E. M. Gershenzon. *Spectral and Radiospectroscopic Studies of Semiconductors at Submillimeter Wavelengths.*

1. *Introduction.* The submillimeter segment of the electromagnetic wavelength scale (100–1000 μm), which is of substantial interest for radioengineering applications, radioastronomy, and the study of matter, occupies a position intermediate between the longwave infrared and radio bands. Until recently (despite the inroads made by methods and equipment typically used for the optical and millimeter bands), the submillimeter band remained one of the segments for which equipment was poorly developed. In recent years, however, it has been yielding rapidly; for example, the development of monochromatic sources [backward-wave tubes (BWT)][1] has opened fundamentally new possibilities for spectroscopy. The methods usually employed here are optical—the emission of a thermal or gas-discharge source is used and the spectrum is either resolved with diffraction gratings (echelettes) or Fourier-transformed. The spectrometers have relatively low sensitivity and poor resolution. Submillimeter lasers, which combine monochromatic outputs with adequate power levels, are practically unturnable and permit measurements only at fixed frequencies as the magnetic field or other parameters are varied, and this limits research possibilities. BWTs generate radiation at powers of $\gtrsim 10^{-4}$ W and can be tuned in a broad frequency band by varying the supply voltage. This has made possible significant progress in the development of BWT spectral instruments and the investigation of dielectrics,[2] gases,[3] etc.

The present paper is devoted to the results of a study of semiconductors at submillimeter wavelengths using high-resolution BWT spectrometers.[4–14]

We mention first of all some of the aspects from which submillimeter investigation of semiconductors offers promise.

a) Several objects in semiconductors have relatively low binding energies (1–5 meV); therefore their characteristic frequencies fall in the submillimeter band. We refer here, among others, to the entire spectrum of excited states of minor impurities of semiconductors, the binding energies of many impurity complexes, excitons, the activation energies of electron hopping between impurities, which results in conductivity, Zeeman and Stark transitions for excited states of the impurity, etc. Quantum cyclotron and spin resonances, cyclotron resonances of electrons localized on impurities, etc., occur at these frequencies.

b) In the traditional conception, spectroscopy and radiospectroscopy have different fields of application and observing frequencies that are quite far apart. In semiconductors, because of the high permittivities ($\varkappa \gtrsim 10$) and the small effective mass of the carriers ($m^* \lesssim 0.1\ m_0$), these fields of study of the properties of matter at submillimeter wavelengths simply overlap and spectroscopic measurements based on variation of frequency and radiospectroscopic methods, which generally use fixed observing frequencies and magnetic modulation methods, coexist and complement one another. For example, the excited atomic states, Zeeman effect, and others

that typify spectroscopic studies produce the same absorption and conductivity-change effects as the cyclotron and spin resonances usually used in radiospectroscopy.

Such problems of semiconductors physics as carrier heating in the electric field, the action of light, the possibility of emission of high-energy phonons, and several others reappear in a new light under these conditions.

c) In addition to being of independent interest for semiconductors, submillimeter experiments with them are promising as models for the realization of extreme conditions that have not yet been brought about in the laboratory. An example is the action of a magnetic field H on an impurity atom. Even in Ge and GaAs at the quite feasible constant $H \approx 60$ kOe, the equivalent field with respect to the free atom is $H_{eq} \approx 3$ GOe, which is not now attainable even in a pulsed mode; such materials as n-InSb, where the binding energy and effective mass of the electrons are lowest, may become especially promising for experiments of this nature. The problems of investigating the interference of excited atomic states and the influence of H, E, and other factors on it, study of negative ions, etc., can be restated.

d) The practical significance of submillimeter studies of semiconductors can be illustrated even now by such applications as chemical analysis of impurities in semiconductors, determination of various constants for materials, and the development of new types of photoresistors.

2. *Apparatus and methods.* A spectrometer that covers the range from 2000 to 250 μm in the spectrum with several BWT has been developed for the study of semiconductors[5] (Fig. 1). In semiconductors (as distinct, for example, from gases[3]), the narrowest spectral lines have widths $\Delta \mathscr{E} \gtrsim 10^{-3}$ meV even for isolated centers because of the presence of phonons even at helium temperatures, so that the generators do not require special frequency stabilization, and a spectrometer resolution $\lambda/\Delta\lambda = 10^5$ is attained by the use of good power supplies. AFC is used if necessary. Stabilization of radiated power in the tuning range of each BWT is a more difficult matter—the ragged amplitude-frequency characteristic of the BWT is a disadvantage. The radiated power level incident on the sample has
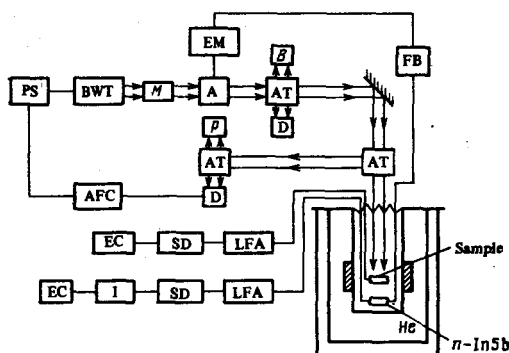

FIG. 1. Block diagram of submillimeter spectrometer for study of semiconductors.

been smoothed by using an attenuator connected into an electromechanical negative feedback circuit. Measurements in the spectrometer are based on the photoconductivity of the sample or on the radiation that it absorbs with the aid of an n-InSb detector placed behind the sample. The sample holder is placed in a helium cryostat with a superconductive solenoid ($H_{max} \approx 60$ kOe). Either the frequency or the magnetic field is scanned. The high-frequency channel is quasioptical.[15] Polarization measurements (including circular polarization), illumination of the samples, application of uniaxial compression P and an electric field E to them, variations of the temperature T, and position adjustments of the samples are provided for. Photoconductivity measurements are made by the standard modulation procedure with synchronous detection; small absorption coefficients (below $\alpha \approx 10^{-5}$ cm^{-1}) are measured by double modulation of the magnetic field with subsequent retrieval of the shape of the spectrum from the derivative signal by an integrator; if necessary, both the photoconductivity and the absorption spectra can be recorded in a single experiment. The sample can also be placed in an open cavity; autodyne reception can be used to obtain especially high sensitivity.[13]

Samples of Ge, Si, GaAs, and n-InSb were used in the experiments as technologically fully mastered materials with which various weakly bound states in semiconductors can be studied.

3. *Impurities in* Ge *and* GaAs.[5] By investigating hydrogen-like impurities in semiconductors, we can not only study the impurity atoms, but also obtain valuable information on the crystal itself.

In contrast to the spectral procedures generally used in the infrared band, in which the electron transitions from the ground state of the impurity atom to excited states are studied, we have usually investigated transitions between excited states. The only exceptions were the experiments with n-GaAs in a magnetic field, where transitions from the ground state also fall in the range of the spectrometer used. In most experiments, the spectra were registered with the photoconductivity due to photothermal ionization[16] of excited states.

By way of example, Fig. 2 shows a segment of the photoconductivity spectrum of Sb in Ge, and Fig. 3 a diagram of the measured transitions between excited states for this impurity. Naturally, identification of the
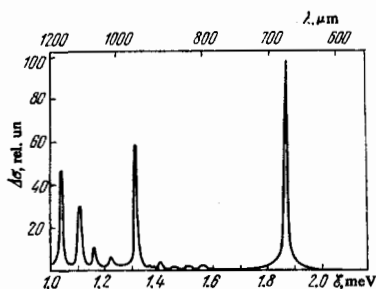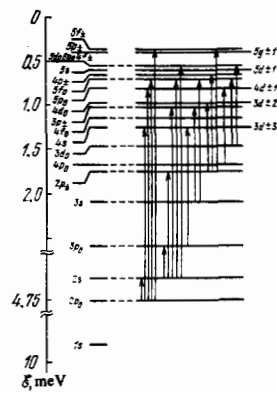

FIG. 3. Energy diagram of the spectrum of Sb in Ge. Left—calculation[27]; center—identified transitions; right—designations of levels obtained experimentally.

transitions becomes more difficult here as compared to the three series, but the opportunities for measurements are also greater in the rich spectrum. Their principal results boil down to the following. We determined the energy spectra of many excited states for various types of donors and acceptors in Ge, studied their Zeeman splitting in a broad range of H from the hundreds of oersteds to 60 kOe, the anisotropy of the Zeeman effect, and the effects of pressure, temperature, illumination, and electric fields on the population of the states; we investigated the intensities and widths of the transition lines and their variations with impurity concentration and experimental conditions, and studied the interaction of the states. Several experiments were made not only in photoconductivity, but also in absorption. Comparisons with theory were made where possible. We also made a detailed study of the submillimeter spectra of impurities in GaAs and compared the experimental results with calculation.

As examples that illustrate the possibilities of the method, Fig. 4 presents the results of a study of the interaction of impurity states in Ge, while Fig. 5 shows the energy spectrum of n-GaAs in a magnetic field. Among other things, we see from the latter figure that GaAs is an excellent model material for study of atoms in strong fields.

Several features of the photoconductivity and absorption spectra due to transitions between excited states

FIG. 2. Segment of photoconductivity spectrum of Ge:Sb at $T = 7\,°K$.
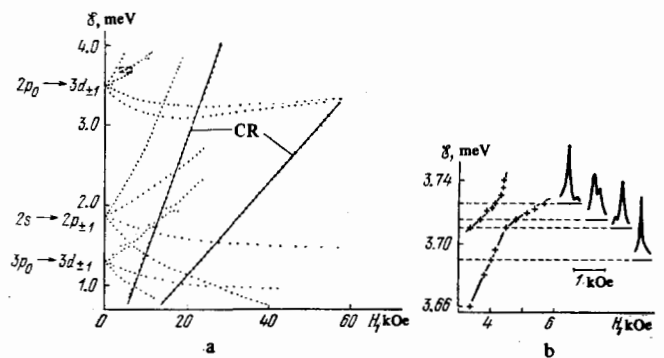

FIG. 4. Transition energy vs magnetic field for three lines of the spectrum of Sb in Ge ($H \parallel \langle 111 \rangle$) (a). (b) shows an enlarged portion of the diagram in Fig. (a) and the shape transformation of the spectrum in the level-anti-intersection region.
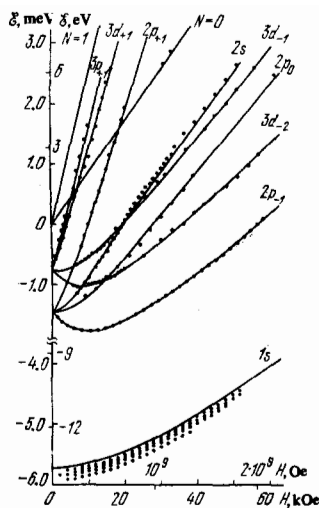
FIG. 5. Energy of donor levels in GaAs vs magnetic field (plotted points) (the solid lines represent the calculation of [28] for the hydrogen atom).

FIG. 7. Dependence of shape of Si:B conductivity spectrum on cencentration of B (cm⁻³): 1—3 · 10^{14}, 2—6 · 10^{14}, 3—1.5 · 10^{15}, 4—3 · 10^{15}, 5—5·10^{15}, 6—8 · 10^{15}, 7—1.2 · 10^{16}, 8—3 · 10^{16}, 9—6 · 10^{16}, 10—10^{17} cm⁻³ at $T = 1.5$ °K ($N_A/N_D < 0.01$).

were observed, and their variations with the chemical nature of the impurity were established; in some cases, significant adjustments were made to literature data and the ranges of validity of existing calculations were determined, etc.

4. **H⁻ centers in Ge and Si.** [6] The problem of negative ions (for example, H⁻) is attracting considerable interest in view of their role in astrophysics, gas-discharge physics, etc. In semiconductors with hydrogen-like impurities, capture of an extra carrier by a neutral impurity center may result in formation of so-called D⁻ (A⁺) centers—negatively charged donors (positively charged acceptors), which are analogs of H⁻. They have a binding energy $\mathscr{E}_i \approx 0.05\mathscr{E}_0$, where $\mathscr{E}_0$ is the ionization energy of the neutral center, i.e., their concentration should be quite high at sufficiently low temperatures ($kT < \mathscr{E}_i$) under the conditions of intrinsic or impurity excitation of free carriers. Peculiarities of the luminescence, scattering, and recombination of free carriers associated with the presence of such centers have been reported in several papers. We performed direct experiments to determine $\mathscr{E}_i$ in Ge and Si with various impurities (the values of $\mathscr{E}_i$ range from 1–3 meV). They are based on determination of the longwave limit of photoconductivity due to photodetachment of carriers from H⁻ centers by submillimeter radiation. The first measurements were made with BWTs; later, they were continued on grating spectrometers. The observed photoconductivity was investigated most thoroughly in Si:B, where there are no other photoconductivity mechanisms in a broad frequency range near $\mathscr{E}_i$. By way of example, Fig. 6 shows a photoconductivity spectrum of Si:B at $T = 1.5$ °K under monopolar excitation conditions at a
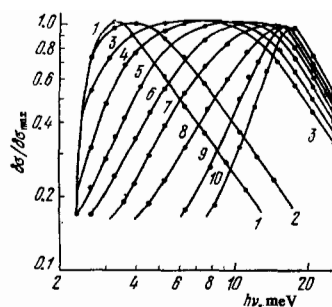
B concentration of ~ 10^{14} cm⁻³. It was shown that the concentration of the A⁺ centers increases exponentially with decreasing temperature; we studied the increase in the concentration of these centers with increasing excitation level, their impact neutralization in an electric field, the influence of a compensating impurity, and various other effects. For example, it was established that the binding energy of the A⁺ centers increases with increasing impurity concentration due to the field effect of the negatively charged acceptors and hopping of holes over neutral impurities (Fig. 7). Subsequently, other authors also undertook to study photoconductivity due to H⁻ centers. [17,18]

5. **H₂⁺ centers.** [7] In lightly doped (Na³ ≪ 1, where N is the concentration of the donors and a is the Bohr radius of the electron on the donor) and strongly compensated semiconductors ($1 - K \ll 1$, where K is the degree of compensation), the energy of electrons localized on impurities depends strongly on small-scale ($R < N^{-1/3}$) fluctuations of the impurity potential. [19] The potential wells formed when two charged donors approach to a distance smaller than the average: $R \lesssim R_m (1 - K)^{1/3} N_D^{-1/3}$ are especially important. At $T \to 0$, most electrons are in such wells. The paper [19] considers only pairs with $R \sim R_m \gg a$.

We assumed that pairs with $R \gtrsim a$ can be regarded as analogs of the molecular hydrogen ion H₂⁺ with characteristic distances R between the nuclei. This assumption was tested on n-InSb with Na³ = 0.05 and $1 - K < 0.15$. On examination of the absorption spectra, we observed peaks (Fig. 8) whose energies were comparable with those in the H₂⁺-center spectrum. Good agreement with theory was obtained (Fig. 8). Later, the presence of H₂⁺ centers in semiconductors was also demonstrated by experiments with n-GaAs. [20]

6. **Free excitons in Ge.** [8] Study of excitons at submillimeter wavelengths instead of in the optical band, which is usually used for this purpose, is of special interest, since transitions of excitons from the ground state to excited states can be observed directly under these conditions. In addition to knowledge of the energy spectra of the free excitons, analysis of these transitions offers new opportunities for investigation of their interactions with one another, with free carriers, etc.

We made a series of photoconductivity and absorption studies of free excitons in the range from optical excitation levels so low that the free-exciton concentration was $n_e \approx 10^{10}$ cm⁻³ (which is inaccessible to other
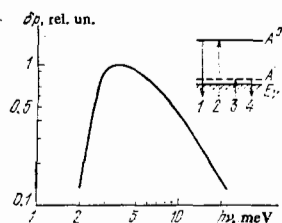

FIG. 6. Spectrum of longwave edge of photoconductivity due to photoneutralization of A⁺-centers in Si:B (the scheme of the energy transitions is indicated).
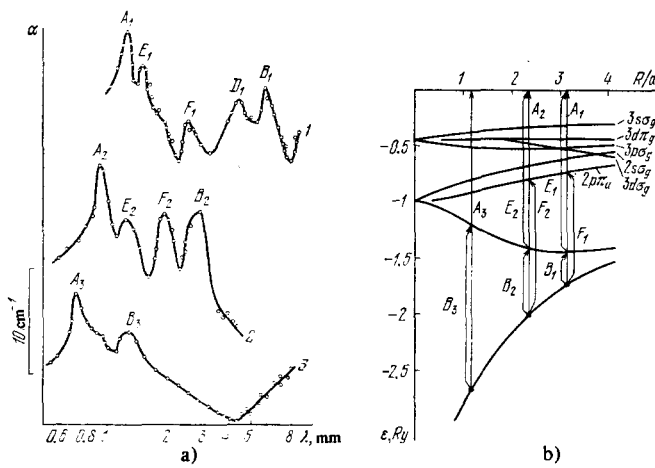
FIG. 8. Absorption spectra in n-InSb for three samples with $N_D \approx 1.2 \cdot 10^{14}$ cm^{-3} and various compensations K (1—0.85, 2—0.89, 3—0.94) at $T = 4.2$°K. The energy levels of H$_2^*$ are given on the right as they depend on distance between nuclei (in units of the Bohr radius). The arrows indicate transitions between H$_2^*$ levels whose energies are close to the energies of the observed peaks for samples 1-3.

methods) to 10^{14} cm^{-3}, at which excitons condense into electron—hole drops.[21] The similar studies of[22], where only absorption was investigated, were carried out concurrently with ours, and the results of the experiments provide an independent confirmation of our results.

As an example, Fig. 9a presents absorption spectra obtained for free excitons; it shows the lines of exciton transitions from two ground-state levels and the photoionization band. When the temperature is lowered from 4.2 to 2°K, the strength of the first series of lines ($\ell = 2.52$, 2.86 meV,...) relative to the second ($\ell = 3.14$, 3.42 meV,...) decreases exponentially with an activation energy $\Delta \approx 0.4$ meV. In addition to the exciton photoexcitation lines, the photoconductivity spectrum (Fig. 9b) shows a longwave photoconductivity limit ($\ell = 3.8$ meV). Another photoconduction band shifted by approximately 0.4 meV toward lower energies makes its appearance at sufficiently high T. The aggregate of these data yielded the energy spectrum of indirect excitons in Ge. The binding energy of the exciton, measured from the longwave limit of nonresonant photoconductivity, is found to be 3.8 meV; the energy position of the second series of exciton photoexcitation lines yields the
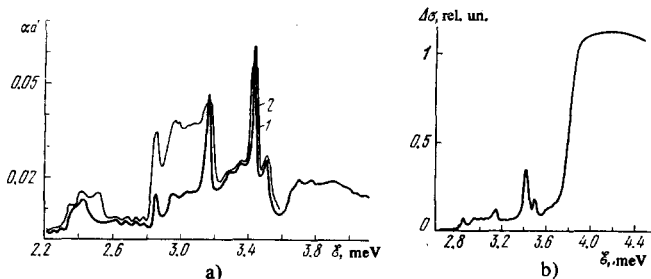
excited-state energies of this series. The energy gap Δ between the levels of the exciton ground state according to the temperature dependence of absorption-line strength and spectral measurements gives $\Delta = 0.4$ meV. However, because of the nonparabolicity of the exciton band,[23] analysis of the results is difficult: the temperature dependences are measured at relatively high $T = 4.2 - 2$°K, and the additional band in the photoconductivity spectrum is observed only at high temperature $T \sim 4$°K), when the photoexcitation lines may distort the form of the nonresonant band.

In addition to these data, we obtained information on the widths of the exciton lines and their temperature dependence and observed their splitting in a magnetic field and their behavior in an electric field.

With lowering of the temperature at a sufficiently high excitation level, a sharp decrease in the strength of the entire absorption spectrum begins at a certain temperature. Analysis showed that the effect is determined by the decrease in the concentration of free excitons in their interaction due to condensation of excitons to form electron—hole drops. The temperature curves of exciton concentration at various excitation levels G are reproduced in Fig. 10; the line drawn through the threshold points separates the region in which the system is single-phase (exciton gas) from the region in which drops and a saturated exciton gas coexist. Also included are the data obtained on the concentration of the electron—hole drops. The sensitivity of the method made it possible to trace the branch of the phase diagram to the lowest temperatures and excitation levels; for example, it was shown that the exciton concentration no longer depends on temperature down to 0.5°K when $n_e \sim 10^{11}$ cm^{-3}. The work function φ of the electron—hole pair from the condensate to the exciton and the dependence of the drop radius R on T and G were calculated from the experimental data, and it was established that the drop concentration depends weakly on excitation level and increases sharply with decreasing temperature; other information was also obtained.

7. *Free carriers in* Ge.[9,10] We illustrate the possibilities for submillimeter study of free carriers with two examples.

a) At helium temperatures, cyclotron resonance (CR) in the submillimeter band corresponds to the quantizing magnetic fields H. Under these conditions, we succeeded in observing a new specific quantum effect.[9] It was

FIG. 9. Free-exciton absorption spectrum in Ge at concentration $n_e = 5 \cdot 10^{11}$ cm^{-3} ($T = 4.2$°K (1) and 2°K (2)) (a) and photoconductivity spectrum of Ge at $n_e = 10^{12}$ cm^{-3} ($T = 2$°K) (b).
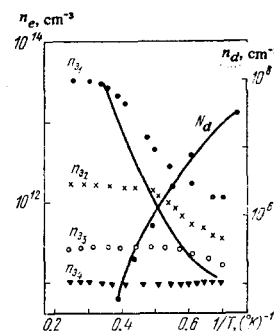

FIG. 10. Temperature dependence of exciton concentration n_e at three excitation levels, and concentrations of electron—hole drops N_d for the highest of them. The solid line represents the segment of the exciton phase diagram obtained from these data.
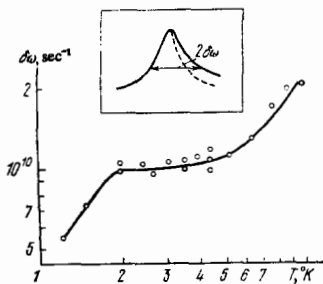
FIG. 11. Temperature curve of CR line width $\delta\omega$ in IB range (428 GHz) and shape of CR line with IB.

FIG. 12. Photoconductivity spectrum of Ge : Sb with $N_D \approx 3 \cdot 10^{15}$ cm^{-3}. The insert shows the electron energy levels in Ge with H $\parallel \langle 111 \rangle$; the observed IVCIR transition (peak A)—3; B are impurity transitions.

found that the width of the CR line may be governed not only by scattering processes, which result in damping ("dissipation") of velocity, as is usually the case, but also by a shift of the electron energy levels due to their interaction with the scatterers. This shift always occurs and is the same for all electrons, and this is why it cannot be observed in the resonant frequency. Under quantum conditions, however, it may depend on the kinetic energy of the electron along H. Then different electrons will have different—"partial"—resonant frequencies, and the line will acquire an additional broadening that might be called inhomogeneous broadening (IB). Such conditions are realized, for example, in scattering by acoustic phonons, when the average energy of the phonon with which the phonon interacts is found to be near kT. Figure 11 illustrates the effect of inhomogeneous broadening on the temperature dependences of the half-width of the CR line in Ge. As the temperature T is lowered, the line narrows to a certain limiting value, the width in the IB range, and then, beyond the plateau determined by the IB, it narrows further as T is depressed further. Among other things, this experiment explained the considerable differences between data in the large body of literature on quantum CR.

Interesting results are also obtained for the quantum CR in scattering on ionized impurities. [14]

b) Another example is detection of the new resonance predicted in[24] and due to transitions of electrons interacting elastically with impurities between the Landau levels of different valleys. [10] This resonance is possible in multivalley semiconductors in a quantizing magnetic field, when, "simultaneously" with absorption of a photon, the electron experiences an intervalley transition as a result of interaction with the impurity. This resonance, which we have named "intervalley cyclotron-impurity resonance" (IVCIR) should occur at frequencies $\omega = [l_2 + (1/2)]\,\omega_2 - [l_1 + (1/2)]\,\omega_1$, where ω_1, ω_2, l_1, and l_2 are the cyclotron frequencies and Landau-level numbers of the two valleys involved. It is similar in nature to the cyclotron–phonon and intervalley cyclotron-phonon resonances, [25] but differs from them in that its frequency is not governed by the phonon spectrum of the crystal.

We observed and studied it in Ge : Sb (N_{Sb} less than 10^{15} cm^{-3}). As an example, Fig. 12 shows a diagram of some of the possible IVCIR transitions and the photoconductivity spectrum with the sought resonance at $H_{res} = 0.77 H_{CR}$. The same peak is also reproduced in Fig. 12 on an enlarged scale.
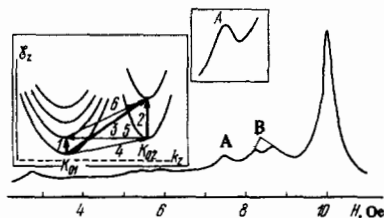
The correctness of the identification was confirmed by study of the anisotropy of the effect, the temperature dependences of intensity (the intensity of the IVCIR peak increases with decreasing T as compared to the photo-thermal-ionization lines of the impurities), and the dependence of intensity on N_{Sb} (the resonance increases with increasing N_{Sb} and does not appear on pure samples with $N_{Sb} \sim 10^{12}$ cm^{-3}).

8. *Certain applications.* [11,12] a) One of the high-priority problems at the present time is to design sensitive and fast-responding radiation detectors for the submillimeter band. Figure 13 shows characteristics of Ge (curve 5) and n-InSb (1) photoresistors that have now been developed for this segment of the spectrum (to avoid complicating the figure, data on the narrow-band n-GaAs detector, which operates near 300 μm, have been omitted). Also shown are the characteristics of new photoresistors whose development was made possible by the submillimeter-photoconductivity effects described above.

1) *The H$^-$ photoresistor.* [11] Since the binding energy of an electron (hole) on a D$^-$(A$^+$) center is lower than that at a neutral impurity atom, the longwave photoconductivity limit due to ionization of these centers (neutralization of D$^-$(A$^+$)) is accordingly shifted to longer wavelengths as compared to ordinary impurity photoconductivity. This makes it possible to use photoresistors based on this principle to cover the entire submillimeter region of the spectrum, using such well-developed semiconductor materials as doped Ge and Si. We have developed a photoresistor based on Si : B (its spectral sensitivity characteristic with $N_B = 4.10^{14}$ cm^{-3} and $K = 0.005$ at $T = 1.6$ °K and $E = 10$ V/cm is represented by curve 2 in Fig. 13); its sensitivity in the 100–500 μm range is on a par with that of ordinary impurity photoresistors, and its spectral range supplements those of known photodetectors. By increasing N_B, we can shift the photo-
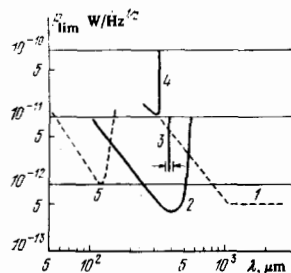

FIG. 13. Maximum sensitivities of known (dashed) and new (H$^-$ (2), impurity (3), and exciton (4)) submillimeter photoresistors.
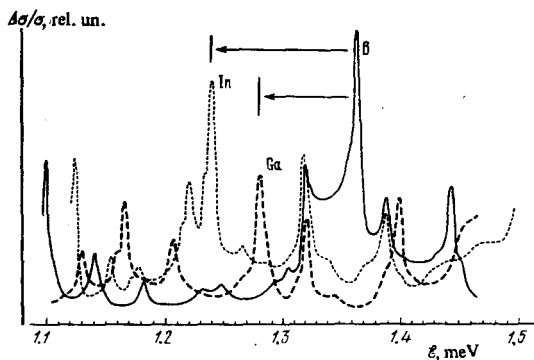
$\Delta\sigma/\sigma$, rel. un.

FIG. 14. Segment of photoconductivity spectrum of Ge doped with B, Ga and In.

resistor's sensitivity range to shorter wavelengths (see Fig. 7). A similar photodetector based on Si:P was proposed independently in[18], but its characteristics are still inferior to those of Si:B.

2) *The exciton photoresistor.*[11] Since submillimeter radiation with photon energies higher than the photo-ionization energy of the excitons ($\lambda \lesssim 300$ μm) causes excitons to decay into free electrons and holes, the photoconductivity that arises can be used to indicate the emission. Our maximum-purity Ge ($N_D + N_A \lesssim 10^{12}$ cm^{-3}) should cover the band from 120 to 300 μm (curve 4 in Fig. 13; while not as sensitive as other photoresistors at $n_e \sim 10^{14}$ cm^{-3}, it has superior response speed ($\tau \sim 10^{-8}$ sec).

3) *The narrow-band impurity photoresistor.*[11] Like the ground state of an impurity,[16] excited states can be ionized photothermally to create narrow-band submillimeter detectors with several photosensitivity bands. Thus, the wavelengths for donors in Ge are $\lambda = 318$, 354, 658 μm, etc.; $\Delta\lambda/\lambda$ is $\sim 0.3\%$. Application of a magnetic field retunes the detector, but the richness of the photoconductivity spectrum makes it difficult to use it as a single-band device. These difficulties can be overcome in the relatively narrow band from 350 to 400 μm (curve 3 in Fig. 13). A sensitivity band ~ 1 μm wide can be tuned in this range by varying H from 0 to 17 kOe.

b) Another important application of submillimeter spectroscopy was made possible by its appearance on the scene: chemical analysis of impurities in semiconductors, including maximum-purity Ge.[12] Here the method is entirely based on the photoelectric spectroscopic procedure developed in detail in[26], which uses the differences in the ground-state ionization energies of impurities that differ in chemical nature. The shortwave limit of the BWTs that have been developed does not permit study of the ground state, but the spectrum of the excited states also carries information on the chemical nature of the impurity. By way of example, Fig. 14 shows parts of the spectrum of Ge doped with B, Ga, and In. Even though the chemical shift of the excited states is smaller than that of the ground state, the high resolution of the BWT spectrometer makes up for this in full; the high sensitivity compensates for the lower population of the excited states. Thus, for the

segment of the spectrum shown in Fig. 14, the line of the $G^* - E^*$ transition (notation of[5]) corresponds to $\mathcal{E}_B = 1.365$ meV for Ge:B and $\mathcal{E}_{In} = 1.24$ meV for Ge:In. Therefore the relative chemical shift is $\sim 10\%$; the chemical shift of the ground state for this case is $\sim 15\%$, but the widths of the lines corresponding to transitions between excited states with $N_A \lesssim 10^{14}$ (less than 0.01 meV) is four times narrower than the ground-state lines obtained with the best infrared spectrometers. We note that it is preferable to use the line broadening of the transitions between excited states to determine concentration as the impurity concentrations increase.

9. *Conclusion.* The experimental results reported above testify to the new opportunities that have appeared with the advent of high-resolution submillimeter spectroscopy. Obviously, the subject has not in any way been exhausted by the problems discussed above. It is sufficient to mention the prospects for study of narrow-band and gapless semiconductors. We should also note the prospects for study of superconductors, since the energy gaps of all of the presently most interesting materials correspond to the submillimeter wave band.

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