

Optical Properties II

An Old Defect is Finally Understood and a New One (Invited) Rises to Challenge Us: Part I Experiment and Theory of the Anharmonic Effect in C-H and C-D Vibrations of SiC / Part II Spectra Associated with Stacking Faults in 4H SiC Grown in a Hot Wall CVD Reactor

W. J. Choyke

University of Pittsburgh, USA

Characterization of Bulk and Epitaxial SiC Material Using Photoluminescence Spectroscopy

A. Henry[1], A. Ellison[2], U. Forsberg[1], E. Janzén[1]

[1]Linköping University, Sweden; [2]Okmetic AB, Sweden

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Linköping University, Sweden

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INSA Lyon, France

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Y. Koshka, M. S. Mazzola

Mississippi State University, USA

Properties of the UD-1 Deep Level Center in 4H-SiC

B. Magnusson[1], A. Ellison[2], E. Janzén[1]

[1]Linköping University, Sweden; [2]Okmetic AB, Sweden

An Old Defect is Finally Understood and a New One Rises to Challenge Us

Part 1. Experiment and Theory of the Anharmonic Effect in C-H and C-D Vibrations of SiC

W.J. Choyke¹, R.P. Devaty¹, S. Bai¹, A. Gali², P. Deák², G. Pensl³

1. Dept. of Physics and Astronomy, Univ. of Pittsburgh, Pittsburgh, PA 15260, USA

Phone (412) 624 9251, FAX (412) 624 1479, E-Mail <choyke@imap.pitt.edu>

2. Budapest University of Technology and Economics, Budapest, Hungary

3. Univ. of Erlangen-Nürnberg, Erlangen, Germany

Almost thirty years ago the C-H and C-D vibrations in polytypes of SiC were discussed in some detail [1-4]. At that time hydrogen in semiconductor was more of a curiosity than an important issue. Today, hydrogen in semiconductors in general and in SiC in particular is an area of important experimental and theoretical study [5,6]. With improved methods of crystal growth, ion implantation, spectroscopy and theoretical machinery we now revisit the anharmonic nature of the C-H and C-D vibrations in SiC.

Theory [6] has indicated that the $V_{Si} + H$ complex is likely to be formed only in crystals where the Fermi level extends from the middle of the band gap to somewhere close to the valence band. This has not been fully confirmed by experiment but we believe that all the SiC used in these experiments was slightly p-type. Data is obtained from CVD films as well as boules and Lely crystals.

The C-H and C-D fundamental stretch modes and their higher harmonics observed here as vibrational replicas of bound exciton recombination can be discussed using a model based on the one dimensional Morse potential used by Fowler et al. [7] to analyze infrared OH stretch modes in insulators. Table I shows the measured energies of the fundamental and second harmonic of C-H and C-D stretch vibrations measured in a 6H SiC sample implanted with both hydrogen and deuterium. In the table the transitions are labeled $\Delta E_{nm} = E_n - E_m$ where the subscripts are the indexes of the energy levels of the anharmonic Morse potential. The two parameters ω and x are adjusted to precisely account for the energies of the fundamental and second harmonic of the C-H vibrations of H_3 . The predicted values for the C-D fundamental and second harmonic are shown in Table I. The Morse potential accounts quite well for both the anharmonicity and the isotope effect. Table II lists the measured and predicted values for the C-H vibrations for the H_3 spectrum.

Table I

6H SiC:D,H Spectrum	Data (meV)		Model (meV)	
	ΔE_{10}	ΔE_{21}	ΔE_{10}	ΔE_{21}
H_3	369.5	353.6	369.5	353.6
D_3	273.7	264.7	273.9	265.3

Table II

6H SiC:H	ΔE_{10} (meV)	ΔE_{21} (meV)	ΔE_{32} (meV)
Data	369.1	353.5	337.7
Model	369.1	353.5	337.9

We have also carried out an ab initio density functional calculation for the $V_{Si} + H$ complex in 3C SiC. The details of the calculation method and tools can be found in a paper by Aradi et al. [6]. To calculate the anharmonicity of the C-H stretch frequencies we use the method explained in Jones et al. [8]. The calculated energies of the second and third harmonics of the C-H vibrations are 349.6 meV and 335.1 meV in good agreement with the experimental values. The calculated anharmonicity parameter as it was defined in [8] is 14.5 meV, close to the measured value of 15.6 meV (using Table II). The calculations strongly support the fact that the measured H_1 , H_2 and H_3 spectra in 6H SiC are indeed related to the fundamental and higher harmonic vibrational modes of the C-H bond in the $V_{Si} + H$ complex.

Part 2. Spectra Associated with Stacking Faults in 4H SiC Grown in a Hot Wall CVD Reactor

S. Bai¹, G. Wagner⁴, E. Shishkin¹, W.J. Choyke¹, R.P. Devaty¹, M. Zhang⁵, P. Pirouz⁵, T. Kimoto⁶

4. Institut für Kristallforschung, Berlin, Germany

5. Case Western Reserve Univ., Cleveland Ohio, USA

6. Kyoto University, Kyoto 606-8501, Japan

Bergman et al. [9] reported an interesting study on the effect of stressing hot wall CVD grown 4H SiC P-N junctions during extended high voltage testing. Of particular import to the work to be reported here is their finding of a new set of photoluminescence lines associated with areas of the junctions which are also found to have stacking faults after a long period of high voltage operation. We now report our findings of variations of this P-N junction spectrum in as grown films of 4H SiC and further evidence for their association with stacking faults.

The undoped epitaxial films of 4H SiC were grown at IKZ-Berlin in a hot-wall CVD reactor at about 1550°C and were about 30µm thick and in a vertical hot-wall reactor at Kyoto University and were about 70 µm thick.

How do our results compare with those of Bergman et al [9] in P-N junctions produced from hot-wall 4H SiC epitaxial films and also of approximately 30µm thickness? Dr. Bergman very kindly sent us a spectrum of the photoluminescence from a P-N junction with stacking fault features presumably induced by long term testing at high voltage. The spectrum was taken at 2K using a FreD laser. In our spectra, taken with a FreD laser we are in better agreement with the Bergman spectrum than those obtained using a He-Cd laser. We are in good agreement as to the temperature variation of this spectrum as well as to the relative decrease of room temperature lifetime in regions decorated by stacking faults.

We conclude that a number of spectra associated with stacking faults can be seen in hot-wall CVD grown 4H SiC. Possibly, the similarly observed spectra in P-N junctions exposed to long term high voltage operations were made on similar material as ours and the regions with spectra associated with stacking faults had these features amplified by the stress induced by the high voltage electrical testing. Finally, an interpretation of the nature of seven of these stacking fault spectra will be given.

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Characterization of bulk and epitaxial SiC material using photoluminescence spectroscopy.

A. Henry¹, A. Ellison², U. Forsberg¹ and E. Janzén¹

¹ Department of Physics and Measurement Technology, Linköping University, 581 83 Linköping, Sweden

² Okmetic Hans Meijersväg 2, 583 30 Linköping, Sweden

tel: +46 13 28 24 14, fax: +46 13 14 23 37, e-mail: ahy@ifm.liu.se

We are using low temperature photoluminescence (LTPL) to evaluate the quality of SiC wafers and are able to characterize up to 2 inch diameter wafers (with or without epilayers) at low temperature (2K).

Up-to six full wafers can be placed in a bath cryostat, which is mounted on a movable support. The luminescence excited either by the 244nm line or the 351 nm of an Ar⁺ laser is dispersed by a single monochromator on which a UV sensitive CCD camera is mounted to detect rapidly the PL spectra.

At 2K sharp PL spectral lines are observed and their energy position depends on the particular polytype, which allows polytype identification (Fig.1) enabling us to draw some "polytype" map of wafers as shown in Fig.2. This technique is very useful when applied to low-doped semi-insulating wafers which are color-less, as opposed to highly doped material.

The dopants and residual impurities related LTPL lines (as such as Ti in Fig.1) can also be detected. For the nitrogen donor a quantitative analysis can be made.

For low doped material ($n < 3 \cdot 10^{16} \text{ cm}^{-3}$) the relative intensity between the nitrogen bound-exciton no-phonon line (such as Q₀ in 4H-SiC, see Fig.1) and one of free-exciton line (such as I₇₆) has allowed a quantitative estimation of the doping concentration [1]. This behavior has

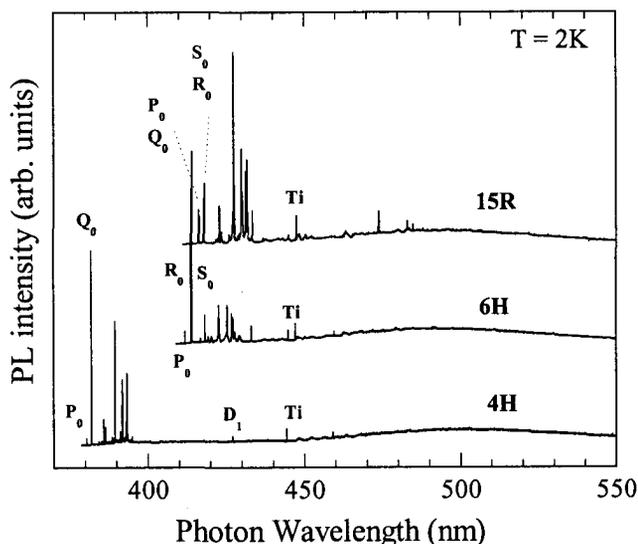


Fig.1 : PL spectra recorded on three bulk samples, at 2K showing three polytypes of SiC with low doping level ($n < 5 \cdot 10^{16} \text{ cm}^{-3}$).

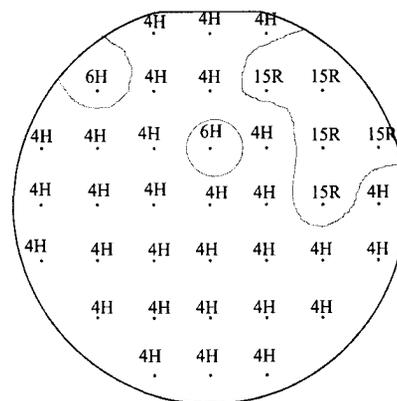


Fig.2: Polytype map of a substrate as determined from PL spectra at 2K.

been used to draw doping maps of 4H-SiC layers [2]. However this is limited to nitrogen concentration below $3 \cdot 10^{16} \text{ cm}^{-3}$. In this work we will show that for medium doped samples ($1 \cdot 10^{16} < n < 3 \cdot 10^{18} \text{ cm}^{-3}$), the relative intensity between the two nitrogen bound-exciton no-phonon lines is correlated to the nitrogen doping concentration (see Fig.3). For highly doped 4H-SiC ($n > 5 \cdot 10^{18} \text{ cm}^{-3}$) a broad PL band (Fig.4) is observed which we recently have shown to be related to the heavy nitrogen doping itself [3]. The energy position of this band can thus be used for calibration of the doping concentration by comparison with data obtained by Secondary Ion Mass spectrometry. This behavior can also be used for bulk material to perform a nitrogen-doping map of the substrate.

This complete understanding of the near band gap emission for 4H polytype can be used and enables the determination of the nitrogen concentration by measuring the LTPL in the very large range of doping available today, e.g. from low 10^{14} to 10^{19} cm^{-3} .

Similar behavior is also observed for 6H-SiC material and will be presented.

Preliminary results for heavily Al-doped (p-type) epilayer show also the presence of a large band in the near band gap emission. Its energy position shifts towards lower energy with increasing doping, as in the case of n-type.

We will show that this knowledge is very useful for the understanding of the photoluminescence from thin epitaxial layer structures with various different epilayers such as MESFET structures, or from low doped epilayers when contribution from the substrate appears in the PL spectrum.

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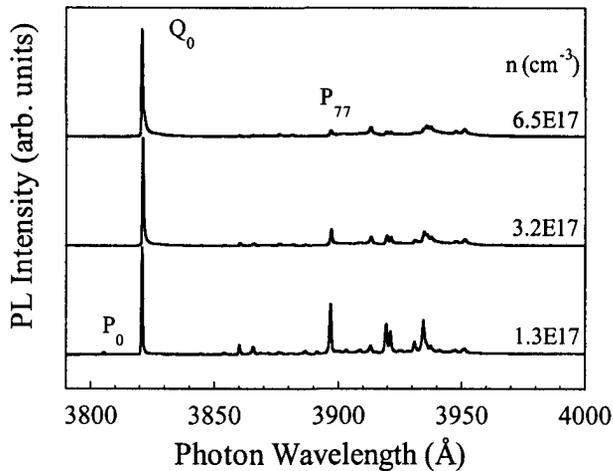


Fig.3 : PL spectra of medium doped 4H-SiC epilayers recorded at 2K

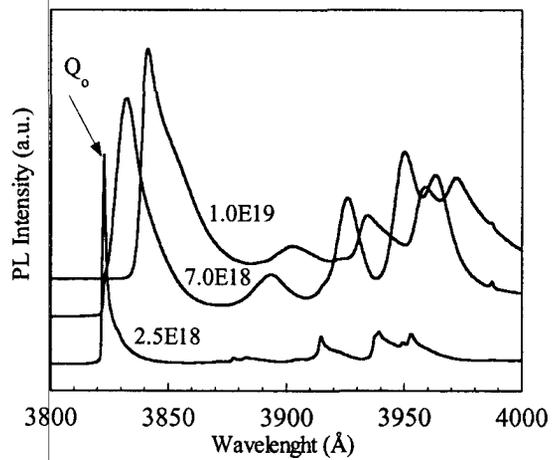


Fig.4 : PL spectra of heavily doped 4H-SiC epilayers recorded at 2K

Photoconductivity of Low-Doped and Semi-Insulating 4H-SiC and the Free-Exciton Binding Energy

I.G. Ivanov, J. Zhang, L. Storasta and E. Janzen

*Department of Physics and Measurement Technology, Linköping University,
S-581 83 Linköping, Sweden*

Phone: +46 13 - 28 25 32

Fax: +46 13 - 14 23 37

E-Mail: iiv@ifm.liu.se

The free-exciton (FE) binding energy, E_{bx} , is a fundamental parameter, however, its exact value is far from being well established in any of the SiC polytypes. Values obtained in previous studies often do not agree (see Ref. [1] for a review), which determines the need in further experimental work employing techniques different from those used before. In a recent paper (Ref. [2]) we demonstrated the use of photoconductivity for studying the structure of the exciton absorption edge, and reported on the determination of E_{bx} in 6H-SiC. The purpose of the present work is to use similar approach for studying the structure of the excitonic bands in 4H-SiC, including the determination of the FE binding energy, as well as to develop further the interpretation of the photoconductivity spectrum near the fundamental absorption edge.

The basic idea behind the application of photoconductivity for finding the position of the conduction band edge (which coincides with the edge of the exciton continuum) is the electroneutrality of the excitons. Therefore, no contribution to the photocurrent from free excitons is expected, if the excitons are created by the exciting light in bound states (below the exciton continuum), whereas some current is anticipated if excitons are created in non-bound states (above the continuum). In this latter case, the electron and hole can be separated in the applied electric field before they form an exciton in a bound state, and thus contribute to the photocurrent. The threshold of this intrinsic photocurrent is at the conduction band edge E_g plus the lowest energy ($\hbar\Omega_0$) of a phonon insuring the momentum conservation in the light absorption process.

However, in real samples the threshold of the photocurrent occurs at the fundamental absorption edge (i.e., the excitonic bandedge plus $\hbar\Omega_0$). Thus even excitons created in bound states contribute to the photocurrent due to Auger recombination at impurities, as discussed in detail in Ref.[2]. Furthermore, this extrinsic photocurrent usually is much larger than the intrinsic one, so the latter is obscured (see Fig.1, sample B). However, in samples of exceptionally low residual doping, the extrinsic photocurrent is seen to saturate, and even decrease at higher photon energies, as shown in Fig.1 (sample A). This enables the observation of the intrinsic counterpart in the current, and also of additional thresholds in it due to involvement of different momentum-conserving phonons in the photon absorption process, as denoted by arrows in Fig.1. From the spectra, the free-exciton binding energy in 4H-SiC can be estimated to be in the interval 20 - 27 meV. This is close to the value of 20 meV, estimated in Ref.[3] from electroabsorption measurement. An estimation of this

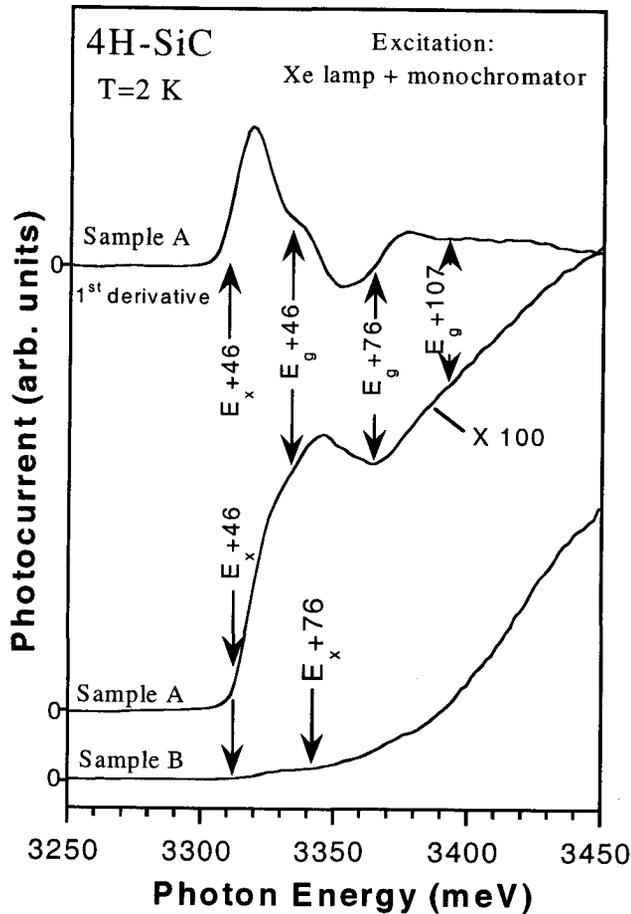


Fig.1. Comparison of the photoconductivity spectra of very low doped sample (A), showing saturation of the extrinsic photocurrent and contribution from the intrinsic one, and higher doped sample (B), dominated by the extrinsic photocurrent. The numbers above the arrows are the energies (in meV) of the main phonons assisting the light absorption. The derivative of spectrum A (on top) is provided in order to make more prominent the thresholds. Note the scale change for sample A.

the paper. The temperature dependence of the photoconductivity spectra of various samples is also considered.

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quantity based on the isotropic hydrogenic model for 4H-SiC yields the value 34 meV, however, such calculation has very limited accuracy due to the high anisotropy of the electron and hole effective masses in 4H-SiC. Precise calculation accounting for this anisotropy are not available by now.

The reasons for saturation (and following decrease) of the extrinsic photocurrent are not simply in saturation of only residual impurity (as suggested previously in Ref. [2]), but are closely related to the presence of compensation. This is confirmed by the observation of saturation also in higher-doped (but compensated by deep levels) semi-insulating bulk 4H-SiC. The near-bandgap photoconductivity spectra of some highly doped semi-insulating samples do not show contribution from the intrinsic photocurrent because of the much higher rate of capturing of excitons. However, semi-insulating samples which exhibit free-exciton related emission in the low temperature photoluminescence spectrum, show also contribution from the intrinsic photocurrent. The photoconductivity properties of various semi-insulating samples is discussed in

UV Scanning photoluminescence spectroscopy investigation of 6H and 4H SiC

L. Masarotto, J.M. Bluet, and G. Guillot

Laboratoire de Physique de la Matière - CNRS (UMR5511)
INSA de Lyon - Domaine Scientifique de la Doua -Bâtiment Blaise Pascal
7, avenue Jean Capelle, 69621 Villeurbanne Cedex - FRANCE
Tel : 33 4 72 43 87 32 ; Fax : 33 4 72 43 85 31 ; e-mail : bluet@insa-lyon.fr

Great progress have been made in the recent years in SiC bulk growth and epitaxy. Nevertheless, some problems still limit the rise of high performance and high reliability devices. In the case of SiC wafers, for instance, while the micropipes density has been strongly reduced down to 10 cm^{-2} , the dislocation density is still of typically 10^4 cm^{-2} . These defects, reproduced in the epitaxial layers, are deleterious for high power devices. Additionally, in the case of semi-insulating substrates, deep levels acting as carrier traps affect the devices performance [1]. For epitaxial layers, the main problem resides in the diminution of doping inhomogeneities exceeding 20% on a two inches wafer. The presence of polytypes inclusions in substrates as well as in epitaxial layers is also a recurrent problem in SiC wafers. In order to analyse these defects, to understand their origin and their impact on devices performance, non destructive and few time consuming characterization tools are strongly needed by the material growers.

For this tight quality control of the wafers, we have developed and adapted for the UV excitation a scanning photoluminescence (SPL) apparatus, initially conceived for III-V compounds analysis. Indeed the first reported results [2, 3] indicate that SPL is a very promising tool for SiC material characterization. In our equipment, the PL imaging is obtained by scanning the sample, fixed to an x-y stage with $1 \mu\text{m}$ minimal step. The excitation is provided by a doubled Ar^+ laser beam (244 nm) focused by an achromatic microscope objective (x52). With this excitation the spot diameter is about $2 \mu\text{m}$ and the penetration depth is below $1 \mu\text{m}$ in 4H-SiC. Nevertheless, one must keep in mind that the diffusion length of photoexcited carriers can be much more larger. In this case the spatial resolution is not limited by the apparatus but the material himself. The PL signal can be either directly collected, giving integrated PL intensity, either dispersed using a monochromator, giving spectrally resolved PL (1 nm of spectral resolution in the range 300 nm – 800 nm).

The optical signature of different defects have been investigated. For example, an exhaust of the photoluminescence intensity near the dislocations has been evidenced. This effect comes from the gettering effect of non radiative traps around dislocations which results in a denuded zone in the vicinity of the defect. From this observation, the density on epitaxial layers can be obtained without using chemical etching. The presence of micropipes can also be detected by photoluminescence mapping without using KOH etching. We will show that non emergent micropipes (not visible with optical microscope focussed on the surface) can be revealed. Some examples of polytypes mixtures on epitaxial layers (cubic inclusions) and bulk samples (4H and 6H distinct zone) will also be presented. An other application of SPL is the analysis of sample surface after different technological steps. Indeed, the PL signal intensity is very sensitive to the non radiative recombination at the surface. For example, we

have observed contrast inversion on etched surface (using RIE etching) before and after a $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$ 2:1 + HF cleaning. Before the cleaning, the integrated PL intensity is stronger on the etched zone. After cleaning, the PL intensity is strongly reduced and becomes lower than in the rest of the sample (Figure 1). The understanding of this effect is not clear yet. A possible explanation is that after etching, we collect a strong fluorescence from etching residue (polymerised carboxide group) which disappears after the cleaning.

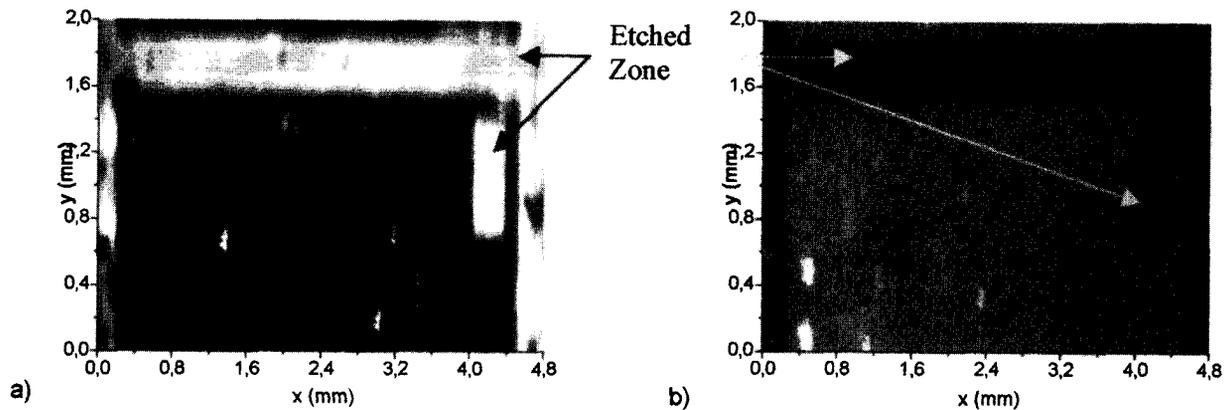


Figure 1 : a) Integrated PL intensity image of a MESA structure. The etched zone surrounding the central rectangle shows higher luminescence than the rest of the sample. b) Same image, after chemical cleaning. The etched zone has now a weaker luminescence.

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Photoluminescence Investigation of Hydrogen Interaction with Defects in SiC.

Yaroslav Koshka, Michael S. Mazzola

Department of Electrical & Computer Engineering, Mississippi State University, Box 9571, Mississippi State, MS 39762

phone: +1-662-325-2411 fax: +1-662-325-9478 email: ykoshka@ece.msstate.edu

Hydrogen interaction with donors and acceptors in SiC has been extensively investigated in the past. Hydrogen was shown to electrically passivate acceptors in 4H and 6H SiC.^{1,2} Results of thermal admittance spectroscopy (TAS) measurements have shown that hydrogen can form complexes with both Al and B acceptors.³ Hydrogen incorporation and out-diffusion were also investigated by monitoring changes in a PL emission attributed to a hydrogen-vacancy complex.^{2,4} However, there were no successful attempts to correlate hydrogen-related processes in SiC with changes in acceptor-related photoluminescence. A possible passivation of lattice damage related defects with hydrogen also was suggested in the past⁵ however no experimental data confirming this has been reported.

In this work, we investigated changes in hydrogen, aluminum, and lattice damage related PL lines in 4H and 6H-SiC samples subjected to hydrogenation in hydrogen plasma. Hydrogen incorporation in the epilayers was evidenced by appearance of H-related PL peaks (Fig.1). In addition a significant reduction in a relative intensity of Al bound exciton (Al-BE) photoluminescence in p-type epilayers occurred after hydrogenation (Fig. 1). It was also observed that the intensity of Al-BE PL in the hydrogenated sample was decreasing with time during the measurements. Prolonged excitation with above band-gap light caused a gradual reduction and finally a complete disappearance of Al-BE emission, which was accompanied with a slight increase of R_0+S_0 N-BE photoluminescence as shown in the bottom spectrum of Fig. 1. The kinetics of quenching of the Al-BE PL is shown in the inset to Fig. 1.

The possibility for passivating lattice damage related defects with hydrogen was investigated by hydrogenating samples that had been implanted with nitrogen and annealed at 1500°C to form D_I and D_{II} centers.^{6,7} After the hydrogenation was performed under conditions that allowed the hydrogen incorporated in the epilayer to reach the depth of the maximum concentration of D_I centers, a significant reduction of the intensity of D_I photoluminescence with respect to the intensity of N-BE peaks was observed (Fig. 2). The stability of the passivated D_I complex was investigated by performing annealing at different temperatures. An annealing at 540°C for 2 hr caused only partial recovery of D_I PL intensity (Fig. 2). The released hydrogen contributed to an increase of H-related PL as shown in the figure. A partial recovery of Al-BE PL was also observed after annealing at this temperature (not shown). Annealing at the higher temperature of 1100°C for 1 hr caused further recovery of D_I PL, which was accompanied by a complete disappearance of H-related PL due to hydrogen outdiffusion from the epilayer (the bottom spectrum in Fig. 2). Also, the shape of Al-BE region of the spectrum was completely restored to its pre-hydrogenation form due to Al dehydrogenation.

The observed changes in the Al-related and the D_I photoluminescence are attributed to a passivation of corresponding defects with hydrogen after the hydrogenation and to subsequent de-passivation of these complexes after the annealing. Possible mechanisms for the observed transient decay of the remaining Al-BE PL after hydrogenation caused by the light excitation are discussed in relation to

the recombination enhanced defect reaction.⁸ Additional experiments are conducted to verify also a possible passivation of D_{II} centers that were formed after ion implantation and annealing.

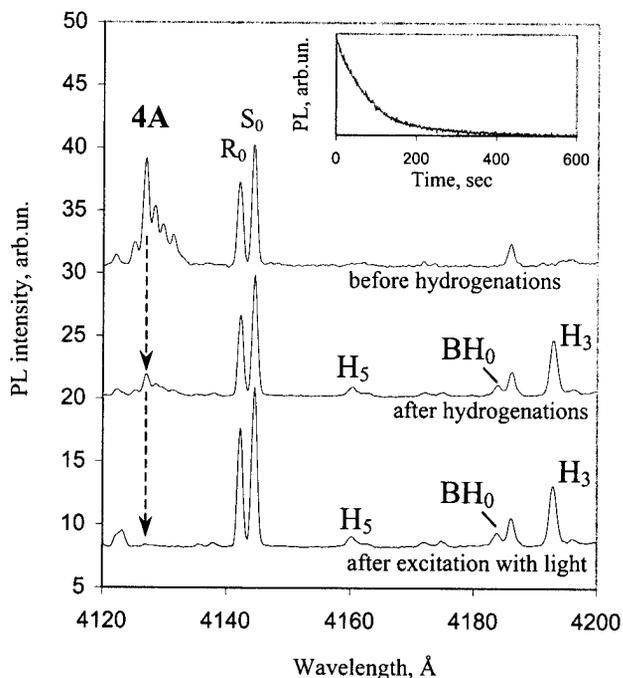


Fig. 1 Photoluminescence spectra before and after plasma hydrogenation, and after 40 min of excitation with above band-gap light. A reduction in 4A Al-BE PL line can be observed after hydrogenation. Light excitation caused further gradual reduction and disappearance of Al-BE PL line 4A. Quenching kinetics for 4A line under excitation is shown in the inset.

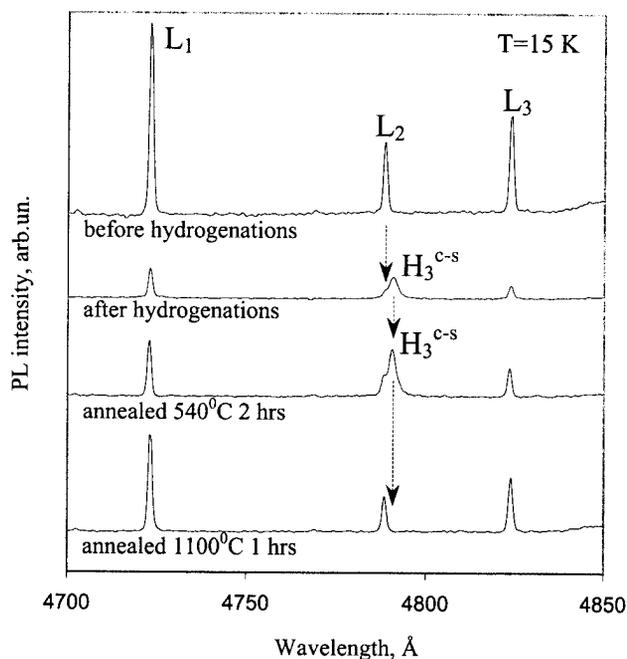


Fig. 2 Changes in D_1 photoluminescence (lines L_1 , L_2 , and L_3) stimulated by hydrogenation and annealing. Hydrogenation caused significant reduction of D_1 PL intensity and an appearance of the H-related PL (H_3^{c-s}). 540°C anneal stimulated partial de-hydrogenation of D_1 centers accompanied with an increase of H-related PL. More D_1 centers are de-passivated after 1100°C anneal, which is accompanied with a complete disappearance of H-related PL due to H outdiffusion from the epilayer.

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Properties of the UD-1 deep level center in 4H-SiC

B. Magnusson^{1*}, A. Ellison², and E. Janzén¹

¹Department of Physics and Measurement Technology
Linköping University, SE-581 83 Linköping, Sweden

²Okmetic AB, Hans Meijers väg 2, SE-583 30 Linköping, Sweden

*Phone: +46 13 282476, Fax: +46 13 142337, E-mail: bjmag@ifm.liu.se

Deep levels have large impact on the electrical and optical properties of SiC. Besides the known deep levels with optical transitions in the infrared region such as vanadium, chromium and the silicon vacancy, there are a number of so far unidentified deep level centers. The UD-1 center (unidentified defect 1) in 4H SiC is one of the most interesting ones, since it gives rise to specific dominating infrared absorptions lines in semi-insulating HTCVD substrates[1]. This lines can also be found in the luminescence from commercial p-type substrates. The properties of the semi-insulating substrates have been studied after different annealing steps [2] and it is shown that the intensity of the UD-1 absorption increases after a 1600°C but it is unchanged after an 800°C annealing. Some of the optical properties and the electronic structure of the UD-1 defect in 6H-SiC were presented earlier [3] and indicate that the defect is either a substitutional defect or a complex along the c-axis.

More information of the defect is needed to understand the mechanism for how the semi-insulating material is compensated and which defect is the active deep level. Therefore we have in this study concentrated on the UD-1 defect in 4H-SiC. The measurements which earlier have been made on the 6H polytype are now also done in 4H together with new experiments such as photoluminescence excitation and absorption measurement at different temperature (from 2 K and up). The optical transitions of the UD-1 defect can be studied with both absorption and luminescence measurements. It consist of two sharp no phonon lines at 1.0585 and 1.0595 eV (Fig. 1.), the line width is less than 0.2 meV at low temperature.

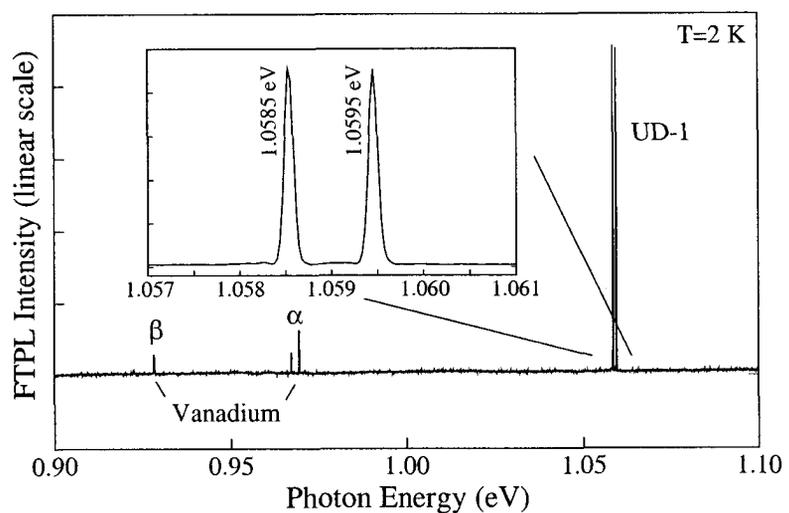


Fig. 1. FTPL spectrum of a semi-insulating 4H-SiC HTCVD sample annealed at 1600°C. Note that the vanadium FTPL is a factor 7 weaker than the UD-1 luminescence.

To be able to understand what role the UD-1 defect plays, the temperature dependence of the UD-1 absorption is studied. The absorption intensity is found to decrease with

increasing measurement temperature. At 80 K the absorption is completely quenched. From an Arrhenius plot of the temperature dependence of the intensity of the UD-1 absorption we can deduce that the thermal activation energy is less than 100 meV.

The Zeeman splitting of the UD-1 defect lines are presented in Fig. 2. The high energy line split into two lines and the lower energy line into four lines. The g values and the electronic structure for the defect will be presented.

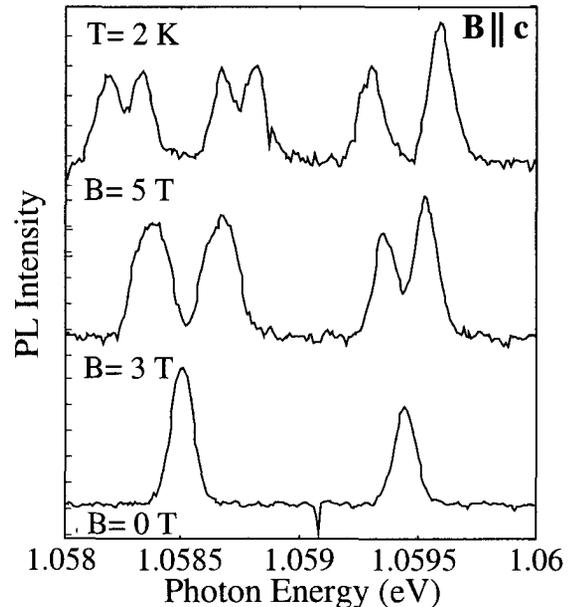


Fig. 2. The splitting of the two lines in the UD-1 defect at different magnetic field, with B parallel to the c -axis.

An isochronal annealing study of the UD-1 center will also be presented. The annealing behavior of the defect is investigated in detail between 800°C and 1600°C. This is important information to compare to how other defects in SiC behave at different annealings. It is also a result that could be used to compare the optical absorption signal to other measurement methods, such as EPR or DLTS.

The level scheme of the UD-1 defect and the annealing behavior of the defect will be discussed in detail in order to understand the origin of the absorption increase after high temperature annealing.

References:

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