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Investigation of Isoelectronic Impurity Absorption in n-GaP

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The momentum conserving indirect excitonic transitions from the valence band maximum to the conduction band minima close to the X_1 points in the Brillouin zone have been investigated for moderately doped n-type GaP using a novel depth- and time-resolved excess free-carrier-absorption (FCA) technique. Different excitonic singularities due to band-to-band and excitonic absorption are observed near the indirect band gap edge at 77 K. Observations clearly yield signatures for sharp no-phonon absorption lines of excitons bound to neutral sulfur (S) and isoelectronic nitrogen (N) impurities, their complexes and phonon replicas. A parallel investigation also reveals emission lines for exciton bound to S and N as well as to their phonon sidebands. The characteristic energies obtained in our experiments are compared with those reported in literature. Optical nonlinearity at the principle no-phonon exciton absorption A line of isoelectronic nitrogen N is observed.

Keywords: fundamental absorption, excitons, isoelectronic S and N impurities, band structure and phonons, carrier recombination and trapping.

INTRODUCTION

A number of its unique material properties and physical characteristics singles out tetrahedrally coordinated gallium phosphide (GaP) from other crystals. The band gap of GaP is equal to 2.33 eV at zero temperature; the indirect conduction band minima is along (100) direction close to the *X* points of the Brillouin zone. Conduction band exhibits so called *Camel's Back* feature [1 – 3]. As in other indirect semiconductors, optical absorption edge can be characterized by a typical phonon-assisted step-like behavior in nominally undoped GaP crystals. However, optical properties change considerably if GaP crystal is doped by nonisovalent impurities from IV group (C, Si), from VI group (O, S, Se) and, particularly, by isovalent impurities from the V group, such as N and Bi. In contrast to monotype semiconductors Ge, Si, the doping by these impurities in GaP not only generate a series of hydrogen-like donor-acceptor (DA) bound states in the host semiconductor but also creates a very effective bound exciton states. This is produced by the interplay and the change of the charge distribution of the short-range impurity potential, the adjacent strain field and the electronic polarization [4 – 8]. For this reason isovalent N and Bi impurities sometimes are viewed as an alloy even in the case of a very dilute doping. It was discovered that N doping leads to a quasidirect band gap emission with enhanced optical functionality. From the other hand, for sufficient quantities of N impurity, it exhibits transform of the impurity band and performs a huge band gap bowing in mixed GaPN and GaPAs compounds [9 – 10]. The striking difference from the conventional behavior of donors and acceptors is one of the reasons for the great interest in GaP investigations that have enriched the physics of semiconductors considerably. Despite a more than 30 year study of the GaP:N system, the key problems of the isovalent centers have still remained an unsolved mystery. Particularly the detail lattice structures of these states are not known [8]. From now on, novel investigation of isoelectronic impurity systems can disclose quantitative basis for understanding and open new ways to control the color and efficiency of GaP for light emitting diodes and lasers.

The present work addresses the study of fundamental absorption of moderately doped n-GaP wafers with isoelectronic impurities. We employ a novel time- and depth-resolved free carrier absorption (FCA) technique to extract important details in the absorption spectrum. The same technique has been recently proposed by our group to study nominally undoped and doped Si and 4H-SiC semiconductors [11, 12]. The concept assumes that fundamental absorption coefficient, α_{bb}, can be distinguished from the nonequilibrium free-carrier density distributions when absorbed quanta generate e-h pairs. The method requires employment of the impulsive laser excitation at a variable wavelength and a fast excess carrier density probing [13]. The major advantages of the method are as follows. (i) A rather wide range of fundamental absorption in a material can be detected by using so called perpendicular pump-probe geometry. Albeit indirectly, α_{bb} values in the range as low as 10^{-2} cm^{-1} and as high as 10^{3} cm^{-1} can be easily extracted with high accuracy. (ii) The measurement can be performed independently of a rather strong background of absorption caused by equilibrium free carriers, extrinsic donor-acceptor (DA) or intrasubband absorption. Later processes do not alter the number of excess pairs and are relatively invisible in FCA signals on a sub-to-nanosecond time scale.

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SAMPLES

The studied GaP samples were commercial moderately doped double side optically polished n-type wafers of the 350 μm thickness and (100) orientation. They were deliberately doped by S and N to different levels and also contain unknown amount of other donors and acceptors.

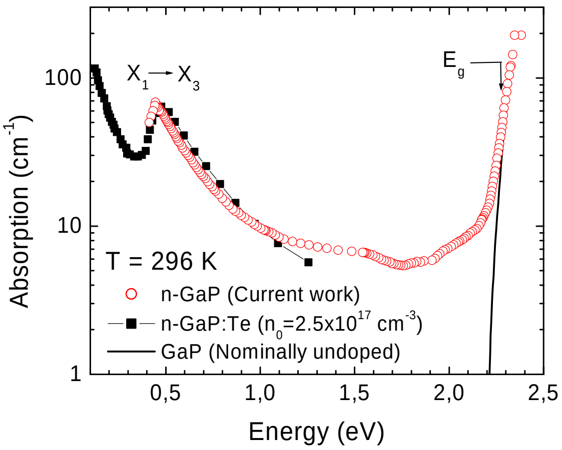

Fig. 1. The absorption spectra of investigated moderately doped n-GaP wafers (open circles) in comparison with a typical n-GaP:Te (solid squares) [15] and a nominally undoped GaP sample (solid line) [2] at room temperature. Energy of indirect band gap *Eg* and the peak caused interconduction free-electron transitions are indicated

Fig. 1 shows absorption spectra at 296 K obtained from the conventional transmitted light measurements by a double beam spectrometer at the normal incidence angle. The absorption coefficient is calculated assuming multiple internal reflections and taking into account reflectivity variation with photon energy from published indexes of refraction data [14]. The absorption values in the infrared range are compared with a typical n-GaP:Te sample shown by solid squares. The characteristic absorption peak at 0.45 eV arises due to free electron vertical transitions between conduction subbands at X_1–X_3 points of Brillouin zone [15]. The similar height of this peak in our wafers allows us to estimate uncompensated free electron concentration $n_0 = 2.5 \cdot 10^{17}$ cm^{-3}.

Another characteristic observed feature is an absorption plateau below the indirect band gap energy in the range 1.3 – 2.1 eV. It shows that samples actually contain compensated donors and acceptors. The absorption generally can be ascribed to nearest-neighbor impurity related absorption bands like O (at 1.75 eV), Zn–O complex (at 2.1 eV) Cd–O complex and other inadvertent impurities (e.g. Si, Cu, S, and C) [16]. The large degree of DA absorption in GaP was previously attributed to enhanced phonon-coupling oscillator strength and, in a few cases, to bound excitonic effects [16]. The DA impurities produce the smoothing of the lower-energy side of the band edge and the higher-energy side of the free-electron spectrum.

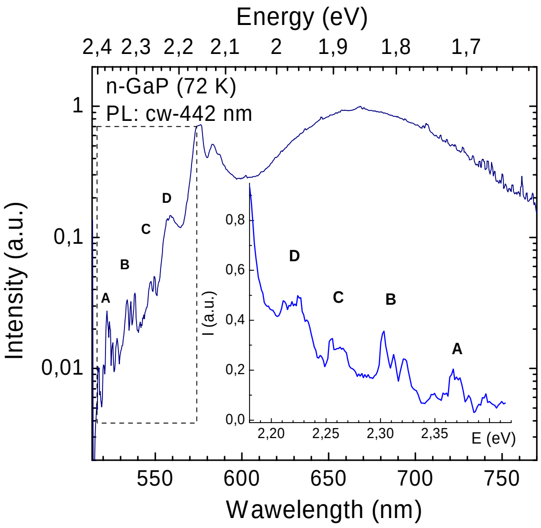

Fig. 2. Spectrally normalized and stray-light corrected PL spectrum of investigated n-GaP wafers under 442 nm cw excitation at 72 K. The inset shows near band-edge emission region

Fig. 2 shows spectrally normalized and corrected photoluminescence (PL) spectrum at 72 K obtained from our samples under cw-excitation with HeCd laser line. The broad emission band at 1.8 – 1.9 eV can be attributed to various deep DA pairs (for example, Zn,O), the bands at 2.13 eV and 2.16 eV to emission of Si–S, S–C, Si–C, Zn–S pairs and their replicas [17, 18]. In the near band-edge emission region (see the inset) we attribute the peaks labeled D at ~2.22 eV and C at ~2.26 eV to S and N phonon replicas, respectively [19]. The peaks labeled B are related to no-phonon S and N excitons [5], while the peaks labeled A are probably due to emission into excited levels of S and N with zone center phonons [6]. It is clear, however, that the richness of PL spectrum structure prohibits separation of the no-phonon sharp bound-exciton lines related to isoelectronic N and S impurities.

PRINCIPLE OF THE FCA TECHNIQUE

Let us assume that a monochromatic beam pulse of pump light of intensity I'_{ex} is normally incident on the flat surface of a semi-infinite solid. A fraction *R* of the light will be reflected from the surface, where *R* is the reflectivity of the medium. The remaining part of the light will penetrate into the medium, and the intensity of the light at the surface will be $I_{ex}(0) = (1 - R)\, I'_{ex}$. As the light travels through the medium, it interacts with it, exchanging energy, and the intensity at a distance *z* from the surface is expressed as $I(z) = I_{\mathrm{ex}}(0) \cdot \exp(-\alpha_{tot} z)$. Here α_{tot} is the total absorption coefficient for all available absorption processes, i.e. the interband fundamental and all extrinsic optical transitions such as doping related free-carrier or impurity-to-band. Let us assume that a single type carrier, which is excited to the band by extrinsic optical transitions do not create exciton and return its energy to the lattice during relaxation time much faster the total duration of the pump pulse. The instant excess *e-h* pairs concentration ($\Delta n = \Delta p$) thus is created only by the fundamental band-to-band absorption process. The concentration follows the profile of local light intensity in the medium [13]:

$$\Delta n(z) = \Delta n(0) \cdot \exp(-\alpha_{tot} z)\,; \qquad (1\ \mathrm{a})$$

$$\Delta n(0) = (1 - R)\left(\frac{I_{ex}(0)}{\hbar\omega}\right)\alpha_{bb}\,. \qquad (1\ \mathrm{b})$$

In Eqs. (1) it is assumed low photon re-emission along the optical path of the incident light. This condition is well satisfied for an indirect semiconductor due to a weak coupling to the radiation field (weak emission rate).

If an excess pair depth-distribution $\Delta n(z)$ is detected, there are two ways to extract α_{bb}: i) from the exponential profile according Eq. (1 a), or ii) from the excess carrier concentration $\Delta n(0)$ at the illuminated surface according to Eq. (1 b). The first way is a good approximation in the case of intrinsic or nominally undoped material when a condition $\alpha_{tot} \cong \alpha_{bb}$ is satisfied easily. An advantage of this way is that only a measurement of the carrier concentration distribution in arbitrary units is needed. Limitations, nevertheless, arise for small α_{bb} values, where exponential profiles (slopes) become too obscure to be resolved experimentally [11, 12]. The second way is valid for both intrinsic and extrinsic material. Moreover, this approach substantially lowers the detection limit of α_{bb}. As shown by Eg. (1 b) extraction of absolute α_{bb} values requires knowledge of the pump intensity value $I_{ex}(0)$. This requirement, however, can be avoided using relative $\Delta n(0)$ measurements at various excitation wavelengths with a constant photon density and providing calibration to a known α_{bb} value in the high energy region of the spectrum [12]. To satisfy the semi-infinite sample approximation in Eq. (1) one needs to neglect multiple reflections if absorption is week. This is obtained by sharpening of the back side of the samples and was justified for all cases in the present experiment.

EXPERIMENTAL

In the time- and depth-resolved FCA experiments, the n-GaP samples were excited from the lateral cross-cut side as shown in Fig. 3. The excitation was performed with a 2.5 ns duration pulse from a tunable wavelength optical-parametric-oscillator pumped by a Nd:YAG laser (Coherent *Infinity*). Basically, the whole lateral side is homogeneously excited in order to minimize the effects of spatial averaging. The injected excess carrier concentration was monitored using a stable cw IR probe beam (λ_{pr} = 1.3 μm) of a weak power LED (4 mW), which propagates into the sample parallel to the excited surface. The probe beam is *p*-polarized and strikes the sample at an angle incidence close to the Brewster angle φ_B to avoid multiple reflections. During the measurement samples were placed on a cold finger of optical cryostat (Oxford) translatable by a μm-step motor, enabling measurements of the depth profile of the excited carrier distributions along the *z*-direction perpendicular to the excited surface. The spatial resolution is defined by the probe beam focusing within the sample and reaches 5 μm [13]. Photo-induced transients of the probe intensity were recorded by a 0.5 ns rise-time amplified InGaAs photo-receiver with subsequent signal processing by a digital 1 GHz bandwidth oscilloscope (LeCroy 9347).

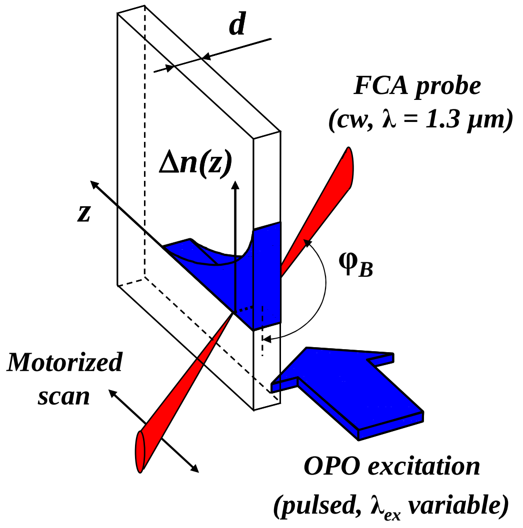

Fig. 3. The pump-probe geometry of FCA experiment

Excited carrier concentration was determined from the recorded transient of the probe beam intensity according to the relation:

$$\Delta n(t) = (d\sigma_{eh})^{-1} \ln[I_p(0) / I_p(t)]\,, \qquad (2)$$

where *d* is the sample thickness along the probe path and $I_p(0)$ and $I_p(t)$ are transmitted probe intensities before and after excitation pulse, respectively. The parameter σ_{eh} represents the FCA cross-section, which is specified to be $5 \cdot 10^{-17}$ cm^2 for GaP [15]. The detected $\Delta n(t)$ transient were obtained by averaging about 10^3 signal traces. The typical absolute error of measured fundamental absorption coefficient is 5 %.

RESULTS

A typical carrier density decay record in n-GaP is presented in Fig. 4. The photon energy of 2.5 eV is chosen to exceed the excitonic band gap (E_g = 2.322 eV for 77 K), however, similar results are obtained in a wide spectral range of energies. Only a slow decay component was observed for moderately and a week injection levels. A characteristic lifetime of the slow component increases from 10 μs to 10 ms with decreasing temperature. The amplitude of slow signal linearly increases up to the free carrier density of about $2 \cdot 10^{16}$ cm^{-3} and then saturates. A fast transient is observable at higher injections. The characteristic decay time of this component is 4 ns and is nearly independent of the temperature and the excitation intensity.

Typical depth profiles of the excess carrier concentration from the slow time decay component are shown in Fig. 5. The optical excitation pulse impinged into the sample from the left side. With increasing the excitation pulse intensity the carrier concentration distribution shifts deeper into the sample. Evidently, carrier concentration saturates at $4 \cdot 10^{16}$ cm^{-3}. For lower intensities, a characteristic distribution slope can be extracted, which, according to Eg. (1 a) is caused by an exponential factor $(-\alpha_{bb} \cdot z)$. In Fig. 5, the slope with a value α_{bb} = 120 cm^{-1} is detected for 2.48 eV photon energy. The $\Delta n(0)$ concentration (extrapolated to the depth = 0) is proportional to the incoming intensity, as prearranged by Eq. (1 b).

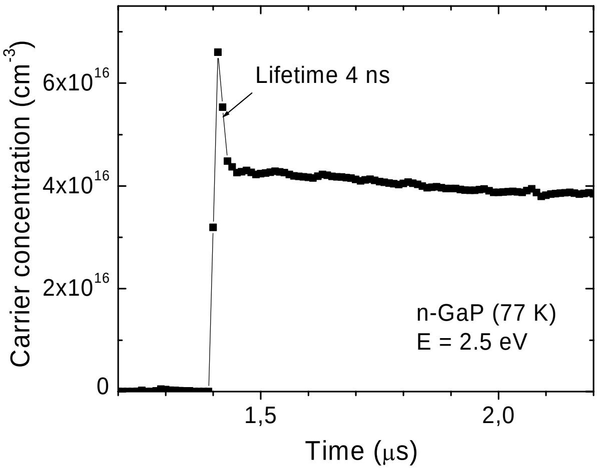

Fig. 4. Typical excess carrier decay obtained in n-GaP wafer at 77 K after 2.5 ns pulse excitation with above band gap energy (2.5 eV). Lifetime of the fast component is indicated

These results we explain in the following way. Free-carrier pairs are photogenerated through the fundamental band-to-band absorption processes including bound excitons to impurities. The minority holes are quickly trapped by defects, which possesses large capture cross section in the minority half of the band. The defects are negatively charged before capture, i.e. acceptor-like. There is strong evidence that such defects often dominate the recombination and hence controls the minority carrier lifetime in n-GaP [20]. From the saturation of the slow component we estimate trap defect density to be $4 \cdot 10^{16}$ cm^{-3}. Using the published correlation between trap concentration and the trapping lifetime (e.g. Fig. 8 in Ref. [20]), we count the hole lifetime to be of about 10^{-10} s. This trapping lifetime is sufficiently shorter of the excitation pulse duration used in the measurement. So, the generated electrons do not recombine during the excitation pulse and their concentration is proportional to α_{bb} value. At higher injections, the above described hole traps become filled and the generated but not captured free holes recombines with electrons through other deep recombination centers (probably of O origin [20]). They are acceptor-like and free holes are needed for efficient electron recombination with characteristic lifetime of 4 ns.

The α_{bb} values obtained in the current work have been extracted from the slow decay component amplitude at excitation levels not exceeding $2 \cdot 10^{16}$ cm^{-3}. Absorption values greater than 20 cm^{-1} were extracted from the depth profiles of the excess carrier as shown in Fig. 5 (the first approach discussed above, Eq. (1 a)). While for lower absorption α_{bb} values were counted from the $\Delta n(0)$ quantity (the second approach, Eq. (1 b)) providing careful calibration to absorption values in the high absorption region of the spectra (α_{bb} >> 20 cm^{-1}) [11, 12].

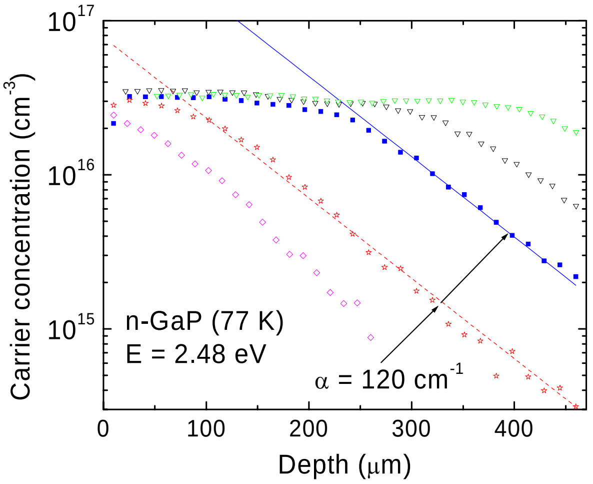

Fig. 5. Depth-resolved FCA concentration measurements in n-GaP wafer excited with 2.48 eV pulses obtained with five different initial excitation intensities. The optical excitation pulse impinged onto the sample from the left. FCA concentration is recorded 25 ns after ending the excitation pulse (in the slow decay component). Two lines indicate characteristic carrier profile with an absorption coefficient of 120 cm^{-1}

In Fig. 6 (a) the obtained absorption spectrum (77 K) is displayed in comparison with published ones for heavily doped GaP:N [4] and nominally undoped GaP [2, 3]. The zoom-in portion of those spectra is shown in Fig. 6 (b). Below the indirect excitonic band gap $E_g(X_1)$ we were able to extract absorption tail up to the value of α_{bb} = 0.03, more than an order below previously determined absorption in doped GaP wafers. The characteristic shape of the observed features provides a clue to understanding of their nature. Phonon-assisted steps of TA, LA and LO phonons determines the absorption in undoped sample. In doped samples, several sharp structures are seen at or below the band gap. It is clear that absorption processes, forbidden in the perfect GaP crystal, are possible in the vicinity of N and S atoms. The line A is related to no-phonon excitonic absorption at nitrogen substituting phosphor atom [4]. In this process crystal momentum is conserved by the impurity itself. Indeed, the cross section for this process is reasonably large. Each N atom also induces about 7000 times more free particle absorption at ~0.04 eV above $E_g(X_1)$ than the phosphorus atom it replaces. Also rich phonon structure is seen above the A absorption line. The line A_x is denoted to no-phonon creation of a free indirect exciton. Due to momentum conservation reasons this transition is forbidden in a perfect crystal but becomes partly allowed because nitrogen perturbs the translation symmetry of the lattice [4, 7].

In our wafers we clearly observe A line. From linear correlation between absorption in the peak of the A line and N concentration [21] (α_{bb} = 4900 cm^{-1} for N = $7 \cdot 10^{18}$ cm^{-3} in doped sample [4]) we established nitrogen concentration in our wafers to be about $5 \cdot 10^{16}$ cm^{-3}. Other prominent structures in our wafers are S_0 and S_0' peaks

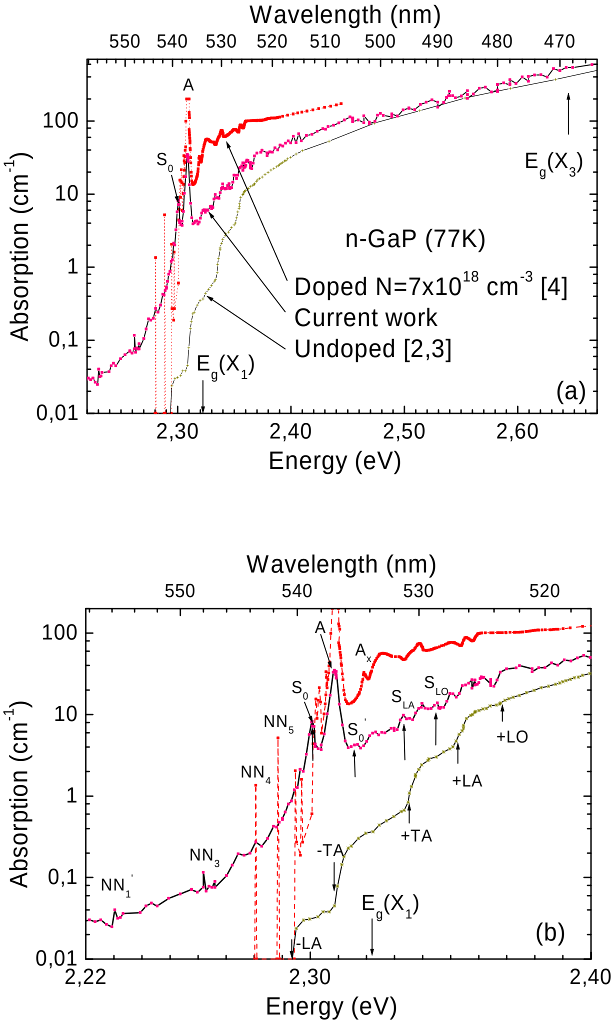

Fig. 6. (a) Absorption spectrum obtained in n-GaP wafers at 77 K (current work) in comparison with absorption of heavily N doped GaP [4] and nominally undoped GaP [2, 3]. The spectrum of GaP : N was measured at 2 K and is shifted in the plot to the left by 8.8 meV to align the same excitonic band gap value $E_g(X_1)$ for 77 K [3]. Band gap energies and two peaks of nitrogen (A line) and sulfur (S_0) impurity bound excitons are indicated. The absorption peak at A line in heavily doped sample reaches 4900 cm^{-1} and is not displayed in the plot as a whole. (b) The zoom-in spectra in the lower energy region. The characteristic features related to different phonon involvement in nominally undoped GaP and to various NN_i complexes and S phonon-assisted replicas are indicated

denoted to no-phonon ground and first excited exciton state at neutral S donor, respectively [5, 7]. Extra absorption in comparison with undoped GaP also occurs above $E_g(X_1)$ including features observed in heavily N doped GaP. In addition to this, in the first 0.05 eV above the S_0 absorption line, the structures marked S_{TA} and S_{LO} could be indicated as phonon-assisted transitions to the S_0 bound exciton previously observed in Ref. [5]. However, we do not observe indirect A_x exciton possibly because nitrogen concentration is too low. The additional absorption at high energies is due to presence of various replicas overlapping with excitonic continuum. We observe that deviation from undoped GaP sample increases also around the second indirect band gap $E_g(X_3)$ (see Fig. 6 (a) the top-right corner).

In heavily N-doped GaP specimens, some excitons can be bound to the pairs of nitrogen atoms, located sufficiently close to each other. These exciton produce a series of absorption NN_i lines in accordance to possible internuclear distances between nearest neighbors of N atoms. Such structures are clearly visible in doped GaP and few of them (NN_1, NN_3 and NN_4) can be identified in our spectrum in Fig. 6 (b). So far, such high resolution was not obtained in GaP samples with $N < 2 \cdot 10^{17}$ cm^{-3}.

OPTICAL NONLINEARITY

Optical nonlinearity was studied at normal incidence of the laser pulse through the n-GaP wafer. The results are shown in Fig. 7 for two photon energies 2.31 eV and 2.37 eV. These energies have been selected to account the same absorption at 77 K for A peak and in the above band gap region. It is shown that increasing of excitation intensity provides gradual reduction of transmission at 2.37 eV because of the plasma-induced band gap narrowing.

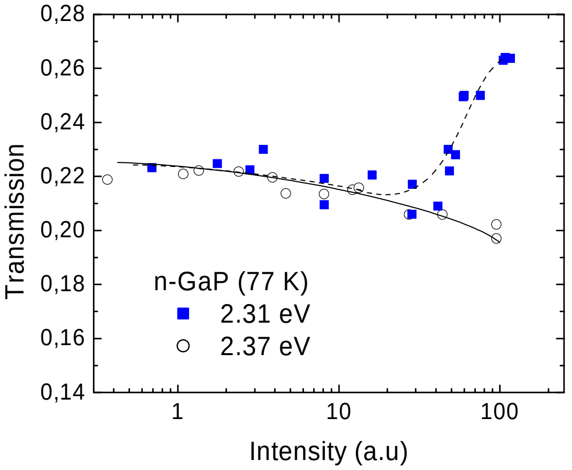

Fig. 7. Transmission of the optical pulses through n-GaP wafers at 77 K as a function of intensity. The photon energy 2.31 eV is tuned to the no-phonon exciton absorption A peak, the energy 2.37 eV is tuned to the same absorption value in the above the band gap region (see Fig. 6 (b)). The lines are guide to the eye

However, we observe quite sharp increase of the transmission at higher intensities with 2.31 eV. This can be explained by a shift of the A peak to lower energies together with overall absorption spectrum. Possibly the bound exciton at N impurity obey high Mott transition density and is stable under plasma injection. This can be used for nonlinear optical elements construction in GaP:N waveguides.

In summary, we have shown effectiveness of FCA technique to study different features and optical properties of fundamental absorption in n-GaP crystals.

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