



## Full Length Article

## Probing carrier concentration of doped GaN single crystals from LO phonon-plasmon coupled modes

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## ABSTRACT

Raman and infrared reflectance spectroscopy were used to study GaN single crystals with different doping types and carrier concentrations. In different samples, the characteristic spectra related to LO phonon-plasmon coupled (LOPC) mode are observed as changing regularly with carrier concentration. Furthermore, to obtain the carrier concentration and mobility of GaN crystals, Raman and infrared reflectance spectra with LOPC modes were fitted. The carrier concentration obtained agrees well with that obtained from the Hall measurement, which confirms that these non-contact and non-destructive spectroscopy methods with high spatial resolution are reliable to probe the carrier concentration of doped GaN single crystals.

## 1. Introduction

As a third-generation semiconductor, gallium nitride (GaN) has received extensive attention in recent years. Compared with traditional semiconductor silicon, GaN owns the characteristics of a larger bandgap (3.39 eV), higher critical breakdown electric field ( $2.5 \text{ MV}\cdot\text{cm}^{-1}$ ), and greater electron saturation drift velocity ( $2.5 \times 10^7 \text{ cm}\cdot\text{s}^{-1}$ ) [1,2], which endows it with unique advantages in ultraviolet optoelectronics [3–6] and high-power [7,8] and radio-frequency [9] electronics applications.

To design an electronic device with required performance, carrier concentration and mobility should be precisely measured. In the polar semiconductor GaN, when longitudinal optical (LO) phonons and plasmons have the same electric field direction and similar frequencies, an obvious coupling effect will occur to form a LO phonon-plasmon coupled (LOPC) mode, which can be observed in infrared reflectance or Raman spectra [10]. When carrier concentration increases, both peak position and linewidth of LOPC mode in spectra will change simultaneously, which can assist in extracting carrier concentration and mobility [11–15]. Compared with the widely-used Hall measurement, these spectroscopy methods own the advantages of being non-contact and damage-free since the ohmic electrodes are unnecessary. Therefore, for the measurement of the electronic parameters of GaN single crystals, spectroscopy methods are expected to be more applicable than electrical methods [10,16].

There have been some researches using Raman spectroscopy to

characterize the electrical properties of GaN, but most studied samples are thin films [1,17–19] or have a narrow carrier concentration range or single doping type (Si-doped mainly) [18,20–22]. Raman spectroscopy characterization of electrical properties of bulk GaN crystals with a wide carrier concentration range and diverse doping types is still worthy of research. In addition, studies that apply infrared reflectance spectroscopy to characterize the carrier concentration and mobility of GaN are rarely conducted [10,19], which also proves the necessity of further study.

In this article, both Raman and infrared reflectance spectra are applied to measure bulk GaN single crystals with different doping types (Fe-, Si-, Ge-doped and undoped) and carrier concentrations ( $10^{16}$ – $10^{18} \text{ cm}^{-3}$ ). In the analysis of Raman spectra, the dependence of LOPC mode frequency on carrier concentration is established according to the dielectric function, which can effectively predict the behavior of LOPC modes in different carrier concentration samples. Furthermore, the lineshape of LOPC<sup>+</sup> mode is fitted through a Raman intensity relationship, based on which we get the mobility and carrier concentration. The electrical properties are also extracted from the infrared reflectance spectra through the relationship between reflectivity and dielectric function. Comparing the results obtained from Raman and infrared reflectance spectra with those from the Hall measurement, it is found that the carrier concentrations achieved from these three ways agree well while the mobility does not. The possible reasons for the mobility discrepancy are discussed.

Abbreviations: LOPC, LO phonon-plasmon coupled.

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## 2. Materials and methods

Four bulk GaN single crystals were grown by hydride vapor phase epitaxy (HVPE) method with the same size of  $10 \times 10 \times 0.35 \text{ mm}^3$  and different doping types (Fe-, Si-, Ge-doped and undoped), all of which were from Suzhou Nanowin Science Technology Co., Ltd. These samples grew along the *c*-axis with (0001) plane and were epitaxially polished to achieve optical quality. Carrier concentration and mobility of these four samples were measured by the Hall measurement at room temperature (Table 1). It is noted that valid data of GaN: Fe sample cannot be obtained due to its low carrier concentration ( $n \sim 10^{16} \text{ cm}^{-3}$ ).

Raman spectra were measured through a Renishaw spectrometer (InVia Reflex) with a resolution of  $1.5 \text{ cm}^{-1}$ , during which an Ar-ion laser at  $488 \text{ nm}$  was used for excitation. The laser was incident perpendicularly on the (0001) plane of the crystals with the backscattering geometric configuration. Infrared reflectance spectra were obtained via a Fourier transform infrared spectrometer (Shimadzu IRAffinity-1S). This measurement was performed on the (0001) plane of the crystals at  $10^\circ$  incidence. A gold mirror was applied for background scanning. Both the Raman and infrared measurements were performed at room temperature.

## 3. Results and discussion

### 3.1. Analysis of Raman spectra

Fig. 1(a) shows Raman spectra of the four bulk GaN crystals with different doping concentrations, where both  $A_1(\text{LO})$  and  $E_2(\text{high})$  modes can be observed [23]. Since GaN: Fe sample has a relatively low carrier concentration ( $n \sim 1 \times 10^{16} \text{ cm}^{-3}$ ),  $A_1(\text{LO})$  mode will not couple with plasmon or shift to high frequency [24]. Therefore, the frequency of  $A_1(\text{LO})$  Raman peak ( $731 \text{ cm}^{-1}$ ) obtained by Lorentz fitting of GaN: Fe can be taken as that of uncoupled  $A_1(\text{LO})$  mode, whose value corresponds with that of the reported research [24]. In addition, the  $A_1(\text{LO})$  mode coupled with plasmon forming  $\text{LOPC}^+$  mode exhibits blueshift and broadening with increasing concentration in undoped, Ge-doped, and Si-doped GaN, as shown in the inset of Fig. 1(a). To study the lineshape of this coupled mode, we started from the dielectric function as follows [25]:

$$\varepsilon_r(\omega) = \varepsilon_\infty + \varepsilon_\infty \frac{\omega_{\text{LO}}^2 - \omega_{\text{TO}}^2}{\omega_{\text{TO}}^2 - \omega^2 - i\omega\Gamma} - \varepsilon_\infty \frac{\omega_p^2}{\omega(\omega + i\gamma)} \quad (1)$$

where  $\varepsilon_\infty$  is the high-frequency dielectric constant.  $\gamma$  and  $\Gamma$  are the plasmon and phonon damping constants, respectively.  $\omega_{\text{TO}}$  ( $533 \text{ cm}^{-1}$ ) and  $\omega_{\text{LO}}$  ( $731 \text{ cm}^{-1}$ ) are transverse optical (TO) and LO phonon frequencies [26].  $\omega_p$  is the plasmon frequency. The second term in Eq. (1) comes from lattice vibration and the third term is from plasmon.  $\omega_p$  is related to the free carrier concentration  $n$ , which can be expressed as:

$$\omega_p = \left( \frac{4\pi n e^2}{\varepsilon_\infty m^*} \right)^{1/2} \quad (2)$$

where  $m^*$  is the effective mass, and  $m^* = 0.19 m_0$  for GaN [27]. If only consider the excitations near the Brillouin zone center ( $q \cong 0$ ) and neglected damping terms  $\gamma$  and  $\Gamma$ , Eq. (1) can be simplified and written

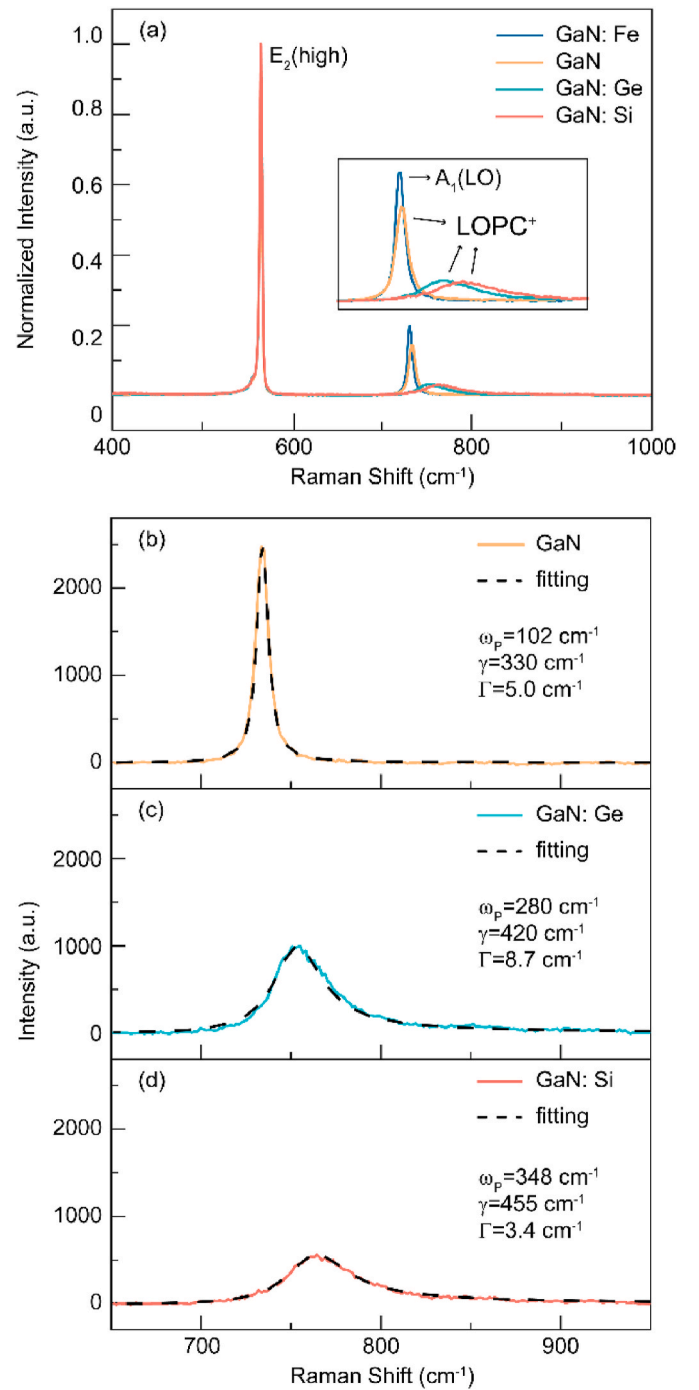


Fig. 1. Analysis of Raman spectra of GaN single crystals with different carrier concentrations. (a) The experimental Raman spectra of samples; The experimental (solid line) and fitting (dash line) curves of (b) undoped GaN, (c) GaN: Ge, and (d) GaN: Si.

Table 1

Carrier concentrations and mobility of the four bulk GaN single crystals obtained from the Hall, Raman, and infrared reflectance measurements.

Sample	Hall		Raman ( $A_1$ mode)		Infrared reflectance ( $E_1$ mode)	
	Carrier concentration ( $\text{cm}^{-3}$ )	Mobility ( $\text{cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$ )	Carrier concentration ( $\text{cm}^{-3}$ )	Mobility ( $\text{cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$ )	Carrier concentration ( $\text{cm}^{-3}$ )	Mobility ( $\text{cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$ )
GaN: Si	$1.36 \times 10^{18}$	355	$1.37 \times 10^{18}$	108	$1.72 \times 10^{18}$	98
GaN: Ge	$7.01 \times 10^{17}$	407	$8.89 \times 10^{17}$	117	$9.53 \times 10^{17}$	123
GaN	$1.18 \times 10^{17}$	777	$1.18 \times 10^{17}$	149	$1.37 \times 10^{17}$	175
GaN: Fe	—	—	—	—	—	—

as  $\varepsilon(\omega) = 0$  [2]. The frequency of LOPC mode  $\omega_{\text{LOPC}}$  is the root of this equation. Solving this equation, we can get the relationship between  $\omega_{\text{LOPC}}$  and  $\omega_p$  [15]:

$$\omega_{\text{LOPC}}^2 = \frac{1}{2} \left\{ \omega_p^2 + \omega_{\text{LO}}^2 \pm \left[ (\omega_p^2 + \omega_{\text{LO}}^2)^2 - 4\omega_p^2\omega_{\text{TO}}^2 \right]^{\frac{1}{2}} \right\} \quad (3)$$

Eq. (3) indicates that the LOPC mode in GaN can split into two modes, that are,  $\text{LOPC}^+$  and  $\text{LOPC}^-$ . Fig. 2 shows the relationship between LOPC mode frequency  $\omega_{\text{LOPC}}$  and carrier concentration  $n$ . From the red solid lines calculated by Eq. (3), as carrier concentration increases, for  $\text{LOPC}^+$  mode, it moves to the high frequency and gets away from  $\omega_{\text{LO}}$ ; for  $\text{LOPC}^-$  mode, it also moves to high frequency but approaches  $\omega_{\text{TO}}$ . In this work, aside from the  $\text{LOPC}^+$  mode, the weak  $\text{LOPC}^-$  mode is observed in the spectra with logarithmic coordinates in GaN: Si and GaN: Ge samples. Both  $\text{LOPC}^+$  and  $\text{LOPC}^-$  mode frequencies shown in Fig. 2 are in line with the theoretical equation when  $n < 10^{18} \text{ cm}^{-3}$ ; however, when  $n > 10^{18} \text{ cm}^{-3}$ , a slight discrepancy is observed.

The above results verify that the correlation between LOPC Raman shift and carrier concentration shown in Eq. (3) can be used to estimate samples' carrier concentration, especially for those with a low carrier concentration, of which the plasmon damping is low enough to be ignored. For samples with a high carrier concentration, since LOPC mode in Raman spectra (Fig. 1(a)) is greatly affected by plasmon damping, merely using  $\omega_{\text{LOPC}}$  as the input parameter is not sufficient to obtain a reliable estimate of carrier concentration, and lineshape fitting taking account of plasmon damping must be applied instead.

An intensity equation is constructed to extract the carrier concentration of GaN by fitting the LOPC Raman peak, which can also obtain carrier mobility  $\mu$ . In the Raman scattering of polar semiconductors, there are three mechanisms for LOPC mode to interact with incident light: deformation-potential + electro-optic (DP + EO) mechanism, charge density fluctuation (CDF) mechanism, and impurity induction (IIF) mechanism [14]. The DP + EO mechanism describes how atomic displacements and macroscopic longitudinal field modulate the polarizability; the CDF mechanism describes the scattering caused by the fluctuation of charge density; the IIF mechanism describes how the effect between electrons and phonons caused by impurities affects scattering process. Among these three mechanisms, the contribution of DP + EO mechanism is predominant in GaN, according to the relevant research [1]. Considering this mechanism only, the intensity of LOPC peak can be expressed as [28,29]:

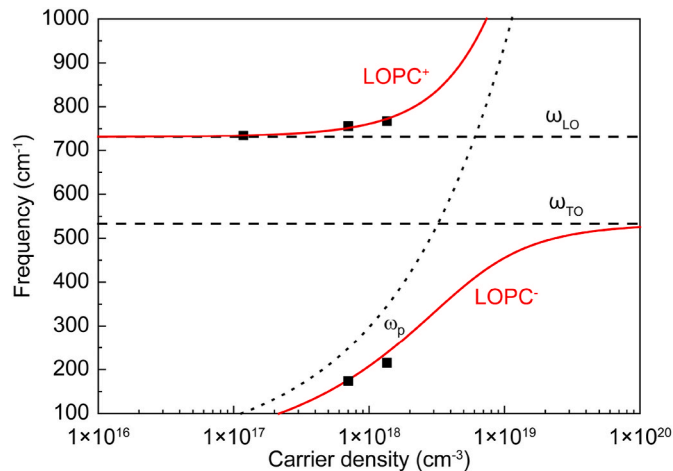


Fig. 2. The LO phonon-plasmon coupled interaction in GaN. The red solid lines indicate that the LOPC mode depends on carrier concentration, which are calculated by Eq. (3). The dotted line shows the plasmon frequency  $\omega_p$ , which is calculated from Eq. (2). The black solid dots come from the Hall and Raman measurements.

$$I(\omega) = \text{const} \cdot A(\omega) \cdot \text{Im}[-\varepsilon_r(\omega)^{-1}] \quad (4)$$

where  $\omega$  is the Raman shift, and  $A(\omega)$  is given by:

$$\begin{aligned} A(\omega) = & 1 + 2C \frac{\omega_{\text{TO}}^2}{\delta} [\omega_p^2 \gamma (\omega_{\text{TO}}^2 - \omega^2) \\ & - \omega^2 \Gamma (\omega^2 + \gamma^2 - \omega_p^2) \\ & + C^2 \{ \omega_p^2 [\gamma (\omega_{\text{LO}}^2 - \omega_{\text{TO}}^2) + \Gamma (\omega_p^2 - 2\omega^2)] \\ & + \omega^2 \Gamma (\omega^2 + \gamma^2) \left[ \frac{\omega_{\text{TO}}^4}{\delta (\omega_{\text{LO}}^2 - \omega_{\text{TO}}^2)} \right] \\ & \delta = \omega_p^2 \gamma [(\omega \Gamma)^2 + (\omega_{\text{TO}}^2 - \omega^2)^2] \\ & + \omega^2 \Gamma (\omega^2 + \gamma^2) (\omega_{\text{LO}}^2 - \omega_{\text{TO}}^2) \end{aligned} \quad (5)$$

where  $C$  is the Faust-Henry coefficient. The electron mobility  $\mu$  can be obtained from plasmon damping constant  $\gamma$  by the following equation:

$$\gamma = \frac{e}{m^* \mu} \quad (6)$$

It should be noted that the mobility is anisotropic since GaN is a uniaxial crystal. The mobility obtained through Hall measurement was in-plane. As for the spectroscopy methods, the direction of the fitting mobility depends on the phonon vibration direction, as the measured phonon is coupled with plasmon. In the Raman measurement, the  $A_1(\text{LO})$  mode vibrates along the  $c$ -axis, so we got the out-of-plane mobility. In the infrared measurement, the  $E_1(\text{LO})$  mode coupled with the plasmon vibrates perpendicular to the  $c$ -axis, so the obtained mobility is the in-plane one (see details in section 3.2). For GaN, its effective mass can be written as  $m^* = [(1/3)(1/m_{\parallel}) + (2/3)(1/m_{\perp})]^{-1}$  ( $m_{\parallel}$  and  $m_{\perp}$  are the parallel and perpendicular effective mass, respectively). However, since there is little difference between the perpendicular  $m_{\perp}$  and parallel effective mass  $m_{\parallel}$  of GaN [30,31], we considered the mobility of GaN as isotropic and simplified the mobility calculation in Raman and infrared spectra by using the same effective mass, that is  $m^* = 0.19 m_0$  [27].  $C = 0.48$ ,  $\varepsilon_{\infty} = 5.35$  are taken for the Raman fitting [18,24,27]. Based on Eqs. (1), (2), (4)-(6) and choosing  $\gamma$ ,  $\Gamma$ , and  $\omega_p$  as fitting parameters, the free carrier concentration and mobility can be obtained.

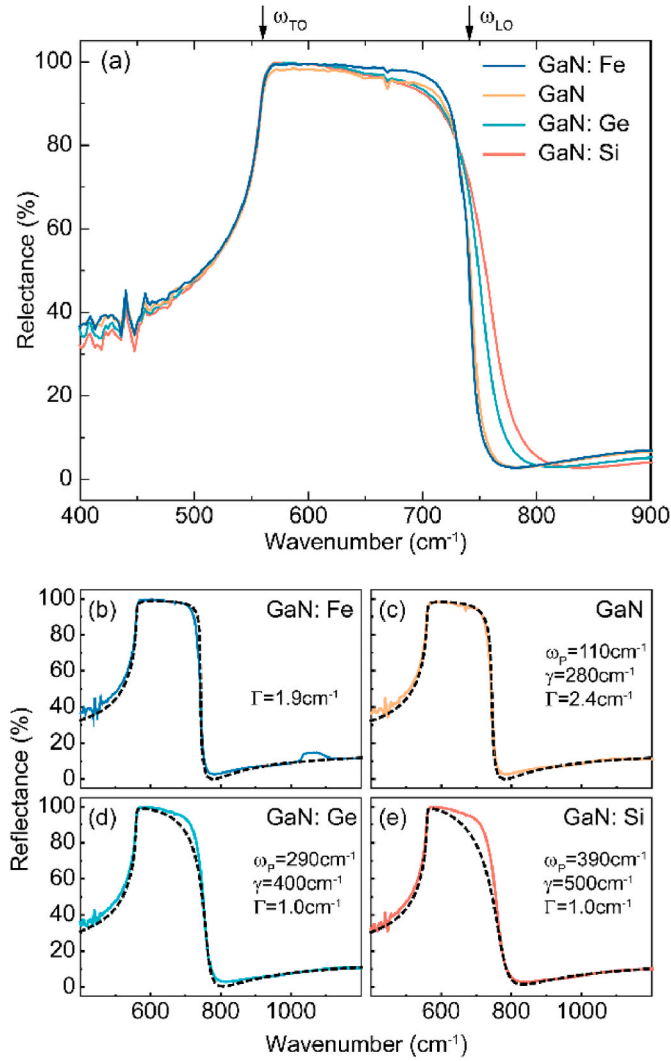
In this research, we focus on  $\text{LOPC}^+$  branch. Since the carrier concentration of sample GaN: Fe is too low that the  $A_1(\text{LO})$  mode is uncoupled, it cannot be fitted by this way. The  $\text{LOPC}^+$  peaks and fitting curves of undoped GaN, GaN: Ge, and GaN: Si samples are shown in Fig. 1(b)–(d). As carrier concentration increases, the  $\text{LOPC}^+$  mode shows blueshift and broadening, which means LOPC effect is strengthened. The fitting curves well correspond to the experimental results. The carrier concentrations and mobility gotten from Raman spectra are shown in Table 1.

### 3.2. Analysis of infrared reflectance spectra

Fig. 3(a) shows the infrared reflectance spectra of the four bulk GaN crystals. Since the light is incident from the (0001) plane, the observed phonons in infrared reflectance spectra are  $E_1$  modes [10]. Phonon polaritons formed by coupling the  $E_1(\text{TO})$  phonons and the photons result in a reststrahlen band with strong reflection, whose frequency is between  $\omega_{\text{TO}}$  and  $\omega_{\text{LO}}$  [32]. The high-frequency edge of reststrahlen band becomes smooth as the carrier concentration increases, for LOPC mode formed by the coupling of  $E_1(\text{LO})$  and plasmon will affect the behavior of phonon polaritons. Thus, we can get the carrier concentrations of GaN by fitting the infrared spectra.

At the junction between vacuum or air and solid surfaces, vertical and parallel reflectivity are considered respectively as follows [33,34]:

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**Fig. 3.** Analysis of infrared reflectance spectra of GaN single crystals with different carrier concentration. (a) The experimental infrared reflectance spectra of samples; The experimental (solid line) and fitting (dash line) curves of (b) GaN: Fe, (c) undoped GaN, (d) GaN: Ge, and (e) GaN: Si.

$$R_s = \frac{|\cos\theta_i - \sqrt{\epsilon - \sin^2\theta_i}|^2}{|\cos\theta_i + \sqrt{\epsilon - \sin^2\theta_i}|^2} \quad (7)$$

$$R_p = \frac{|\epsilon\cos\theta_i - \sqrt{\epsilon - \sin^2\theta_i}|^2}{|\epsilon\cos\theta_i + \sqrt{\epsilon - \sin^2\theta_i}|^2}$$

where  $\theta_i$  is the incident angle, which is  $10^\circ$  in the infrared measurement. Here, it can be approximated to normal incidence ( $\theta_i = 0^\circ$ ) within allowable error range. The reflectivity of light at normal incidence is:

$$R(\omega) = R_s = R_p = \frac{[\eta(\omega) - 1]^2 + \kappa(\omega)^2}{[\eta(\omega) + 1]^2 + \kappa(\omega)^2} \quad (8)$$

where  $\kappa(\omega)$  and  $\eta(\omega)$  refer to extinction coefficient and refractive index, respectively, which are related to the dielectric function as:

$$\epsilon_r(\omega) = [\eta(\omega) + i\kappa(\omega)]^2 \quad (9)$$

The infrared reflectance spectra of GaN, GaN: Ge, and GaN: Si can be fitted using Eqs. (1), (8) and (9) as fitting functions with  $\omega_p$ ,  $\Gamma$ , and  $\gamma$  as fitting parameters. However, it should be noted that this method cannot obtain a valid carrier concentration for GaN: Fe with a low carrier

concentration ( $n \sim 10^{16} \text{ cm}^{-3}$ ) since  $E_1(\text{LO})$  and plasmon are weak coupling. The dielectric function of GaN: Fe should omit the carrier term, yielding the equation [35]:

$$\epsilon_r(\omega) = \epsilon_\infty + \epsilon_\infty \frac{\omega_{\text{LO}}^2 - \omega_{\text{TO}}^2}{\omega_{\text{TO}}^2 - \omega^2 - i\omega\Gamma} \quad (10)$$

The infrared reflectance spectra of samples with low carrier concentration can be fitted by using Eqs. (8)–(10) with  $\Gamma$  as the only fitting parameter. The frequencies of  $E_1(\text{TO})$  and  $E_1(\text{LO})$  are taken as  $560 \text{ cm}^{-1}$  and  $741 \text{ cm}^{-1}$  in the fitting of four samples [36–38]. The fitting curves are shown in Fig. 3(b)–(e) by dashed lines, and the obtained results are summarized in Table 1. The fitting curves are consistent with the experimental curves in the range of  $\omega > 750 \text{ cm}^{-1}$ . It is worth noting that as carrier concentration increases, the fitting curve shows a certain deviation from the experimental curve around  $700 \text{ cm}^{-1}$ . The main reason is that the dielectric function in Eq. (1) is simplified by assuming TO-phonon damping equals LO-phonon damping [39]. In fact, for doped semiconductors, LO-phonon damping will increase with impurity concentration while TO-phonon damping is almost independent of that [16, 40].

### 3.3. Comparison

Table 1 summarizes the carrier concentrations and mobility tested by Hall, Raman, and infrared reflectance measurements. For the range of  $n > 10^{17} \text{ cm}^{-3}$ , a good consistency is shown among the carrier concentrations obtained from these three methods. However, the mobility obtained by spectroscopy methods is lower than that obtained by the electrical method, which the following reasons may cause. One reason may be that the assumption that TO-phonon damping is equal to LO-phonon damping in the dielectric function model does not reflect the actual situation well. Especially when the doping concentration increases, LO-phonon damping increases significantly while TO-phonon damping does not [40]. Also, when considering the distribution of carrier velocities, Hall mobility is no longer equal to drift mobility obtained from Raman and infrared measurements, and the ratio of Hall mobility and drift mobility depends on the scattering mechanism [41]. Another possible reason is that the carriers are in an equilibrium state in the Hall measurement, while in Raman or infrared reflectance measurement, they are in a non-equilibrium state due to light excitation [19]. This may cause a difference in the predominant carrier scattering mechanism among different methods, resulting in higher plasmon damping constants of Raman and infrared reflectance measurements than that of Hall measurement. Thus, the optical mobility will be lower.

## 4. Conclusions

In conclusion, Raman and infrared reflectance spectroscopy are used to study GaN single crystals with different doping types (Fe, Ge, Si-doped, and undoped) and carrier concentrations. The relationship between Raman shift of LOPC mode and carrier concentration is studied, which is suitable for preliminarily estimating the carrier concentration of low carrier concentration samples. For the samples with a carrier concentration greater than  $10^{17} \text{ cm}^{-3}$ , the Raman and infrared reflectance spectra with LOPC mode of GaN crystals are fitted to obtain their carrier concentrations and mobility. Comparing the Hall, Raman, and infrared results, it is found that the carrier concentrations extracted from these three ways show a good consistency while the mobility does not. The low mobility in spectroscopy methods may come from the carrier scattering mechanisms and the adopted simplification made in the dielectric function. These results show that Raman and infrared reflectance spectroscopy can be confirmed as reliable methods for measuring the carrier concentration of GaN single crystals.



## Author Contributions

**Linxuan Li:** Investigation, Formal analysis, Methodology, Writing – original draft; **Siqi Zhu:** Writing – review & editing; **Lu Cheng:** Data curation, Methodology. **Hongsheng Qi:** Methodology, **Yu Fan:** Methodology, **Wei Zheng:** Conceptualization, Funding acquisition, Supervision.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

Data will be made available on request.

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