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Borgh, Giovanni; Bongiorno, Corrado; La Magna, Antonino; Mannino, Giovanni; Patanè, Salvatore; Adam, Jost; Puglisi, Rosaria Anna

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Surface Plasmons in Silicon Nanowires

Giovanni Borgh, Corrado Bongiorno, Antonino La Magna, Giovanni Mannino, Salvatore Patanè, Jost Adam, and Rosaria Anna Puglisi*

Plasmon resonances (PRs) in metallic nanostructures have been extensively studied, whereas reports on PR in silicon nanowires (Si NWs) are very few, partial, and they refer to structures larger than 100 nm. Discrete resonances in Si NWs with core sizes as small as 30 nm at high resolution are observed. They are attributed to plasmonic resonances identifying two groups, the traveling waves, exhibiting discrete modes along the NW length for several orders of harmonics, and the localized waves, generated by transverse oscillations along the NW diameter, observing them to the best of our knowledge for the first time in silicon NWs (SiNWs) of every size. The experimental findings are coupled to modeling, confirming the data and adding further insights into the Si NW’s embedding medium role. A plasmon-induced resonant cavity in Si NWs opens markets in material processing, photodetectors, and novel plasmon-based nano-optics, thanks to the intense optical energy delivery below the diffraction limit and the addition of the exceptional integration capacity of silicon.

1. Introduction

Semiconductor nanowires (NWs) and their exceptional properties are well known.[1–5] In the past years, scientists have devoted significant efforts to study silicon NWs (SiNWs), a pivotal material to develop the next generation of low-dimensional devices for nanoelectronics and nanophotonics applications. Their unique geometry and optical properties render them ideal scaffolds to develop performant devices in the fields of lasers, sensors, solar cells, photonic devices, optical processors, and other novel plasmonic devices.[6–11] The NWs’ plasmonic properties have attracted particular attention with the recent development of multicolor plasmonic lasers showing huge threshold reduction and ultrafast modulation.[12,13] In this frame, SiNWs play a strategic key role due to Si’s well-known broad industrial compatibility. In literature, while metallic nanostructures have been the subject of extensive work,[14–22] reports on plasmon resonances (PRs) in SiNWs are very few. Understanding low-energy plasmonic resonances in semiconductor nanostructures is, in fact, not trivial. Factors intrinsic to the phenomenon and related to the challenging experimental investigation hinder their study and their understanding.

Several conditions have to be fulfilled, from the choice of the proper material properties to appropriate characterization techniques and simulation tools. As electron energy loss spectroscopy (EELS) allows to trigger plasmons by electron impact, it enables experimental spectral profile characterization of a complete set of plasmon modes. Previous studies, conducted by EELS on SiNWs with diameter larger than 100 nm, demonstrate that they support PRs at energies higher than 11 eV.[23–26] Peaks revealed at ≈4–5 eV have been previously attributed to interband transitions,[23,24] whereas in a later paper to plasmon polarization without discerning between longitudinal or transversal oscillations.[27] There is, however, no direct observation, nor deep understanding in the current literature on low-energy PRs for isolated SiNWs smaller than 100 nm. This lack is mainly due to the phenomenon difficulty and the high spectral and spatial resolution needed for the characterizations. Moreover, to our best knowledge, the transverse oscillation mode has never been visualized nor identified.

In this work, we studied SiNWs with Si core diameter of 30 nm, 420 nm length, and an oxide shell thickness of about 5 nm, with the purpose of monitoring and understanding their plasmon behavior. Spatially resolved scanning transmission electron microscopy (STEM–EELS) is the primary technique chosen for this purpose. The 2D high-resolution characterization allows identifying the PR, as a function of the position inside the SiNWs, at a subnanometric level. We characterized and identified the EELS features in the low-loss region, from 2 to 20 eV. We acquired the EELS scans along the SiNW’s axial and radial direction, and we identified the most significant signal contributions at several energies. Our numerical modeling...
results, based on finite-element method (FEM) analyses in conjunction with first-principles material models, verify our experimental findings. We observe a surface plasmon energy and intensity dependence on the position inside the NW. We distinguish two groups of separate PRs: the traveling surface waves, influenced by the cavity imposed by the NW longitudinal dimension, and the localized waves. Our results highlight the PRs profoundly different nature caused by the different dimensionality involved along with the two nanostructure directions. We visualize the discrete modes arising from the surface plasmon polariton (SPP), characteristic of the traveling waves, for several orders of harmonics, and experimentally image the localized surface plasmon (LSP) on the SiNW walls, also exhibiting peculiar fine-scale features. The results of this work increase the knowledge on the low-energy electron phenomena occurring in small SiNWs.

By controlling these PRs, it will be possible to create resonant cavities with nanometric dimensions, matching those of modern transistors, thus further advancing the field of nanooptics.\textsuperscript{[9,10,13,28]}

2. Results and Discussion

Mapping the localized modes in semiconductor nanostructures, as SiNWs, helps reveal their localized nature and monitor the nanostructure geometry role. Nevertheless, their experimental investigation in the low energy range has long been a technical challenge due to the lack of suitable techniques. The possibility to acquire EELS spectra with high energy resolution and from nm-sized sample portions allowed us to overcome the old limitations and obtain new information, previously inaccessible. Figure 1a

![Figure 1](image.png)

**Figure 1.** a) EELS spectra acquired on two different areas of the SiNWs, imaged in the inset (vertically shifted for a clear view). We acquired the blue curve in the NW central region (blue rectangle) and the red one in the surface region close to the NW base (red rectangle). To optimize the analyzed area, we chose rectangle sizes of $63 \times 18 \text{ nm}^2$. The vertical lines depicted on the curves indicate three main peaks. Inset: EFTEM image obtained using the Si bulk plasmon energy loss at $25 \text{ eV}$. b) EELS spectra, acquired in several positions along the NW growth axis. We acquired every curve in the corresponding box with the same color as indicated in the NW EFTEM image on the right. To properly investigate the spectrum spatial dependence, we chose a box size of $12 \times 3 \text{ nm}^2$ as a compromise between the spatial resolution and the signal noise. Magenta and ocher arrows indicate a peak at $8.5 \text{ eV}$ always present inside the NW; a peak at about $4.5 \text{ eV}$ disappears (grey arrow) in the area near the wire end (gray box in the EFTEM). c) Spectra from radial scans acquired at two different heights: positions 1 and 2, corresponding to regions 1 and 2 in the EFTEM map reported on the right. The vertical lines highlight the significant contributions.
shows two EELS spectra, vertically shifted for a clear view, extracted from two different SiNW regions. The images only show half of the SiNW, being resonance symmetric. The blue curve is an extract from the SiNW internal area (blue box) and the red curve from the surface region (red box). The dominant signal in both cases is the one centered at about 17 eV, related to the Si volume PR. This peak intensity in the blue curve is relatively greater than that of the red one due to the greater Si bulk fraction sampled during the surface area acquisition. A signal at 8.5 eV is present in both cases, whereas at 4.5 eV another signal arises only in the case of the SiNW core scanning (vertical line). From this result, we observe that the low-energy EELS spectrum characteristics vary according to the sampled region; hence, they spatially depend on the SiNW’s spectral behavior during the interaction with the electromagnetic field. Moreover, the spectral shape, especially in the low-energy region, suggests multiple contributions underneath the one indicated. Therefore, we chose an investigation methodology consisting of the acquisition of two types of EELS scans along the SiNWs’ axial and radial directions, using larger acquisition boxes. Figure 1b shows these axial scan results. The curves are correlated with the equally colored boxes in the energy-filtered transmission electron microscopy (EFTEM) map at 17 eV on the right. To verify the absence of parasitic signals, we started the scan by extracting the data from an external spot (red box lower left and red curve).

The red curve marks the background noise. The green curve, acquired in the external areas of the SiNWs (green box), shows the absence of the 17 eV peak, as expected, and two peaks at energies lower than 6 eV, as discussed later. The two low-energy contributions, at around 4.5 and 8.5 eV (Figure 1a), are now clearly distinguishable, as soon as the scan enters the NW, with modulation in intensity in some cases. The 8.5 eV signal is present, with an almost invariant intensity, for the entire wire length (magenta and other arrows), whereas the 4.5 eV signal undergoes an intensity drop near the tip at 55 nm of height taken from the SiNW base (gray arrow). The 4.5 eV signal decreases, confirming a spatial dependence on the spectral behavior. The signal intensity at 8.5 eV appears greater than the 4.5 eV signal intensity all along the NW, also due to the tails of the 17 eV peak. As anticipated above, some signals at energies lower than 6 eV are detectable even when the spectrum is acquired 10 nm away from the NW tip (green curve). This phenomenon is related to the large PR cross section, increasing the probability to generate plasmons also relatively far away from the wire edge and to the delocalized nature of electrons, producing the so-called scattering delocalization. We expect its coupling efficiency to be frequency dependent, with larger extension for lower energies. Indeed, as intensity (black to light-blue curve), whereas the 4.5 eV signal exhibits an almost invariant intensity. At position 2, the disappearance of the 4.5 eV signal confirms the spatial dependence of the spectral behavior. Figure 1c, green lines, corresponding to the green boxes in the relative EFTEM, acquired radially outside the wire, shows the delocalization effect. The data shown in Figure 1 indicate that the peaks, confined in a small energy range, are not energetically isolated from each other. We pursued a profound peak analysis through fitting procedures to gain further information about both physical and practical significance. After normalization and background removal, the EELS spectra extracted pure signals revealed their peak position, amplitude, and line width. We chose Lorentzian fitting because it is a widely accepted empirical approach. Figure S1, Supporting Information, shows the experimental spectra with the relative peak fit results.

After peak analysis, EELS maps were acquired to visualize the EELS signal spatial distribution with subnanometric resolution within and around the nanostructure at relevant energy values. Figure 2a shows the energy-filtered spectroscopic image (EFSI) maps acquired at four different energies. The yellow regions, indicated by the white arrows, and corresponding to the higher electron intensity, showcase discrete spots attributable to a nano-resonator’s harmonic behavior. They reveal the fundamental harmonic at 1.7 eV with one spot (and the other one symmetrically at the other tip, not imaged), the second harmonic at 4.5 eV with three spots (close to the ends and at the center of the SiNW), and the third one at 5.8 eV with four spots in total. The spots are located along the long NW axis, indicating the waves traveling back and forth between the NW ends. Hence, the surface PR modes represent an interplay between a traveling surface plasmon, also referred to as the SPP, and the resonator, induced by the NW length. The positions of all the experimental spots appear slightly shifted toward the NW end. We address this effect to the reflection mechanism of the SPP wave at the NW ends, as observed in silver nanorods. At the tip, supposedly playing the oscillator node’s role, there is an omnipresent, low, but nonzero signal that we address to the well-known point effect causing field enhancement.

At 8.5 eV, a strong signal located at the Si–SiO2 interface along the NW walls is evident. We attribute this resonance to the transverse PR, also referred to as LSP, as occurring in spherical particles, too. The interface plays the role of a confining well for radial resonance. The comparison between the first three images and the last one teaches the difference in the spatial charge distributions between longitudinal and transverse modes. The map at 8.5 eV also reports a graph with a red curve showing the signal profile intensity acquired in correspondence to the black box along the indicated portion of the NW wall. The signal profile intensity shows peaks and valleys. We observed this result systematically on all of the NWs analyzed. This phenomenon has never been reported nor theorized so far in the literature, and it suggests a discrete modal behavior also for the LSP. It could be due to periodic charge distribution along the NW walls, probably related to a constructive/destructive interference mechanism; so, it is interesting to conduct deeper investigations in the future. The colored rectangles in Figure 2a indicate the regions from which the EELS signal profiles reported in (b) are radially extracted, at the significant heights along the NW. As is evident, all the profiles are flat except the 8.5 eV.
corresponding to LSP, which present two peaks at the NW walls, corresponding to the harmonic oscillator wells. Figure 2c shows the EELS signal profiles acquired axially at the same energies as in (a). It displays the signal profiles mediated on the entire NW surface (white rectangle) along the axial direction for fixed energy values. Several intensity spots appear at different positions for several energies, clearly indicating the harmonic peaks, whereas, as expected, the intensity remains constant all along the SiNW for transverse excitation. Our experimental data on the propagating SPPs match well with the harmonics observed by STEM in silver nanoparticles. The interplay between the SPPs and the resonator observed in our SiNWs is similar to the observations in metallic NWs. When analyzing metallic nanostructures, however, the density of free electrons is very high, and as a consequence, the plasmon signals, both in energy and in the maps, are intense and well separated. In semiconductors, on the contrary, one expects the resonance spots to be less intense and more spatially and energetically diffused as the signal comes from the valence band electrons. Our experimental findings confirm these expectations. Some literature findings regarding PR in semiconductor NWs anticipated the longitudinal modes’ appearance. Nevertheless, these studies highlighted only two peaks in the spectra, at 4.2 and 8.2 eV, with the data not resolved enough to identify other peaks. Moreover, the spot visualization in the maps was not clear for low spatial resolution of the technique used, so the article discusses a uniform intensity along the longitudinal direction. Our work demonstrates that it is possible to identify the resonance spots using proper characterization, acquisition scanning, and data analysis. Moreover, the literature attributes a peak at 8.2 eV to the multipolar mode of a longitudinal oscillation with the charge distributed in alternate ways along the radial direction, even though the authors attributed the peak to a longitudinal wave. Due to our observations, the 8.5 eV mode has a maximum intensity in correspondence with the NW walls, and we consequently ascribe it to the LSP. In contrast, the multiple lower-energy modes’ intensity is distributed along the axial direction, pertaining them to the longitudinal waves. This evidence indicates that the harmonics originate from traveling charge oscillations along the longitudinal direction on the SiNW surface. On the contrary, the 8.5 eV mode originates from localized charge oscillations oriented transversely and then blocked by the NW walls, acting as quantum wells.

These results evidence the fundamentally different nature of these two classes of resonances, correlated with these Si nanostructures’ two orthogonal dimensions. At the end of the SiNWs’ growth process, there may be gold atoms coming from the catalyst residues inside the NWs. For this reason, to exclude false conclusions from our energy maps, we subjected the materials to a chemical removal process of gold, referred to as the Au–Si plasmon, is found at 11–12.5 eV, significantly different than that at 4 or 8.5 eV, as visible in our experiments. Moreover, previous authors expected that the PR effect is due to an extended interface between a metal film and the semiconductor. Here we did not observe any metallic films on the SiNWs, and we expect any gold residuals to take
the form of dispersed atoms exclusively. Therefore, it could be excluded that the observed signals are due to gold.

We conducted numerical simulations to deeply understand and verify our experimental findings, modeling the optical dispersion data and the SiNW’s resonant behavior under optical excitation. Due to a lack of reliable material data for Si at high energies in the literature, we first carried out first-principles density functional theory (DFT) calculations on bulk Si. Subsequently, we extracted the corresponding complex optical dispersion data (Figure 3, see Experimental Section for DFT details). The calculated complex relative electric permittivity \( \varepsilon \) reveals a negative real part for energy levels beyond 4 eV (Figure 3b, blue curve), a well-known prerequisite for the existence of surface plasmons. \(^{[14]}\) At the same time, its imaginary part (orange curve), corresponding to optical losses, decays toward higher energies.

Based on these dispersive material data, our electrodynamic simulations reveal optically excited, discrete surface energy states, corresponding to the earlier-discussed PR: under a longitudinally polarized impinging plane wave excitation, the structure comprises longitudinal standing wave patterns corresponding to the longitudinal SPP resonance harmonics at discrete energies, with two, three, and four intensity lobes at the interface between the Si core and the SiO\(_2\) shell, at 4.0, 5.0, and 6.5 eV, respectively. The structure also comprises the omnipresent strong field enhancement spots located on the NW tips, extending through the shell and the close surroundings. Under transversely polarized excitation, the structure exhibits the LSP mode at higher energy (9.1 eV), which appears uniform also inside the nanostructure. We remind that, when observed by TEM (Figure 1), the signal intensity is higher at the SiNW borders than at the center due to the integration effect at the borders. No other resonances are apparent (between 1 and 11 eV) under this polarization. Our numerical results strongly suggest that our experimental observations arise from the (weighted) integration of field profiles perpendic-ular to the main wire axis, including the evanescent fields stemming from the Si core and the SiO\(_2\) shell and mainly confined inside the latter. The full-spectral extinction profiles for both excitations further support this claim (Figure S2, Supporting Information). We address the slight mismatch in resonance ener-gies, as compared to our experimental findings, to the introduced uncertainty via the material models and the shell structure: first, the DFT-calculated Si data refers to bulk Si, neglecting confinement effects arising from the NW geometry and lattice effects arising from the specific NW axis growth. Second, the SiO\(_2\) shell parameter choices (stoichiometry, morphology) might further influence the resonance positions. For a more detailed discussion of optically excited near-field maps, we refer to Supporting Information (Figure S3, Supporting Information).

While further investigations might lead to a higher-grade quantitative match, our numerical findings qualitatively prove the experimental claims. The experimental and theoretical results illustrated so far and in mutual agreement point to the conclusion that SiNWs exhibit PRs, similar to those found in metallic NWs. The second evidence is that these electronic oscillations fall in two categories, longitudinal and transversal, confined by the nanostructure geometry.

### 3. Conclusion

We investigated the plasmon occurrence and behavior in 30 nm-wide SiNWs, through the combination of two powerful characterization techniques, STEM and EELS, providing high spatial and energy resolution. We acquired the spectra as functions of the position inside the SiNWs by scanning the beam along the axial and radial direction. This high-resolution scanning allowed us to identify the spectral response for both traveling and localized resonant modes for these SiNWs in the energy range between 3.7 and 8.5 eV. We classify the observed signals as low-energy plasmonic resonances, further by our dispersive material data calculations. The energy maps clearly show discrete intensity spots with axial symmetries for the longitudinal oscillations. In an energy region well separated from the longitudinal propagating waves, we demonstrated the transverse LSP oscillations’ existence, visualizing them for the first time to our knowledge. Our electromagnetic simulations on the optical properties and plasmon responses of the SiNWs strongly support...
our experimental findings of resonant longitudinal surface plasmon states and well-separated transverse localized plasmons. Furthermore, our study unexpectedly revealed fine-scale features, such as intensity modulation along the NW walls, resembling the longitudinal waves’ quantization. Finally, our high-resolution methodology revealed that the longitudinal plasmons’ wavelength shows a compression toward the NW tips.

The possibility of exploiting these SiNWs as resonant cavities represents a tremendous potentiality for advancing nano-optics, and the excellent integration capacity offered by silicon for broad on-chip optoelectronic applications practically sustains this potentiality.

4. Experimental Section

SiNWs Growth: We grew SiNWs by the induced plasma chemical vapor deposition (IP-CVD) technique through the well-known vapor–liquid–solid (VLS) process11 using gold (Au) as metal catalyst.12,34 To this end, we first cleaned the substrate (CZ < 100 > 13 Ω-cm p-type 6° Si wafer) in sonating baths of acetone, ethanol, and water for about 5 min each. Then, a one-minute dip in HF (1%) removed the native oxide film. Once dried, we loaded the substrate into the sputter chamber for the Au deposition at a pressure of 5 × 10⁻⁶ mbar and a current of 10 mA for 60 s. The Au thickness deposited was ≈2 nm (monitored by in situ quartz balance). Before the depositions, a CVD chamber heated the substrate at 380 °C for 1 h under vacuum conditions to allow the coalescence of the Au dots, whereupon the growth took place at 380 °C for 30 min with a pressure equal to 20 mTorr and a plasma power of 20 W. Silane (SiH₄) was the Si gas source, and Ar the plasma ignition, with the SiH₄/Ar ratio fixed to 30. Once the SiNW growth was complete, exposing the sample to the atmosphere immediately created a compliant, ≈5 nm-thick SiO₂ layer surrounding the wires. Finally, the SiNWs underwent a procedure allowing the Au removal. First, a 5 min dip in an HF-buffered solution diluted in H₂O (1:10) etched the SiO₂ shell, and then a 4 min exposure to a gold-etched solution (Fujifilm gold etch II w/OHS) removed the residual gold.

STEM Analysis: After the synthesis, a mechanical stripping procedure collected the SiNWs from the wafer onto a metallic meshed grid. We conducted our STEM/EELS analyses at 200 kV using a JEOL JEM-ARM200F cold FEG microscope equipped with a Ceos sextupole Cs aberration corrector on the probe and EELS by a Gatan Quantum ER spectrometer equipped with a fast shutter and 2 × 10³ pixel acquisition camera. The inherent spectrum image acquisition mode provided a 3D datacube containing the 2D dark-field STEM image of the NW and the low-loss spectrum point by point simultaneously. In our experimental acquisitions, we conducted a 2D primary beam scan across a single NW, using a pixel size of 1.3 nm and 0.25 eV per pixel in the 2 K Gatan camera. The EELS energy range of the EELS spectrum resulted as 50 eV with an energy resolution of 0.45 eV. We applied a standard power-law fitting procedure with slit placed between 1.5 and 2.5 eV to remove the zero loss from all raw spectra.

Figure 4. Optical simulations of isolated SiNWs confirm the experimental findings: a plane wave, impinging perpendicularly to the long NW axis (light green arrows), excites the structure. Displayed here are the resulting simulated longitudinal and transverse resonant energy states (E-field magnitude [V m⁻¹]), between 4.0 and 9.1 eV. The images show the a) resulting electrical field magnitudes in and around the nanostructure, in vertical cross sections (half NW length) displaying the Si core and the surrounding SiO₂ shell and vacuum b) as well as 3D field intensity plots on the interface between Si core and SiO₂ shell. Under longitudinal polarization of the impinging wave (light blue arrow, black box), the excited structure comprises longitudinal standing wave patterns corresponding to the longitudinal harmonics of SPP resonances at discrete energies, with two (4.0 eV), three (5.0 eV), and four (6.5 eV) intensity lobes on the Si–SiO₂ interface, plus a pronounced field enhancement on the NW tips inside the shell region. Under transversely polarized excitation (light blue arrow, 9.1 eV), the structure exhibits the transverse LSP mode at higher energy, well separated from the longitudinal traveling surface modes.
A 21 pixel-wide binning furthermore reduced the EELS spectra signal-to-noise ratio. The 2D energy-filtered spectrum images (EFSI) consisted of a slice of the background-corrected datacube using a slit of 0.3 – 1 eV. 

Energy Spectrum Peak Fit: We conducted the EELS spectra peak fitting with the freely available software Fityk,[40] using standard Lorentzian model functions. To improve the statistics, we set the peak energy by conducting several samplings in each wire’s significant areas, with the peak widths and intensity as the free parameters. We furthermore converted the spectra from energy to wavelength to better discriminate adjacent signals better and increase the choose of choice. We reported the results of the peak fitting procedure in Figure S2, Supporting Information.

Optical Simulations: We conducted FEM simulations to model the optical response of isolated Si NWs. We used the wave optics module of the commercially available FEM solver COMSOL Multiphysics.[41] We modeled the experimentally investigated NW by a 30 nm-diameter Si cylindrical core, surrounded by a 5 nm-thick silicon dioxide (SiO2) shell. We assumed a total NW length of 420 nm, with slightly rounded tips to avoid exaggerated edge effects. The NW was embedded in a spherical vacuum domain, surrounded by perfectly matched layer (PML) boundaries to mimic an infinite outer domain. We excited the structure with a background electromagnetic plane wave (wavelengths between 110 and 550 nm, corresponding to energies between 11.27 and 2.25 eV, respectively), with an electrical field amplitude of 1 V m⁻¹, impinging perpendicularly to the long NW axis (green arrows in Figure 3). We conducted separate simulation sweeps with the electrical field polarization in parallel and orthogonal to the long NW axis (blue arrows in Figure 4). The final setup comprised 6 635 940 degrees of freedom (per wavelength). We modeled the crucial Si and SiO2 material influence on the optical simulations via complex-valued, dispersive refractive index data. The SiO2 data set was based on literature values.[42] Due to a lack of literature reports on critical Si data set via in-house Si dispersion data for energy levels beyond 4 eV, we generated the more

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

Research data are not shared.

Keywords

plasmon resonances, scanning transmission electron microscopy, silicon nanowires, simulations

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