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Carrier-induced change due to doping in refractive index of InP: Measurements at 1.3 and 1.5 μm

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Accurate measurements of the InP refractive index as a function of free-carrier doping are reported at 1.3 and 1.5 μm , the two strategic wavelengths for optical communications. A total of 21 samples with different *N*- and *P*-doping levels have been measured using a novel and simplified grating-coupling technique. In contrast to the conventional method, this only involves the use of a directly etched diffraction grating on the sample surface, thereby avoiding the necessity of a specific guiding layer. The measured index, in agreement with earlier predictions, decreases by more than 0.05 when the *N* doping is increased from below 10^{15} to about 10^{19} electrons per cubic centimeter. This effect, however, is much less pronounced with *P* doping than with *N* doping. © 1996 American Institute of Physics. [S0003-6951(96)00146-5]

Indium phosphide is the basic semiconductor material for long-wavelength optoelectronic and photonic device fabrication devoted to future optical communication systems. Several modern applications in this field such as, wavelength-division multiplexing, design of Bragg reflectors, or modal calculations require an accurate knowledge of InP refractive index in the 1.3–1.5 μm wavelength range. Such considerations have revived the field of InP refractive index experimental determination^{1,2} with the goal of increasing the accuracy of previously available reference data^{3,4} up to, at least, 10^{-3} . Evidently, such accuracy necessitates a thorough knowledge of free-carrier induced change in refractive index due for instance to voluntary impurity doping. Unfortunately, the few available experimental data^{5–7} show marked discrepancies. One frequently resorted in a theoretical model⁸ to evaluate doping influence.

One of the most accurate techniques for refractive index measurements employs a grating coupler etched on the surface of a guiding layer.^{1,9} It provides the effective guided-mode indices, n_{eff} , of allowed propagating modes within a planar waveguide. Very sharp peaks are observed either on the reflected or guided signals as the input angle of incidence, θ , is varied. Effective guided-mode indices, n_{eff} , are determined from the resonant-coupling angles according to the formula $n_{\text{eff}} = \sin \theta + m\lambda/\Lambda$, where λ is the wavelength in vacuum, Λ the grating period, and m the grating order. The range of λ and Λ are kept so that $m = 1$ in our experiments. This procedure has been employed to determine the refractive index of a thin unintentionally doped InP layer grown by molecular beam epitaxy (MBE).¹

In the present work, the InP substrate index is obtained from a slightly different scheme which in fact is implicit in our earlier work. For example, as we have remarked in Ref. 1, **a guiding layer is not required** to observe a sharp peak, the substrate index, n , then being given by $n = \sin \theta_0 + \lambda/\Lambda$, where θ_0 is the angle of peak position. The physical origin of this somewhat unexpected¹⁰ peak resides in the experimental setup (see the inset of Fig. 1) used for guided-mode observation.¹¹ Clearly, rays having incidence angles between $\theta_0 - \Delta\theta$ and θ_0 can only give rise to an optical signal detected by the photodiode. Provided that the detector aperture d is small compared to its distance L to the sample exit edge, the angle straightforwardly follows from grating diffraction theory and Snell's law according to

FIG. 1. Measured optical intensity in the $m = +1$ diffracted order with the experimental arrangement given in the inset. Sample under test corresponds to No. 7 of Table I, grating period and working wavelength are $\Lambda = 510.2$ nm and $\lambda = 1.3159$ μm .

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FIG. 2. Calculated optical intensities in the $m = +1$ diffracted order with and without absorption ($k = 2 \times 10^{-4}$, thick line and $k = 0$, thin line, respectively). Bell shape curves account for a Gaussian apparatus response centered at θ_0 with a $\sigma = 0.02^\circ$ standard deviation. Calculations are performed at $\lambda = 1.5577 \mu\text{m}$ assuming a semi-infinite InP substrate whose real part of the refractive index is $n = 1.553$. Grating period and depth are $\Lambda = 530.1$ and 250 nm, respectively.

$$\sin(\theta_0 - \Delta\theta) \approx n \left(1 - \frac{d^2}{2n^2 L^2} \right) - \frac{\lambda}{\Lambda}. \quad (1)$$

A first-order expansion yields an estimate of $\Delta\theta$ (Fig. 1),

$$\Delta\theta \approx \frac{\sin \theta_0 - \sin(\theta_0 - \Delta\theta)}{\cos \theta_0} \approx \frac{d^2}{2 \cos \theta_0 n L^2}. \quad (2)$$

Applying Eq. (2) to our experimental conditions, i.e., $d = 0.5$ cm, $L = 20$ cm, and with the InP substrate of Ref. 1 at $\lambda = 1.5577 \mu\text{m}$, we obtain $\Delta\theta = 6 \times 10^{-3}$ degrees. In practice, the measured peaks are wider as seen in Fig. 1 for a typical measurement at $\lambda = 1.3159 \mu\text{m}$ with sample No. 7. The measured peak extends over $\Delta\theta \approx 0.05^\circ$, corresponding to five steps of the motorized rotating sample holder. Accordingly the standard derivation is $\sigma = 0.02^\circ$. This value is about three times greater than the $\Delta\theta$ -value calculated from Eq. (2). Such a broadening of the response comes from the beam divergence of our illuminating laser, which is of about the same order of magnitude. This 0.02° angle uncertainty corresponds to a 3×10^{-4} uncertainty on the substrate refractive index.

Because of the novelty of our measurement technique, we had to ensure that the above crude ray analysis agrees well with the rigorous electromagnetic theory. Therefore, we calculated the optical intensity in the $m = +1$ diffracted order using Moharam's formalism¹² without ($k = 0$) and with ($k = 2 \times 10^{-4}$) absorption. The results are given in Fig. 2 together with their convolution with a Gaussian experimental apparatus response having the standard deviation $\sigma = 0.02^\circ$. Without absorption, the optical intensity reaches zero with an infinite slope exactly at $\theta_0 = 12.521^\circ$. This angle is directly related to the prescribed substrate index $n = 3.1553$, grating period $\Lambda = 530.1$ nm, and wavelength $\lambda = 1.5577 \mu\text{m}$. Consequently, there is no path for a light ray emerging exactly parallel to the grating surface. The resulting signal accounting for the apparatus response exhibits in that case a maximum noticeably shifted from θ_0 . However, with a realistic absorption of $k = 2 \times 10^{-4}$, the optical intensity decreases smoothly without the previous singular point occurring at θ_0 . Accounting for a Gaussian apparatus response yields a signal with a nearly Gaussian shape whose maximum occurs

TABLE I. Sample characteristics and measured refractive indices at $T = 300$ K.

Sample No.	Doping type	Doping level ($\times 10^{18} \text{ cm}^{-3}$)	$n \sim 1.3159 \mu\text{m}$	$n \sim 1.5577 \mu\text{m}$
1	n.i.d. ^a	$N \approx 0.001$	3.2081	3.1722
2	Fe	$N \approx 0.001$	3.2052	3.1683
3	Fe	$N \approx 0.001$	3.2046	3.1679
4	n.i.d.	$N = 0.0042$	...	3.1701
5	n.i.d.	$N = 0.0054$	...	3.1679
6	n.i.d.	$N = 0.0061$	3.2041	3.1688
7	...	$N = 0.074$	3.2050	3.1685
8	Sn	$N = 1.1$	...	3.1624
9	...	$N = 1.2$	...	3.1622
10	...	$N = 2.0$	...	3.1585
11	...	$N = 3.1$	3.1909	3.1530
12	...	$N = 3.2$	3.1940	3.1553
13	S	$N = 4.0$	...	3.1430
14	...	$N = 4.4$	...	3.1461
15	...	$N = 5.2$	...	3.1455
16	S	$N = 8.2$	3.1671	3.1251
17	S	$N = 8.5$	...	3.1268
18	S	$N \approx 10$	...	3.1170
19	S	$N = 12$	3.1638	...
20	Cd	$P = 1.7$	3.2062	3.1634
21	Zn	$P = 3.6$	3.2021	3.1616

^aEpitaxial layer grown by MBE (see Ref. 1).

at 12.513° . The systematic error made by assuming that the substrate index corresponds to this 0.008° -shifted peak maximum is much smaller than the angle accuracy of 0.02° quoted above. Consequently, this method appears to be the best tradeoff between accuracy and simplicity.

Gratings were defined on each substrate sample by means of a holographic technique using calibrated fringes obtained with an argon laser. They were further etched in a Plasmalab 80 chamber with SiCl_4 . Resulting grating profile was therefore nearly square. Calibration procedures either in gas flow and rf power have allowed a perfect control of grating depth that was always kept close to ≈ 100 nm. Owing to this low value, we could eliminate any secondary effect resulting from deep gratings. Experiments were conducted with two distributed feedback (DFB) semiconductor lasers emitting at 1.5577 and $1.3159 \mu\text{m}$. Both these devices were operated at a ≈ 1 mW output power that corresponds to the calibrated wavelength given above. The sample temperature was stabilized at 300 K using a thermoelectric cooler, ensuring a negligible sample heating. As is well known, this is required for accurate index measurements.^{1,2}

Experimental results are listed in Table I and plotted in Fig. 3. The data of sample No. 1, shown in Table I for comparison, are taken from Ref. 1. This value, in fact, has been measured using the conventional grating coupling technique and belongs to the previously mentioned nonintentionally doped MBE grown InP layer on an $N = 3.2 \times 10^{18} \text{ cm}^{-3}$ doped substrate. The mean accuracy of our refractive index data from the modified grating-coupler method described here is estimated to be $\approx 5 \times 10^{-4}$ when all contributions are considered. The doping level was measured from the Hall effect on all samples. The typical accuracy on this measurement being estimated to be of the order of $\approx 20\%$. For the three nonintentionally doped samples No. 1–3, we have as-

FIG. 3. Measured InP refractive index as a function of doping level and type for the two wavelengths used in experiments. Square: $\lambda=1.3159 \mu\text{m}$, circles: $\lambda=1.5577 \mu\text{m}$, filled symbols: N doping, open symbols: P doping. Dark and gray solid lines are guidelines to the eyes for N and P doping, respectively.

sumed a doping level of $N=10^{15} \text{ cm}^{-3}$. It appears from Table I that InP samples having very similar doping levels exhibit the same refractive index values to within a few times 10^{-3} . This discrepancy is probably a consequence of substrate manufacturing conditions and/or the nature of dopant atoms and/or different densities of growth-induced defects. One can expect that a more accurate determination of the free-carrier concentration together with a thorough knowledge of the sample crystalline quality would further reduce data dispersion. Nevertheless, the most important conclusion of our study is the large decrease of the n -value, $\sim 4.5 \times 10^{-2}$ at $\sim 1.3 \mu\text{m}$ and $\sim 5.5 \times 10^{-2}$ at $\sim 1.5 \mu\text{m}$, when the N -doping is increased from a low level (non-intentionally doped, $\sim 10^{15} \text{ cm}^{-3}$) up to about $\sim 10^{19} \text{ cm}^{-3}$. Also worth indicating is the reduced effect in the case of P doping.

A simple comparison of our results with those previously published, both experimental⁵⁻⁷ or calculated,⁸ show a general agreement. The overall tendency for a rapid decrease of n as soon as doping level exceeds 10^{18} cm^{-3} can be clearly evidenced from Fig. 3. Its origin is mainly the intra-

band free carrier absorption also known as the plasma effect.^{5,6,8} From a quantitative point of view, we can compare our measurement results to calculations. For instance at $\lambda=1.5577 \mu\text{m}$, the mean measured decreases of n -values are 7×10^{-3} , 3×10^{-2} , and 5.5×10^{-2} for $N=10^{18} \text{ cm}^{-3}$, $N=3 \times 10^{18} \text{ cm}^{-3}$ and $N=10^{19} \text{ cm}^{-3}$, respectively. At the same doping levels, the calculations reported in Ref. 8 predict 7×10^{-3} , 3×10^{-2} , and 8×10^{-2} , respectively. Although a slight discrepancy appears at the highest doping level, this very good agreement comforts the model proposed in Ref. 8.

In conclusion, accurate measurements at $1.3159 \mu\text{m}$ and $1.5577 \mu\text{m}$ of InP refractive indices have been performed as a function of doping type and level. A new measurement method derived from the grating coupler technique has been applied to 21 samples. This large number of samples covering a wide range of free-carrier concentrations must be of great use to those involved in the design of InP-based photonic devices operating in the $1.3\text{--}1.5 \mu\text{m}$ wavelength range.

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