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Comparison of the results of optical and electrophysical measurements of the concentration of conduction electrons in n-InSb samples

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The infrared reflection spectra of *n*-InSb single-crystal samples doped with tellurium were studied at room temperature. Using dispersion analysis, the spectral dependences of the real and imaginary parts of the permittivity were obtained and the loss function was constructed. The values of the characteristic wave number corresponding to the high-frequency plasmon-phonon mode were determined and the values of the optical electron concentration, N_{opt}, were calculated. Electrophysical measurements were performed on the same samples using the man der Pauw method at room temperature and the values of the Hall concentration, N_{Hall}, were determined. It was shown that for all the studied samples, the optical concentration exceeds the Hall concentration. It was suggested that the surface layers of the samples are enriched with free electrons. The thickness of the surface layer of the sample, in which the reflected light signal is formed, was estimated and shown to be no more than $1\,\mu$m.

Keywords: *n*-InSb, n-InSb, reflectance spectra, plasmon-phonon interaction, van der Pauw method, free electron concentration.

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Introduction

Studies have been carried out at JSC „Giredmet“ for a number of years aimed at developing a non-contact and non-destructive method for determining the free charge carriers concentration (FCCC) in various semiconductor materials. The method is based on the mathematical processing of the reflectance spectrum recorded in the mid-infrared (MIR) and far infrared (FIR) regions. The dependencies of real ε_1 and imaginary ε_2 components of complex dielectric constant$\varepsilon = \varepsilon_1 + i\varepsilon_2$ on the wavenumber were calculated using the dispersion analysis based on the experimentally found reflectance spectra; after that the so-called „loss function“ was plotted:

$$LF = \operatorname{Im}(-1/\varepsilon) = \frac{\varepsilon_2}{\varepsilon_1^2 + \varepsilon_2^2}.$$

Next, the value of the characteristic wavenumber ν_+ was determined to find FCCC based on numerical calibration curve. Though this method for determination of FCCC was developed for $T = 295$ K, it can be applicable for other temperatures, as well, e.g.,$T = 77$ K.

Since in semiconductor compounds A_2B_6 and A_3B_5 there is a noticeable proportion of ionic bond, in calculations it is necessary to allow for the interaction of plasma vibrations with longitudinal optical (LO) phonons (plasmon-phonon interaction). In this case, instead of earlier existed independent vibrations (plasmons and LO-phonons) the mixed plasmon-phonon modes with frequencies of ν_- (low-frequency, primarily phonon) and ν_+ (high-frequency, primarily plasmon) occur [1,2]. To find FCCC the high-frequency mode ν_+ was used with a large contribution of plasmonic vibrations the frequency of which directly depends on FCCC value. The values ν_- and ν_+ were identified with the wavenumbers corresponding to the maxima of the loss function.

This approach was used to define the concentration of free electrons in *n*-InSb [3], *n*-InAs [4], *n*-GaAs [5], *n*-GaSb [6], as well as holes in *p*-GaAs [7]. If only one type of free charge carriers was identified in the studied samples, the optical data were compared with the results of traditional Hall measurements using Van der Pauw method [3–5]. If the free-charge carriers of two types (*n*-GaSb [6], *p*-GaAs [7]) were found, then, by comparing optical and Hall data it was possible to easily find the mobility ratio of either„light“ and „heavy“ electrons [6], or „light“ and „heavy“ holes [7].

This study continues the investigations previously published. The results of measuring the conductivity electrons concentrations by optical method N_{opt}, and electrophysical method, N_{Hall}, on Tellurium-doped *n*-InSb samples are compared.

Experiment procedure

Single antimonide crystals were grown using Czochralski method. The initial components In and Sb (purity 6N), as well as the doping impurity (Te), were placed in a quartz filter crucible, which was installed in the operating crucible of the growth chamber. InSb compound was synthesized in a filter crucible at a temperature of $\approx 750\,^\circ$C in vacuum. After filtering the melt into a operating crucible, the temperature of the melt decreased to $\approx 525\,^\circ$C. The single crystal was grown on a seed oriented in the crystallographic directions [100] or [111] (table). The grown single-crystal was annealed in the heater zone according to a special thermal condition selected experimentally [8].

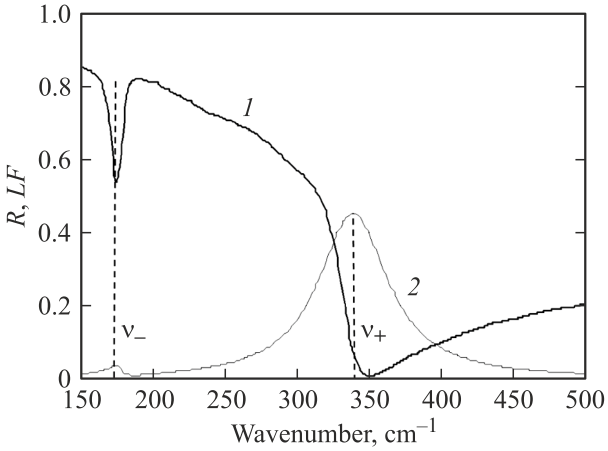
Reference plates were cut perpendicular to the growth axis from the upper and lower parts of the ingot. The plates were ground using M14 powder and etched in a polishing solution to remove the damaged layer. Samples of near square shape with linear dimensions of 8–12 mm were cut out from the plates; samples thickness varied within 0.78–1.98 mm (table). Reflectance spectra were recorded using Fourier transform spectrometer Tensor 27 within the wavenumbers $340 < \nu < 1500\,\mathrm{cm}^{-1}$ (resolution $2\,\mathrm{cm}^{-1}$) and vacuum Fourier transform spectrometer BRUKER IFS66v/s within the wavenumbers $50 < \nu < 1200\,\mathrm{cm}^{-1}$ (resolution $4\,\mathrm{cm}^{-1}$); incidence angle was close to the normal incidence angle and was no more than 13° to the normal.

Electrophysical measurements were carried out using the Van der Pauw method. The contacts were soldered with indium into the end face at the corners of the sample. A holder with two samples attached on opposite sides was placed in the gap between the poles of the electromagnet: the samples were positioned perpendicular to the direction of magnetic field induction. Electric resistivity was measured in the absence of magnetic field, and the Hall coefficient was measured in a magnetic field with induction $B = 0.5\,\mathrm{T}$ and value of current going through the sample of 200 mA.

The reflectance spectra were processed using dispersion analysis. ε_1 and ε_2 versus wavenumber curves were defined, the loss function LF was plotted, the characteristic wavenumber ν_+ was found; after that, using the calibration curve the concentration of conductivity electrons N_{opt} was calculated. The theoretical model used in the calculations, as well as algorithm for determiningN_{opt} are outlined in details in paper [3]. Value N_{opt} was compared with equivalent value obtained through Hall measurements, N_{Hall}.

Experimental results and discussion

The Figure illustrates the typical reflectance spectrum $R(\nu)$ (curve *1*) and the loss function (curve *2*) corresponding to this spectrum; sample №1 — see the table.



The reflectance spectrum of the sample №1 (curve *1*) and loss function (curve *2*). Vertical dashed lines indicate ν_- and ν_+.

As seen from the Figure, the loss function has two maxima: the left (low-frequency), due to the LO-phonon, is weakly pronounced, while the right (high-frequency) is more pronounced. For the mentioned sample $\nu_- = 175\,\mathrm{cm}^{-1}$, and $\nu_+ = 338\,\mathrm{cm}^{-1}$. The value N_{opt} was found from the known value ν_+ using the calibration curve expressed as a cubicle parabola [3]:

$$N_{\mathrm{opt}} = 1.90 \cdot 10^{10}(\nu_+)^3 - 6.90 \cdot 10^{12}(\nu_+)^2 + 3.54 \cdot 10^{15}(\nu_+) - 5.06 \cdot 10^{17}. \quad (1)$$

The table below shows the parameters of the examined *n*-InSb samples, the results of optical and electrophysical measurements, as well as values

$$\delta = \frac{N_{\mathrm{opt}} - N_{\mathrm{Hall}}}{N_{\mathrm{opt}}}.$$

The samples are arranged in ascending order of N_{opt} values.

It should be noted that resolution of the recorded reflectance spectra was no worse than $4\,\mathrm{cm}^{-1}$ (see above), so that a random relative error for calculation of ν_+ doesn't exceed ±1.2% (sample №1, for which $\nu_+ = 338\,\mathrm{cm}^{-1}$). For all other samples, the mentioned error is even smaller. According to specially conducted metrological studies of the Hall methodology, the random relative error of determining N_{Hall} with a confidence probability of $P = 0.95$ does not exceed ±6%. Accordingly, in the table, the values of N_{opt} are given with two decimal places, and the values of N_{Hall} — with one decimal place. The values of parameter δ are rounded.

As can be seen from the table, for all the examined samples $N_{\mathrm{opt}} > N_{\mathrm{Hall}}$, and this difference is irrespective of the electron concentration and crystallographic orientation of the sample and varies widely: from 1% (sample №9) to 23% (sample №5). We observed a similar pattern with respect to the samples of *n*-InAs doped with sulfur and tin. Concentration by optical method was higher than that obtained by the Hall method, and the value δ reached 20% [4].

Parameters of the studied samples

№ No.	Sample thickness, d, mm	Crystallographic position	ν_+, cm^{-1}	Electron concentration, cm^{-3}		δ,%
				N_{opt}	N_{Hall}	
1	1.11	< 100 >	338	$6.36 \cdot 10^{17}$	$5.5 \cdot 10^{17}$	14
2	1.40	< 100 >	351	$7.08 \cdot 10^{17}$	$6.3 \cdot 10^{17}$	11
3	0.89	< 100 >	356	$7.39 \cdot 10^{17}$	$6.5 \cdot 10^{17}$	12
4	1.92	< 111 >	362	$7.73 \cdot 10^{17}$	$6.9 \cdot 10^{17}$	11
5	1.92	< 111 >	386	$9.25 \cdot 10^{17}$	$7.1 \cdot 10^{17}$	23
6	0.86	< 100 >	395	$9.87 \cdot 10^{17}$	$9.2 \cdot 10^{17}$	7
7	0.78	< 100 >	397	$1.00 \cdot 10^{18}$	$9.8 \cdot 10^{17}$	2
8	1.29	< 100 >	408	$1.08 \cdot 10^{18}$	$9.7 \cdot 10^{17}$	10
9	1.36	< 100 >	414	$1.21 \cdot 10^{18}$	$1.2 \cdot 10^{18}$	1
10	0.98	< 100 >	474	$1.64 \cdot 10^{18}$	$1.5 \cdot 10^{18}$	9
11	1.54	< 100 >	502	$1.94 \cdot 10^{18}$	$1.5 \cdot 10^{18}$	22
12	1.55	< 100 >	536	$2.33 \cdot 10^{18}$	$2.2 \cdot 10^{18}$	5
13	1.66	< 100 >	537	$2.35 \cdot 10^{18}$	$2.0 \cdot 10^{18}$	16
14	1.98	< 111 >	555	$2.58 \cdot 10^{18}$	$2.0 \cdot 10^{18}$	22

For n-GaAs samples doped with tellurium and silicon, a reverse pattern was obtained: concentration by optical method was lower than that obtained by the Hall method [9].

Thus, it can be stated that concentrations of the conductivity electrons obtained through optical and Hall methods differ, and the difference can be in both directions depending on the sample material. A fundamentally crucial aspect should be stressed here. Since the reflected light signal is formed in a narrow near-surface layer of the sample, the values N_{opt} obtained from optical measurements will relate specifically to this layer, whereas the values N_{Hall} — will apply to the entire volume of the sample. This may be the cause of the difference betweenN_{opt} and N_{Hall}.

What is the thickness of the near-surface layer? Let's try to define it by assuming that the skin layer acts as the layer in which the reflected light signal is formed. Let's use the known formula here

$$l = c\sqrt{\frac{2\varepsilon_0}{\omega\eta e N\mu}}. \quad (2)$$

Here l — thickness of the skin-layer, $c = 3 \cdot 10^{10}$ cm/s — light speed in vacuum, $\varepsilon_0 = 8.85 \cdot 10^{-14}$ F/cm — dielectric constant, $\omega = 2\pi c\nu(s^{-1})$ — angular light frequency, η — dimensionless permeability of material, $e = 1.6 \cdot 10^{-19}$ C — charge of an electron, N — concentration of conductivity electrons, μ — their mobility.

If we use formula (2) let's select the values of parameters relating to sample №1 (table): $\nu \approx \nu_+ = 338\,\text{cm}^{-1}$ ($\omega = 6.37 \cdot 10^{13} c^{-1}$), $\eta = 1$ (non-magnetic material), $N = N_{\text{Hall}} = 5.5 \cdot 10^{17}\,\text{cm}^{-3}$, $\mu = 2.7 \cdot 10^4\,\text{cm}^2/(\text{V·s})$. Then, according to formula (2) we'll obtain $l \approx 3.2 \cdot 10^{-5}\,\text{cm} = 0.32\,\mu\text{m}$. This is the thickness of layer where intensity of electromagnetic wave drops by $e \approx 2.71$ times. Having taken the thickness of the required layer as $3l$, we'll get that N_{opt} belongs to the sample's near-surface layer with a thickness of about $1\,\mu$m. For other studied samples this value will be even lesser.

It follows from the data obtained that for all the samples studied, the near-surface layer is enriched in free electrons (as well as in the samples n-InAs [4]). Among the possible reasons, two major errors can be listed: topological effects and uneven distribution of the doping impurity across the crystal volume. As for the first assumption, we should pay attention to paper [10], which predicts the occurrence of a topologically nontrivial phase in a stressed InSb. In its turn, doping leads to specific stresses in the material. In our opinion, the assumption of an uneven distribution cannot explain the effect of a different sign in different samples, since when the plate is cut out, a fragment of a single crystal with both increased and decreased concentrations of tellurium may appear on the surface. The available information is insufficient to make any conclusions about the nature of the mentioned layer. This issue shall be studied independently.

Conclusions

1. IR-spectra of n-InSb samples doped with tellurium at room temperature were obtained. Loss functions are constructed using dispersion analysis and the values of the concentration of conductivity electrons are determined. N_{opt}.

2. Electrophysical measurements were performed on the same samples using Van der Pauw method at room temperature and Hall concentrations N_{Hall} were obtained.

3. It is shown that for all studied samples optical concentration exceeds the Hall concentration. An assumption is made that the near-surface layers of the studied samples are enriched with conductivity electrons.

4. The thickness of the near-surface layer of the sample in which the reflected light signal is formed is estimated. It doesn't exceed $1\,\mu$m.

Conflict of interest

The authors declare that they have no conflict of interest.

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