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In-situ Si doping of heteroepitaxially grown c-BN thin films at different temperatures

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Abstract

Phase pure cubic boron nitride (c-BN) films have been epitaxially grown on (001) diamond substrates at 420 °C, 600 °C and 900°C. Si was added during film growth at these different deposition temperatures in order to achieve *in-situ* *n*-type doping. The Si concentration increases while the growth temperature decreases. Effects of the different deposition temperatures and the silicon concentration on the transport properties of these c-BN epitaxial films have been systematically investigated. The results demonstrate that the film doped at 420 °C has lower resistivity than the ones doped at elevated temperatures. The temperature dependence of electron transport property measurements and their corresponding theoretical fittings reveal that activation energy of Si doped films is about 0.3 eV and the increased Si concentration could improve the compensation from the deep-level acceptors.

Key words: IBAD, c-BN films, epitaxial, *in-situ* doping, *n*-type conduction, electronic application.

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1. INTRODUCTION

As the simplest III-group Nitrides, cubic boron nitride (c-BN) has driven worldwide attentions for over half centuries.[1] Besides the superhardness and excellent chemical inertness, about 6eV wide band gap, possibility for both *n*- and *p*-type doping, high thermal conductivity and wide optical transmittance over a large spectral range from UV to visible make it not only applicable as hard, protective coating and tooling, but also a very promising candidate in high temperature, high power and high frequency microelectronic and optoelectronic industry. In fact, a c-BN junction diode has been fabricated by growing Si-doped c-BN on a Be-doped seed crystal under high pressure high temperature (HPHT), which exhibited good rectification at 530°C.[2] Unfortunately, the small crystal size of c-BN prepared by HPHT limits its application in large-area electronic devices. Thus it is indispensible to develop a controlled growth of high quality c-BN films in an epitaxial manner as well as with appropriate electronic properties for the purpose of realistic industry.

However, c-BN films have mostly been of poor structural quality due to the applied energetic ion bombardment during deposition, which is necessary to the formation of cubic phase. Consequently, the majority of these films are of mixture phases (amorphous BN (a-BN), turbostratic (t-BN), and c-BN) containing high densities of defects and grain boundaries, severally prohibiting the electronic applications. Significant progresses in synthesis of high quality c-BN films have been achieved, among which the advent of epitaxial c-BN films on appropriate substrates marked a milestone.[3, 4] These epitaxial films are robust, phase pure and singly crystal in large area, which opens exciting opportunity for electronic application of c-BN films. A recent report demonstrated a way to grow single-crystal c-BN film by MBE, possibly allowing to precise control of thickness and doping in the near future.[5] Further attempts on the effective doping of these films towards *p*- and *n*-type conductivity have been made including ion implantation and *in-situ* doping respectively.[6-9]

Ion implantation can effectively turn the intrinsic insulating c-BN films to be semiconducting even semi-metallic.[8] However, ion energies and doses during

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implantation process need to be precisely controlled because the defects inevitably accompanied by radiation damage will probably lead c-BN eventually to the destruction of the cubic phase and to a transformation into mechanically soft hexagonal BN (h-BN). Consequently, the high ionized impurity scattering will result in the low hall mobility far compatible to the industry level.[10] On the other hand, *in-situ* doping can genteelly introduce the desired dopants into the films without over disruption of the matrix structure. However, the incorporation of Si into c-BN film during deposition was restraint by segregation to the surface at high temperatures from previous reports.[7] The effective donor concentration of the resultant *n*-type film is very limited due to the high compensation ratio at elevated temperatures. Moreover, the behavior of impurities in the host matrix of c-BN is considerably complicated, which mainly reflects in two or more conduction mechanisms dominating at different temperature regions.[11-13]

Recently doping of c-BN films have been extensively investigated including theoretical and experimental efforts.[14-18] However, doping of the epitaxial c-BN films has been rarely present. Hence the purpose of the present study is to experimentally investigate the role that doping temperature plays on the electron transport properties of *in-situ* Si doping of c-BN epitaxial films. c-BN films have been epitaxially grown on (001)diamonds at different temperatures following the previous recipe and Si has been incorporated into c-BN films while film deposition. Temperature dependent resistivity and Hall measurements have been taken to systematically investigate the transport behavior of these doped films.

2. EXPERIMENTAL

2.1. Deposition. All the c-BN films studied in the present work were heteroepitaxially grown on HPHT-synthesized Ib-type (001)diamond single crystals (3×3×1 mm^3) by ion beam assisted deposition (IBAD) using two separate ion sources (dual beam technique). Source I delivers a 1.3 keV Ar^+ ion beam used for sputtering the water cooled boron target. During the deposition of the B atoms onto the substrate, the growing film is bombarded with a mixture of 280 eV N_2^+ and Ar^+ ions provided

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by source II. As has been reported previously[19], epitaxial c-BN growth can be obtained under optimized bombarding conditions if diamond single crystals ((001)-oriented, size 3x3x1 mm^3) held at 900°C are employed as substrates. Complementary to the already published recipe for preparation, we have installed the possibility of adding small concentrations of another element like Si during the c-BN film growth in order to test the corresponding doping behavior. For the purpose of *n*-type doping, a small strip (1.5×60 mm^2) of an undoped Si wafer was inserted through a linear feedthrough allowing to expose different amounts of its area to the primary ion beam. In this way, the concentration of the Si in the deposited c-BN films can be varied thereby controlling the flux of the additionally sputtered Si atoms. In order to maximize the Si concentration in c-BN films, the Si flux has been precisely controlled.

2.2. Characterization. The obtained films were characterized by FTIR, AES, XPS and EELS. FTIR spectroscopy of all the films shows a single narrow peak at c-BN To mode position through 400 ~ 4000cm^{-1} range, indicating a 100% cubic content. AES shows a 1:1 stoichiometry of B:N, while EELS gives a primary plasmon energy of 31eV, indicating a rather good quality. All the films used in this work are around 300 nm thick confirmed by Taly Step. For the purpose of electrical measurements, Au/Cr was fabricated on the top of c-BN films as electrodes by Pulsed Laser Ablation (PLD) though a four-point mask, exhibiting a perfect Ohmic characteristics (for more detail, please see the reference[20]). Four 30μm diameter gold wires were bonded onto these contacts and connected with measurement housing, while all the undoped diamond substrates about 1mm thick act as an insulator between c-BN films and the housing. All the electrical transport measurements were carried out through room temperature to about 500 °C by using Van Der Pauw method.

3. RESULTS AND DISCUSSION

As reported previously[7, 9], the introduction of Si will be restrained to be 200 ppm at 900 °C due to the surface segregation effect. As a consequence, Si might be preferably incorporated at lower deposition temperature regions where the segregation effect is

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not dominating. In order to study the influence of the doping temperature, a series of Si doped c-BN films were prepared on top of (001) diamond substrates at 420 ^{o}C, 600 ^{o}C, and 900 ^{o}C respectively. The corresponding Si concentrations in c-BN films grown at 420 ^{o}C, 600 ^{o}C and 900 ^{o}C are 465, 300 and 200 ppm accordingly, determined by time of flight secondary ion mass spectrometer (ToF-SIMS) (see supporting information). As expected, the Si concentration increases while the growth temperature decreases. The incorporation of more Si atoms would benefit the electronic transport properties of the c-BN films, on the other hand it may probably disturb the cubic structure replaced by hexagonal phase.

FTIR measurements were performed to evaluate the volume fraction of the cubic phase by subtracting the background due to the diamond substrate from the transmission spectrum and the compressive stress by estimating the TO-position of c-BN[21]. The corresponding FTIR spectra have been present in Fig.1. Clearly the FTIR spectrum is dominated by only one peak, which is characteristic for the transverse-optical (TO) mode of cubic phase[22]. The absence of h-BN characteristic peaks strongly indicates 100% cubic phase without any h-BN interface, although some of the films have been grown at lower deposition temperatures on diamond substrates. To further demonstrate the temperature effect on the film quality, the cubic TO mode peak position and full width at half maximum (FWHM) are plotted as a function of deposition temperature, as shown in Fig.2. It is obvious that as the deposition temperature increases from 420 ^{o}C to 900 ^{o}C the c-BN peak position shifts from 1097 cm^{-1} to 1075 cm^{-1}, suggesting that the built-in compressive stress decreases due to the elevated temperatures.[23] On the other hand, as the deposition temperature is increased, the FWHM value decreases from 190 cm^{-1} to 110 cm^{-1}, indicating an improved crystalline quality. For the Si doped c-BN films at 900 ^{o}C with 200 ppm concentration the cubic phase can be preserved simultaneously while no indication of h-BN interface layer. However, it is noteworthy mentioning that for the other doped films the unique cubic phase remains stable even though the Si concentration is significantly increased as well as the deposition temperature decreases. Thus, it is indispensible to search a matching point between Si concentration and the film

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quality.

The electronic transport properties have been thoroughly investigated by the temperature dependent sheet resistance and Hall effect measurements. The sheet resistance as a function of temperature was measured for the Si doped c-BN films grown at 420 ^{o}C, 600 ^{o}C and 900 ^{o}C, respectively, using the van der Pauw method from 300 K to 800 K, as shown in Fig.3. Sheet resistances for these three samples are logarithmically plotted as a function of $1/k_BT$ (k_B Boltzmann constant), from which the activation energy can be extracted. Since Si prefers to segregate to the film surface at high temperatures, only sheet resistance values can be shown in Fig. 3 because of lacking knowledge of the segregation layer at different temperatures. It can be clearly seen that the film grown at higher deposition temperatures, which, in turn, containing less impurity concentration as estimated from ToF-SIMS, exhibits a higher resistance as expected. The film doped at 900 ^{o}C exhibits a sheet resistance value of 2×10^{10} Ω at room temperature, while the film doped at 420 ^{o}C has a lower value of 10^{9} Ω, implying a higher Si doping efficiency at lower temperatures. However, two different activation energies are observed within the present temperature range, indicating some complex conduction mechanism. For the doped semiconductors, the resistivity is often represented as a sum of terms $\rho^{-1} = \sigma = \sigma_1 \exp(-E_1/k_BT) + \sigma_2 \exp(-E_2/k_BT)$, where these two terms correspond to two different conductions with activation energies of E_1 and E_2. E_1 and E_2 are the approximately the impurity ionization energy. The data were fitted over a limited range of temperatures, assuming that mobility changes are inconsequential. In the present resistance fitting, the dominant conduction mechanism shifts from one impurity ionization with activation energies of 0.17-0.26 eV at low temperatures to another impurity ionization with activation energies of 0.45-0.89eV at high temperatures. Furthermore, the activation energy reduces as the Si impurity concentration increases from 200 ppm to 465 ppm within both temperature regions.

Unfortunately, based on exclusively resistance measurements this phenomenon cannot be unravelled. Therefore, the conduction mechanism resulting in two

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activation energies will be discussed in detailed combined with Hall effect measurement. Nevertheless, from Fig.3, a clear tendency of lower resistances resulting from more impurities can be seen, suggesting Si was successfully incorporated into the film, lowering the film resistance at least near the surface. In the following section, Hall measurements will reveal more reliable activation energies and provide more information in detail.

In order to identify the conduction mechanism for Si dopants, silicon doped c-BN films grown at 900 ^{o}C, 600 ^{o}C and 420 ^{o}C on (001)diamonds were characterized by Hall measurement using the van der Pauw method from 300 K to 800 K. A clear negative Hall signal could be detected confirming the silicon doped films grown at 600 ^{o}C and 900 ^{o}C to be of *n*-type conduction, distinct from the previous report of hopping conductions occurred in Si contained c-BN polycrystalline films[11]. Unfortunately, it was difficult to detect the Hall voltage from the film grown at 420 ^{o}C due to the higher impurity concentrations. The temperature dependent carrier concentrations for silicon doped c-BN films grown at 600 ^{o}C and 900 ^{o}C are shown in Fig. 4 as well as after H-plasma treatment. For the film doped at 600 ^{o}C, the carrier concentration is measured to be 3×10^{13} cm^{-3} at about 330 K; when adding silicon to the film at 900 ^{o}C, the carrier concentration decreased to 5×10^{12} cm^{-3}, which, again, is consisted with the previously conclusions from ToF-SIMS, the higher deposition temperature has lower impurity concentration. Since Si is tended to segregate to the film surface when doping during the film growth, H-plasma (50 W, $P_{H_2}=10^{-2}$ mbar) was used to remove the segregated surface layer. After H-plasma treatement for 20mins, even lower carrier concentrationshave been revealed by Hall measurement. As the temperature increases, the electron concentrations increase exponentially for all the samples. The exponential relationship between carrier concentration and $1/k_BT$ indicates that the conduction can be attributed to thermal activation of carriers. For a more detailed understanding of the electrical transport, consideration should be taken into the effect of carrier compensation on donor concentration, N_D, due to unintentional acceptor concentration, N_A, originating from other impurities and/or

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defects. The general expression of carrier concentration for an *n*-type semiconductor is given by

$$\frac{n(n+N_A)}{N_D-N_A-n}=\frac{N_C}{g_D}\exp\left(-\frac{E_D}{k_BT}\right) \tag{1}$$

Where g_D is the degeneration factor of donor stats, which equals to 2, k_B the Boltzmann constant, T the temperature, E_D the donor energy level, and N_C the effective density of states in the conduction band. The number of equivalent minima in the conduction band is 6 for c-BN. The density of states effective mass, m_{dos}^* for the electron is given by $m_{dos}^*=\left(m_t^*m_t^*m_l^*\right)^{1/3}$, where m_l^* and m_t^* are longitudinal and transverse electron effective masses, respectively. For this calculation of c-BN, we used $m_l^*=1.2m_0$ and $m_t^*=0.26m_0$, where m_0 is the free electron mass[24].

From Fig. 4, the slopes of logarithm of carrier concentrations versus to reciprocal of k_BT decrased from the film at 600 ^oC, to 900 ^oC and after H-plamsa treatment, which was consisted with that more activated donors could induce lower activation energy. The calculated results are well fitted with the experimental data over the entire temperature reagion. The simiulations confirm the activation energy, E_D, to be 0.3 eV and 0.35 eV for the films doped at 600 ^oC and 900 ^oC, respectively. Furthermore, after H-plasma treatment, the activation enregy increases to 0.4 eV for both of these two films, which is due to the decreasing carrier concentration resulting from H-plasma. The best-fitted values and experimetal results are listed in Table 1. Considering the *n*-type conduction, Si was the only possibility for this *n*-type behavior, consistent with the theoretical calculations[25]. The acceptor concentration as well as the donor concentration increases with Si doping concentration. The compensation ratio, $N_A/(N_A+N_D)$, is almost the same as 49% for the film grown at 900 ^oC before and after H-plasma, while is slightly decreased from 49% for the film grown at 600 ^oC down to 44% after H-plasma treatment. This suggests that the compensating acceptors are increased as well as the donor increases proportionally. The possible candidate for compensating acceptor is the substitutional C in N site, C_N, and/or the complex of C_N with N vacancy, V_N-C_N[26]. The simulations confirm that the activation energy of 0.3 ~

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0.4 eV is in a good agreement with the previous experimental results for Si doped c-BN[27, 28]. However, one may notice that the H-plasma treated sample exhibit higher activation energy than the as-prepared one, which is due to the lower sheet carrier concentration by H-plasma, although so far, proton involved transport mechanism in c-BN is still unknown. Nevertheless, the surface removal by H-plasma is expected. In the lower temperature region, a deviation of the fitting lines from the experimental data can be observed, indicating the highly compensated situation. One may notice that the activation energies extrapolated from Resistance and Hall measurements are not consistent at least E_D is much higher for resistance measurements at higher temperature region than the one from Hall measurement. This is probably because that the mobility at higher temperature region could not be treated as a linear behaviour, which, actually as we will show in the following, obeys the power law.

To further shed the light on the electric properties of *in-situ* doping of c-BN films, Hall mobility has been derived from the temperature dependent Hall measurements, as shown in Fig.5. Hall mobility increased gradually with increasing temperature for both Si doped c-BN films and the values were 20 and 0.5 $cm^2V^{-1}s^{-1}$ at around 300 K, respectively. As a matter of fact, the film grown at 600 ^oC has higher mobility than that grown at 900 ^oC, which is normally related to the impurity and defect densities. Furthermore, it is important to note that Si doped c-BN exhibited dominant *n*-type semiconductor properties and nominally undoped films exhibited *p*-type properties[7, 9]. The latter had a higher resistivity with lower sheet hole density, but larger Hall mobility, which indicates that adding silicon to the film resulted in high defect density thus high scattering centres. Not like nominally undoped films which showed a negative temperature dependent mobility, the positive temperature dependence of Hall mobility is found for these silicon doped c-BN films, indicating additional scattering mechanism occurring in the doped films. According to the fitting results, both of these Si doped films could be fitted to a $T^{1.5}$ law, suggesting impurity ionization scattering effect. However, for the film grown at 900 ^oC, this $T^{1.5}$ fitting curve does not describe the data precisely at high temperatures between 600 K and 800 K. In this range, another T^6- law delivers a reasonable description, which is still unknown.

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4. CONCLUSIONS

High quality c-BN films have been deposited on (001)diamond substrate at 420 ^{o}C, 600 ^{o}C and 900 ^{o}C respectively, while Si was added during the film growth in order to achieve *in-situ* doping. Si concentration increases as the deposition temperature decreases, determined by both SIMS and temperature dependent Hall effect measurements. As the deposition temperature increases from 420 ^{o}C to 900 ^{o}C, the intrinsic stress of the epitaxial c-BN films will release as estimated by the IR characteristic peak shift as well as the film quality will be improved judged by the significant change of the peak width. Negative hall signal was observed confirming *n*-type conduction for all the Si doped c-BN films. c-BN films doped at 420 ^{o}C has lower resistivity than the ones doped at elevated temperatures. The temperature dependence of Hall effect measurements and theoretical fittings reveal that the increased Si concentration could improve the compensation from the deep-level acceptors.

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Figure caption

Figure 1 FTIR spectra for c-BN films grown on diamond substrates at 420 ^{o}C, 600 ^{o}C and 900 ^{o}C respectively.

Figure 2 c-BN peak position and FWHM as a function of deposition temperature.

Figure 3 Temperature dependence of sheet resistance for silicon doped c-BN films grown at 420 ^{o}C (open squares), 600 ^{o}C (open circles) and 900 ^{o}C (open triangles) on top of diamond substrates. The solid lines are fittings.

Figure 4 Temperature dependence of carrier concentration of silicon doped c-BN films grown at 600 ^{o}C and 900 ^{o}C and after H-plasma treatment, respectively. Open squares and triangles show the film grown at 600 ^{o}C and 900 ^{o}C and solid symbols show the corresponding films after H-plasma treatment. The solid lines show the simulation results.

Figure 5 Temperature dependence of Hall mobility of Silicon doped c-BN films grown at 600 ^{o}C and 900 ^{o}C. The open symbols showed the measured mobility; solid lines showed fitting by $T^{1.5}$, dot line showed fitting by T^{6} at high temperature range.

Table caption

Table 1 SIMS and Hall measurement results compared with the simulated parameters.

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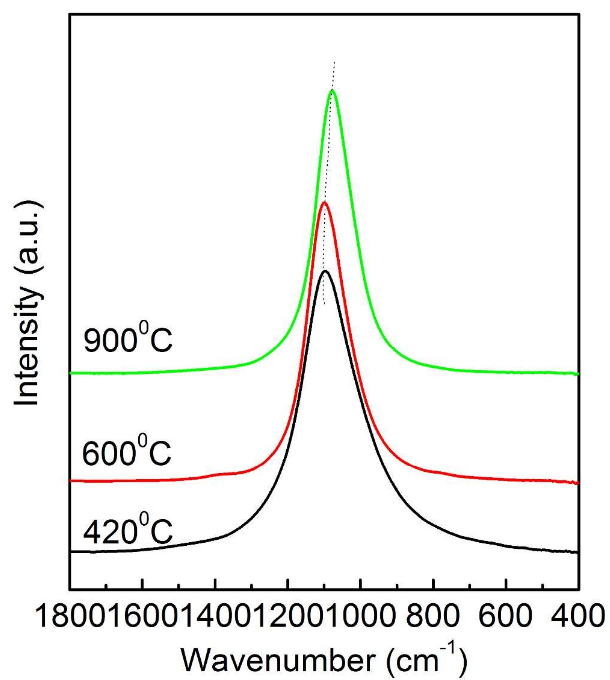

Fig.1 FTIR spectra for c-BN films grown on diamond substrates at 420 ^{o}C, 600 ^{o}C and 900 ^{o}C respectively

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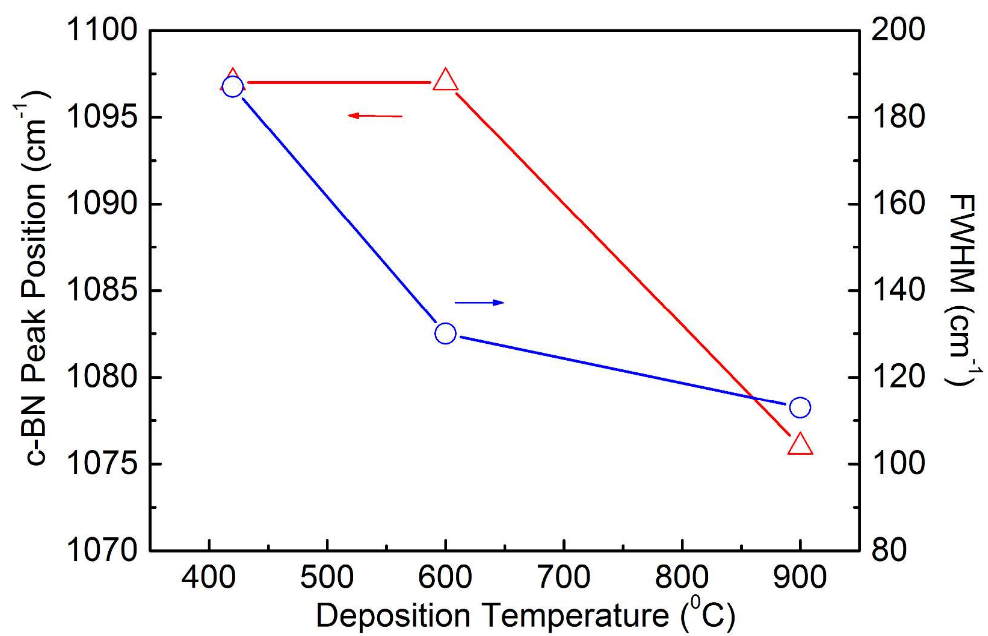

Fig.2 c-BN peak position and FWHM as a function of deposition temperature

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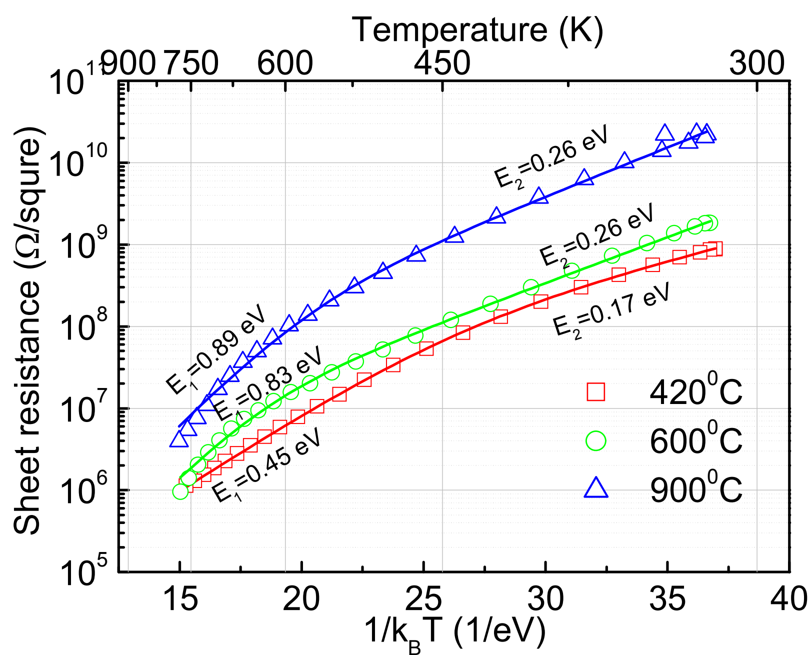

Fig.3 Temperature dependence of sheet resistance for silicon doped c-BN films grown at 420 °C (open squares), 600 °C (open circles) and 900 °C (open triangles) on top of diamond substrates. The solid lines are fittings.

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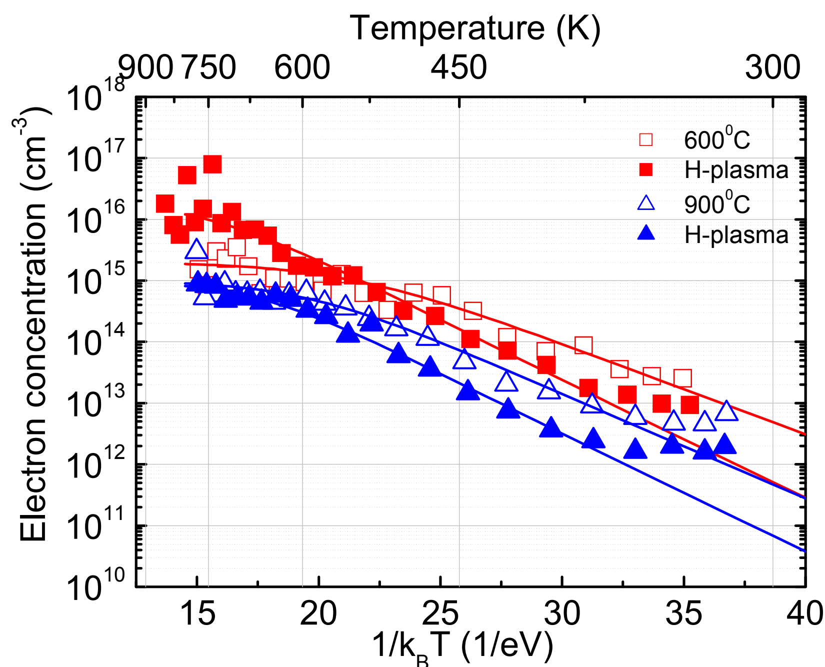

Fig.4 Temperature dependence of carrier concentration of silicon doped c-BN films grown at 600 °C and 900 °C and after H-plasma treatment, respectively. Open squares and triangles show the film grown at 600 °C and 900 °C and solid symbols show the corresponding films after H-plasma treatment. The solid lines show the simulation results.

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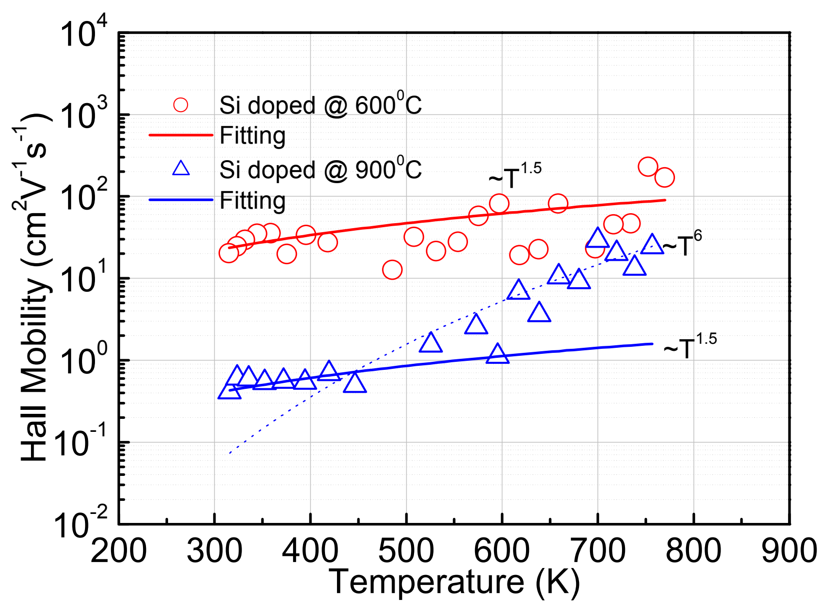

Fig.5 Temperature dependence of Hall mobility of Silicon doped c-BN films grown at 600 °C and 900 °C. The open symbols showed the measured mobility; solid lines showed fitting by $T^{1.5}$, dot line showed fitting by T^{6} at high temperature range.

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Table 1 SIMS and Hall measurement results compared with the simulated parameters.

Parameters	Simulation			SIMS		Hall
	E_D (eV)	N_D (cm^{-3})	N_A (cm^{-3})	C_{Si} (cm^{-3})	C_C (cm^{-3})	n (cm^{-3})
Film grown at 600°C	0.3	4.2×10^{16}	4.0×10^{16}	5.0×10^{19}	1.0×10^{21}	10^{13}~10^{15}
After H-plasma treatment	0.4	1.0×10^{17}	8.0×10^{16}	/	/	10^{13}~10^{16}
Film grown at 900°C	0.35	3.1×10^{16}	3.0×10^{16}	3.3×10^{19}	8.0×10^{20}	10^{12}~10^{14}
After H-plasma treatment	0.4	3.1×10^{16}	3.0×10^{16}	/	/	10^{12}~10^{14}

E_D: Donor energy level; N_D: Donor concentration; N_A: Acceptor concentration; C_{Si}: Si concentration; C_C: C concentration; n: electron carrier concentration.

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