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Properties of the electron-hole condensate in Ge double-injection diodes. I *

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The results of a study of the properties of electrically injected electrons and holes in Ge at liquid-He temperatures are presented. The injected electrons and holes are found to form a condensate with properties like those extensively explored in optical-excitation experiments. Spatial scans of the recombination radiation from the condensate show that it is possible to obtain an approximately uniform distribution of condensate between the p^+ and n^+ contacts. The transient response shows that the radiation from the condensate decays exponentially at 3.2°K, with a decay time of approximately 40 μsec. A number of features of the condensate radiation are found to be in agreement with the properties observed in material doped with Li.

I. INTRODUCTION

The properties of the condensate of electron-hole pairs in germanium and silicon at low temperatures have been explored in a number of experiments.[1,2] In all of these experiments, the pairs are produced by either electron-beam or optical excitation of the Ge crystal. These experiments have established the physical properties of the condensate,[1,2] the phase diagram governing the formation of the condensate,[3] and the existence of droplets of condensate.[4]

Most studies have been performed on high-purity Ge and Si. However, there have been studies of the effects of a number of different impurities on the recombination spectrum from both Ge and Si.[5–13] These studies show that impurities may produce a number of distinctly observable features in the recombination spectra. The impurities are responsible for additional lines in the spectra due to recombination of excitons bound to impurities[10,12,13] and a concomitant reduction in the intensity of the free-exciton line.[9] The condensate line itself is also changed. At impurity densities greater than about 10^{15} cm^{-3}, the linewidth of the condensate recombination radiation is perturbed from the value observed in the pure material[5] and the linewidth becomes dependent on the level of excitation.[8]

Both optical and electron-beam excitation of the crystal produce pairs over a narrow region near the surface. The thickness of this layer depends on photon or electron energy. For many of the energies used, the layer is on the order of 1000 Å thick. The electron-hole pairs are driven further into the crystal by surface electric fields and density gradients. However, this produces a rather nonuniform density of pairs[4] in which the condensate phenomenon is being observed.

In this paper we report on a series of experiments in which the condensate phenomenon is explored in a Ge double-injection diode. In the following paper[14] (henceforth referred to as II), we compare the characteristics of the recombination radiation spectra for excitation by double injection and laser illumination.

In the double-injection experiments the electron-hole pairs are injected electrically to produce an approximately uniform density of pairs.[15] Electrically injected electrons and holes have been observed to condense into a plasma which has properties similar to those found in the optical-excitation experiments. In previously reported experiments, the device structures were such that device heating was a potential problem.[16] In the present work, the device structures have been modified to minimize the influence of heating. To minimize contamination of the sample by deep level impurities introduced during high-temperature processing, we utilize process temperatures less than 325 °C. In particular, we use a Li diffusion to form the n^+ contact. A discussion of the influence of Li on the recombination spectrum from Ge is contained in II.

Section II of this paper describes the fabrication of the Ge double-injection devices. Section III presents the experimental procedures used. The experimental results are given in Sec. IV. Finally, Sec. V contains the conclusions drawn from these results.

II. DEVICE FABRICATION

The primary goal in fabricating a device for these experiments is to obtain broad-area electrically injecting contacts which will function at liquid-He temperatures. In addition the physical configuration of the device must be such that large currents can be passed through the device without excessive heating. The structure and fabrication process investigated here were chosen to meet these goals.

Double-injection diodes were fabricated from high-purity Ge slices with net carrier concentration on the order of 10^{11} cm^{-3}. The slices were

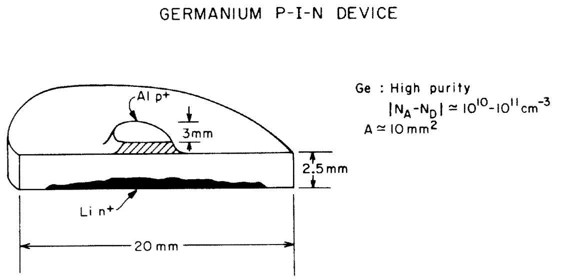

FIG. 1. Typical Ge double-injection diode of the mesa type used in electron-hole condensate experiments.

100–200 mm^2 in area and 0.6–6 mm thick. They were lapped with 5-μm size grit and cleaned sequentially in trichloroethylene, acetone, and methanol ultrasonic baths and then dried. One face of the Ge slices was masked with Scotch plastic tape, and then the slices were chemically etched in three parts HNO_3 to one part HF for 2 min. The etch was quenched with methanol, plastic tape removed, and the slices were placed in HF. They were rinsed with methanol and dried prior to loading them into a vacuum deposition system. An Al film ~0.5 μm thick was evaporated on the chemically etched side of the slices. The opposite side of the slices was painted with a Li in mineral oil suspension and the slices placed in a quartz tube furnace having a N_2 atmosphere. They were heated at 325 °C for 20 min and then slowly cooled to room temperature (~1.5 °C/min). This procedure yields a Li-diffused n^+ contact one side and a solid-phase Al p^+ contact on the other side of the Ge slices.[17] The diffusion length expected from these process conditions is approximately 0.2–0.3 mm and the concentration at the surface is $(1-2)\times 10^{17}$ cm^{-3}.[18]

A black wax mask was used to protect the entire Li-diffused side of the Ge slices and 2–10 mm^2 Al contacts on the other side. Some Al contacts were located at the edge of the Ge slices while others were located away from the edge. The Al mesas were defined using a three parts HNO_3 to one part HF etch. The Li-diffused side was lightly lapped and contacted with In; and Cu wires were soldered with In to the Al contacts. A schematic sketch of a typical double-injection diode of the mesa type is shown in Fig. 1. The Ge double-injection diodes were attached mechanically to a Cu block, rinsed with methanol, and dried prior to performing experiments.

III. EXPERIMENTAL PROCEDURE

The experimental setup used in this work is shown schematically in Fig. 2. A Janis variable temperature Dewar with an operating range of 2–300 °K was used in these experiments. For temperatures below 4.2 °K, a vacuum-regulated liquid-He bath was used. Above 4.2 °K, the temperature was controlled using heated He vapor and an Artronix model 5301 temperature controller. The double-injection (DI) diode temperature was

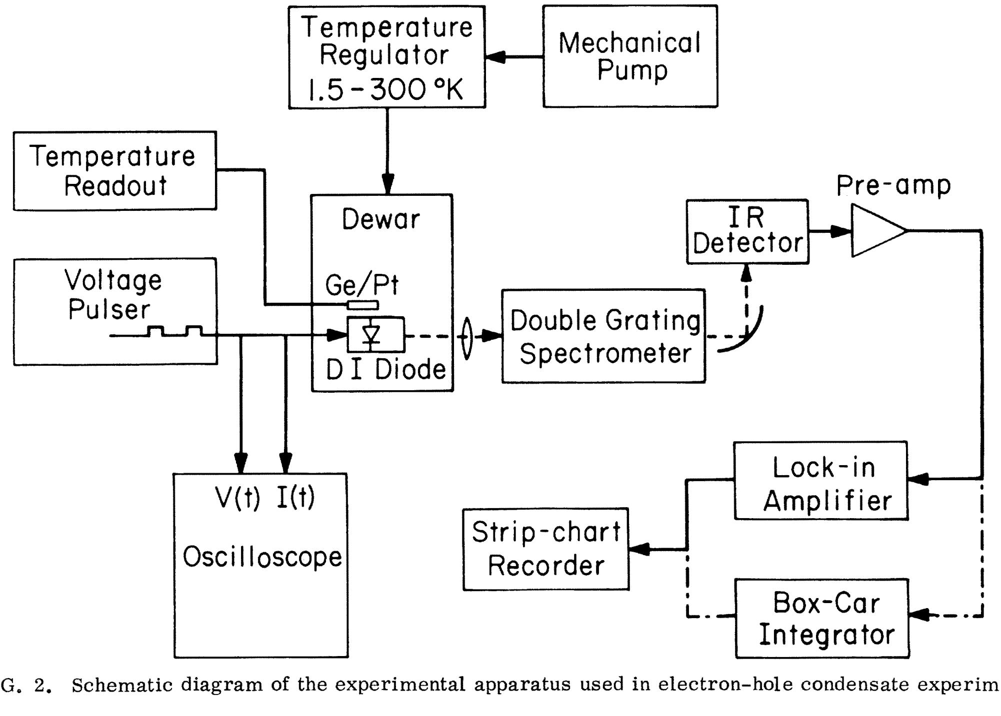

FIG. 2. Schematic diagram of the experimental apparatus used in electron-hole condensate experiments.

measured using a calibrated Ge or Pt resistor attached to the Ge structure close to the diode.

The electrical and optical properties of the diode were measured using direct and pulsed currents. For direct current measurements, a current source was used and the voltage measured by a digital voltmeter. For pulsed currents measurements, a voltage pulser was used. The current was measured using either a current probe or by measuring the voltage across a known resistance in series with the diode. The diode voltage was measured with an oscilloscope.

The radiation emitted from the region between the n^+ and p^+ contact of the double-injection diode was imaged onto the entrance slit of a Spex 1400-II spectrometer. For direct current injection, the radiation was mechanically chopped and detected with a cooled InSb photodetector. For pulsed current injection, the recombination radiation was detected with a cooled InAs detector. The detector and preamplifier had a (0–63%) response time of 1.7 μsec. The signal from the detector was processed either by a lock-in amplifier or box-car integrator, which were synchronized with the current pulse.

The spatial distribution of the radiation was obtained by mechanically scanning an InAs detector through the image of the region between the p^+ and n^+ contact. The active region was imaged with a magnification of approximately three onto the plane of the photodetector. The detector was equipped with a mask having an aperture of 0.3 by 1 mm which was positioned near the middle of the image and mechanically scanned between the n^+ and p^+ contacts.

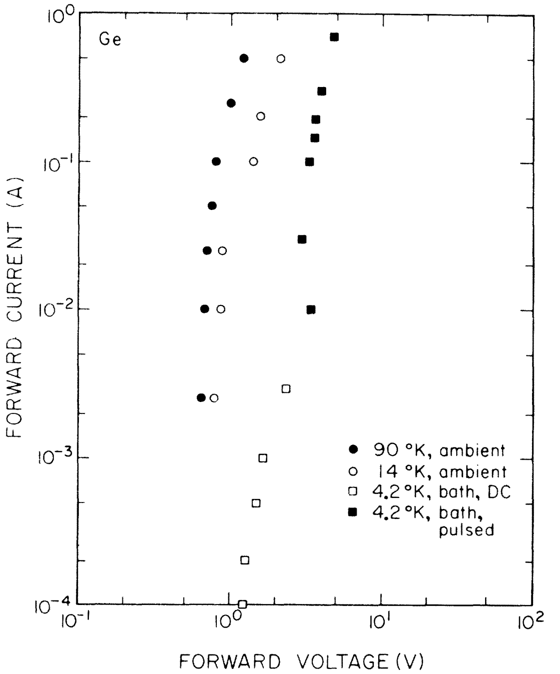

FIG. 3. Forward biased, current-voltage characteristics of 6 mm^2 by 2.4-mm-long mesa-type double-injection diode made on a slice of Ge with ~200 mm^2 area. The dots, circles, and squares are the results for temperatures of 90°, 14°, and 4.2°K, respectively.

IV. RESULTS

A. Electrical characteristics

The measured steady-state forward current-voltage characteristics of a typical diode for temperatures between 4.2 and 90 °K is shown in Fig. 3. This particular mesa-type device was 2.4 mm long with a mesa area of 6 mm^2 and located near the edge of the slice. At 90 °K, the forward current increases rapidly for applied voltages near 0.5 V. This behavior is in agreement with the prediction of standard double-injection theory for diodes operating in the diffusion regime.[19]

The data at lower temperatures indicate a similar current-voltage characteristic with the voltage at which the current rises rapidly shifted to slightly higher values. This shift to higher voltages may be due to increased values of the voltage drop at the contacts. An additional possible complication in obtaining a qualitative understanding of the diode characteristics is the presence of excitons or condensate. The mass-action law and the observation of recombination radiation from excitons (see Sec. IV B) indicate that excitons are present in the double-injection device for temperatures between about 7 and 77 °K. In principle, one of the ways in which the excitons can contribute directly to the steady-state current is through the necessity of supplying electrons and holes to replace those excitons which have recombined. The current contribution due to exciton recombination may be estimated using the law of mass action to determine the exciton density for a free carrier density sufficient to carry the observed current. From this estimate of exciton density and the observed exciton lifetime of 6 μsec (see Sec. IV D), the current due to exciton recombination is found to be typically four orders magnitude smaller than the device current in the present structures.

At temperatures where the condensate is observed (see Sec. IV B), the condensate may influence the current-voltage characteristics. Again one possible way that this may occur is through the need to supply electrons and holes to replace those that recombine in the condensate. This contribution to the current can be estimated using the estimated fill factors $\lesssim 1\%$ (see Sec. IV C) and the observed decay time in the condensate of 40 μsec (see Sec. IV D). This estimate

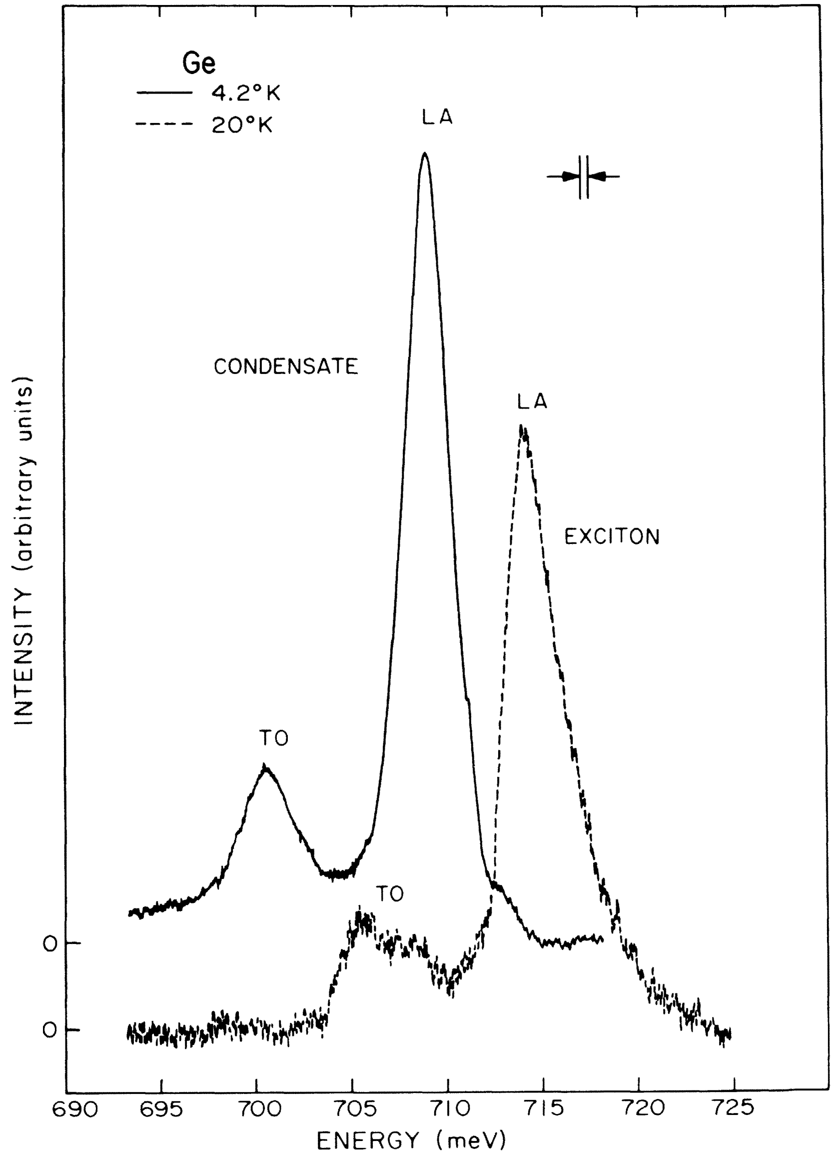

FIG. 4. Phonon-assisted recombination radiation spectra of a Ge double-injection diode at 20 °K (dashed line) for a device current of 800-mA (11-msec pulse, 22% duty cycle) and at 4.2 °K (solid line) for a device current of 300 mA (1-msec pulse, 9% duty cycle). The vertical scales are not the same for the spectra.

leads us to conclude that the contribution to the current due to recombination in the condensate is typically three orders of magnitude smaller than the device current. We are presently investigating a number of other ways in which the excitons and condensate may influence the current-voltage characteristics.

B. Recombination radiation spectra

The recombination radiation spectra of a double-injection diode are shown in Fig. 4. The spectrum at 20 °K shows two peaks at the well established LA- and TO-phonon-assisted exciton recombination lines. The spectrum from the same device at 4.2 °K shows distinct peaks at 709 and 700 meV which have been previously identified with the recombination radiation from the electron-hole condensate.[20]

Evidence for the interpretation of the spectrum at 4.2 °K in terms of electron-hole condensate is given by the fitting of the line shape to a simple model of the recombination process from the condensate. This simple model was first suggested by Pokrovskii[21] and consists of assuming that the electrons and holes occupy states with densities of states which are the same as obtained from the standard band structure. Further, one assumes that the band gap is shifted to a lower value, and that the matrix element for the transition is not subject to any momentum selection rule and is energy independent. The results of such a fit are given in Fig. 5. The dots are the experimental

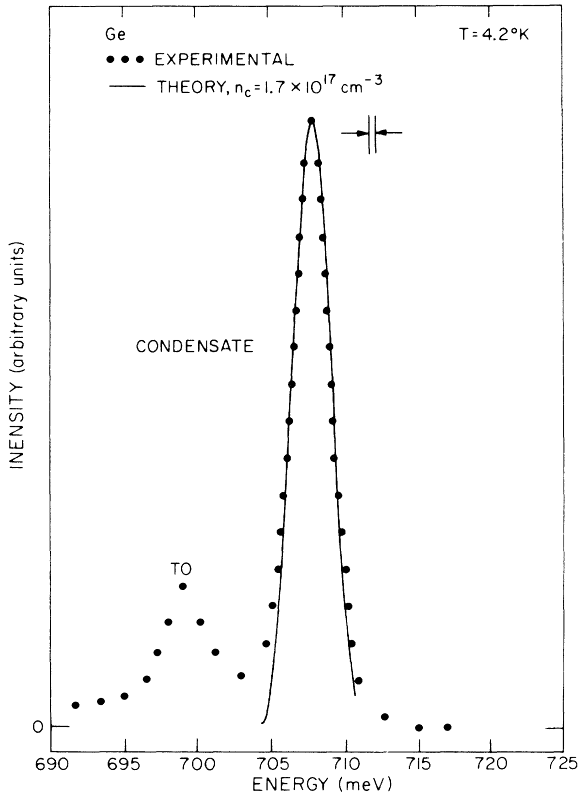

FIG. 5. Condensate recombination spectrum of a Ge double-injection device. The points are the experimental spectra obtained at 4.2 °K when current pulse with a duration of 1 msec, and an amplitude of 300 mA were passed through the device with a duty cycle of 9%. The solid line is the theory obtained from the free-electron and hole model with a density of 1.7×10^{17} cm^{-3} electron-hole pairs.

spectrum; the solid line is obtained from the model with a temperature of 4.2 °K, a density of electron-hole pairs of 1.7×10^{17} cm^{-3}, corresponding to the full width at half-maximum, linewidth, of 3.0 meV, and a line position of 709 meV.

In a previously published laser excitation experiment on pure Ge at 4.2 °K,[22] a high-resolution recombination radiation spectrum was obtained. We extract from that spectrum a linewidth of 3.4 meV. From the published phase diagram,[3] this linewidth corresponds to a density in the condensate of 2.1×10^{17} cm^{-3}. Laser excitation experiments on Ge doped with impurities to concentration of $2 \times 10^{15} - 10^{17}$ cm^{-3} have shown a narrowing of the line and reduction in the density[8] similar to that observed in the spectrum of Fig. 5.

Aside from the narrowing of the line, the spectrum observed from the double-injection devices differs in another way from those obtained in laser excitation of pure Ge. No exciton line is observed in the double-injection experiments while the exciton line is observed in the laser excitation experiments on high-purity Ge.[1,22] Experiments using laser excitation on Li-doped Ge (as presented in II) show a decrease in the radiation intensity of the exciton line similar to the double-injection case. For Ge doped with other shallow impurities, the absence of the exciton line has been noted.[9]

In measurements of the recombination spectrum, we observed electron-hole condensate radiation up to a temperature of 5 °K. Above 5 °K the spectrum changes and two sharp lines develop. The transition in the spectral shape between 4.8 and 7.2 °K is shown in Fig. 6. The sharp line at 714 meV has been identified as the LA exciton line[23] (the exciton spectrum at 20 °K is included in Fig. 6 for comparison). The other line at 712 meV we attribute to the bound exciton caused by the presence of Li. Bound-exciton lines have been observed in Li-doped Si,[13] and in Li-doped Ge.[24]

In optical excitation of high-purity Ge, it has been reported that electron-hole condensate radia-

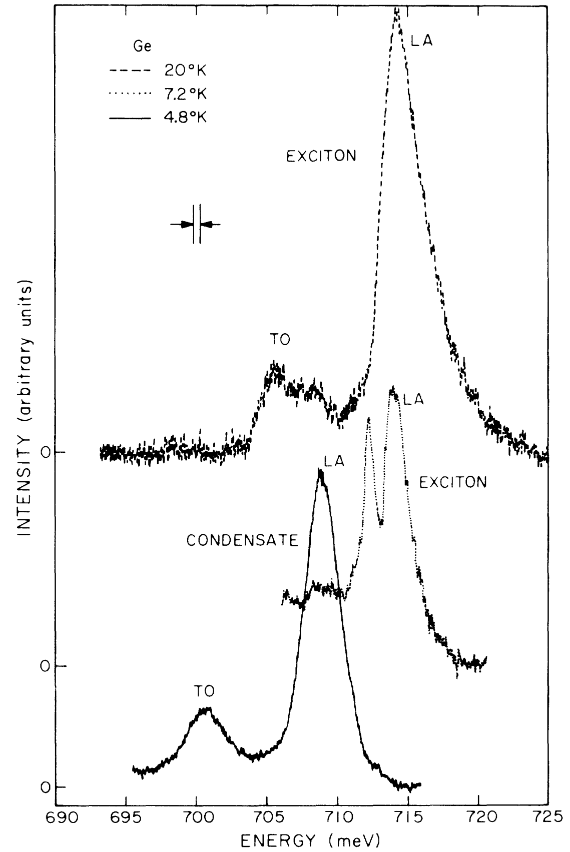

FIG. 6. Phonon-assisted recombination radiation spectra for a Ge double-injection diode at 20 °K (800-mA, 11-msec pulse, 22% duty cycle, solid line), 7.2 °K (800-mA, 11-msec pulse, 22% duty cycle, dashed line), and 4.8 °K (330-mA, 1-msec pulse, 9% duty cycle, dotted line). The vertical scales are not the same for the spectra.

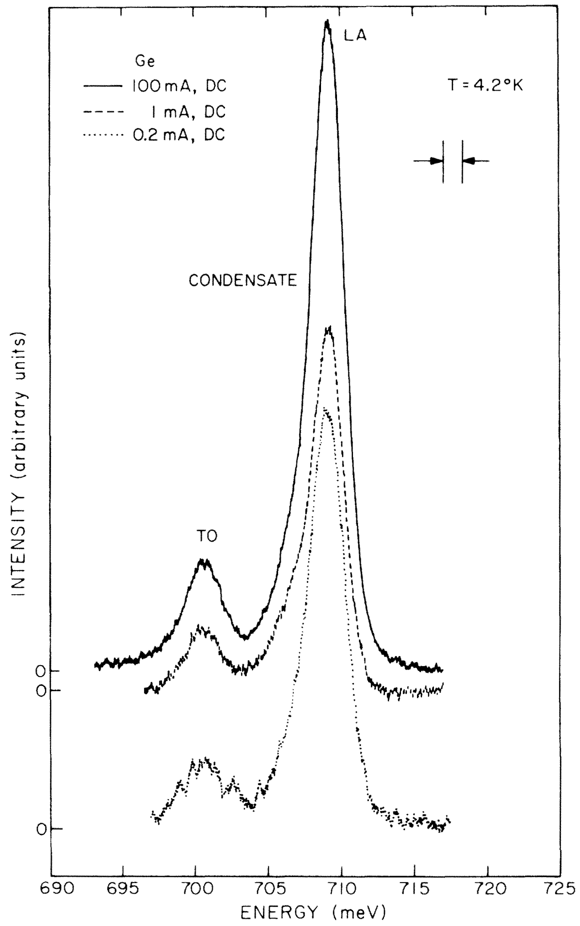

FIG. 7. Phonon-assisted condensate recombination radiation spectra of a Ge double-injection diode at 4.2 °K with direct currents of 0.2 mA (dotted line), 1.0 mA (dashed line), and 100 mA (solid line). The vertical scales are not the same for the spectra.

tion appears only for excitation values above a threshold value.[1] Below this threshold value, only free-exciton radiation is observed. In current excitation used in the present work, we have found no evidence for free-exciton radiation even at the lowest current levels. As the injection current is decreased, the electron-hole condensate signal disappears into the noise without any indication of a free-exciton line. In measurements presented in paper II[14] of laser excitation of high-purity Ge at 4.2 °K, a clear threshold is observed. On Li-diffused Ge of the same original purity, no free-exciton line is seen even at the lowest levels of laser excitation.

There are three differences in the character of the recombination spectra at temperatures of 4.2 °K from these double-injection devices when compared to laser excitation on pure Ge: narrower linewidths, absence of the free-exciton line, and absence of a threshold. Since these same three features are observed in laser excitation of Li-doped Ge as reported in II, we attribute these features to the presence of Li within the active volume of this double-injection structure.

C. Dependence of recombination radiation intensity on current and temperature

In this section, the dependence of the recombination radiation from the condensate on current and temperature is presented. The phonon-assisted recombination radiation spectra from the condensate are shown in Fig. 7 for several device currents at a temperature of 4.2 °K. The spectra shown were obtained for a device currents of 0.2 mA (dotted line), 1 mA (dashed line) and 100 mA (solid line). Note that even for the lowest device current, no evidence of an exciton line is observed. Further, there is no noticeable shift in the LA-phonon-assisted condensate line at 709 meV as a function of current, but an increase in linewidth is observed. The linewidth as a function of current at 4.2 °K for the same device is shown in Fig. 8. These data show that linewidth is independent of current at low current levels. The value of linewidth of 2.5 meV corresponds to a density of 1.2×10^{17} cm^{-3}. At higher current levels, the linewidth increases to a value of 3.0 meV which corresponds to a density of 1.7×10^{17} cm^{-3}. Similar variations of linewidth with excitation intensity has been observed in laser excitation experiments of doped Ge.[9]

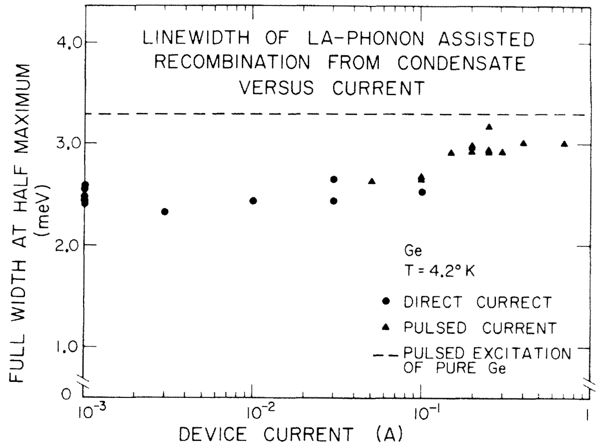

FIG. 8. Full width at half-maximum, linewidth of the LA-phonon-assisted recombination line of the condensate vs current through the double-injection diode. The bath temperature was 4.2 °K. The data in this figure were taken under a number of different current conditions. The solid circles are data taken with direct-current injection. The solid triangles are data taken with pulsed current injection. The pulse length was between 1 and 4 msec. The duty cycle ranged between 6.8 and 36%.

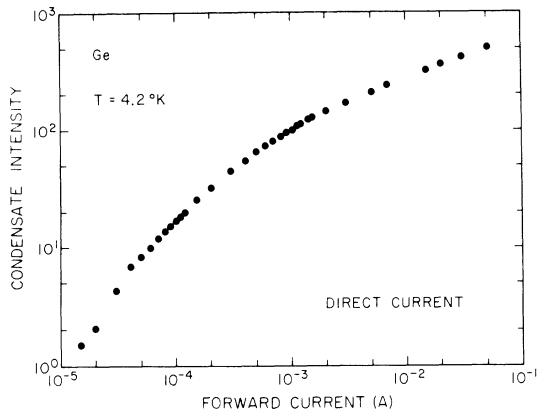

FIG. 9. Recombination radiation intensity of the 709-meV condensate peak from a Ge double-injection diode vs direct current. The temperature of the bath was 4.2 °K. The radiation was passed through a spectrometer centered at 709 meV with a bandwidth of 1.4 meV.

The intensity of the condensate radiation as a function of current was determined in two different ways. In the first procedure, the radiation was passed through a spectrometer centered at 709 meV with a band pass of 1.4 meV and detected by an InAs or InSb detector. In the second procedure, the radiation was passed through a low-wavelength pass filter ($\lambda_0 \leq 1.95$ μm) and detected without use of the spectrometer. The slight broadening of the line with increasing current level implies that a small correction should be applied to the results obtained using the first procedure to obtain a relative integrated intensity. However, this correction is small and will be neglected.

The radiation intensity for direct current excitation of the device shown schematically in Fig. 1 at a temperature of 4.2 °K is given in Fig. 9. The procedure involving the spectrometer was used. These results show that the intensity increases monotonically with current. Above 500 μA, the intensity increases sublinearly with current. The sublinear increase in the radiation intensity with device current at higher currents may be due to device heating[16] or to impact ionization[25] by energetic free carriers.

For higher current levels, pulsed currents were applied to minimize heating. The intensity versus current was measured using both the spectrometer and low-wavelength pass filter. In both experiments 100-μsec current pulses were applied at 2% duty cycle. The output of the detector was processed using a boxcar integrator with a 20-μsec-wide gate positioned 10 μsec before the end of the current pulse. The results from the two experiments coincide in the current range where both experiments could be performed.

In Fig. 10, we present the results for the intensity versus pulsed current at temperatures of 2.3, 3.0, 3.5, and 4.0 °K measured using the filter. These results show that at constant device current the radiation intensity increases as the temperature decreases. The intensity continues to increase with increasing current up to about 250 mA and then begins to decrease. This decrease may again be due to device heating or to impact ionization by energetic free carriers. Increasing current is associated with an increase in the free-carrier density. This in turn should be associated with an increase in the amount of condensate at constant temperature. Since the condensate is a lower-energy state than free electrons and holes, one expects more condensate as the temperature is decreased.

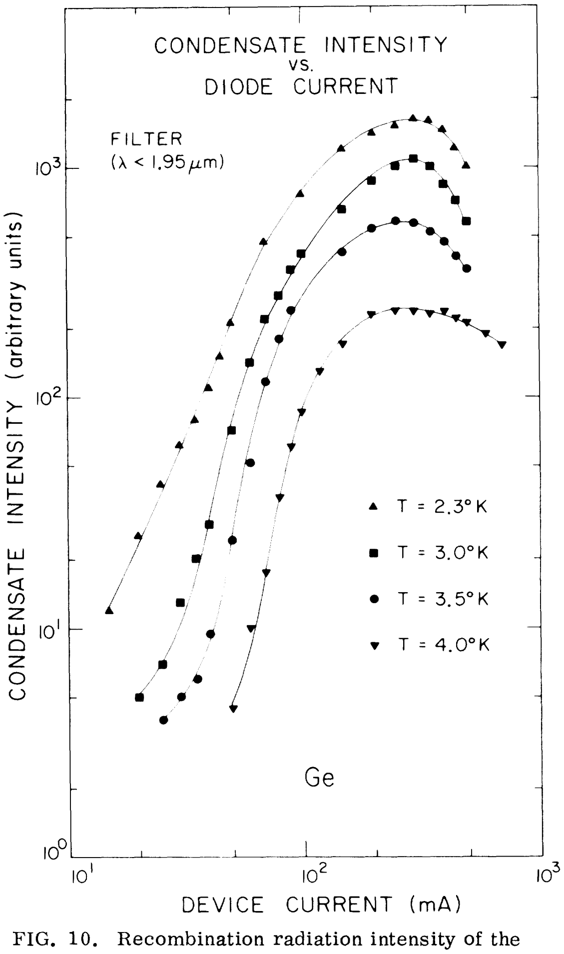

FIG. 10. Recombination radiation intensity of the condensate from a Ge double-injection diode vs pulsed device current. The bath temperatures were 4.0 °K (circles), 3.5 °K (triangles), 3.0 °K (squares), and 2.3 °K (dots). The radiation was passed through a low-wavelength pass filter ($\lambda \leq 1.95$ μm). The intensity was measured over a 20-μsec gatewidth positioned 10 μsec from the end of a 100-μsec-wide current pulse at 2% duty cycle.

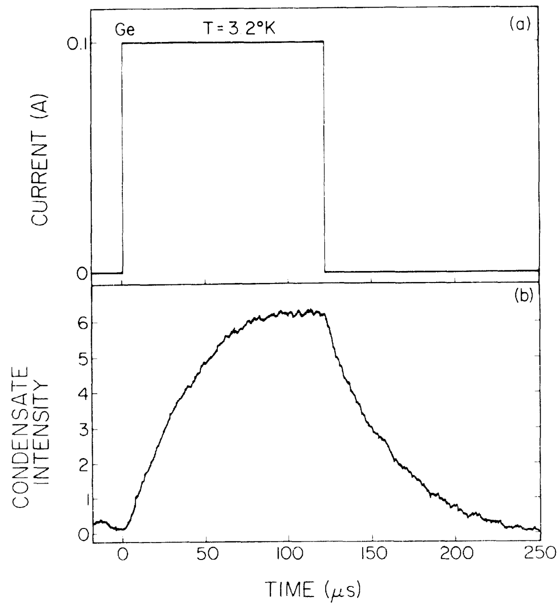

FIG. 11. Time trace of (a) current pulse and (b) condensate radiation intensity for a Ge double-injection diode with a pulsed current of 100 mA (120-μsec pulse, 24% duty cycle) at 3.2 °K bath temperature.

An estimate of the condensate fill factor can be made assuming the radiative efficiency is independent of temperature. This assumption is consistent with data presented in Sec. IV E where the total recombination lifetime of 40 μsec is found to be independent of temperature between 2 and 3.2 °K. At the highest intensity shown in Fig. 10, we estimate that somewhat less than 1% of the active volume of the device is filled by the electron hole condensate. This fill-factor estimate was based on the detector efficiency quoted by the manufacturer and solid-angle correction for an isotropic source.

D. Kinetics of electron-hole condensate

The kinetics of electron-hole condensate liquid formation has been studied by measuring the variation of intensity of the recombination radiation as a function of time at temperatures between 2 and 3.2 °K. In this temperature range, the intensity was measured without a spectrometer since only condensate radiation was observed. A typical result obtained at 3.2 °K is given in Fig. 11 where part (a) gives the current pulse and part (b) gives the intensity as a function time. The rise in the condensate radiation is considerably slower than the onset of the current pulse. The relatively slow rise of the condensate radiation observed here is in direct contrast with the very rapid increase in the recombination radiation from the electron-hole condensate observed by optical excitation.[26] Note, however, that in the published laser excitation transients, pulse widths short compared with these rise times have been used. Under these conditions, the slow rise time as shown in Fig. 11 would not be observed.

The decay is exponential over the range measured as shown in Fig. 12. The characteristic time for the decay of the radiation from the electron-hole condensate is 40 μsec at 3.2 °K. The same value of 40 μsec was also obtained down to 2 °K. This value for the decay time agrees rather well with the values reported in optical excitation experiments on pure Ge.[27–31] However, the observation of an exponential decay characteristic directly is in disagreement with the decay characteristic observed in optical excitation experiments.[27–29,31] In these experiments at a temperature of 3.2 °K evaporation from the droplet is thought to play an essential role in the decay of a drop and to modify the characteristics from a standard exponential form.[29,31] No observation of droplet decay using laser excitation has been reported for Ge doped heavily with an impurity. Theoretical calculations of the influence of impurities on the electron-hole condensate suggest that impurities will increase the work function as compared to the work function in pure Ge.[32] An increase in the work function would tend to diminish the importance of evaporation in the decay of the condensate. For this reason, we would expect the decay in doped Ge to exponential at higher temperatures than those found in laser excitation

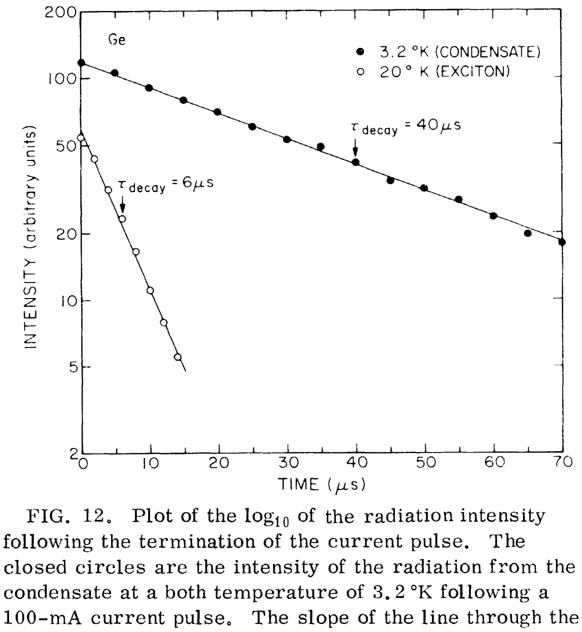

FIG. 12. Plot of the $\log_{10}$ of the radiation intensity following the termination of the current pulse. The closed circles are the intensity of the radiation from the condensate at a both temperature of 3.2 °K following a 100-mA current pulse. The slope of the line through the points gives a decay time of 40 μsec. The open circles are the intensity of the radiation from excitons at a bath temperature of 20 °K following a 500-mA current pulse.

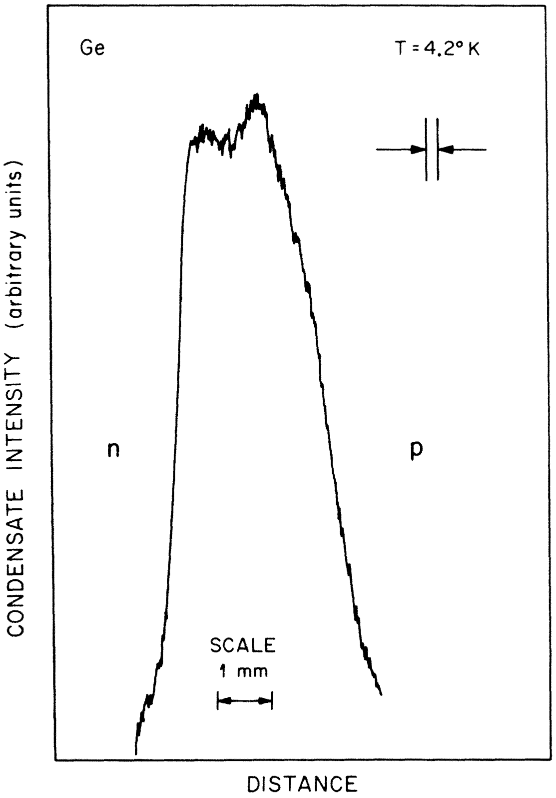

FIG. 13. Spatial distribution of the condensate radiation from the region between the p^+ and n^+ contacts of a Ge double-injection device. The device was at a temperature of 4.2 °K. Pulses of current of 1-msec duration with an amplitude of 100 mA were applied with a duty cycle of 9%.

of pure Ge. We are presently pursuing the decay of the condensate radiation in doped materials.[33]

For completeness, we have also included in Fig. 12 the decay of exciton radiation at 20 °K. This decay is also exponential with a characteristic time of 6 μsec. This value of the time is in good agreement with some of the values reported for optical excitation experiments.[1,31,34]

E. Spatial variation of recombination radiation

An example of the spatial variation of the radiation between the n^+ and p^+ contacts for a temperature of 4.2 °K is shown in Fig. 13. This scan indicates that the radiation is emitted approximately uniformly from the region between the contact. Radiation spectra from the regions near the n^+ contact, the p^+ contact, and the region between the contacts are characteristic of the electron-hole condensate. The radiation intensity decreased when one moved the detector to a region which was not between the contacts. These results indicate that, for the case of electrical excitation of the Ge, it is possible to have an approximately uniform region over which to study the condensation phenomena. At other current levels, we see more structure indicating that some spatial variation exists in the condensate distribution.

V. SUMMARY

In summary, electron-hole condensate can be produced by electrical injection of electrons and holes. The condensate radiation intensity is found to increase with increasing current at fixed temperature and decreasing temperature at fixed current. There is some evidence of impact ionization or heating at high current levels. When a sharp current pulse is applied to the device, the recombination radiation from the condensate is found to grow at a slower rate than the onset of the current pulse. The decay following the turn off of the current is exponential with a characteristic time of 40 μsec between 2 and 3.2 °K.

The recombination radiation spectrum from the double-injection structures is similar to that obtained by optical excitation of doped Ge. The line position 709 meV is the same as that observed in optical excitation of pure and doped Ge. The linewidth is dependent on current and is smaller than that found in optical excitation of pure Ge but in close agreement with that found in doped Ge. Another important difference in the double-injection spectrum from that found in pure Ge is the absence of an exciton line in the temperature range where the condensate is observed. At higher temperatures, the double-injection spectra clearly show radiation from excitons and bound excitons. These features of the double-injection spectra are like those obtained by optical excitation on pure Ge in which Li has been diffused in a manner similar to that used in forming the n^+ contact. Hence, we attribute these features to the presence of some Li in the active region of the double-injection devices. This conclusion is supported by data presented in the following paper (II).

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