Excitation of surface phonon polariton modes in gold gratings with silicon carbide substrate and **their potential sensing applications**

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Abstract:

We demonstrate the excitation of surface phonon polaritons (SPhPs) in the mid-infrared (mid-IR) Reststrahlen band (10.288 μm -12.563 μm) on patterned surfaces with silicon carbide (SiC) substrate and gold (Au) gratings. The very large negative permittivity of Au limits its applications in the mid-IR range, to couple incident light to SPhPs modes, their momentum mismatch can be compensated by patterning Au grating onto the surface of SiC substrate. Samples were fabricated and characterized experimentally by the Fourier transform infrared reflection (FTIR). The optical properties were also simulated by the rigorous coupled wave analysis (RCWA) method. Reflection dips are observed for light polarized vertical to the grating lines (TM-polarized), which are attributed to the coupling of electromagnetic (EM) waves into the SPhP modes. In addition, we present small-volume index sensing with analyte specificity based on mid-IR SPhPs in the fabricated configuration.

Keywords: Surface phonon polaritons (SPhPs); Gold gratings; Mid-infrared (Mid-IR); Rigorous coupled wave analysis (RCWA)

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1. Introduction

Strong enhancement of the electromagnetic (EM) field, subwavelength scale confinement, sensitive to changes in the environment and optical non-linearity are among the remarkable effects related to surface plasmons (SPs) [1, 2]. These remarkable effects bring many advancements, such as subwavelength waveguiding and modulating, light trapping in solar cells, superlensing, near-field optical microscopy, tip and surface-enhanced Raman scattering, analysis for the label-free detection, as well as enhanced quantum efficiency for detectors in the ultraviolet (UV), visible (VIS) and near-infrared (NIR) spectral ranges [3-15]. Surface plasmon polaritons (SPPs), evanescently confined in the perpendicular direction, are EM excitations propagating at the interface between a dielectric and a conductor. These EM surface waves arise via the coupling of the EM fields to oscillations of the conductor's electron plasma. SPPs can be excited by illuminating the surface under the conditions that provide momentum-matching, called surface plasmon resonance (SPR) conditions, which is shown as a dip in the reflectance spectrum [3]. When the surface mode is excited, the EM energy comes to be strongly confined in the proximity of the surface, with the fields evanescently probing to the surrounding environment. Any change in the refractive index (RI) of outer medium has an effect on varying the propagation constant of SPPs, making it naturally to be exploited as probes for surface analysis and sensing devices. The metal-based plasmonics has been successfully demonstrated in the sensing application with high performance from UV

to NIR, however, **their very large negative permittivities at longer wavelengths limit their usefulness beyond the NIR** [16-18].

Fortunately, polar dielectrics offer an opportunity to simultaneously achieve sub-diffraction confinement, low optical losses and operation in the mid-IR to THz spectral ranges through the stimulation of SPhP mode, which is the coupling between the EM fields and optical phonons (lattice vibrations) of the polar crystal [19-25]. A high reflectivity and negative real part of permittivity are also observed for polar dielectric crystals within a spectral range referred to as the Reststrahlen band which gives rise to the SPhP phenomenon [26-30]. Within this spectral band, the reflection of incident light approaches **to 100%, but unlike that in metals**, this is realized without free carriers. SPhPs are studied less extensively than metal-based SPPs due to the fact that few materials can support SPhPs [31].

Due to the low intrinsic loss, SPhPs, especially on silicon carbide (SiC) based structures, examples of various applications including enhanced near-field light-matter interaction [32], high-sensitivity mid-infrared index sensing [23, 33, 34], mid-IR perfect absorption [35], extraordinary optical transmission and absorption in perforated membranes [36] have been reported. Similar to the case of SPPs, there exists a momentum mismatch between the incident photons and the SPhP modes, which can be **overcome** by using the coupling of the incident field to surface modes through the diffraction gratings [20, 24] and **high-index prism** [21, 37]. The existence of magnetic polariton within the phonon absorption band in **SiC-based** slit array and deep gratings has been revealed by Z. M. Zhang group [38], and the corresponding

applications as an IR filter and a coherent thermal emission source were considered.

In this paper, we theoretically describe and experimentally demonstrate the coupling of free-space radiation to SPhPs on Au gratings with SiC substrate. The TM-polarized infrared attenuated total reflection measurements are performed by using transform infrared (FTIR) spectroscopy. Based on the RCWA calculations, the mechanisms of the observed modes are revealed, which offers insights into the anomalous response of the variation of the geometrical parameters. Furthermore, a reflection-type sensor based on an angle scanning method was achieved. The proposed concept can be extended to different wavelength region by using other polar dielectrics that support SPhPs, e.g., AlN (11.2-16.4 μm), GaP (24.8-27.4 μm), GaAs (34.2-37.2 μm), CdTe (59.1-70.9 μm), and PbSe (49.2-256.5 μm) [24].

2. Structure and theoretical calculation

The majority of SPhP-based phenomena have been investigated on SiC surfaces because the high-energy phonons of SiC lie in the mid-IR and are thus accessible using standard IR sources and detectors [19, 21, 22]. In the absence of free carriers, the frequency-dependent dielectric permittivity of SiC is given by: [39, 40]

$$\varepsilon_{\text{SiC}}(\omega) = \varepsilon'_{\text{SiC}} + i\varepsilon''_{\text{SiC}} = \varepsilon_{\infty} \frac{\omega^2 - \omega_{\text{LO}}^2 + i\gamma\omega}{\omega^2 - \omega_{\text{TO}}^2 + i\gamma\omega} \quad (1)$$

where the longitudinal optical phonon frequency $\omega_{\text{LO}}=972 \text{ cm}^{-1}$ at $\lambda_{\text{LO}} = 10.288 \text{ }\mu\text{m}$, the transverse optical phonon frequency $\omega_{\text{TO}}=796 \text{ cm}^{-1}$ at $\lambda_{\text{TO}} = 12.563 \text{ }\mu\text{m}$, the damping rate due to vibrational anharmonicity $\gamma=3.75 \text{ cm}^{-1}$, and $\varepsilon_{\infty}=6.5$. The Reststrahlen band defined by $\varepsilon'_{\text{SiC}} < 0$ corresponds to $\omega_{\text{TO}} < \omega < \omega_{\text{LO}}$, within which SPhPs can be excited. In order to couple the incident light to SPhP modes, their

momentum mismatch can be bridged by patterning a grating onto the materials' interface [20, 24, 38].

Fig.1. (a) The schematic image of the Au grating patterned on the SiC substrate. The angle θ denotes the incident angle to the sample surface. (b) Real (blue) and imaginary (green) parts of the permittivity of SiC around its Reststrahlen band.

The structure used and coordinates system in the calculations are shown in Fig. 1 (a), where the x -direction is parallel to the surface and perpendicular to the ridges of the grating. The y -direction is assumed to be infinite along the grating grooves in this study, the z -direction is set along the surface normal, and θ represents the angle of incidence (from normal). The permittivity of SiC was modeled using the equation developed in Ref. 40 and is shown in Fig. 1(b). The simulation of optical phonons within the Reststrahlen band of SiC induces a negative permittivity which is similar to the plasmonic materials below their plasma frequency. Outside of this band, SiC behaves as a typical and transparent dielectric material mainly defined by its index of refraction.

Two-dimensional rigorous coupled-wave analysis (RCWA) algorithm [41-43], a frequency domain technique which utilizes the Floquet theorem to calculate the

amplitudes of a large number of spectral orders of diffraction is employed to study the spectral property of the **proposed structure with a plane wave incident on the grating**. Although no approximations to Maxwell's equations are made in the RCWA method, the number of spatial harmonics retained in calculations must be truncated in order to obtain a solution. For 2D grating problems, the number of equations to be solved increases with the square of the number of orders retained, and the tradeoff between speed and accuracy becomes much severer. **To ensure the numerical accuracy of the calculation with reasonable computational time, all the calculations have been tested for convergence and calculated with the number of diffracted orders equal to 203.**

The frequency-dependent dielectric constant of Au is expressed by Lorentz-Drude model [44]:

$$\varepsilon_{\text{Au}}(\omega) = \varepsilon_{\infty} - \frac{\omega_p^2}{\omega^2 + i\Gamma_p\omega} - \frac{f_1\omega_1^2}{\omega^2 - \omega_1^2 + i\Gamma_1\omega} \quad (2)$$

where ω_p and ω are the frequencies of the plasma and interest respectively, Γ_p is the damping (or relaxation) rate, i denotes the complex number ($i = \sqrt{-1}$), and the contribution from interband transitions at infinite frequency is ε_{∞} . The Lorentz term includes the Lorentz oscillator damping rate Γ_1 , the Lorentz resonance width ω_1 and the weighting factor f_1 . The values of different parameters for Au are given in Table 1.

Table 1. Values of Lorentz-Drude parameters for Au

Parameters	Values
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ϵ_{∞}	6.8890
$\omega_p(\text{ev})$	8.9601
$\omega_1(\text{ev})$	2.9715
$\Gamma_p(\text{ev})$	0.0723
$\Gamma_1(\text{ev})$	0.9503
f_1	1.7857

Reflectance of the designed sample as a function of grating period and incident wavelength is shown in Fig. 2(a), where only the zero order mode is propagating and the other modes are evanescent. The parameters are chosen as $f=0.22$, $d=0.28 \mu\text{m}$, and $\theta=45^\circ$. It is understood that the dark area is caused by propagating SPhP which is generated by different diffracted orders from the grating. Or in other words, the k -vector of a diffracted order for normal incidence equals to an integer multiple of the grating vector. As a SPhP wave propagates at the interface, the conservation of wave vector is provided by diffraction on a periodic structure [3]:

Fig. 2. Reflectance as functions of wavelength and grating period Λ (a), grating thickness d (c) and incident angle θ (d). (b) Zero order reflectance versus Λ and wavelength with normal incidence, the other parameters are the same as used in Fig. 2 (a). **The color scale shows reflectivity from 0 to 1.**

$$\mathbf{k}_{\text{SPhP}} = \mathbf{k}_0 \sin \theta \pm i \left(\frac{2\pi}{\Lambda_x} \right) \mathbf{u}_x \pm i \left(\frac{2\pi}{\Lambda_y} \right) \mathbf{u}_y \quad (3)$$

where $\mathbf{k}_{\text{SPhP}}$ indicates the SPhP wavevector, $\mathbf{k}_0$ denotes the wavevector of light, θ is the angle of incidence, Λ_x , Λ_y are the periodicity of the structured film in the x - and y -directions respectively, $\mathbf{u}_x$ and $\mathbf{u}_y$ are the reciprocal lattice vectors, while i and j are integer numbers corresponding to different directions of SPhP. In addition, the magnitude of SPhP wave vector is given by

$$|\mathbf{k}_{\text{SPhP}}| = \frac{2\pi}{\lambda_0} \sqrt{\frac{\epsilon_{\text{SiC}(\lambda_0)} \epsilon_d}{\epsilon_{\text{SiC}(\lambda_0)} + \epsilon_d}} \quad (4)$$

where λ_0 is the wavelength of incident light, $\epsilon_{\text{SiC}(\lambda_0)}$ is described as the complex permittivity of SiC, and ϵ_d means the permittivity of the adjacent medium. **For**

comparison, the reflectivity of the grating structure with the same parameters used in Fig. 2(a) is calculated and shown in Fig. 2(b). Obviously, within the Reststrahlen band, the real part of ϵ_{SiC} is negative, leading to a dramatic increase in reflectivity for TE-polarization.

SPhPs can be generated by the diffracted orders of light scattered from a metallic grating [45]. This scattered radiation has a larger momentum than that of the incident radiation by an integer number of reciprocal lattice vectors. To better understand the SPhP phenomenon, we plot the reflectivity contour map versus wavelength and grating thickness with the electric field parallel to the grating vector, as depicted in Fig. 2(c). In practical calculation, the adopted parameters are same as those used in Fig. 2(a) except for the thickness of grating (d). A fascinatingly well-behaved independence on grating thickness has been revealed. Usually, the spectral response of grating-based devices is highly dependent on the structural parameters [46]. It is difficult to fabricate an ideal structure with the same parameters as the designed one. During the process of grating fabrication, the most difficult thing is to control the thickness. Thus, we evaluate the response of the structure under variation in the grating thickness. Keeping other parameters as constants, it is found that the resonant wavelength almost does not change for $d < 0.5 \mu\text{m}$. Therefore, this type of grating is much easier to achieve compared with previous designs. Figure 4(d) shows that the SPhP only exists for large incident angles. The grating structure was assigned the following parameters: $f=0.22$, $d=0.28 \mu\text{m}$, and $\Lambda=7 \mu\text{m}$. Under the situation with small θ , the evanescent wave emerging from the grating is very weak

when it arrives at the SiC, so there is little observable SPhP excitation.

3. Experiments

The preparation of substrates must be extremely careful. After cleaning in a mixed solution containing hydrochloric acid, hydrofluoric acid, and deionized water with the a mixing ration (volume ratio) of 1:1:4, the substrate was dried by N₂ and baked in quartz tube at 150°C for 1h. Chrome (Cr) thin films with a thickness of 10 nm was deposited and used as interlayer between SiC wafer and gold film to promote adhesion and decrease the internal stresses. Gold film with a thickness of 280 nm was also deposited using electron beam evaporation. The system was pumped to a base pressure of 4×10^{-6} mbar at 20°C. The films were deposited by using electron beam evaporation under the oxygen partial pressure of 5×10^{-4} mbar at 0.1 nm/s. Then the silicon wafers were patterned by electron beam lithography on AZ5214 photoresist (with a thickness of about 1.5 μ m) with the inverse pattern of the desired grating. The sample was baked at 85°C for 30 mins. The plasma of a CF₄/Ar gas mixture was applied to the grating-patterned surface for 12-18 min at room temperature to leave the grating nanolines through removal of the metallic coating. The plasma power, chamber pressure, and the gas flow rates were adjusted to 100 W, 10 Pa, and 30 sccm and 5 sccm, respectively. O₂ plasma was then applied to the top surface to expose the head portion of the Au grating. The plasma power, the gas flow rate and etching time were tuned to 150 W, 30 sccm and 5.5 mins, respectively. Figure 3 presents a micrograph by scanning electron microscopy (SEM) for the fabricated gold grating.

Fig. 3. A SEM micrograph for the fabricated gold grating on a SiC substrate.

Reflection spectra were measured with a Bruker V80v Fourier transform infrared (FTIR) vacuum spectrometer by using an internal room-temperature pyroelectric deuterated triglycine sulfate far-infrared detector [24]. The experiment uses a PIKE Technologies 10Spec specular reflection accessory, giving reflection spectra at an angle of incidence of 10° within the vacuum sample compartment, thus minimizing effects of atmospheric absorption. The KRS-5 far-IR beam-splitter was used to select the polarization of the incident light. SPhPs can only be excited by incident light with a component of the electric field perpendicular to the grating ridge. All reflection data were normalized to the reflection through a gold mirror.

Fig.4. Modeled (red, calculated from the permittivity in Eq.1) and experimental (blue diamond) reflection from a flat SiC surface.

Using the permittivity described in Eq.1, the reflectivity from a flat SiC surface is calculated for TE and TM- polarized light at a zero angle of incidence. The calculated and experimental reflection spectra (TM-polarized) are shown in Fig. 4 and they agree well with each other. It is noted that the experimental reflection somewhat smaller than the predicted reflection in short wavelength region of the “Reststrahlen” band, perhaps due to either (i) imperfect reflection from the Au mirror used for normalization or alternatively (ii) the difficulty in fitting the highly dispersive portion of the SiC permittivity using Eq.1.

A TM-polarized plane wave was launched within the xz -plane at an incident angle of 45° in the simulation to approximate the experimental conditions. A typical pair of reflection spectra from the experiment and RCWA calculation are shown in Fig.5 with diameters of $\Lambda = 7.149 \mu\text{m}$, $h = 0.28 \mu\text{m}$ and $f = 0.296$. The measured reflection spectrum displays double pronounced minima, which do not appear in the transmission spectrum for the case of the plane structures. These dips in the reflection are due to

the excited phonon resonances. The differences between the experimental and simulated spectra can be ascribed to the additional induced surface damage caused by the reactive-ion etching process during the experiment. Such a discrepancy could be an indication of the difficulty in accurately modeling the SiC permittivity closer to the phonon resonance [19]. In addition, this discrepancy could also result in a greater sensitivity of our response, in this frequency region, even to slight changes in beam collimation, incidence angle, or sample position [19, 24]. Although the intrinsic optical losses associated with the fast scattering lifetimes pose an inevitable limitation on the performance of metals in the mid-IR wavelength range, the occurrence of the negative permittivity within the underlying SiC substrate results in the observation of dips.

Fig. 5. Experimental and modeled reflection spectra from patterned Au grating on SiC substrate.

In addition, to experimentally demonstrate SPhPs, the FTIR measurements were used to detect the resonant angles (θ_{res}). All scans of wavenumber display minimal R and maximal SPhP excitation, which are shown in Fig. 6. As can be seen, θ_{res} shifts

toward a smaller angle as the wavenumber increases. Another important quantity to describe the quality of the SPhP is the Q factor (ω_0/FWHM , full-width at half maximum) of its resonances [47]. The Q factor of SPhP resonance is larger than that of the dipole-like slot antenna resonance ($Q \sim 10\text{-}20$), Mie scattering ($Q \sim 10\text{-}30$) of the SiC whiskers [28]. We also notice that the Q factor of our SPhP resonance is higher compared to other theoretically proposed localized SPhP resonances ($Q \sim 50\text{-}60$) of the SiC nanodisks [48] and in the same order as the localized SPhP resonance ($Q \sim 40\text{-}135$) of the SiC pillar arrays [49].

Fig. 6. Experimental plots of resonant angle and Q factor versus wavenumber.

4. Potential sensing applications

Molecules exhibit unique spectroscopic signatures in the mid-IR fingerprint region that are manifested with larger index (n) differences compared to materials operating in the visible range [23]. The requirement of small-volume sensitivity explains the widespread use of SPPs. SPhPs hold great potential for small-volume mid-IR sensing because of the high sensitivity to analyte permittivity ($\epsilon_a = n_a^2$).

The change in the resonance curve caused by a change in the RI Δn can be characterized either by an angular shift of the curve $\Delta\theta_{\text{res}}$ (sensing by angular modulation) or a change in the reflectance ΔR at a fixed angle (sensing by intensity modulation). According to previous papers on the performance of SPR sensors [50, 51], sensitivity by intensity is expressed by:

$$S_I(\theta) = \lim_{\Delta n \rightarrow 0} \frac{\Delta R(\theta)}{\Delta n} = \frac{\partial R(\theta)}{\partial n} \quad (5)$$

Fig. 7. Theoretical reflectivity with 2 μm of analytes 1, 2 contained on the top of the structure.

We theoretically investigate a reflection-type sensor based on an angle scanning method. The sensing element consists of Au grating embedded in a SiC substrate in which excitation of SPhPs was achieved by illumination with TM-polarized light. We characterized the sensor response by the RI changes in the environment. Using a wavelength of $\lambda=12 \mu\text{m}$, we calculated the reflectivities for analytes 1 ($n=1.33$) and 2 ($n=1.334$), which are shown in Fig. 7. Here, $\Delta n=0.004$ RIU, but R increases by 34.45%. The ratio of $\Delta R/\Delta n$ is 86.125 RIU^{-1} for the SPhR sensor. The value suggests at least qualitatively that the present sensor offers a high sensitivity by intensity.

Similar to SPPs, SPhPs can only be excited through evanescent field coupling under TM-polarization. In order to ensure evanescent field coupling, the obvious choices can be employed either Kretschmann configuration or Otto configuration or a grating [20, 21, 24, 35, 40]. The Kretschmann configuration requires thin film deposition on a high index prism. The quality of resonance response is quite poor and the property of the phononic material is dependent on the deposition conditions, such as temperature and pressure. For the Otto configuration, the method does not require thin film deposition, however, the results published so far do not show as good a phononic response as expected. Another method is to employ a grating to increase the k -vector of the incident photons, which can be achieved by electron beam lithography patterning directly onto the phononic material surface as the authors did in this work.

5. Conclusion

We have experimentally demonstrated excitation of SPhPs in the mid-IR range on patterned structures with SiC substrate and Au gratings. The presence of the negative permittivity substrate (SiC) enables the stimulation of SPhPs. The SPhP mode supported in the fabricated structures was studied through both the FTIR spectroscopy measurements and numerical simulations. The results show the spectral dependence of the SPhP mode on the geometry. The TM-polarized spectrum shows two prominent reflection dips at 820 and 948 cm^{-1} , the experimental observations are in good agreement with the RCWA predicted results. Finally, small-volume index sensing with analyte specificity based on mid-IR SPhPs in the fabricated configuration is also presented. It is important to note that this method is not limited to SiC-based SPhPs,

but also applicable to other types of polar dielectrics.

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