Localized surface plasmon resonance frequency tuning in highly doped InAsSb/GaSb one-dimensional nanostructures

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Abstract
We report a detailed analysis of the influence of the doping level and nanoribbon width on the localized surface plasmon resonance (LSPR) by means of reflectance measurements. The plasmonic system, based on one-dimensional periodic gratings of highly Si-doped InAsSb/GaSb semiconductor nanostructures, is fabricated by a simple, accurate and large-area technique fabrication. Increasing the doping level blueshifts the resonance peak while increasing the ribbon width results in a redshift, as confirmed by numerical simulations. This provides an efficient means of fine-tuning the LSPR properties to a target purpose of between 8–20 μm (1250–500 cm⁻¹). Finally, we show surface plasmon resonance sensing to absorbing polymer layers. We address values of the quality factor, sensitivity and figure of merit of 16 700 nm RIU⁻¹ and 2.5, respectively. These results demonstrate Si-doped InAsSb/GaSb to be a low-loss/high sensitive material making it very promising for the development of biosensing devices in the mid-infrared.

Keywords: localized surface plamons, nanoribbons, highly doped semiconductors, mid-infrared, sensing

(Some figures may appear in colour only in the online journal)

1. Introduction
The interaction between polarized-incident light and free electrons in materials gives rise to a subwavelength oscillating mode referred to as plasmons. By combining the properties of such electronic oscillation and its nano-scale integration many nanophotonic devices for different applications may be designed. Over the last few years, surface plasmon polaritons (SPP) in extended surface, and localized surface plasmons (LSP) in single or low periodicity grating resonators [1–3], have indeed attracted extensive studies for plasmonic applications, such as nanophotonic integrated circuits [4], nano-bio-photonics [5] and biosensing [6] among others. The most appropriate candidates for plasmonic material are those presenting a large density of free electrons and thus providing a negative real permittivity [7]. This is why noble metals are the most commonly used. In particular, Ag and Au exhibiting a low damping coefficient (Γ, Γ = 1/τ, being τ the relaxation time) compared to Cu or Al, are generally chosen for visible and near-infrared (near-IR) spectral range applications [8–10]. Nonetheless, they show limitations at longer wavelengths for mid-infrared (mid-IR) applications where many molecules present their main vibrational absorption resonances. Despite the high density of free electrons, and therefore the high electron mobility, their small band-splitting causes these materials to exhibit a high density of losses arising mainly from interband transitions. In turn, these losses are detrimental for the performance of plasmonic devices [11], and low-loss materials are therefore required.

Recently, highly doped semiconductors (HDSC) [12] have emerged as an alternative for low-loss plasmonic materials in mid-IR allowing for the tuning of the plasma
constant pitch
controlled by the etching time.

...doped and easily fabricated using epitaxial growth techniques... 

...molecular sensing applications since they can be heavily... 

...been demonstrated to be promising plasmonic materials for... 

...doped group-IV nanostructures, including doped-Si gratings... 

...enhancing resonances... 

...enabling band-gap engineering for electrically activated LSP... 

...avoiding interband losses and band offset with GaSb of 1 eV... 

...applications. We have developed a simple low-cost technique... 

...periodic nanostructures of highly Si-doped InAs$_{0.91}$Sb$_{0.09}$... 

...InAs$_{0.91}$Sb$_{0.09}$ was grown at 450°C. Then, a 100 nm-thick layer of... 

...the plasma frequency, was determined by an optical technique... 

...photolithography and chemical wet etching for tailoring... 

...which allows large scale surface processing by means of... 

2. Experimental and theoretical methods

...The epitaxial structures were grown by molecular beam epitaxy (MBE) on Te-doped (100)-GaSb substrates. After oxide desorption, a 500 nm thick GaSb buffer layer was grown at 500°C. Then, a 100 nm-thick layer of Si-doped InAs$_{0.91}$Sb$_{0.09}$ was grown at 450°C. Four samples with different doping levels of 1.5 x 10$^{19}$ cm$^{-3}$, 2.2 x 10$^{19}$ cm$^{-3}$, 4.7 x 10$^{19}$ cm$^{-3}$ and 5.5 x 10$^{19}$ cm$^{-3}$ labeled as A, B, C and D respectively, were selected to evaluate the role of the doping level on tuning the LSPR. The doping level, and thus the plasma frequency, was determined by an optical technique based on the Brewster mode [25].

...Structural and optical characterizations of the nanoribbons were performed by means of scanning electron microscopy (SEM), atomic force microscopy (AFM) and reflection spectroscopy experiments. Plan- and cross-sectional view images were obtained using a SEM-Inspec S-50 with a typical electron-beam energy of 20 kV. AFM measurements were done in tapping mode with standard Si tips in 2.5 μm x 10 μm surface at 1 μm s$^{-1}$ scanning speed. Reflectance measurements were performed under quasi-normal incidence with a Bruker Vertex 70 V Fourier transform infrared spectrometer, equipped with a Hyperion 3000 microscope, a reflector objective with an N.A. of 0.4 in a reduced area of 100 μm x 100 μm and in the 450–5000 cm$^{-1}$ spectral range. A KBr beam splitter, the internal mid-IR optical source and a MCT detector (detectivity, $D > 5 \times 10^9$ cm Hz$^{1/2}$ W$^{-1}$) with BaF$_2$ windows, were used. A gold mirror sample was used as the reference. The microscope was covered with a Plexiglas housing and purged with nitrogen. An automatic stage controls the movement and the accurate setting of the measurement location. The measurement protocol consisted of an average of 4 spectra taken in the same spot with 8 cm$^{-1}$ resolution and 1000 scans with the same background reference.

...To obtain LSPR in the mid-IR, the highly doped InAsSb layer is engineered into low periodicity nanoribbon gratings. Figure 1 schematizes the process flow of the grating fabrication. The sample surfaces were first cleaned with organic...
The gratings with a complete surface of 5 \( \times \) 5 mm\(^2\), a constant periodicity (pitch, \( \Lambda = 2 \mu m \)) and ribbon width (\( w = 1 \mu m \)) were patterned by photolithography using a MJ4-SÜSS mask aligner. The exposed resist was cured at 110 °C for 1 min and developed for 40 s. Afterwards, wet etching was conducted with a citric acid (C\(_6\)H\(_8\)O\(_7\)) and hydrogen peroxide (H\(_2\)O\(_2\)) solution with a 2:1 ratio at 20 °C to define the grating, as detailed below. The etching time was controlled to obtain different ribbons widths. The resulting nanoribbons present high verticality of the side walls and great structural quality as can be observed in the cross-sectional SEM images of a grating with 500 nm and 150 nm ribbon widths shown in figures 1(e) and (f) respectively.

Numerical simulations were carried out by solving the Maxwell equations by finite-difference time-domain (FDTD) method (Lumerical FDTD Solutions 8.12.631 from Lumerical SolutionsInc.) using the model reported in [24]. The nanoribbons were assumed as infinitely extended with an InAsSb thickness of 100 nm and variable width using boundary conditions where the 2 \( \mu m \)-pitch was included. The dielectric permittivity (\( \varepsilon(\omega) \)) of the semiconductor layer was modeled using a Drude function, see equation (1)

\[
\varepsilon(\omega) = \varepsilon_{\infty} \left(1 - \frac{\omega_p^2}{\omega(\omega + i\gamma)}\right),
\]

where, \( \varepsilon_{\infty}, \gamma \) and \( \omega_p \) correspond to the dynamic dielectric permittivity, the scattering rate and the plasma frequency defined as \( \omega_p = \sqrt{\frac{N e^2}{m_{\text{eff}} \varepsilon_{\infty} \varepsilon_0}} \), with \( m_{\text{eff}} \) the effective mass and \( N \) the doping level. The parameters used for the simulation were a scattering rate \( \gamma \), \( 1 \times 10^{13} \text{rad s}^{-1} \) and the plasma frequency directly obtained from the Brewster mode measurements [25]. The GaSb substrate was described by \( n_{\text{GaSb}} = 3.77 \) [26], and the dielectric permittivity, \( \varepsilon_{\infty} \), of the nanoribbons was used as a free parameter to fit the experimental results.

### 3. Results and discussion

The nanoribbon engineering with such small lateral sizes (\( w < 150 \mu m \)) was achieved thanks to the development of a simple manufacturing technique which allows large area processing and highly accurate structural definition. It consists of a key combination of micrometric UV patterning and chemical-wet etching. After UV lithography and resist development, 1 \( \mu m \) wide windows are opened to etch the InAsSb layer. The high etching selectivity of about 100 between GaSb and InAsSb [27] allows the accurate control on the definition of this nanoribbon geometry. The vertical etching of GaSb and InAsSb are well characterized [27, 28]. After 70 s etching at 20 °C with a gentle stirring movement to favor homogeneous etching (see schematic in inset figure 2),

![Figure 2. 3D-AFM images (2.5 \( \mu m \) \( \times \) 10 \( \mu m \)) corresponding to different etching times (a) 70 s (b) 120 s and (c) 180 s. (d) Evolution of the ribbon width with etching time. The dot-dash line corresponds to the linear fit. Bottom left inset: schematic of the wet etching procedure at 20 °C with stirring movement.](Image)

the whole 100 nm-thick InAsSb layer is etched away, regardless of its doping level. This results in 100 nm high nanoribbons with \( w = 1 \mu m \) and \( \Lambda = 2 \mu m \). Any additional etching results in the lateral etching of the ribbon and this can
therefore be used to control the widths of the structures to the nanoscale range until ~100 nm, as shown in figure 2.

Figures 2(a)–(c) show the AFM images for three different etching times of 70 s, 120 s and 180 s, corresponding to ~1000, 700 and 350 nm measured nanoribbon widths, respectively. After etching, the nanoribbons present a good surface quality and well-defined walls. The final top-down surface and lateral roughness are on average: (0.71 ± 0.04) nm, (comparable to the roughness of as-grown GaSb, <0.5 nm) and (5 ± 2) nm, respectively. This lateral roughness represents a deviation of less than 1% with respect to the ribbon width. These values correspond to the mean value and statistical deviation measured after etching the four different samples. Figure 2(d) summarizes these results, where a linear relationship between ribbon width and etching time is obtained. Furthermore, the lateral etching rate (150 ± 18 nm min⁻¹) resulted in being faster than the vertical etching (89 ± 3 nm min⁻¹). This significant difference between in-plane and transversal etching rates may be attributed to different factors such as the fact that vertical etching first involves removing the native oxide layer and then opening trenches, and the variation of the etching rates due to the different crystalline directions [28]. The linear relationship between the nanoribbon width and chemical wet-etching seems to be independent of the doping level and offers an efficient means of controlling the nanoribbon width.

Figure 3 shows the dependence of the LSPR properties on the doping level and the ribbon width of the plasmonic resonators. The normalized reflectance spectra for the different doping concentrations and the same grating geometry (Λ = 2 µm, t = 100 nm and w = 500 ± 20 nm) is illustrated in figure 3(a). Increasing the Si-concentration in the InAsSb layer increases the LSPR wavenumber, as expected, which shows that for a given ribbon width, the LSPR wavenumber can be tuned by changing the doping level of the sample. Similar results have already been shown in Si NWs and InAs nanoplats [19, 29]. Slight changes in the doping level (from 4.7 × 10⁻¹⁹ to 5.5 × 10⁻¹⁹, sample C and D respectively) already provoke a significant peak position shift of the LSPR equivalent to ~100 cm⁻¹. Furthermore, increasing the doping level threefold, from sample A to sample D, induces a resonance shift of about 390 cm⁻¹. Additionally, the increase of the Si-concentration broadens the spectra, as can also be seen in figure 3(a). This broadening is closely related to the losses when comparing plasmonic properties of different samples, which will be discussed later.

Figure 3(b) illustrates the influence of the nanoribbon widths on the LSPR properties for a given doping level. Correspondingly, figures 3(c)–(e) and (f)–(h) show plan view and cross section SEM images of the three samples (D1, D2, D3), with Nd = 5.5 × 10⁻¹⁹ cm⁻³ and nanoribbons widths of ~670, 325, 105 nm, respectively. The nanoribbon-width control for different gratings allows the fine-tuning the LSPR frequency for a given doping level. Figure 3(b) illustrates the reflectance spectra difference under p- and s-IR light polarization. Under s-polarization, an almost constant reflectance signal is measured in the whole wavenumber range, since LSPR are not excited by this electric field orientation (parallel to the ribbons). In contrast, under p-polarization, we observe a well-defined LSPR reflectance peak for each sample and the maximum reflectance position and the reflection intensity strongly depend on the nanoribbon size. When decreasing the ribbon width, the LSPR wavenumber is blueshifted and the reflectance intensity decreases as theoretically proposed [24]. The attenuation of the reflection signal is attributed to the decrease of the total surface of reflection, therefore, decreasing the nanoribbon size results in weaker reflection spectra. The resonance wavenumber shift is ascribed to the delay retardation of the depolarization field inside the nanoribbon, similar to the one in nanoparticles [30]. The impact of the size of nanoparticles on plasmon properties has already been reported for other materials [13, 24, 31–34]. For such understanding, it is necessary to compare the wavenumber of the incoming wave with the dimensions of the nanostructures. For small enough particles, w < λ or w ≪ 100 nm in an electrostatic electric field, the quasi-static approximation can be used. However, for larger nanoparticles, typically w > 100 nm, additional terms must be taken into account. In particular, the retardation effect has been demonstrated to cause a wavenumber shift of the resonance peak [30, 35, 36].

Taking into account a Drude-model for the InAsSb layer and using the model proposed by Barho et al [24], we have performed FDTD numerical simulations to obtain the electrical field profile of LSPR and the reflectance of the nanoribbons and compared them to the experimental data. Figure 4(a) shows the wavenumber dependence of the LSPR reflectance peak maximum (νR) on the ribbon width for each doping level (A, B, C and D). As a first approximation, the nanostructured system has been modelled using the parameters of InAs since the percentage of Sb in the alloy is low. The experimental data is redshifted by about ~5% with respect to the simulation, in that case (not shown here). Generally, this shift is attributed to the inexact values of the permittivity of such HDSC epilayers [37]. In fact, our experiments fit perfectly (figure 4(a)) by assuming a value of ε∞ = 10.4 for the Si doped-InAsSb alloy, which is slightly different from the established values for InAs (12.3) and InSb (15.7) [38]. This points to limits in the Drude approach to model complex systems. Indeed, the physical parameters of these compound semiconductors strongly depend on the dopant concentration and alloy composition making an approximation of their properties necessary to be able to closely model the experimental results.

The quality factor (Q), determined as the ratio between the maximum resonance peak wavenumber and the full width at half maximum (FWHM) expressed in cm⁻¹ (see equation (2)), is also an indicator of the losses and sensitivity of a plasmonic system [7]

$$Q = \frac{\nu_R}{\text{FWHM}}.$$  (2) 

A large Q indicates a low-loss material and high local field strength, and thus, benefits for sensitive sensor fabrication in a single energy, i.e., wavelength on a certain range. Figure 4(b) illustrates the evolution of the Q as a function of the different doping levels (solid arrow) and ribbon width (dash arrow). In
this mid-IR energy range, the values for FWHM and \( Q \) are between 60–170 cm\(^{-1}\) and 3–16, respectively. Additionally, a general tendency for both magnitudes has been found: increasing the doping level, and consequently the LSPR wavenumber, results in a reduction of the FWHM when decreasing the ribbon width and therefore, in an enhancement of \( Q \) (solid guideline in figure 4(b)). Such tendency has already been reported in the visible energy range, for Au-spheres but with lower \( Q \) value (2–8), and for Au-nanorods with similar ones (8–21) [39], comparing to InAsSb nanoribbons. On the other hand, decreasing the ribbon width results in a narrower FWHM and a higher \( Q \) (follow dash guideline in figure 4(b)). For a given ribbon width, increasing the doping level results in a spectral broadening and a nearly constant \( Q \).

These results demonstrate the possibility of tuning the LSPR wavenumber, by either changing the doping level or designing the appropriate ribbon width. Furthermore, they also point out that the best approach to obtain a plasmonic system with the lowest losses is to tailor the nanoribbon widths as narrow as possible and then to adjust the doping level to tune the LSPR to the required wavenumber for a certain application. Besides, FDTD simulations were conducted without changing the damping coefficient, \( \Gamma \), and consequently, considering the same losses in the material. The calculations reveal an excellent agreement with the experiment.

The relevance of plasmonic systems to sensing applications is usually discussed in terms of sensitivity (5) and figure of merit (FOM). The sensitivity is defined as the ratio between the maximum peak resonance shift and the refractive index variation and it is expressed in units of nm RIU\(^{-1}\) [2]. The FOM is calculated as the ratio between the sensitivity and the FWHM of the LSPR reflection spectra [40], see equations (3) and (4) respectively

\[
S = \frac{\Delta \lambda}{\Delta n},
\]

\[
\text{FOM} = \frac{S}{\text{FWHM}}.
\]

To demonstrate the environmental sensing of highly doped InAsSb nanoribbons in the mid-IR, samples D1, D2 and D3 were coated with polymethylmethacrylate (PMMA-A4). Figure 5(a) presents the SEM cross section image of a 325 nm nanoribbon width (sample D2) coated with PMMA. The PMMA nominal thickness is 200 nm, with a refractive index of 1.49 in the mid-IR [41]. The solution was spun at 2000 rpm for 30 s and heated at 120 °C for 1 min. The resulting PMMA thickness on the nanoribbons is to the order

\[
L = \frac{D \times D}{S_{\text{LSPR}}}, 3
\]
of \( \sim 170 \) nm, while the thickness on the substrate corresponds to the nominal value, as measured from the SEM images. Figure 5(b) shows the reflectance spectra without (lines) and with (symbol lines) PMMA coating in the whole region. In presence of PMMA, the resonance peak maximum is red-shifted by \( \sim 18 \) cm\(^{-1}\), \( \sim 24 \) cm\(^{-1}\) and \( \sim 19 \) cm\(^{-1}\) for samples D1, D2 and D3, respectively, while their reflection intensity is maintained basically unchanged. The redshift is attributed to the change in the refractive index of the environment and the invariant reflection intensity is ascribed to the constant scattering cross-section since the area of the nanoribbons is not modified by the deposition of PMMA. In the proximity of the nanoribbon resonances, two absorption lines of the PMMA are found at 1150 and 1195 cm\(^{-1}\), shown clearly in figure 5(c). The latter shows the presence of the PMMA layer on the samples directly in the optical measurement.

Considering the definitions of \( S \) and FOM shown in equations (3) and (4), and counting the field enhancement confinement is larger in the interface between substrate and resonator [22], and hence assuming the sensing at such hot spots, the samples D1, D2 and D3 exhibit sensitivities and FOMs of 694 nm RIU\(^{-1}\) and 1.3, 550 nm RIU\(^{-1}\) and 2.5, and 245 nm RIU\(^{-1}\) and 2.1, respectively. It has been noted that the sensitivity decreases when decreasing the scattering area of the nanoribbon which corroborates the blueshift found due to smaller nanoribbon-widths. These large sensitivity values outperform those reported for other materials in the visible range such as WO\(_2\) 2.83 (280 nm RIU\(^{-1}\)), Ag nanoprisms (200 nm RIU\(^{-1}\)), Au nanoshells and Cu\(_2\)S\(_x\) (350 nm RIU\(^{-1}\)) [32]. In mid-IR, we have extracted a value of sensitivity of 1510 nm RIU\(^{-1}\) for InAs nanopillars [19]. This value is in the same order of magnitude that the one reported for InAsSb nanoribbons, assuming both resonators effective surface density. The InAs nanopillars geometry has an effective surface approximately 2.5 larger, consequently a higher sensitivity, and making both sensing systems equivalent.

4. Conclusions

To conclude, we have defined a simple yet controlled procedure for high quality InAsSb nanoribbon fabrication for plasmonic applications, by means of chemical-wet etching and UV photolithography. The grating nanoribbon widths are

![Figure 4](image-url)  
**Figure 4.** (a) Wavenumber of the LSPR peak maximum (scattered symbols) for the different doping concentrations (A)-(D) and FDTD simulation (solid lines) with \( \varepsilon_{\infty} \) adjusted to 10.4. Evolution of the \( Q \) as a function of the resonance wavenumber for different doping levels (solid guideline) and ribbon widths (dash guideline).

![Figure 5](image-url)  
**Figure 5.** (a) SEM cross-sectional image of a \( \sim 200 \) nm PMMA-coated nanoribbon with \( w = 325 \) nm (sample D2). Reflectance spectra of (b) samples D1, D2 and D3 coated with and without a layer of PMMA. (c) Close-up of the region where PMMA presents its high absorption lines (1151 and 1192 cm\(^{-1}\)) due to ester group vibrations.
reduced to ~100 nm with a lateral error of 1%, showing high homogeneity and reproducibility in large areas. The doping level and nanoribbon width highly influences the LSPR frequency giving rise to two different approaches to fine-tune the resonance. For fixed grating geometry, increasing the doping level is found to blueshift the LSPR reflection peak. Moreover, by setting the doping level, a reduction of the ribbon width blueshifts the LSPR peak and enables the fine adjustment of the resonance. After coating the grating with PMMA, the system reacts to the new environmental conditions: the LSPR resonance redshifts while the reflectance intensity is maintained. A quality factor of 16 comparable to those reported for Au in the visible spectrum, a sensitivity of ~700 nm RIU−1 and a FOM of 2.5 are achieved with the highly doped engineered InAsSb nanostructures, demonstrating well-suited plasmonic properties: low-losses and reliability. These results demonstrate that InAsSb nanoribbon gratings are promising candidates for the development of mid-IR biosensing plasmonic applications.

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