the transmission values between samples with single undoped and stacked doped–undoped ZnSe films. Since analytical expressions for transmission and reflection for a single or two stacked absorbing films on a thick absorbing substrate are not known, a simple approximation of the transmission has been used over the entire doping range of 3×10^{16} – $3.82 \times 10^{18} \text{ cm}^{-3}$. Absorption coefficients α_f have been determined to be between 5×10^2 and $1.44 \times 10^4 \text{ cm}^{-1}$, depending on the wave number and the doping level. While the error on the α_f values for lower doping densities is expected to be low, the values reported for the $3.82 \times 10^{18} \text{ cm}^{-3}$ sample may be as much as $5 \times 10^3 \text{ cm}^{-1}$ too large. The absorption coefficient increases monotonically as the doping level increases. Doping levels as low as $3 \times 10^{16} \text{ cm}^{-3}$ can be measured since the absorption coefficient is $9.2 \times 10^2 \text{ cm}^{-1}$ at $W = 100 \text{ cm}^{-1}$. At this wave number, higher doping, ($3.82 \times 10^{18} \text{ cm}^{-3}$), gives $\alpha_f = 1.44 \times 10^4 \text{ cm}^{-1}$. At $W = 100 \text{ cm}^{-1}$, when we include our previous set of samples,⁵ we show with good accuracy that α_f is proportional to $n^{1/2}$. This allows the determination of n from α_f from Fig. 6. Over the two W ranges, 40–150 and 350–1100 cm^{-1} , α_f decreases monotonically as W increases. Over limited ranges $\alpha_f \propto W^{-p}$. In the mid infrared, $p_2 = 2.60 \pm 0.4$ for $3 \times 10^{17} \text{ cm}^{-3}$ and increases to 4.7 ± 0.3 for $3.82 \times 10^{18} \text{ cm}^{-3}$. In the

far infrared, p_1 decreases as the doping increases, yielding $p_1 = 0.66 \pm 0.03$ for $3 \times 10^{16} \text{ cm}^{-3}$, 0.25 ± 0.02 for $3 \times 10^{17} \text{ cm}^{-3}$, and is nearly 0 for $3.82 \times 10^{18} \text{ cm}^{-3}$. These results are in good agreement with our previous results on another set of samples. In the mid infrared region, they also agree with the predictions of the theoretical model of Ruda,⁴ where the absorption is ascribed to free carrier absorption. In the far infrared, there is clearly another absorption process, which might originate from plasmon phonon coupling.

ACKNOWLEDGMENT

This work was supported by DARPA Grant No. MDA 972-88-J-1006.

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