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Optical detection of microwave-induced impact ionization of bound excitons in silicon

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In this paper we present the first experimental evidence for the cause of the nonresonant background signals commonly observed in optically detected magnetic resonance (ODMR) experiments in silicon. By making a direct comparison of bound-exciton (BE) photoluminescence (PL) intensity changes in an electric field with microwave-field-induced similar PL changes in *n*-type Si, we show that the main physical mechanism causing the ODMR background signal at lower microwave frequencies is impact ionization of free and bound excitons. We show that the efficiency of the microwave-induced impact ionization of bound excitons (BE's) is very strong, and it causes complete quenching of PL intensity for BE's associated with shallow donors. As a result the PL intensity can be enhanced by more than 150% for isoelectronic BE's. The change in PL intensity with microwave power shows a characteristic threshold, which was typically 20 mW at 9 GHz microwave frequency in our case.

I. INTRODUCTION

The technique of optical detection of magnetic resonance (ODMR) was originally introduced for photoluminescence (PL) studies of electron-spin resonance (ESR) in the excited states of defect systems, and has become very powerful for the identification of complex defects in semiconductors.[1] The power of ODMR has often been limited by a commonly observed strong and broad nonresonant background signal, especially in the technological important materials GaAs (Ref. 2) and Si.[3] In some cases these background signals show structure attributed to cyclotron resonance (CR), which has been utilized to measure optically detected CR (ODCR). ODCR was first observed[4] in Ge and has then been applied to a variety of compound semiconductors.[5-7] The first observation of ODCR in silicon was reported by Weber and Watkins,[3] and the first application of ODCR in silicon has recently been published, presenting studies of surface damage.[8]

The mechanisms responsible for optical detection of the CR were first discussed by Romestain and Weisbuch.[5] When the CR condition is fulfilled microwave energy is resonantly absorbed, thus resonantly increasing the effective temperature of free carriers. This in turn affects the PL intensity, and several possible mechanisms for this have been considered.

The application of microwaves in PL studies other than ODCR is by no means a new concept. Microwave radiation has found a range of applications in the studies of electron-hole droplets (EHD's) in Ge, as reviewed by Markiewicz and Timusk.[9] Dimensional resonances (Alfvén wave resonances) have been studied for EHD's.[10] Another application of microwave radiation was in measurements of the Auger emission probability by observing the CR of the created free carriers.[11-13] The first studies of microwave-induced exciton breakdown in Ge were due to Manenkov, Keldysh, and co-workers.[14-18] These authors have studied the impact ionization of excitons by the microwave-field-accelerated free carriers in the presence of EHD's and found that the heating of carriers was due to the electric part of the microwave field. In the first study on Ge a rapid increase of free-carrier concentration following a microwave pulse of power as low as 5 mW was shown.[16]

Up to now, no detailed study has been made to clarify the cause of the nonresonant background signal observed in ODMR experiments. Obviously much more sensitive defect studies could be done in ODMR, if the background signal was properly understood, and thereby possible to reduce or eliminate. Also, an understanding of the physical mechanism could provide new information on, e.g., recombination properties in the material.

We have recently proposed that the mechanism responsible for the broad nonresonant background signal in ODMR at lower microwave frequencies (9 GHz) in silicon, and most probably also for other compound semiconductors, is the impact ionization of excitons by hot free carriers.[19] In this paper we present a first direct proof that the cause of nonresonant PL intensity changes in ODMR experiments is related to the impact ionization mechanism, which leads to large changes of relevant PL emission intensities. From here on this optically detected microwave-induced impact ionization signal will be abbreviated as the ODMII signal. We utilize here also the results of recent studies of electric-field-induced quenching of bound excitons (BE's) in silicon.[20-22]

II. EXPERIMENTAL SETUP AND PROCEDURE

A range of different Czochralski-grown Si samples, *p*-type (B doped) as well as *n*-type (P doped), have been used in this study. The silicon samples have usually been annealed at higher temperatures in order to study different carbon-oxygen related bound excitons. The concentration of interstitial oxygen and substitutional carbon in the as-grown samples were around 7×10^{17} cm^{-3} and below 5×10^{15} cm^{-3}, respectively, derived from room-temperature Fourier-transform infrared data using the American Society for Testing and Materials (ASTM) standard.

The measurements of electric field-induced impact ionization of BE's were performed at 2 K, with both electric field and laser (Kr$^+$ ion laser) excitation pulsed, typically at 100 Hz with a pulse length less than 50 μs. The electric field was applied parallel to the sample surface along the [110] direction. The laser beam was defocused and illuminated the (111) surface of the sample. Other details on the experimental setup for the electric field measurements can be found elsewhere.[20]

For the ODMII experiments we used a modified Bruker 200 D-SRC 9 GHz spectrometer, equipped with an Oxford Instrument ESR 10 continuous flow He cryostat, and a cylindrical optical cavity. Microwave power up to 600 mW was available and samples could be cooled down to 2 K. For the optical excitation Ar$^+$ and Kr$^+$ ion lasers were used. Optical detection was done with a North-Coast EO 817 Ge detector and a phase sensitive lock-in amplifier (NF electronic instruments LI-575), in phase with either chopped microwaves or laser excitation. The laser beam could be chopped with either a mechanical chopper, or an acousto-optic modulator (Intraaction AOM-125). The luminescence was in all cases observed in the Faraday configuration. A 0.25-m Jobin Yvon grating monochromator was used for the studies of the spectral dependence of the ODMII signal. This was measured as follows: The magnetic field was set at the signal maximum, and the signal intensity versus PL photon energy was measured in phase with the chopped microwaves at 6 kHz. In such an experiment the microwave-power related changes of the intensity of a given PL line can be measured directly, which is a real advantage of the ODMII method over previous studies of microwave-induced exciton breakdown in Ge of Manenkov, Keldysh, and co-workers.[13–18] (In their studies the efficiency of the Auger or exciton impact ionization processes has been estimated indirectly from the size of the free-carrier-induced microwave absorption and resonance cavity detuning, and therefore it was not possible to separately follow the different recombination channels in the impact ionization breakdown.)

III. EXPERIMENTAL RESULTS

As a background to the ODMII studies is shown in Fig. 1 (a) a low-resolution PL spectrum at $T=2$ K, measured in the ODMR setup with ≈1.5 W/cm^2 excitation intensity from the 5145-Å line of an Ar$^+$ ion laser. The signal was in this case detected in phase with the laser chopped at 200 Hz. This spectrum consists of the transverse optical (TO)-phonon assisted BE related to neutral phosphorus [$P_{\rm TO}$(BE)] at 1.092 eV.[23] Two other distinct features of the PL spectrum are the two no-phonon lines at 0.903 eV and 1.119 eV. These two PL lines have been discussed previously[24–27] and have been labeled in Ref. 27 as line 9 and line 5, respectively. Both these PL lines are due to BE recombination at unidentified isoelectronic hole-attractive centers introduced by the sample annealing.

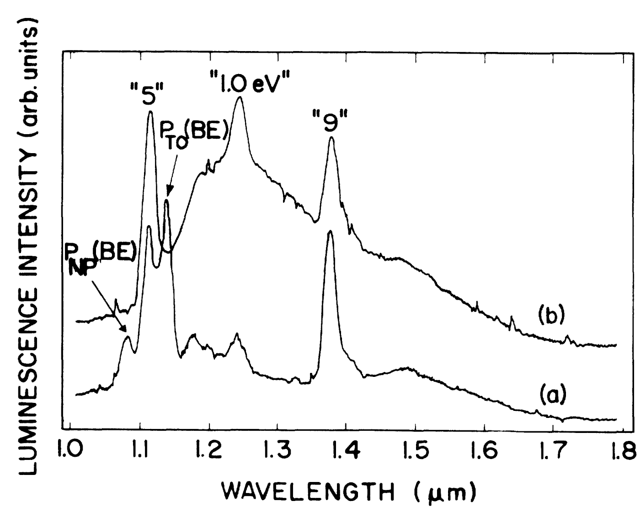

FIG. 1. Photolumincesence spectrum of phosphorus doped n-type (11 Ω cm) Czochralski-grown Si, annealed at 450 °C during 180 h and then isochronally annealed in cycles of 15 min every 25 °C between 475 and 650 °C. This spectrum was measured in a low-resolution ODMR setup at 2 K, with excitation from the 5145-Å line of an Ar$^+$ ion laser (≈1.5 W/cm^2). The spectrum was measured in phase with chopped laser excitation at 200 Hz. (Sample NA in Ref. 22.) (b) Photoluminescence spectrum as in (a) but with microwave power applied (577 mW) chopped at 6 kHz, and the magnetic field (500 G) set at the background signal maximum. The spectrum was measured in phase with chopped laser excitation at 200 Hz.

Figure 2(a) shows a broad featureless ODMII signal, observed in the experiment as a change of the total PL emission intensity in phase with chopped microwaves at 6 kHz as a function of applied magnetic field. The laser excitation intensity was set at ≈1.5 W/cm^2, and the ap-

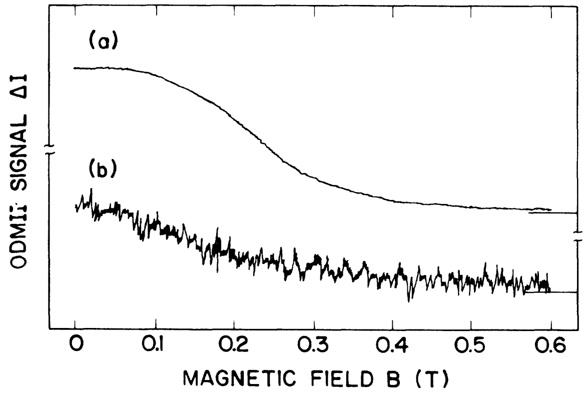

FIG. 2. (a) The broad nonresonant ODMII signal observed as the change in total PL intensity as a function of applied magnetic field. The PL signal was measured in phase with the applied microwave field (577 mW) chopped at 6 kHz. (b) As in Fig. 2(a), but with the monochromator set at 1.119 eV for detection of the isoelectronic bound exciton line 5 (Ref. 27).

plied microwave power was 577 mW. The microwave-induced change in total PL intensity is rather constant up to a magnetic field of about 1000 G and then gradually decreases. It was found that for a specific PL emission the critical magnetic field for the decrease in total PL intensity was increasing with increasing microwave power. The change in PL intensity (i.e., the ODMII signal) for the isoelectronic bound exciton (IBE) line 5 as a function of applied magnetic field can be seen in Fig. 2(b), under the same experimental conditions as in Fig. 2(a).

In Fig. 1(b) the PL spectrum is shown under the same condition as used to get the spectrum in Fig. 1(a), but with the applied microwave field chopped at 6 kHz as in Fig. 2(a). The magnetic field was set at 500 G which is at the maximum of the total PL intensity change from Fig. 2(a). To *maximize* eventual effects of the temperature rise due to the lattice heating, the PL spectrum was in this particular case measured in phase with chopped laser excitation at 200 Hz, and the microwave power was set at the maximum power available (577 mW). By measuring phase sensitively at a rather low chopping frequency (200 Hz), we are "looking" at the slow-varying components (in the ms range) of the PL intensity, induced by the microwaves chopped at 6 kHz. It can be seen that the different bound excitons are strongly affected by the presence of the microwave field, especially near 1 eV a strong enhancement of PL intensity can be observed. The high PL intensity change obtained under such conditions has been advantageous for the measurements at selected photon energies.

In order to study the spectral dependence of the ODMII signal the magnetic field was set at 500 G, and the signal intensity versus PL photon wavelength has been measured in phase with chopped microwaves at 6 kHz (with stationary laser excitation). In this case we are looking at the fast components of the microwave-induced changes of PL intensity and *minimizing* any temperature effects due to lattice heating. The spectral dependence of the ODMII signal (versus photon energy) has then been measured as a function of the applied microwave power from 577 mW down to 99 dB reduction in steps of 1 dB.

In Fig. 3 the spectral dependence of the ODMII signal is shown in phase with chopped microwave power at 577 mW(0 dB). In this measurement the microwave-induced change of the PL intensity can be observed as either a positive (enhancement of PL) or a negative (reduction of PL) ODMII signal. We have also measured the time-resolved ODMII signal and it was found that the ODMII signal was exactly in phase with the applied microwave pulse, with a time constant limited by the response time of the Ge detector (≈ 20 μs). This is an important observation since the bolometric effect is known to be a slow process (in the ms range[28]), and should therefore be phase delayed with respect to the microwave field.[28] This means that a comparison of the PL and ODMII spectra directly demonstrates the influence of microwave field and magnetic field effects on PL intensity. From Fig. 3 it can be seen that there is a dramatic increase of a broad band near 1 eV, IBE lines 5 and 9 are also enhanced, whereas the P_{TO}(BE) PL intensity is totally quenched.

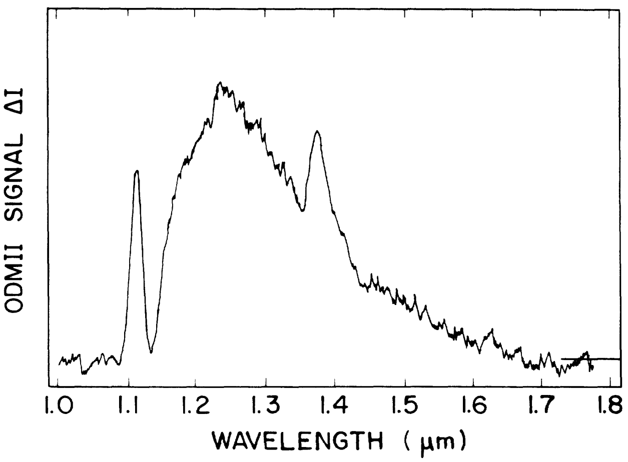

FIG. 3. Spectral dependence of the ODMII signal, measured in phase with chopped microwave field (577 mW) at 6 kHz, and magnetic field (500 G) set at the background signal maximum.

In Fig. 4 the changes in the ODMII signal for IBE lines 5 and 9 and P_{TO}(BE) relative to the PL intensity at zero field, are plotted as a function of the square root of the applied microwave power. The relative changes have been obtained by taking the integrated area of each emission in the ODMII spectra, subtracting the broad spectrum of the superimposed background signal. The absolute changes of the PL intensity have then been obtained by comparing with PL spectra measured at 6 kHz. From the PL spectra we also get the line shape of the broad background signal spectrum, even though the signal-to-noise ratio is rather low. As an effect of this broad background emission, the P_{TO}(BE) emission appears close to the zero level near 1.092 eV in the ODMII spectrum in

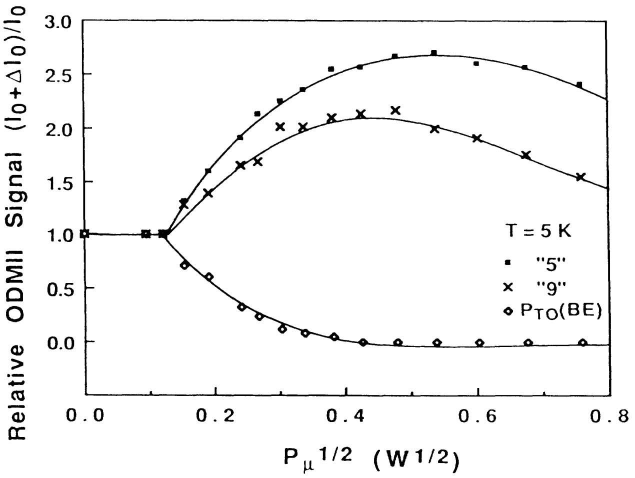

FIG. 4. Relative photoluminescence intensity vs the square root of the applied microwave power at 6 kHz for the phosphorus BE P_{TO} at 1.092 eV and for two isoelectronic bound excitons line 5 at 1.119 eV and line 9 at 0.903 eV (Ref. 27).

Fig. 3, (and not observed as a negative ODMII signal as expected), even though the P_{TO}(BE) emission is totally quenched at this microwave power (577 mW). These reference PL spectra were measured with the applied microwave pulse in phase with the chopped (with an acousto-optic modulator) laser excitation.

It is found that there is a threshold in microwave power close to 20 mW(14 dB) before any change in PL intensity can be observed. Above this energy the shallow bound exciton P_{TO}(BE) decreases and the two IBE's increase in intensity. At around 230 mW (4 dB) the P_{TO}(BE) is totally quenched, whereas IBE line 9 achieves the maximum change in PL intensity corresponding to an enhancement of 100%, followed by a decrease. IBE line 5 has a maximum change in PL intensity at 290 mW (3 dB), corresponding to an enhancement of about 150%, and thereafter decreases.

We have recently performed an investigation of these BE lines in an applied dc electric field,[22] where it-was shown that the PL changes were due to impact ionization. Since the microwave measurements were done on the same Si sample, a direct comparison of PL changes due to dc electric field and microwave electric field and lattice temperature is very relevant. The different behavior of these BE lines under electric-field-induced impact ionization is shown in Fig. 5, while the temperature dependence can be found in Fig. 3 in Ref. 22.

IV. DISCUSSION

In this paper we utilize quite a different dependence of the PL intensity of some deep IBE's, under impact ionization and rise in temperature, respectively, to verify directly that impact ionization of excitons is responsible for nonresonant background signals in ODMR experiments.

The detailed results of the dc electric field-induced impact ionization of IBE's will be presented elsewhere,[22] but some results are discussed here since they are important for the interpretation of our ODMII data. In Fig. 5 it can be seen that the threshold for impact ionization of the shallow donor related BE's is the same as the threshold for increase of PL signal for the two IBE's studied. This is due to an enhancement of recombination via a deep isoelectronic center when shallow predominantly nonradiative BE's are quenched. In the case of deep IBE's it is believed that only one carrier, the one loosely bound, is released as a free carrier during impact ionization. This leaves behind a charged center with a Coulomb-attractive potential, and probably explains how the appearance of new, otherwise not observed broad band(s), attributed to free-to-bound recombination between free electrons and holes localized at the isoelectronic defects.[22] The quite different dependences of PL intensity of IBE's under impact ionization conditions and with a variation in lattice temperature, respectively, have

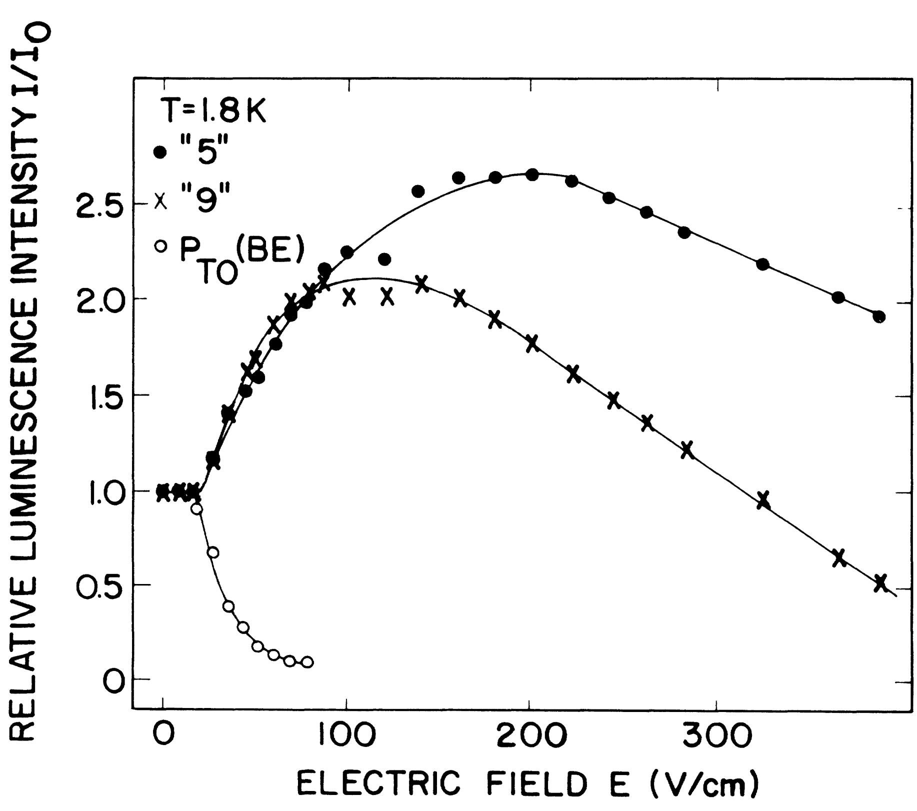

FIG. 5. Relative photoluminescence intensity vs applied electric field for the phosphorus BE P_{TO} at 1.092 eV and for two isoelectronic bound excitons line 5 at 1.119 eV and line 9 at 0.903 eV.

been attributed partly to the existence or nonexistence of the free excitons (FE's).[22] By increasing the electric field in the impact ionization process FE's and shallow BE's are removed, promoting recombination via deeper centers such as IBE's, and also free-to-bound recombinations.

The new ODMII experiments and the results discussed above allow for direct verification which is the mechanism responsible for the common background signal in ODMR in Si. The experimental results shown in Fig. 3 are already consistent with an important contribution of the impact ionization mechanism to the quenching of shallow BE's and enhancement of deep IBE's. We observe an appearance of a strong broad PL band which is another fingerprint of the impact ionization mechanism, the $P_{\mathrm{TO}}(\mathrm{BE})$ is totally quenched, and IBE lines 5 and 9 enhanced. An increase of another PL line at 1.0 eV is also observed. The identity of this PL line is not known, but was also observed in the same spectrum as IBE line 9 in Ref. 27.

The comparison of the data shown in Figs. 1(a) and 1(b) shows, however, that some lattice heating can occur with a low chopping frequency (200 Hz) and a large magnitude of microwave power (577 mW). The intensity of line 9 is reduced when the PL intensity is measured in phase with chopped laser excitation at around 200 Hz (laser pulse length 2.5 ms). This line is decreasing monotonically with an increase in temperature, as was observed previously.[22] This lattice temperature rise is certainly less than 1 K, but is difficult to estimate by just comparing Figs. 1(a) and 1(b), since also the impact ionization rate is temperature dependent.

However the ODMII signals have been measured in phase (synchronously) with a short microwave pulse. Therefore the spectral dependence of the ODMII spectrum in Fig. 3 clearly shows that the impact ionization mechanism dominates the synchronous changes with microwave pulses at 6 kHz (microwave pulse length 83 μs). The PL intensities of the broad band and the two IBE's, lines 5 and 9, are both observed as an *increase* of their intensity. This result is *only* consistent with the impact ionization mechanism, whereby the competing recombination channels via BE's related to shallow donors and acceptors are quenched. By comparing the relative PL intensities of $P_{\mathrm{TO}}(\mathrm{BE})$, IBE lines 5 and 9 as a function of the square root ($P_\mu \alpha E^2$) of the applied microwave power (Fig. 4), with the corresponding dc electric field dependence (Fig. 5), it is obvious that the impact ionization caused by a dc electric field and caused by a microwave field is quite similar.

Different mechanisms can be proposed to explain the dependence of the ODMII signal on the magnetic field, in the presence of free carriers. Possible mechanisms are a magnetic field dependence on free-carrier capture rates, magnetic-field-induced depopulation of light electrons (as observed[9] in Ge), or thermalization between Zeeman sublevels. Another possibility is the magnetoresistance effect, similar to what we have observed in the dc electric-field-induced impact ionization with a magnetic field applied perpendicular to the applied electric field.[29] The threshold for impact ionization was found to increase with magnetic field strength and the measured electrical current (an indication of the impact ionization efficiency) was reduced in accordance with the magnetoresistance change.[29] It is still premature to conclude which mechanism that dominates the magnetic-field-dependent ODMII signal.

There are some specific conditions that must be achieved in order to observe the ODMII signal in ODMR experiments at higher microwave frequencies, where instead CR-induced PL changes can be observed. At CR conditions the free carriers are accelerated in spiral orbits about the axis of a static magnetic field H. The angular rotation frequency is

$$\omega_c = \pm eH/m^*c \ , \tag{1}$$

where m^* is the effective mass of the free carrier.[30] To obtain a distinct resonant absorption of microwave energy at this frequency it is necessary that $\omega_c\tau \geq 1$, where τ is the carrier scattering time. When a 9 GHz microwave frequency is used, this means that free-carrier scattering times ≥ 20 ps must be obtained in order to observe sharp CR lines. For low-doped Si at low temperatures (≈ 4 K) this is usually obtained ($\tau \approx 70$ ps for electrons and holes was obtained in the early study of CR in Si).[30] For higher-doped or annealed Si samples (as used for the present studies) this is usually not the case. Instead the free carriers will be nonresonantly heated from the electric part of the microwave field and at a certain microwave power the energy is sufficient to cause impact ionization of excitons, which in turn causes an avalanche increase in the free-carrier density.

In the high-frequency electric field breakdown experiments of excitons in Ge reported by Manenkov *et al.*,[17] it was found that the threshold for breakdown was independent of the frequency of the electric field. So the reason why no ODMII signal is observed near zero magnetic field at higher microwave frequencies (≥ 23 GHz) could be due to the shorter effective drift length of the carriers, which might not be long enough to effectively reach the excitons, even if the kinetic energy of the carriers is sufficient to cause impact ionization.

At higher microwave frequencies, however, the CR condition $\omega_c\tau \geq 1$ is usually fulfilled, which causes resonant PL changes observed as the ODCR signal.[4-8] We have made some preliminary experiments on low-doped float-zone Si, where we have observed strong ODCR resonances in the ODMII background signal at 9 GHz microwave frequency, after a threshold in microwave power. Therefore we believe that also the ODCR signal is caused by impact ionization, as suggested by Pakulis and Northrop,[8] who also observed a threshold in microwave power for the observation of resonant PL changes due to electron CR at 22 GHz. This shows that the microwave power is usually enough to cause impact ionization at higher microwave frequencies but then it is only effective when the magnetic field is at or near the condition to fulfill CR.

Therefore studies of impact ionization of excitons in semiconductors can be improved when ODCR signals are observed. By determining the relative intensities of the hole and electron resonances in the ODCR spectrum it should be possible to determine which carrier *type* is mainly released as a free carrier in the bound-exciton breakdown.

V. CONCLUSIONS

In conclusion we have investigated the microwave-induced PL changes of bound excitons in Si. By making a direct comparison of the PL intensity of bound excitons in an electric field with the corresponding PL intensities in a microwave field we show that the main mechanism responsible for the microwave-induced PL changes is impact ionization of excitons. The decrease of the PL intensity has a characteristic threshold which is a fingerprint for the impact ionization mechanism and this was close to 20 mW at 9 GHz microwave frequency in our case. The efficiency of the microwave-induced PL changes was very strong, and caused complete quenching of BE's related to shallow donors, while it typically enhanced the PL intensity of IBE's, in one case by as much as 150%.

More studies are needed to fully understand the microwave-induced PL changes in both nonresonance (ODMII) and under cyclotron resonance (ODCR) for different excitonic systems as a function of, e.g., angular direction of magnetic field, microwave frequency, microwave power, and laser excitation intensity.

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