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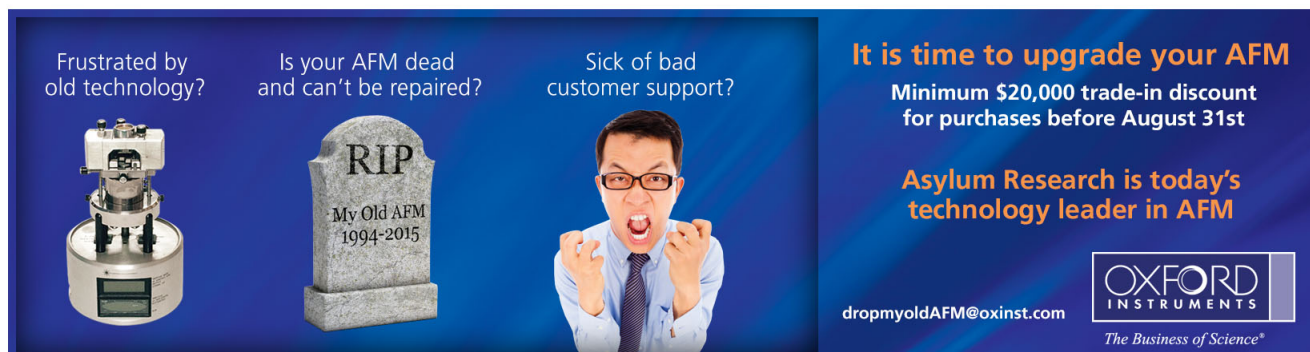
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Extraction of infrared optical constants from fringing reflectance spectra

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We propose a simple and reliable method for extracting the optical constants of homogeneous dielectrics which can be pure or impure, and polar or nonpolar. The extraction is made from fringing reflectance spectra of slab samples with and without metal on the backside. The method is demonstrated to work well for polar semiconductors GaAs and InP in the infrared regime. The extracted extinction coefficient spectra exhibit plenty of features which correspond well to those in absorption and Raman spectra. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4898037>]

I. INTRODUCTION

Knowledge of the complex refractive index of a dielectric slab functioning as substrate is important for investigation of optical properties of a film deposited on the substrate.^{1–3} For a slab of weak optical absorption, one usually considers only the real part of the index (called the refractive index) and neglects the imaginary part (called the extinction coefficient) for simplicity in analysis.^{2,4–9} However, the latter becomes important when a more accurate analysis is required.² It can reveal spectral features behind which there are interesting physical processes.¹⁰ In general, there are two ways to obtain the refractive index and the extinction coefficient of the slab material. One is to measure the incoherent transmittance and reflectance spectra without interference fringes;^{11–13} the other is to measure the coherent transmittance or reflectance spectrum with interference fringes.^{1,4,7,14} The latter is preferable because only a single spectrum is needed for extraction of the complex index and the thickness of the slab. Theoretically, the complex index can be extracted by using the upper and the lower envelopes of the fringes. In practice, however, the use of the lower envelope may cause significant error since it is much more sensitive to the experimental resolution which may result from various factors, such as the experimental setup, the sample surface roughness, the sample surface parallelism, the incident light beam size, and the light collection for detection.

In this work, we propose a method of extracting the optical constants from the fringing reflectance spectra of slab samples with and without metal on the backside. The reflection measurement scheme is employed because it can be applied to a sample backed with opaque material. The method is demonstrated to work well for polar semiconductors GaAs and InP in the infrared regime. The refractive index is first determined by the single oscillator model, and the extinction coefficient is then extracted from the upper envelope of the fringing reflectance spectrum. Also, the sample thicknesses are determined precisely from the periods of oscillating fringes. Our scheme can be extended to any homogeneous dielectrics under certain available conditions.

This paper is organized as follows. We describe sample preparation and spectrum measurement in Sec. II, and the method of extracting the optical constants in Sec. III. We then demonstrate and discuss in Sec. IV the extraction of the optical constants of samples of semiconductors GaAs and InP. Finally, we draw a conclusion in Sec. V.

II. SAMPLE PREPARATION AND REFLECTION SPECTRUM MEASUREMENT

We prepared four two-side polished samples of $10 \times 10 \text{ mm}^2$ in area and about $360 \mu\text{m}$ in thickness; two were cut from an undoped semi-insulating (SI) GaAs wafer and the other two from an iron-doped SI InP wafer. One of the GaAs samples and one of the InP samples were coated with a 100 nm gold film on one surface by E-gun evaporation. In addition, a gold-coated mirror was prepared to serve as the reference. The reflection spectra of the samples and the reference mirror were measured by a Fourier transform infrared (FTIR) spectrometer (Bruker IFS 66v/S). In the measurement, the light of the source was directed by a module into the samples and the mirror at an incident angle of 11° . All the four samples were arranged that the light was incident on uncoated surfaces. The reflectance spectrum was obtained for each sample by dividing the raw reflection spectrum by the reference spectrum. The spectral resolution was set at 0.25 cm^{-1} and ensured being high enough to give steady fringing patterns. These spectra ranged from about 85 to 700 cm^{-1} , limited by optical components in the spectrometer, such as a $6\text{-}\mu\text{m}$ thick Mylar beamsplitter and a deuterated-triglycine-sulfate (DTGS) detector with a polyethylene window. The experiment was conducted with the samples at room temperature and a constant pressure of 3 mbar.

III. METHODS OF EXTRACTING OPTICAL CONSTANTS

A. Extracting optical constants by single-oscillator model

The far-infrared properties of pure polar crystals containing two atoms per unit cell are generally described by the single-oscillator model which yields the dielectric function¹⁵

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$$\begin{aligned} \varepsilon_{\text{so}}(\omega) &= \varepsilon_{\infty} + \frac{\omega_{\text{TO}}^2}{\omega_{\text{TO}}^2 - \omega^2 - i\gamma_{\text{TO}}\omega} (\varepsilon_s - \varepsilon_{\infty}) \\ &\equiv [n_{\text{so}}(\omega) + i\kappa_{\text{so}}(\omega)]^2, \end{aligned} \quad (1)$$

where ω_{TO} is the transverse optical (TO) phonon frequency and also the natural frequency of the oscillator, γ_{TO} the damping factor, ε_s the static dielectric constant, ε_{∞} the high-frequency dielectric constant due to the electronic polarization, and n_{so} and κ_{so} the refractive index and the extinction coefficient, respectively, associated with the oscillator. The model can give a refractive index n_{so} that is valid in a wide spectral region as long as the frequency is well below the electronic transition resonance frequency. However, the given κ_{so} is proper only in a narrow region (approximately the reststrahlen band) about the resonance frequency ω_{TO} , where the optical absorption is strong enough and the single TO phonon process dominates the optical properties.¹⁵ Beyond the region where the absorption is weak, multiphonon processes, especially the two-phonon processes, become important and responsible for most absorption features.¹⁰

The parameters in formula (1) can be extracted by fitting the theoretical reflectance spectrum yielded by the model dielectric function to the measured reflectance spectrum. Figure 1 shows the reflectance spectra of the GaAs samples without and with gold. The spectra exhibit prominent fringing oscillations due to wave interference except where the absorption is strong. As can be seen from the figure, in the

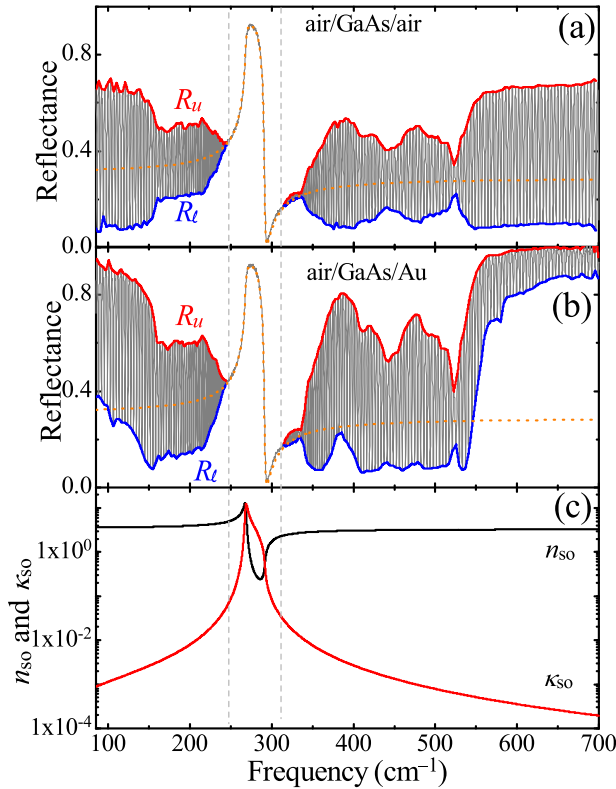


FIG. 1. The measured reflectance spectra of GaAs samples (a) without and (b) with metal on the backside and (c) the spectra of the refractive index n_{so} and the extinction coefficient κ_{so} extracted with the single-oscillator model. The red and blue solid curves in (a) and (b) are the upper envelopes R_u and lower envelopes R_l of the fringes, respectively. The orange dotted curves are the calculated reflectance spectra with the single-oscillator model.

strong absorption region ranging from 250 to 310 cm^{-1} , the reflectance spectra are characteristic only of GaAs, no matter if the sample is coated with gold on the backside. This portion of spectra can be excellently fitted, as shown in the figure, by the theoretical spectrum

$$R_{\text{so}} = \frac{(n_{\text{so}} - 1)^2 + \kappa_{\text{so}}^2}{(n_{\text{so}} + 1)^2 + \kappa_{\text{so}}^2}, \quad (2)$$

with the parameters listed in Table I, provided the light is normally incident on a semi-infinite material. The values of the parameters are in excellent agreement with those in the literature.¹⁵ With these parameters, the spectra of n_{so} and κ_{so} are plotted in Fig. 1(c). As expected, κ_{so} is highest about the resonance frequency $\omega_{\text{TO}} = 268 \text{ cm}^{-1}$ and declines monotonically to lower than ~ 0.03 as ω is away from the strong absorption region. Such simply varying κ_{so} cannot explain the complex fringing patterns in Fig. 1. Here, the multiphonon processes play an important part,¹⁶ and the fringing patterns contain relevant information that enables us to extract the real extinction coefficient κ . In contrast, n_{so} always remains high (> 0.3) and approaches either $\sqrt{\varepsilon_s}$ or $\sqrt{\varepsilon_{\infty}}$ as ω is away from the reststrahlen band. Any correction due to multiphonon processes to the refractive index is expected to be insignificant.

B. Extracting optical constants from fringing reflectance spectra of uncoated samples

We are now in a position to extract κ of the sample material from the fringing patterns. To this end, we assume the real refractive index n to be n_{so} over the whole spectral range. We also neglect any correction to κ_{so} (i.e., $\kappa = \kappa_{\text{so}}$) in the strong absorption region where fringing is unobservable. Provided that an electromagnetic plane wave is normally incident on an uncoated sample, elementary electromagnetics derivation leads to the reflectance formula

$$R(\omega) = \frac{\rho^2(1 - \gamma)^2 + 4\rho^2\gamma \sin^2\phi}{(1 - \rho^2\gamma)^2 + 4\rho^2\gamma \sin^2(\phi + \theta)}, \quad (3)$$

where ρ and θ are the modulus and the angle, respectively, of the one-interface reflection coefficient $r = (1 - n - i\kappa)/(1 + n + i\kappa) = \rho e^{i\theta}$, $\gamma = e^{-2\kappa k_0 d}$, $\phi = nk_0 d$, $k_0 = \omega/c$ (c being the light speed in vacuum), and d is the sample thickness. In the strong absorption region where $\kappa k_0 d \gg 1$ or $\gamma \ll 1$, the reflectance reduces to the simple form $R = \rho^2$. The spectrum exhibits fringing only when the absorption is sufficiently weak that γ is not too small, say, $\gamma > 0.01$. Under the condition of weak absorption such that $\kappa \ll n - 1$, $\rho \approx (n - 1)/(n + 1)$, $\theta \approx \pi$, and Eq. (3) gives a fringing spectrum with

TABLE I. Parameters for the single-oscillator model.

	ε_{∞}	ε_s	$\omega_{\text{TO}} (\text{cm}^{-1})$	$\gamma_{\text{TO}} (\text{cm}^{-1})$
GaAs	11	13.01	268	2.2
InP	9.5	12.27	304	0.7

$$R_u = \rho_u^2, \quad \rho_u \equiv \frac{\rho(1+\gamma)}{1+\rho^2\gamma}, \quad (4)$$

as the upper envelope and

$$R_\ell = \rho_\ell^2, \quad \rho_\ell \equiv \frac{\rho(1-\gamma)}{1-\rho^2\gamma}, \quad (5)$$

as the lower envelope. Either Eq. (4) or Eq. (5) permits determination of γ

$$\gamma = \frac{\rho_u - \rho}{\rho(1 - \rho\rho_u)} = \frac{\rho - \rho_\ell}{\rho(1 - \rho\rho_\ell)}, \quad (6)$$

if ρ has been given by the single-oscillator model ($n = n_{so}$) and the envelope ρ_u or ρ_ℓ has been known from the fringing spectrum. Even without the knowledge of ρ in advance, Eqs. (4) and (5) can jointly give

$$\rho = \frac{\Gamma_+}{1 + \sqrt{1 - \Gamma_+^2}}, \quad \Gamma_+ = \frac{\rho_u + \rho_\ell}{1 + \rho_u\rho_\ell}, \quad (7)$$

and γ by Eq. (6) if both ρ_u and ρ_ℓ have been known. Further determination of κ is then allowed if d has been known.

Notice that Eqs. (4), (5), and (7) are also applied to the case of strong absorption. The envelopes merge, $\rho = \rho_u = \rho_\ell$, in this case because $\gamma \rightarrow 0$ and the angles θ and ϕ become irrelevant. However, Eq. (6) cannot be used to determine γ and κ because of the difficulty in distinguishing ρ_u from ρ_ℓ by the simple measurement. The single-oscillator model is therefore required for determining κ in the strong absorption region where $\kappa = \kappa_{so}$, as has been described in Sec. III A.

C. Extracting optical constants from fringing reflectance spectra of gold-coated samples

For an electromagnetic wave normally incident on a sample coated with gold on the backside, the reflectance formula is

$$R(\omega) = \frac{(\rho - \gamma)^2 + 4\rho\gamma \sin^2(\phi - \frac{1}{2}\theta)}{(1 - \rho\gamma)^2 + 4\rho\gamma \sin^2(\phi + \frac{1}{2}\theta)}, \quad (8)$$

where the symbols are defined as for Eq. (3). In derivation of Eq. (8), we have assumed the metal to be perfectly conducting. Similar to Eq. (3), Eq. (8) reduces to $R = \rho^2$ under the strong absorption condition ($\gamma \rightarrow 0$). Under the condition of either weak absorption ($\kappa \ll n - 1$) or strong absorption ($\gamma \ll 1$), the reflectance spectrum has an upper envelope

$$R_u = \rho_u^2, \quad \rho_u \equiv \frac{\rho + \gamma}{1 + \rho\gamma}, \quad (9)$$

and a lower envelope

$$R_\ell = \rho_\ell^2, \quad \rho_\ell \equiv \frac{|\rho - \gamma|}{1 - \rho\gamma}. \quad (10)$$

If ρ has been given by the single-oscillator model, the upper envelope can determine γ and hence κ using

$$\gamma = \frac{\rho_u - \rho}{1 - \rho\rho_u}. \quad (11)$$

In contrast, the lower envelope by itself cannot determine γ uniquely in some case even with the knowledge of ρ . From Eq. (10), γ is either

$$\gamma = \frac{\rho - \rho_\ell}{1 - \rho\rho_\ell}, \quad (12)$$

if $\gamma \leq \rho$, or

$$\gamma = \frac{\rho + \rho_\ell}{1 + \rho\rho_\ell}, \quad (13)$$

if $\gamma \geq \rho$. Only when $\rho_\ell > \rho$, γ can be uniquely determined; in this condition, only Eq. (13) is acceptable because Eq. (12) would yield a negative value of γ .

D. Extracting the sample thickness from fringing reflectance spectra

Extracting the value of κ from γ requires the knowledge of the sample thickness d . Such information can be obtained by an independent measurement or directly from the fringing oscillation period of the reflectance spectrum. The angle difference $\Delta\phi$ is π between two nearest oscillating peaks in the weak absorption region according to Eqs. (3) and (8). Considering the contribution to $\Delta\phi$ of the changes in k_0 and n with ω , we have the expression for d

$$d = \pi \frac{c}{\Delta\omega} \left(\frac{\partial\omega n}{\partial\omega} \right)^{-1}, \quad (14)$$

where $\Delta\omega$ is the frequency difference between two nearest peaks (i.e., the period of oscillating fringes), and use has been made of the fact that n slowly varies with ω in the interval between the two successive peaks. For the data of $\partial\omega n / \partial\omega$, we appeal to the dielectric function of the single-oscillator model, Eq. (1), or the data in Fig. 1(c). Under the weak absorption condition where $\gamma_{TO}\omega / |\omega_{TO}^2 - \omega^2| \ll 1$, there is a closed-form but complicated expression for approximation of $\partial\omega n / \partial\omega$.

IV. DEMONSTRATION AND DISCUSSION

Let us first analyze the reflectance spectrum for the free-standing GaAs sample as has been shown in Fig. 1(a). The fringing period gives an average sample thickness of $d = 367 \mu\text{m}$ according to Eq. (14) with n given by Fig. 1(c), in agreement with the nominal thickness of $360 \mu\text{m}$. With $d = 367 \mu\text{m}$ and n_{so} given by Fig. 1(c), we extract the spectra of the extinction coefficient κ from the upper and the lower envelopes according to Eq. (6). The results are shown in Fig. 2. Ideally, the κ spectra should be in consistency over the whole spectral range. Practically, they are somewhat different. The difference is especially remarkable where κ is low and the refractive index n is high. For illustration, we plot in the inset of Fig. 2 the zoom-in oscillation pattern of Eq. (3) in the spectral region from 602 to 611 cm^{-1} where the absorption is weak and n_{so} and κ_{so} in Fig. 1(c) are used in

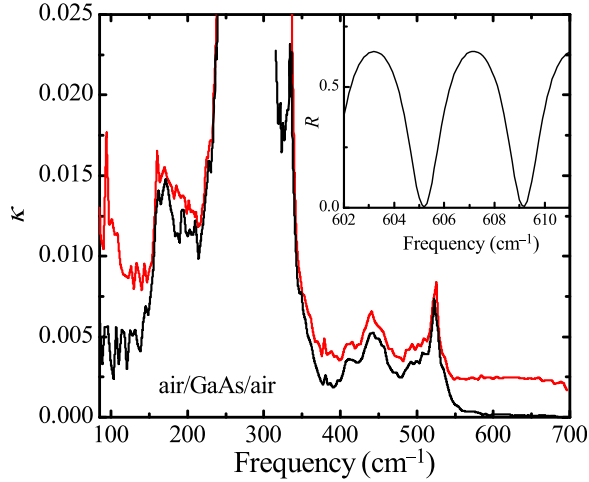


FIG. 2. The extinction coefficient κ spectra of the GaAs sample without metal. The black and the red curves are extracted from the upper and lower envelopes in Fig. 1(a), respectively. The inset shows a zoom-in oscillation pattern of the theoretical reflectance spectrum of the sample in $602 \leq \omega \leq 611 \text{ cm}^{-1}$.

the calculation. We see that the curvature of the R versus ω curve is much larger at the minima than at the maxima. This implies that the measured lower envelope is more sensitive to the spectral resolution than the upper envelope in the region where the absorption is weak. A precise measurement for the lower envelope requires a higher resolution than for the upper envelope. The argument can be further justified by Eq. (3) from which we obtain the curvature at minima

$$\left(\frac{\partial^2 R}{\partial \omega^2}\right)_{\sin^2 \phi=0} = 8 \left(\frac{nd}{c}\right)^2 \frac{\rho^2}{(1-\rho^2)^2}, \quad (15a)$$

and the curvature at maxima

$$\left(\frac{\partial^2 R}{\partial \omega^2}\right)_{\sin^2 \phi=1} = -8 \left(\frac{nd}{c}\right)^2 \frac{\rho^2(1-\rho^2)^2}{(1+\rho^2)^4}, \quad (15b)$$

where we assume $\gamma=1$ for weak absorption and neglect the dispersion of n . The ratio of curvatures (ROC) [Eqs. (15a) and (15b)] is

$$\text{ROC} = -\left(\frac{1+\rho^2}{1-\rho^2}\right)^4 = -\left(\frac{n^2+1}{2n}\right)^4. \quad (16)$$

For $n = \sqrt{\epsilon_\infty} = 3.32$ and $\sqrt{\epsilon_s} = 3.61$, ROC is -10.7 and -14.2 , respectively. Therefore, the optical constants extracted from the upper envelope are more reliable than from the lower envelope. This is evidenced in Fig. 2 where the upper envelope can give an extinction coefficient as low as zero but the lower envelope cannot. The extraction with the lower envelope is seriously limited by the spectral resolution which arises in practice from various factors such as the experimental settings, the sample surface parallelism, the sample surface roughness, the light beam size, and the collection of light for detection.

We now analyze the reflectance spectrum in Fig. 1(b) for the GaAs sample coated with gold. The fringing period

gives an average sample thickness $d = 366 \mu\text{m}$. In this case, we can only use the upper envelope to extract the extinction coefficient because the lower envelope cannot give a unique result over the whole spectral range. Figure 3 shows the κ spectrum extracted from the upper envelope according to Eq. (11) with $d = 366 \mu\text{m}$ and n given by Fig. 1(c). For comparison, we also plot the κ spectrum obtained from the free-standing GaAs sample, which has been displayed in Fig. 2. As can be seen, the two measured κ spectra are basically coincident under the condition of the present resolution, implying the steadiness of our model for extracting the κ spectrum from the upper envelope of the fringing reflectance spectrum of a slab with or without metal. In fact, the model for samples coated with metal is more reliable, as suggested by the zoom-in oscillation pattern in the inset of Fig. 3. The oscillation pattern is calculated using Eq. (8) with n_{so} and κ_{so} given in Fig. 1(c). Obviously, the curvature at maxima is smaller than that shown in the inset of Fig. 2 for the free-standing sample. The former can be approximated as

$$\left(\frac{\partial^2 R}{\partial \omega^2}\right)_{\sin^2 \phi=1} = -32 \left(\frac{nd}{c}\right)^2 (\kappa k_0 d) \frac{\rho(1-\rho)}{(1+\rho)^3}, \quad (17)$$

where we assume $\kappa k_0 d \ll 1$ for weak absorption. We see that the curvature depends on $\kappa k_0 d$ and hence can be very small. In the extreme case $\kappa k_0 d = 0$, the curvature is zero and the reflectance $R = 1$ without fringes.

The κ spectra in Fig. 3 reveal plenty of features which correspond to those in infrared absorption or Raman spectra.^{16–18} These features have been analyzed and ascribed to infrared absorption associated with two-phonon sum or difference processes.¹⁰

The model can be applied to other polar semiconductors. Figure 4 shows the measured reflectance spectra of the InP samples (a) without and (b) with gold, (c) the n_{so} and κ_{so}

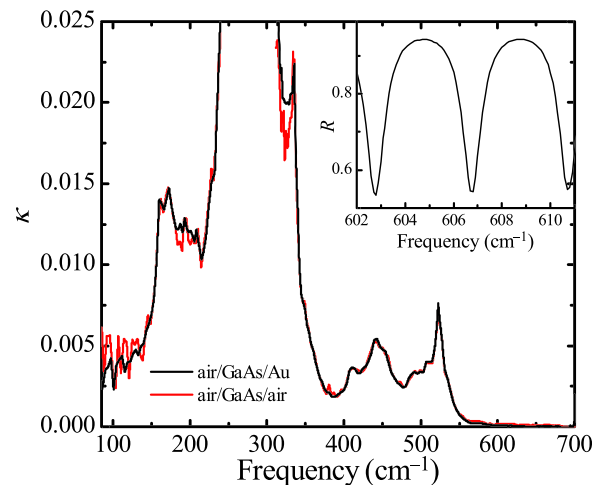


FIG. 3. The extinction coefficient κ spectrum extracted from the upper envelope of the GaAs sample with metal which has been shown in Fig. 1(b) (in black). For comparison, we also show the κ spectrum extracted from the upper envelope of the GaAs without metal, which has been shown in Fig. 1(a) (in red). The inset shows a zoom-in oscillation pattern of the theoretical reflectance spectrum of the sample with metal in $602 \leq \omega \leq 611 \text{ cm}^{-1}$.

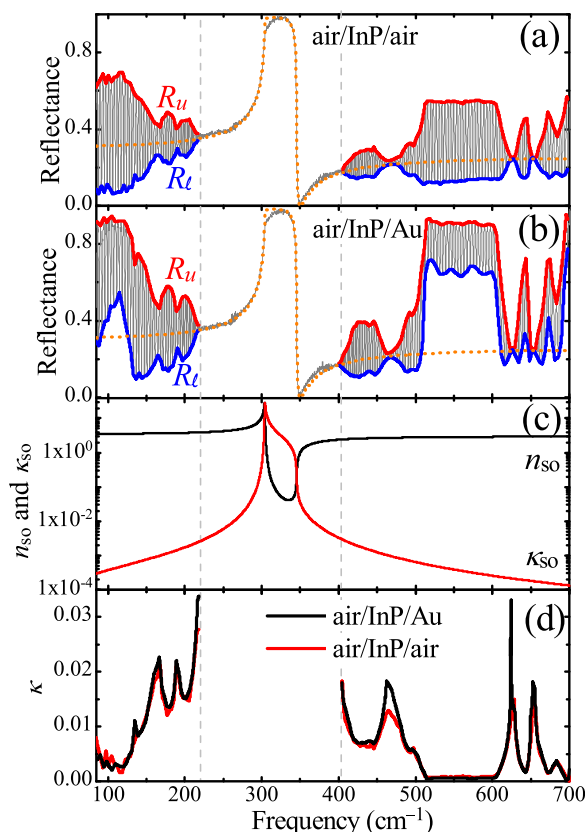


FIG. 4. The measured reflectance spectra of InP samples (a) without and (b) with metal on the backside, (c) the spectra of the refractive index n_{so} and the extinction coefficient κ_{so} extracted with the single-oscillator model, and (d) the extinction coefficient κ spectra extracted from the upper envelopes in (a) (in red) and in (b) (in black). The red and blue solid curves in (a) and (b) are the upper envelopes R_u and lower envelopes R_l of the fringes, respectively. The orange dotted curves are the calculated reflectance spectra with the single-oscillator model.

extracted using the single-oscillator model, and (d) the κ spectra extracted from the upper envelopes in (a) and (b). The parameters for the single-oscillator model are obtained by fitting and listed in Table I. The thicknesses of the samples with and without gold are $d = 374$ and $363 \mu\text{m}$, respectively, extracted from the periods of the oscillating fringes. The features in the κ spectra are consistent with those in absorption and Raman spectra.^{19,20} The window of extremely low absorption ranging from 520 to 600 cm^{-1} results from the small width of the optical phonon branches and the wide gap between the acoustic and the optical phonon branches.

The sample thickness is crucial to the scheme for extraction of optical constants. A proper thickness should be such that the reflectance spectrum can give a correct upper envelope and simultaneously, the extracted κ spectrum has a high resolution to exhibit important features. For a correct upper envelope, the period of oscillating fringes $\Delta\omega$ has to be larger than the experimental resolution δ , i.e., $\delta < \Delta\omega = \pi c/nd$. On the other side, the period should be small enough since it is just the envelope resolution, i.e., the resolution of the extracted κ spectrum. Therefore, an acceptable κ spectrum has a resolution $\Delta\omega$ with δ as the lower bound. Increasing the sample thickness improves the resolution of κ but worsens the reliability of the upper envelope.

For $d \approx 360 \mu\text{m}$ as in our case, the κ resolution is about 4 cm^{-1} .

In practice, the sample thickness d is limited by how high the extinction coefficient κ is when we measure. For the fringing to be observable, γ has to be larger than, say, $e^{-4} \approx 0.02$, corresponding to the condition $d < 2/k_0\kappa$. Since our model is subjected to the condition $\kappa \ll n - 1$, the measure is reliable for κ up to 0.03 . Our extracted κ spectra shown in Figs. 3 and 4(c) are therefore reliable since κ and d both meet the criteria over the whole spectral range.

We have demonstrated successfully the extraction of the κ spectra for pure GaAs and InP in the infrared regime with the refractive index n given by the single-oscillator model. The scheme can be valid also for homogeneous dielectrics, pure or impure, polar or nonpolar, if a reasonable refractive index n has been known in advance and all the requirements described above are satisfied. Steady n is more available from literature than steady κ since the former is much less influenced by impurities. Data of n for a pure material can be used for a sample of the same host material but with impurities. The extracted κ spectrum exhibits features which are characteristic of the impure material.

V. CONCLUSION

We have proposed simple and reliable models for extraction of the extinction coefficient spectra from the upper envelopes of fringing reflectance spectra of slab samples with and without metal deposited on the backside. The models have been demonstrated to be workable for pure GaAs and InP samples in the infrared regime with the refractive index given by the single-oscillator model. The scheme can be extended to any homogeneous dielectrics either pure or impure if a reasonable refractive index has been known in advance.

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