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Citation: Chin. Phys. B . 2019, 28(1): 017101. doi: 10.1088/1674-1056/28/1/017101

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Photoluminescence in fluorescent 4H-SiC single crystal adjusted by B, Al, and N ternary dopants*

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(Received 13 September 2018; revised manuscript received 29 October 2018; published online 30 December 2018)

This paper reports the sensitive effect of photoluminescence peak intensity and transmittance affected by B, Al, and N dopants in fluorescent 4H-SiC single crystals. The crystalline type, doping concentration, photoluminescence spectra, and transmission spectra were characterized at room temperature. It is found that the doped 4H-SiC single crystal emits a warm white light covering a wide range from 460 nm to 720 nm, and the transmittance increases from ~10% to ~60% with the fluctuation of B, Al, and N ternary dopants. With a parameter of C_{D-A} , defined by B, Al, and N concentration, the photoluminescence and transmittance properties can be adjusted by optimal doping regulation.

Keywords: photoluminescence property, luminescence adjustment, N–B–Al codoped 4H-SiC

PACS: 71.20.Nr, 42.70.-a, 78.20.-e, 78.55.-m

DOI: 10.1088/1674-1056/28/1/017101

1. Introduction

In recent years, fluorescent silicon carbide (*f*-SiC) has attracted intense interest for it can be used as a wavelength converter and was expected to substitute the phosphor layers in white light-emitting diodes.^[1–3] Previous researchers reported that *f*-SiC had excellent properties compared to that of red–green–blue three colors phosphor, such as higher thermal conductivity, more easily controlled dopant, higher luminescence efficiency, and smaller lattice mismatch for nitride epitaxy.^[4] In Refs. [5] and [6], researchers suggested a promising conceptual structure which combined with a gallium nitride layer, a nitrogen and boron (N and B) codoped SiC layer, and a N and aluminum (Al) codoped SiC layer to assemble into a new white light-emitting diode. In this structure, the N and B codoped SiC emits orange–yellow light while the N and Al codoped SiC emits green–blue light when they are excited by the gallium nitride ultraviolet light source. A warm white light emission with a color rendering index of 94^[7] can be obtained. For these reasons, the *f*-SiC is regarded as a promising fluorescent material.

As far as we know, the photoluminescence properties of Al, Ga, or B acceptor and N donor doped SiC layer have been studied by Ikeda *et al.*^[8] in 1979. However, this pioneer *f*-SiC compound displayed an extremely inefficient luminescence property. For its indirect band gap property, a chief worry in the study of *f*-SiC is that the quantum efficiency may be very small, estimated to be about 10^{−5}%, which will obstruct its application in LED devices. A breakthrough work was re-

ported by Kamiyama *et al.*^[9] based on donor-to-acceptor pairs (DAPs) recombination mechanism in 2006. In their work, the authors observed a high-efficiency visible light emission (estimated to be about ~95% at 250 K) in B and N codoped 6H-SiC epilayers. This result greatly encouraged the research activities on *f*-SiC material. After that, Jokubavicius *et al.*^[10] studied the effects of source material on epitaxial growth of N and B codoped SiC layers grown by using a fast sublimation growth process method. They demonstrated that high quality N and B codoped SiC layers are suitable to be used as light converter and emission warm white light with a peak located at about 580 nm. Manolis *et al.*^[11] studied the non-radiative decay of majority electrons under a wide temperature range using a time-resolved free-carrier absorption technique. They found that the recombination channel seems to compete with the deep acceptor to donor pair on visible light emission at and below 300 K in N and B codoped 6H-SiC. Besides B and N co-doping, another kind of donor–acceptor co-doping pair, like Al and N codoped SiC, was also been explored. Canassel *et al.*^[12] prepared N and Al codoped 4H-SiC single crystals by using their home-made physical vapor transport reactor. An exciting photoluminescence spectra different to that of a seed with a broad peak located at around 520 nm was found in their work.

Although photoluminescence properties of *f*-SiC regulated by Al–N or B–N binary dopants codoping have been widely studied, few research work has focused on the photoluminescence of *f*-SiC with ternary dopants of B–Al–N. Besides, the influence of dopants on the photoluminescence and

*Project supported by the National Key Research and Development Program of China (Grant Nos. 2017YFB0405700 and 2016YFB0400400), the Young Scientists Fund of the National Natural Science Foundation of China (Grant Nos. 51602331 and 61404146), and the Shanghai Science and Technology Innovation Action Plan Program, China (Grant No. 17511106200).

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transmittance properties is still not clear,^[13] especially for the multi-elements codoped SiC. Furthermore, SiC single crystal is an indirect band gap semiconductor, and has more than 200 crystalline structures, such as 3C-SiC, 4H-SiC, 6H-SiC, and 15R-SiC. For example, the band gaps of 3C-SiC, 4H-SiC, 6H-SiC, and 15R-SiC are 2.41 eV, 3.29 eV, 3.05 eV, and 3.01 eV, respectively. This makes the electron–hole pair recombination quite complicated when introducing deep or shallow donor–acceptor impurity energy levels in the band gap.^[14] Therefore, it is necessary to study the factors which affect the luminescence properties of *f*-SiC before this material is widely used in future.

In this paper, high quality B, Al, and N codoped 4H-SiC single crystals were grown by using the physical vapour transport (PVT) method. The Raman spectroscopy, photoluminescence properties, doping concentrations, and transmission spectra were measured. The relationship between doping concentrations and optical properties of the doped SiC crystal was studied. The purpose is to explore a suitable approach to adjust the luminescence and transmittance of *f*-SiC by B, Al, and N ternary elements co-doping.

2. Experiment

B, Al, and N ternary elements codoped 4H-SiC single crystals were grown by the PVT method. Detailed preparation processes have been described in a previous report.^[15] In this article, the B dopant was introduced and controlled by adjusting the precise weight ratio of boron carbide and SiC powders. The Al dopant was introduced and controlled by adjusting the precise weight ratio of aluminum and SiC powders. The N dopant was introduced and controlled by adjusting the mass flow ratio of N₂ and Ar in the growth atmosphere. For convenience, the samples herein with different proportions of B, Al, and N dopants were labeled as SiC-I, SiC-II, SiC-III, and SiC-IV in the following parts.

The crystalline type of SiC-I to SiC-IV was characterized by Raman spectroscopy excited by a 514 nm laser. The photoluminescence images/spectra of as-grown samples were characterized by using fluorescence spectrometer (Edinburgh FLS980, Edinburgh Instruments Ltd.) excited by a 325 nm laser source at room temperature. In particular, the photoluminescence image of sample SiC-IV, as a representative image, was collected by using 325 nm exciting laser source with a power of 5 mJ. When the sample SiC-IV was exposed to a violet light with a spot diameter of 10 mm, the fluorescence image was captured by a digital camera (G15, Canon digital camera) from opposite direction of the laser. The doping concentrations of B, Al, and N in 4H-SiC crystals were detected by secondary ion mass spectra (SIMS, EAG Laboratories) system. The transmission spectra (Carry 5000 UV-Vis-Nir spectroscopy, Agilent Technologies Inc.) were measured

in a wavelength range from 200 nm to 800 nm at room temperature.

3. Results and discussion

SiC has more than 200 crystalline structures, and each of them has different physical properties, such as lattice constant, band gap, and sublimation temperature. For this reason, it is important to clarify the crystalline type of acquired SiC crystals before studying their photoluminescence properties. Raman spectroscopy is a molecular vibrational spectroscopic technique that can easily identify the crystalline type of SiC. Figure 1 shows the Raman spectra of samples SiC-I, SiC-II, SiC-III, and SiC-IV excited by a 514 nm laser at room temperature. In this figure, five prominent peaks can be clearly observed for all of the four samples. The peaks located at 203 cm⁻¹, 609 cm⁻¹, 777 cm⁻¹, 964 cm⁻¹, and 1172 cm⁻¹ are ascribed to the folded transverse acoustic (FTA (2/4)) mode of 4H-SiC, the folded longitudinal acoustic (FLA (4/4)) mode of 4H-SiC, the folded transverse optic (FTO (2/4)) mode of 4H-SiC, the folded longitudinal optic (FLO (0)) mode of 4H-SiC, and the secondary order of longitudinal acoustic peak (2LA peak) of 4H-SiC,^[16] respectively. No other Raman spectroscopy peak is detected besides these five peaks, which indicates a crystalline type of 4H-SiC for all the samples obtained herein.

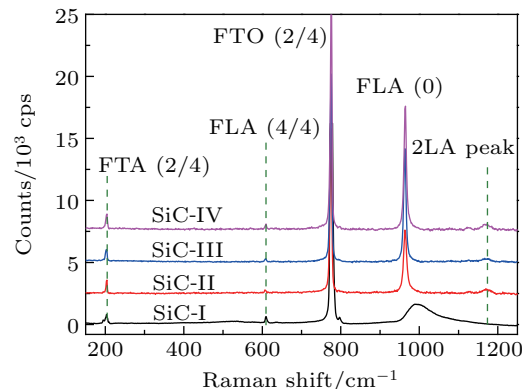


Fig. 1. Raman spectra of samples SiC-I, SiC-II, SiC-III, and SiC-IV excited by a 514 nm laser at room temperature.

Photoluminescence spectra of B, Al, and N codoped 4H-SiC single crystals were measured at room temperature under a 325 nm exciting laser, as shown in Fig. 2(a). It can be clearly seen that the samples SiC-I, SiC-II, SiC-III, and SiC-IV exhibit a main emission peak located at 527 nm. The emission peaks of samples SiC-II, SiC-III, and SiC-IV spectra cover a wide range from about 460 nm to 720 nm, while the emission peak of sample SiC-I is too low to scarcely seen without magnification. To view the luminescence property visually, an emission photograph of SiC-III excited by a 325 nm laser is present on the inset upper right corner of Fig. 2(a). The B, Al, and N codoped 4H-SiC crystal shows a yellow–green emission with a diameter of 10 mm, which can be clearly seen by

naked eyes, when excited by the ultraviolet laser. Other doped 4H-SiC samples also show such yellow–green emission light. The emission light is corresponding to the peak wavelength of photoluminescence spectra. In Ref. [17], Wei *et al.* reported that N and B codoped 4H-SiC single crystal showed a peak wavelength at about 538 nm with full width at half maximum (FWHM) about 104 nm. This result agrees well with the above photoluminescence measurement.

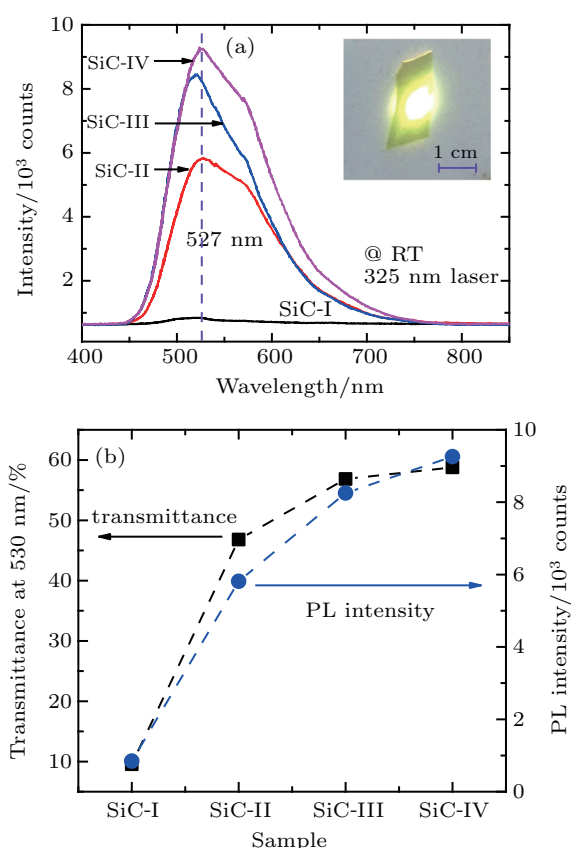


Fig. 2. Photoluminescence spectra and the peak intensity for samples SiC-I, SiC-II, SiC-III, and SiC-IV. (a) Photoluminescence spectra of sample SiC-I to sample SiC-IV excited by a 325 nm laser at room temperature. The inset on the upper right corner is the luminescence of sample SiC-III acquired by digital camera. (b) Peak intensity and FWHM as a function of sample SiC-I to sample SiC-IV. The peak intensity was extracted from the photoluminescence spectra at about 527 nm.

Figure 2(b) shows the peak intensity and FWHM parameter for samples SiC-I, SiC-II, SiC-III, and SiC-IV. The peak intensity is extracted from the photoluminescence spectra at about 527 nm. As shown in Fig. 2(b), an increase trend is obviously observed for the peak intensity with different doping concentration, while the FWHM increases to a maximum value. The FWHM present herein, about 110 nm for B, Al, and N codoped 4H-SiC, is slightly wider than that reported by Wei *et al.* The peak location in photoluminescence spectra also shows a blue-shift phenomenon. These differences in the FWHM and peak location are probably caused by the additional Al doping and the fluctuation in the B and N doping concentrations.

It has been pointed out that the dopants and their concentration play a key role in the luminescence properties of *f*-SiC. Until now, a well-known explanation for the luminescence in *f*-SiC is a donor–acceptor recombination mechanism. In this mechanism, electrons are excited by external ultraviolet light from valence band to conduction band for doped SiC. Thereafter, some of the free electrons and holes occupy the donor states and acceptor states, respectively. These electrons from donor states recombine with the holes from the acceptor states, and release their partial energy as light (other partial energy as phonon for the indirect band gap property of 4H-SiC) in the visible region as seen in the photoluminescence measurement in Fig. 2. From this perspective, the donor and acceptor concentrations is an important factor which will influence the luminescence property of *f*-SiC, because the donor–acceptor recombination rate is proportional to the product of electron density at donor states and hole density at acceptor states.

In order to further analyze the relationship between dopants and the photoluminescence property, the B, Al, and N doping concentration (defined as C_B , C_{Al} , and C_N , respectively) was measured by SIMS under a high precision analysis protocol. The result is listed in Table 1. It can be seen that the concentration of B, Al, and N dopants is about 10^{18} cm $^{-3}$, 10^{16} cm $^{-3}$, and 10^{18} cm $^{-3}$, respectively. In Ref. [18], Ou *et al.* proposed that n-type doping with N concentration larger than 10^{18} cm $^{-3}$ was favorable for observing luminescence in *f*-SiC. They also pointed out that an increase in N concentration could result in stronger luminescence intensity. In our SIMS measurement, C_N satisfies the criteria on lower limit of 10^{18} cm $^{-3}$. However, the N concentrations of sample SiC-I to sample SiC-IV exhibit a decrease trend, which is not consistent with the prediction reported by Ou *et al.* Furthermore, C_{Al} presents a decreasing trend, while C_B shows an irregular change. Therefore, the photoluminescence cannot be explained by the changes of one specific dopant, such as C_B , C_{Al} , or C_N . In other words, all the dopants should be considered together to adjust the photoluminescence of N, B, and Al codoped 4H-SiC single crystal.

Table 1. SIMS measurement results of B, Al, and N concentrations in samples SiC-I, SiC-II, SiC-III, and SiC-IV. In the measurement, the precision is better than $\pm 3\%$. The B, Al, and N concentrations are defined as C_B , C_{Al} , and C_N , respectively. The parameter C_{D-A} is calculated by a function of $C_B - (C_{Al} - C_N)$ and listed in the last column.

Sample ID	C_B/cm^{-3}	C_{Al}/cm^{-3}	C_N/cm^{-3}	$C_{D-A}/\text{a.u.}$
SiC-I	3.00×10^{17}	2.55×10^{17}	1.01×10^{19}	43.921
SiC-II	1.05×10^{19}	8.81×10^{16}	8.49×10^{18}	48.366
SiC-III	1.09×10^{19}	1.77×10^{16}	7.60×10^{18}	49.897
SiC-IV	9.68×10^{18}	3.91×10^{15}	3.58×10^{18}	50.536

In Ref. [19], Syväjärvi *et al.* calculated the concentration difference between N and B dopants, defined as $C_N - C_B$, in their N and B codoped 6H-SiC epilayer. When the integrated photoluminescence peak intensities were normalized to the

strongest sample, they found a relationship between $C_N - C_B$ and the photoluminescence spectra measured at room temperature. The strongest emission was observed in an n-type sample with the largest concentration difference of $C_N - C_B = 4.6 \times 10^{18} \text{ cm}^{-3}$. Ou *et al.*^[20] also found that N and B concentrations larger than 10^{18} cm^{-3} was required to achieve intense DAPs emission. The concentration difference should be larger than $4.6 \times 10^{18} \text{ cm}^{-3}$ in the N and B codoped *f*-SiC. However, the concentration difference between donor and acceptor cannot describe the changes in photoluminescence properties thoroughly in the B, Al, and N ternary elements doping *f*-SiC. In our previous report,^[21] a new parameter of C_{D-A} , defined by a function of $\ln C_B - (\ln C_{Al} - \ln C_N)$, was used to describe the strong correlation between B–Al–N doping concentration fluctuation and photoluminescence properties of 4H-SiC. The parameter C_{D-A} was also calculated and shown in this work, and the result is listed in the last column of the Table 1. It can be seen that, with the fluctuation of B, Al, and N doping in the 4H-SiC single crystals, the C_{D-A} parameter presents an increasing trend, which is consistent with the trend of photoluminescence peak intensity change in Fig. 2(b).

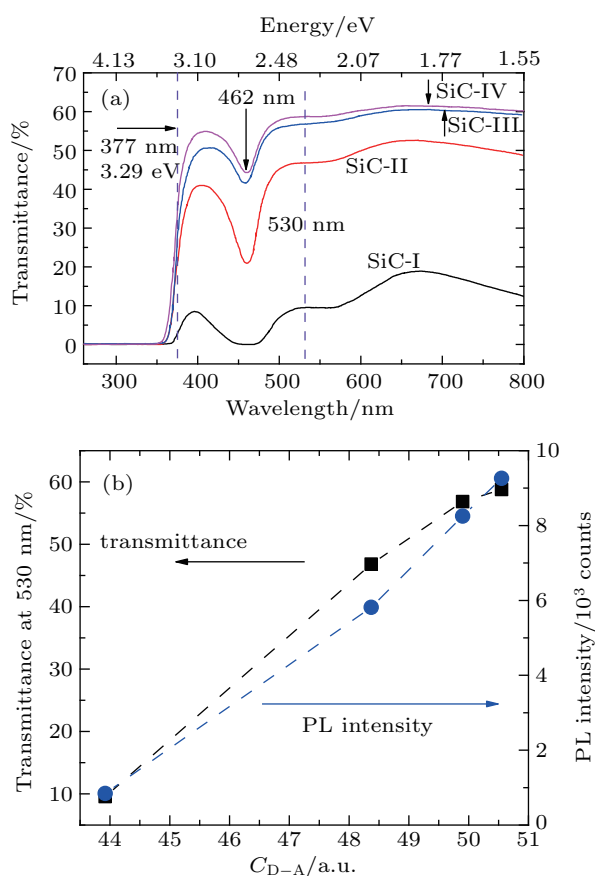


Fig. 3. (a) Transmission spectra of samples SiC-I, SiC-II, SiC-III, and SiC-IV. (b) The transmittance at 530 nm and the photoluminescence peak intensity as function of parameter C_{D-A} . The parameter C_{D-A} is calculated by a function of $C_B - (C_{Al} - C_N)$ and acquired from Table 1.

Besides photoluminescence, transmittance is another optical property that will be affected by B, Al, and N doping in

4H-SiC single crystals. As shown in Fig. 3(a), transmission spectra of SiC-I, SiC-II, SiC-III, and SiC-IV were measured covering the wavelength range from 200 nm to 800 nm at room temperature. The transmission spectra showed similar curves for sample SiC-I to sample SiC-IV that with a steep absorption edge located at 377 nm and an absorption band located at 462 nm. The steep absorption edge is usually considered to be caused by the transition between the bottom levels of the conduction band and the top levels of the valence band, while the absorption band is usually considered to be caused by the induction of N impurity energy level.^[22–24] It is interesting that all the four samples presented distinct transmittances. The transmittances of SiC-I, SiC-II, SiC-III, and SiC-IV are $\sim 10\%$, $\sim 47\%$, $\sim 56\%$, and $\sim 59\%$, respectively. With the fluctuation in B, Al, and N doping, the transmittance of 4H-SiC single crystals also shows an increasing trend.

Due to the similar trend in photoluminescence intensity, parameter C_{D-A} , and transmittance spectra for sample SiC-I to sample SiC-IV, the phenomenon prompts us to consider the association between these three parameters. Following this approach, the functions between C_{D-A} and photoluminescence peak intensity (or transmittance) are constructed and shown in Fig. 3(b). For the changes in transmittance, it can be seen that with the increase in C_{D-A} the transmittance increases firstly, and then suppresses after it reaches 50. Despite the influence of N doping, the transmittance of the doped *f*-SiC can be adjusted by optimal B and Al co-doping. For the changes in photoluminescence peak intensity, with the increase in C_{D-A} , the emission intensity shows a simultaneous increasing trend, which is similar to the above discussion, indicating an encouraging sign that the photoluminescence peak intensity can be enhanced by adjusting B, Al, and N co-doping in fluorescent 4H-SiC single crystal.

In Ref. [25], Sun *et al.* explained the influence of N and B dopants concentration difference on photoluminescence peak intensity in doped 6H-SiC epilayers. They found that the photoluminescence intensity was enhanced with the increase in the difference $(C_N - C_B)$, and would gradually saturate when $C_N - C_B > 2C_B$. In this paper, with the introduction of ternary-element doping, the photoluminescence emission spectra are enhanced. As shown in Fig. 3(b), with the adjustment of B, Al, and N concentrations, the photoluminescence peak intensity can be regulated from 1 to 10 times, and the transmittance value can be increased to about 60% by heavy N doping. The Al has a competition–promotion effect with other dopants. With the substitute of Al on Si site, the N and B dopant will easier to displace the C site in SiC lattice. Obviously, with the increasing of B, Al, and N concentration, the C_{D-A} factor will increase, and the photoluminescence property will also be enhanced. However, excessive Al dopants will suppress the substitution of B and N dopants in SiC lat-

tice, and the C_{D-A} factor will decrease. This adjustment of B, Al, and N dopants is helpful to accelerate the application of *f*-SiC material. However, the detailed recombination of the donor-to-acceptor recombination mechanism is still unclear, and an optimal balance doping of B, Al, and N dopants needs to be explored further.

4. Conclusion

In this paper, the photoluminescence and transmittance properties of B, Al, and N codoped fluorescent 4H-SiC single crystal prepared by the PVT method were studied. The crystalline type of the obtained crystals was characterized by Raman spectroscopy first. Then the doping concentration, photoluminescence spectra, and transmission spectra were measured at room temperature. It was implied that with the fluctuation in B, Al, and N triple dopants the doped 4H-SiC emitted a warm white light covering a wide range from 460 nm to 720 nm when excited by a 325 nm laser, while the transmittance increased from $\sim 10\%$ to $\sim 60\%$. It was experimentally proved that the photoluminescence and transmittance properties could be adjusted by carefully regulating the B, Al, and N doping.

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