Temperature dependence of Raman scattering in bulk 4H-SiC with different carrier concentration

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Abstract: Raman spectra of three bulk 4H-SiC wafers with different free carrier concentration were measured at temperature from 80 K to 873 K. As temperature increases, Raman peaks of most optical phonon modes show monotonous down shift. An anomalous non-monotonous variation with temperature, was observed in the A_1 longitudinal optical (LO) mode from doped samples. Two methods of theoretical fitting, one-mode (LO-plasma coupled (LOPC) mode) and two-mode (A_1(LO) + LOPC) fitting, are employed to analyze this anomalous phenomenon. Theoretical simulations for temperature dependent Raman spectra by using two methods are critically examined. It turns out that one-mode method conforms well the experimental results, while two-mode method is untenable in physics. The non-monotonous variation of blue-red shifts with temperature for LOPC mode from doped 4H-SiC could be explained by the influence from ionization process of impurities on the process of Raman scattering. A quantitative description on temperature dependent Raman spectra for doped 4H-SiC is achieved, which matches well to experimental data.

OCIS codes: (290.5860) Scattering, Raman; (160.6000) Semiconductor materials.

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1. Introduction

Raman spectroscopy is a non-destructive approach for material characterization. Study of Raman scattering has been an attractive topic, especially on the structural and electronic properties of silicon carbide (SiC). SiC, as one of the most promising wide band gap semiconductors, is a kind of ideal material for high voltage and high temperature applications. Its high electron mobility and weaker anisotropy make it suitable for manufacturing of high power devices. Seminal work of Raman scattering of SiC polytypes was done by Nakashima and Harima [1]. Various concepts, including the LOPC mode behaviors at room temperature were discussed comprehensively. As an ideal material for high power and high temperature devices, properties of SiC at different temperatures, especially at high temperautures, are significant to its practical application. Temperature dependent Raman studies on GaN were reported [2,3], in which phonon scattering modes showed monotonous red-shift with temperature increasing. The variation of LO phonon scattering mode with respect to temperature could be explained by higher order phonon process.

For the case of 4H-SiC, we had observed an anomalous temperature behavior of the LOPC mode from highly-doped samples [4], but did not give a good explanation. Recently, R. Han *et al*. studied the temperature dependence of Raman scattering in round pit of 4H-SiC (100-600K) [5] and 4H-SiC with hexagonal defects (100-450K) [6]. Anomalous phenomena of A_1(LO) mode of 4H-SiC were reported, where the A_1(LO) mode showed non-monotonic variation with temperature. The authors gave a qualitative explanation that anomalous variation of A_1(LO) mode stemmed not only from the anharmonic effects of phonons but also from impurity ionization [5,6]. For GaN, references [7,8] regarded A_1(LO) mode as an overlapped mode of A_1(LO) mode and LOPC mode when carrier concentration is high enough to cause asymmetric broadening of A_1(LO) mode, and spectral fittings by two modes, A_1(LO) + LOPC, were applied. In other sense, many author treated the asymmetric A_1(LO) mode as one LOPC mode and did one mode fit for doped SiC [5,6,9] and GaN [10]. Which model is more reliable: two modes of A_1(LO) + LOPC or only one LOPC mode? It should be critically examined.

In this paper, we perform Raman scattering measurements over 80-873 K on a series of 4H-SiC bulk wafers with different nitrogen (N) doping levels. Two theoretical methods, one-mode (LOPC mode) and two-mode (A_1(LO) + LOPC) fittings, are employed comparatively to fit all measured Raman spectra and analyze the temperature dependence of the LOPC mode in doped 4H-SiC. Reliability of each method is evaluated, based upon the fitting results. We corroborate that one-mode method has better accordance with experimental results and reflects clear physical connotation. Two-mode method is untenable because of its contradiction to temperature properties of phonon scattering modes. A clear and good quantitative explanation on the anomalous variation of LOPC mode in 4H-SiC with different carrier concentration is reached.

2. Experiment results

A confocal micro-Raman spectroscope system, Jobin Yvon T64000 with a 2400 l/mm grating, was employed to study Raman mode shift of 4H-SiC samples with temperature varying from 80 K to 873 K. Spectral resolution of the system is up to 0.6 cm^{-1}.A Nd: YOV_4 532 nm laser was utilized for excitation. Three bulk 4H-SiC samples of different doping levels, purchased from CREE company (http://www.cree.com/LED-Chips-and-Materials/Materials), were prepared by physical vapor transport technique: S1 undoped, S2 medium-doped and S3 heavy-doped, with carrier concentration of 9.09×10^{14}, 2.18×10^{18} and 4.87×10^{18} cm^{-3}, respectively.

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4H-SiC has wurtzite structure which belongs to hexagonal symmetry. According to Raman selection rules of hexagonal SiC crystal (C_6^4 space group), 4H-SiC has 24 lattice modes ($6A_1 + 6B + 6E_1 + 6E_2$). Only ten ($3A_1 + 3E_1 + 4E_2$) of the 24 modes are Raman active, and the B modes are silent. The A_1 mode and E_1 mode are also infrared active and they split into longitude optical and transverse optical (TO) branches [1,2,9]. 4H-SiC presents phonon bands with A_1, E_1 and E_2 mode. In the backscattering geometric configuration (*z||c, x,y in c-plane*), A_1, E_1 and E_2, mode can be observed. Raman spectra of each sample are shown in Fig. 1. Red-shift of E_2(TO) and E_1(TO) mode with increasing temperature are clearly observed. Raman mode located at about 970 cm^{-1} should be A_1(LO) mode theoretically. Variation of 970 cm^{-1} peak with temperature for undoped sample S1 is monotonous down shift. However, the two doped samples show non-monotonous variation of the peak at around 970 cm^{-1} (named as A_1(LO)-like mode temporarily). Variation of A_1(LO) and A_1(LO)-like mode peak positions from three 4H-SiC samples are plotted in Fig. 2.

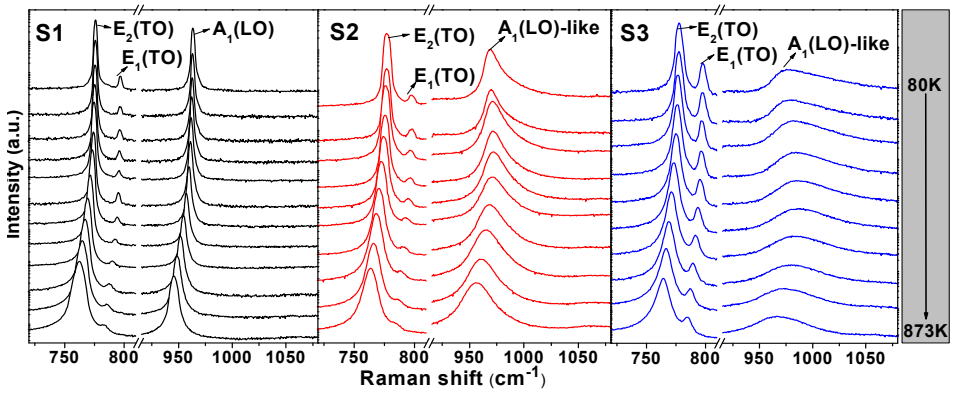

Fig. 1. Raman spectra of three 4H-SiC samples at temperature varying from 80 K to 873 K.

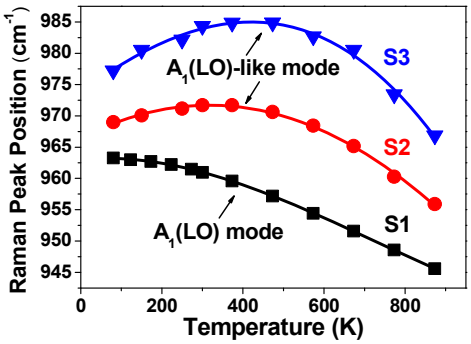

Fig. 2. A_1(LO) (S1) and A_1(LO)-like (S2 and S3) mode peak positions of three 4H-SiC at temperature from 80 K to 873 K. Raman peak positions of S1, S2 and S3 at different temperatures denote by rectangle (black), circle (red) and inverted triangle (blue), respectively Solid lines are introduced to show the trend of temperature dependence of Raman peak position.

3. Analyses of experimental results

3.1 Theoretical simulation of TO phonon frequency

The red-shift of phonon scattering modes with temperature stems from linear thermal expansion and multi-phonon coupling processes. According to Raman scattering model, temperature dependence of phonon frequency $\omega(T)$ in Eq. (1) can be given by [2]

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$$\omega(T) = \Omega_0 + \omega^{(1)}(T) + \omega^{(2)}(T), \tag{1}$$

where $\Omega_0 = \omega_0 - M_1 - M_2$, ω_0 is the harmonic frequency of optical phonon at temperature near absolute zero; M_1 and M_2 are fitting parameters in Eq. (3). $\omega^{(1)}(T)$ is the term caused by linear thermal expansion, and $\omega^{(2)}(T)$ denotes anharmonic phonon coupling contribution to Raman shift. The second term $\omega^{(1)}(T)$ in Eq. (1) can be expressed as

$$\omega^{(1)}(T) = \Omega_0 \left\{ \exp\left[-\kappa \int_0^T \left(\alpha_c(t) + 2\alpha_a(t) \right) dt \right] - 1 \right\}, \tag{2}$$

where κ is the Gruneisen parameter for optical Raman mode, $\alpha_c(t)$ and $\alpha_a(t)$ are coefficients of linear thermal expansion along direction of c and a axis respectively, which were obtained by fitting the data [11]. The third term $\omega^{(2)}(T)$ can be written as

$$\omega^{(2)}(T) = M_1 \left\{ 1 + \sum_{i=1}^{2} \frac{1}{e^{x_i} - 1} \right\} + M_2 \left\{ 1 + \sum_{j=1}^{3} \left[\frac{1}{e^{y_j} - 1} + \frac{1}{(e^{y_j} - 1)^2} \right] \right\}, \tag{3}$$

where M_1 and M_2 are fitting parameters. Values of the exponent x_i and y_j are given as: $\sum x_i = \sum y_j = \hbar\omega_0$. The first term of $\omega^{(2)}(T)$ describes three-phonon process and the second one denotes four-phonon process. The split phonons of the same order are assumed to be of the same frequency. Therefore, it is supposed that $x_1 = x_2 = \hbar\omega_0/2$ and $y_1 = y_2 = y_3 = \hbar\omega_0/3$.

According to Eqs. (1)-(3), fittings of E_1(TO) and E_2(TO) mode shift dependent on temperature (T) are accomplished through the assistance of Matlab software, which is applied to the analyses on T-varied Raman spectra from Fig. 1. These fitting results are shown in Fig. 3. Curves of three 4H-SiC wafers are similar. The effects from free carrier concentration on two TO phonon scattering modes are negligible. Therefore, 4H-SiC samples with different doping levels have the same variation of TO phonon scattering modes with temperature.

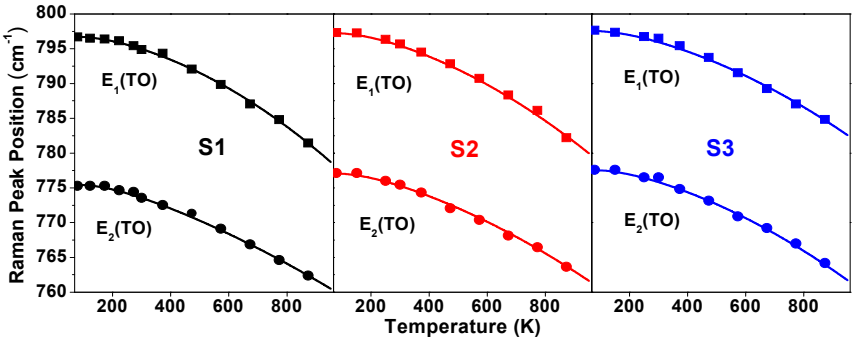

Fig. 3. Fitting result of E_1(TO) and E_2(TO) mode peak positions of three 4H-SiC samples S1, S2 and S3.

3.2 Longitudinal optical phonon-plasma coupled (LOPC) mode

In a doped polar semiconductor, such as SiC, free carrier (plasmon) interacts with the longitudinal optical (LO) phonon to form the LOPC mode. According to theory of scattering cross section, Raman scattering intensity of LOPC mode can be expressed as [1,2,9],

$$I_{LOPC} = \left. \frac{d^2 S}{d\omega d\Omega} \right|_A = \frac{16\pi\hbar n_2}{V_0^2 n_1} \frac{\omega_2^4}{C^4} \left(\frac{d\alpha}{dE} \right) (n_\omega + 1) A \, \mathrm{Im}\left(-\frac{1}{\varepsilon} \right), \tag{4}$$

Where

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$$A = 1 + 2C\frac{\omega_T^2}{\Delta}\left[\omega_p^2\gamma\left(\omega_T^2-\omega^2\right)-\omega^2\eta\left(\omega^2+\gamma^2-\omega_p^2\right)\right]+C^2\left(\frac{\omega_T^4}{\Delta\left(\omega_L^2-\omega_T^2\right)}\right), \quad (5)$$
$$\times\left\{\omega_p^2\left[\gamma\left(\omega_L^2-\omega_T^2\right)+\eta\left(\omega_p^2-2\omega^2\right)\right]+\omega^2\eta\left(\omega^2+\gamma^2\right)\right\}$$

$$\Delta = \omega_p^2\gamma\left[\left(\omega_T^2-\omega^2\right)^2+\left(\omega\eta\right)^2\right]+\omega^2\eta\left(\omega_L^2-\omega_T^2\right)\left(\omega^2+\gamma^2\right), \quad (6)$$

and ω_L is the LO mode frequency; ω_T is TO mode frequency; η is phonon damping constant; γ is plasma damping constant; n_1 and n_2 are refractive indices at incident frequency and scattering frequency ω_2 respectively; C is Faust-Henry coefficient; α is polarizability; E is macroscopic electric field; n_ω is Bose-Einstein factor. Dielectric function is described by

$$\varepsilon = \varepsilon_\infty\left(1+\frac{\omega_L^2-\omega_T^2}{\omega_T^2-\omega^2-i\omega\eta}-\frac{\omega_p^2}{\omega\left(\omega+i\gamma\right)}\right), \quad (7)$$

$$\omega_p^2 = \frac{4\pi ne^2}{\varepsilon_\infty m^*}, \quad (8)$$

where ω_p is plasma frequency; n is free carrier concentration; m^* is effective mass; e is unit charge; ε_∞ is high frequency dielectric constant.

For an intrinsic or an undoped semiconductor, Raman scattering intensity of A_1(LO) mode can also be described by a Lorentz profile function [7,8],

$$I_{A_1(LO)} = \frac{I_0}{\left(\frac{\omega-\omega_s}{\Gamma_s}\right)^2+1}, \quad (9)$$

where ω_s is the LO phonon frequency near the center of Brillouin zone. Γ_s is linewidth of A_1(LO) phonon scattering peak. I_0 is a proportional constant. Equation (9) is applied to fit A_1(LO) band of the undoped 4H-SiC sample S1, which is the same as the fitting results of Eqs. (4)-(6) with $\omega_p = 0$, as shown in Fig. 4 (a) and Fig. 6 (a) in the next two sections. Also, this mode shows normal down shift with temperature increasing (the bottom curve of S1 in Fig. 2), which can be described and fitted well by Eqs. (1)-(3).

3.3 Temperature dependence of A_1(LO)-like mode: two-mode (A_1(LO) + LOPC) fits

Figure 2 shows that the A_1(LO)-like modes of two N-doped 4H-SiC have a blue-shift first and then follow a red-shift in peak frequency with increasing temperature from 80 K to 873 K. This mode frequency variation with temperature is related to doping level of 4H-SiC. Here we first apply the two-mode (A_1(LO) mode and LOPC mode) method, as used in [7,8], to analyze our experimental results.

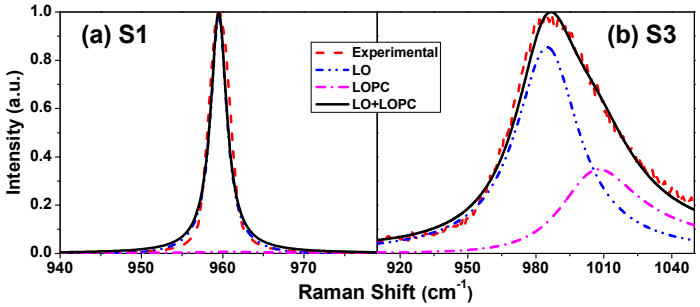

Fig. 4. Fitting results with two-mode method for (a) S1 and (b) S3 at 373 K

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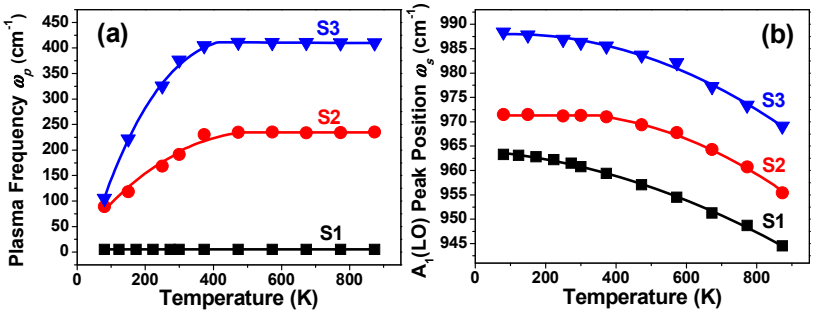

Fig. 5. Temperature dependence of (a) plasma frequency ω_p and (b) A_1(LO) mode peak position ω_s, obtained from the two-modes method. Solid lines are fitting results of three samples, which are introduced to reflect the variation of plasma frequency ω_p and A_1(LO) mode peak position ω_s with respect to temperature.

Using the two-mode method, as shown in Fig. 4 typically, the overlapped mode is entirely composed of A_1(LO) mode in undoped S1, while the highly doped S3 contains two components of A_1(LO) mode and obvious LOPC mode. Values of plasma frequency ω_p and A_1(LO) mode peak position ω_s at each measured temperature for three samples are obtained and plotted in Fig. 5. As we have seen from Fig. 3, the same TO phonon scattering mode of SiC with different doping levels should share about the same peak position at the same temperature. The spectral resolution of the system, up to 0.6 cm^{-1}, ensures the reliability of the results demonstrated in Fig. 3. Previous work reported that difference of the same phonon mode in different doped SiC samples is about 1-3 cm^{-1} at most [12]. Fluctuation of Raman shift within several wavenumbers can be caused by deformed potential from lattice defects. However, fitting results of A_1(LO)-like mode in Fig. 5(b) show that S1, S2 and S3 do not share similar A_1(LO) mode peak position at same temperature. Frequency differences of the A_1(LO) phonon modes in three 4H-SiC samples are up to tens (10-30) of cm^{-1}. This is the dilemma that two-mode method encounters when being used for analyzing the asymmetric broadening peak of A_1(LO)-like mode. Coexistence of A_1(LO) mode and LOPC mode in the same area of material is untenable in physics when doping is uniform. LO phonons and free carriers couple together to scatter photons in doped SiC. So two-mode method is not reasonable in explaining the variation of temperature dependent Raman spectra of A_1(LO)-like mode.

3.4 Temperature dependence of LOPC mode: one-mode (LOPC) fits

Now, we come to utilize the one-mode (LOPC) method to fit the A_1(LO)-like mode. Figure 6 shows such two typical fits, and one LOPC mode fits were done for all measured Raman spectra in Fig. 1 for three 4H-SiC samples at different temperatures. Fitting results of Plasma frequency ω_p are given in Fig. 7(a), in which plasma frequency ω_p of S1 remains less than 5 cm^{-1}, relatively small compared to those of S2 and S3, at temperature ranging from 80 K to 873 K, and plasma frequency ω_p of S2 and S3 increases first and remains almost the same when temperature goes up to a relatively high level. According to Eq. (8), plasma frequency ω_p positively correlates to free carrier concentration n. So the variation of plasma frequency ω_p directly reveals the ionization process of impurities in 4H-SiC. S1 contains relatively fewer impurities than doped samples, so it shows no typical ionization process not as well as S2 and S3 when temperature increases, as what can be seen in Fig. 7(a). S2 and S3 are doped 4H-SiC samples, whose ionization processes are sensitive to temperature. Free carrier concentration n increases when temperature goes up and remains constant after impurities are totally ionized.

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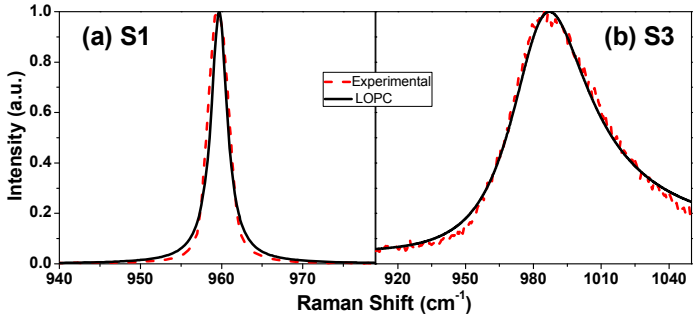

Fig. 6. Fitting results with one-mode method for (a) S1 and (b) S3 at 373 K

3.5 Further discussion on temperature dependence of LOPC mode

By comparing the fitting results of two methods, it turns out that A_1(LO) mode becomes LOPC mode in doped SiC samples. So the asymmetric broadening A_1(LO)-like mode is LOPC mode other than the A_1(LO) + LOPC overlapped mode. Anomalous variation is from temperature behavior of the LOPC mode. Peak position of LOPC mode can be calculated by [9],

$$\omega_{LOPC}^2 = \frac{1}{2}\left\{\left(\omega_p^2 + \omega_{LO}^2\right) + \left[\left(\omega_p^2 + \omega_{LO}^2\right)^2 - 4\omega_p^2\omega_{TO}^2\right]^{\frac{1}{2}}\right\}, \quad (10)$$

where ω_p is plasma frequency. ω_{LO} is the LO phonon frequency, obtaining from the A_1(LO) value of undoped 4H-SiC, and ω_{TO} is the TO phonon frequency, calculated from the Lyddane-Sachs-Teller (LST) relation of $\omega_{LO}^2/\omega_{TO}^2 = \varepsilon_0/\varepsilon_\infty$, where ε_0 and ε_∞ are the static permittivity and the high-frequency permittivity, respectively [13].

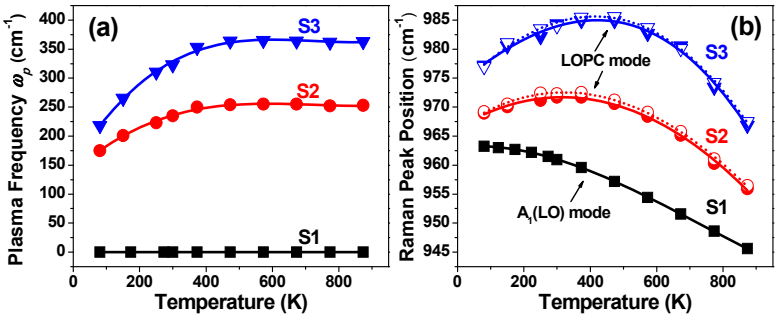

Fig. 7. Temperature dependence of (a) plasma frequency ω_p (b) experimental results (solid symbol) and calculation (hollow symbol) of LOPC mode peak positions, in one-mode method. Solid and short dot lines are fitting results, which are introduced to show the variation of plasma frequency ω_p and Raman peak position with respect to temperature.

Equation (10) reveals the interaction between LO phonons and plasma. Increasing temperature leads to red-shift of LO phonon mode and increase in the degree of impurity ionization, which results in the increment of plasma frequency until complete ionization. Coupling between the LO phonon frequency and plasma frequency contributes to the non-monotonous variation: up shift first and then red-shift with temperature increasing. LOPC mode peak positions can be calculated according to the description of Eq. (10), as what are shown in Fig. 7(b) (short dot lines). Calculation result is consistent with the variation of LOPC mode in experiment (solid lines in Fig. 7(b) and Fig. 2).

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4. Conclusion

Confocal micro-Raman spectroscopy was employed to analyze three pieces of bulk 4H-SiC wafers: undoped, medium-doped and highly doped. All samples were measured at temperature varying from 80 K to 873 K. Three Raman modes, including E_2 (TO), E_1 (TO) and A_1(LO) (or A_1(LO)-like) mode, are clearly observed. The red-shift of E_2(TO) and E_1(TO) phonon scattering modes is explained through phonon frequency function with temperature. Anomalous variation of A_1(LO) mode in doped 4H-SiC stems from the coupling between LO phonons and plasma, which forms the LOPC mode. Two methods, one-mode and two-mode methods, are applied to analyze variation of A_1(LO) mode. These two fitting methods have appeared in literatures for many years but no judgment has been done on the reliability of the two methods. We have demonstrated it clear that the two-mode method is untenable in interpreting anomalous variation of A_1(LO) mode with temperature, which gives rise to the contradiction to temperature properties of phonon scattering modes, while the one-mode simulation is in good accordance with experimental results. Therefore, A_1(LO) mode in doped 4H-SiC is LOPC mode when A_1(LO) mode shows asymmetric broadening. The non-monotonous variation of blue-red shifts with temperature for LOPC mode from doped 4H-SiC could be explained by the influence from ionization process of impurities on the process of Raman scattering. A quantitative description on temperature dependent Raman spectra for doped 4H-SiC is achieved in this paper, matching well to experimental data.

Acknowledgments

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