Plasmon-induced resonant effects on the optical properties of Ag-decorated ZnSe nanowires

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Abstract
In this work we show how the optical properties of ZnSe nanowires are modified by the presence of Ag nanoparticles on the sidewalls of the ZnSe nanowires. In particular, we show that the low-temperature luminescence of the ZnSe nanowires changes its shape, enhancing the phonon replicas of impurity-related recombination and affecting rise and decay times of the transient absorption bleaching at room temperatures, with an increase of the former and a decrease of the latter. In contrast, the deposition of Au nanoparticles on ZnSe nanowires does not change the optical properties of the sample. We suggest that the mechanism underlying these experimental observations is energy transfer via a resonant interaction, based on the fact that the localized surface plasmon resonance (LSPR) of Ag nanoparticles spectrally overlaps with absorption and emission of ZnSe, while the Au LSPR does not.

Keywords: nanowires, plasmons, FRET, luminescence, ZnSe

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

1. Introduction
When photons with a well-defined energy impinge on metallic nanoparticles (NPs), they induce a collective oscillation of the conduction electrons of the NPs, giving rise to a localized surface plasmon resonance (LSPR) producing strong resonant fields in its proximity. Plasmons have applications in several fields, from molecular sensing [1] to biomedicine [2] and optoelectronics [3]. Because of the associated electric field, plasmons induced in NPs placed on semiconductor surfaces may strongly interact with the adjacent material, in particular modifying its optical properties, by means of a Förster interaction (Förster resonant energy transfer, FRET), a non-radiative dipole interaction [4, 5], or by hot carriers transfer from the metallic NPs to the semiconductor [3, 6, 7]. Modifications of the semiconductor optical properties have been observed in several semiconducting materials when coupled to metal NPs, with relevant differences among different material systems [8]. The most common result is enhancement of the intensity of photoluminescence (PL) from the semiconductor, as has been reported for quantum wells [9], Si-based light-emitting diodes [10], metal dichalcogenides flakes [11], quantum dots [12–14], and nanowires [5, 7, 15–17]. Depending on sample preparation, either PL enhancement or quenching has also been observed in a similar system (e.g., Ag-ZnO [18]).

Nanowires (NWs) are characterized by large surface-to-volume ratios, and are a class of materials whose electronic
states can strongly interact with the electric field generated by LSPRs. Metal NPs placed along semiconductor NWs in high-density arrays form a three-dimensional 3D ensemble in a microporous structure that can strongly interact with the environment, thus enhancing the sensing properties of the plasmonic NPs [19]. Moreover, when NPs are obtained by the dewetting of films deposited on the sidewalls of the NWs, the quasi-1D shape of the NW induces NP shapes that differ from the sphere, thus modifying their plasmonic properties [20].

In this work we report the changes induced by Ag NPs on the stationary and transient optical properties of ZnSe NWs. The quasi-stationary and dynamic optical properties of pristine ZnSe NWs were described in our previous publications [21, 22] and represent the starting point of the present work. Ag NPs were grown on the sidewalls of ZnSe NWs by thermal dewetting. The size of the NPs was between 20 nm and 50 nm. The LSPR of Ag NPs of those sizes is in the wavelength range of 400–50 nm. The LSPR and the absorption band of the ZnSe NWs. In particular, we have studied how the PL and the fast transient absorption in ZnSe NWs are modified by the presence of the Ag NPs. To compare the results of the ZnSe NWs/Ag NPs system with those obtained with a material combination where the LSPR of the NPs is not resonant with the ZnSe band gap, we also investigated a ZnSe NWs/Au NPs system, with the Au NPs having a LSPR at about 2.34 eV (530 nm) [20], below the ZnSe band gap. For a similar system (ZnSe: Sb nanoribbons/Si nano-heterojunction), Wang et al [25] reported enhancement of optical absorption when Ag nanospheres are deposited on the ZnSe nanoribbons, with the resulting improvement of a light absorption-based optoelectronic device based on these materials.

2. Experimental

The ZnSe NWs were grown by molecular beam epitaxy on GaAs, quartz and sapphire, at 300 °C. The choice of low-temperature growth was dictated by the resulting superior optical properties [21], as high growth temperatures degrade the optical properties of ZnSe with time because of the presence of point defects [26]. The result of the growth consists of a very high density of randomly oriented, tapered ZnSe NWs. The mean diameter at the tip is 10 nm and the base diameter is about 100 nm, with an average length of about 1 μm. No passivation of the ZnSe NW sidewalls is provided. Details about growth conditions, morphological and structural characterizations, and low-temperature luminescence were published by Zannier et al [21], while a more complete description of the optical properties, including transient absorption, was published by Tian et al [22].

Thin films of silver (and gold, for comparison) with nominal thicknesses below 5 nm were evaporated onto the ZnSe NWs to form NPs after annealing at 280 °C for 10 min. For the comparison between pristine NWs and metal-decorated NWs, we used different pieces of the same as-grown sample. Femtosecond transient absorption spectroscopy (FTAS) measurements were performed on as-grown samples on quartz or sapphire because the substrates are transparent in the spectral region of interest. PL was carried out on NWs grown on all three substrates mentioned above. Several samples were used, to verify the reproducibility of the results after metal deposition. We observed no major dependences on the substrate to be explicitly mentioned; only minor differences will be pointed out.

Figure 1 shows a scanning electron microscopy (SEM) image of (a) pristine NWs and (b) decorated NWs, showing that the Ag NPs have a diameter of 20–50 nm.

In figure 2(a) we report the absorbance of Ag NPs fabricated on the sidewalls of silica NWs that show similar size
and shape distributions to those of the NPs fabricated on ZnSe NWs. In this type of sample, the absorbance is due to the Ag NPs only \[19, 27\]. In figure 2(b) we show the room temperature PL of a typical ZnSe NW sample used for this work. The maximum absorbance for the Ag NPs is very close in energy to the near band edge (NBE) emission of ZnSe. The doublet structure in the Ag NP absorbance is due to the presence of both dipole and quadrupole contributions \[27\].

The PL and time-resolved PL were carried out using a narrow-line laser diode and the second harmonic of a Ti:sapphire laser having a pulse length of 20 fs at 80 MHz repetition rate, respectively. In both cases the excitation wavelength was 405 nm. The PL signal was analyzed using a monochromator 0.35 m in length and a charge-coupled device (CCD) or a Si-APD [full width at half maximum (FWHM) = 150 ps] for quasi-stationary or time-resolved analysis, respectively.

The fast transient absorption (TA) spectra were measured with a pump–probe setup. The pump at 410 nm is generated by an optical parametric amplifier, and the probe is a white light super-continuum (420–750 nm) generated in a commercial femtosecond TA spectrometer (IB Photonics; FemtoFrame II). The experimental set-up is sketched in figure 3. The instrument response function was measured to be approximately 50 fs. The measured quantity is the difference in absorbance (ΔA) between the probe transmitted through the excited sample and that transmitted through the unper- turbed sample as a function of the delay time between pump and probe, obtaining a 2D color map. A reduction of the absorption (bleaching) due to the photoexcitation of electrons from the valence band to the conduction band of ZnSe will result in a negative signal (yellow-red in the 2D false-color map). Changes in the rise and decay times of the bleaching signal between bare and NP-decorated samples give information on the interaction between the different materials.

3. Results

3.1. Absorption

In figure 4 we show the absorbance of a typical pristine sample (red curve) and its Ag-decorated companion obtained from the same growth (black curve). The absorbance of the Ag-decorated samples is higher than that of the pristine samples, and with a line shape of the absorbance that differs from that of the pristine samples, especially in the 2.2–2.6 eV region. The higher absorbance in the decorated samples is due to the added light absorption by the Ag NPs and to an increased light scattering produced by the same NPs.

In figure 5 we show the 2D map in false colors of the TA spectra in pristine (a) and Ag-decorated (b) samples collected as a function of the pump–probe delay and with a pump fluence of 260 μJ cm\(^{-2}\). In both samples, the bleaching of the absorption of the ZnSe NWs (due to the presence of e-h pairs photoexcited by the pump) is observed at an NBE energy of about 2.7 eV and along the defect band \[22\]. The two maps appear to be similar, but with some differences that we will describe. First, however, we wish to point out the absence of any plasmonic signal in the spectra of the decorated sample. Ag NPs of similar size, formed by the same procedure on silica NWs, instead show a clear plasmonic signal in the TA

![Figure 3. Sketch of the experimental set-up used for the pump–probe experiments.](image-url)
spectra, in which the contributions from dipole and quadrupole are clearly distinguished.

The intensity of the TA signal of the NBE region [22] in the Ag-decorated sample is reduced by about 20% with respect to that of the pristine sample, a reduction that we attribute to light absorption and scattering caused by the Ag NPs. The main spectral change observed in decorated samples is the reduction of the relative intensity of the defect-related signal at energies below 2.7 eV. This feature can be better appreciated in figure 6, where we show the normalized TA spectra for both type of samples at the same delay time of 1 ps. The TA signal from the defect bands is weaker in the presence of Ag NPs, also causing an apparent narrowing of the low-energy side of the NBE bleaching signal.

A further difference between pristine and Ag-decorated signals can be observed in the dynamics of the NBE signal. In figure 7 we show the temporal behavior of $\Delta A$ in both types of samples. In figures 7(a) and (b) we show the rise time ($\tau_r$) of $\Delta A$ at the NBE energy in the pristine and Ag-decorated samples, respectively. It increases from $\tau_r = 165 \pm 52$ fs in the pristine sample (within the error bar, this value is the same as that measured in similar samples by Tian et al [22]) to $\tau_r = 250 \pm 95$ fs in the Ag-decorated sample. On the other hand, a reduction is observed in the decay lifetime ($\tau_d$) of the TA signal at the NBE energy in the Ag-ZnSe NWs with respect to the bare ZnSe NWs. In the former case we find $\tau_d = 1.85 \pm 0.05$ ps, as compared with $\tau_d = 3.57 \pm 0.08$ ps in the latter. Finally, figure 7(d) shows the values obtained for the decay time as a function of the probe energy. For the pristine samples, a maximum is observed at energies close to the band gap value. In general, the broad band at low energy exhibits faster dynamics than the band gap region, as seen in the transient maps in figure 5. This observation is in agreement with the work reported in Tian et al [22]. This behavior is also observed in Ag-decorated NWs, but in this case the decay times are lower than their corresponding values in bare NWs over the whole energy range explored in the present work. A similar decrease in the decay time of the absorption bleaching of band-gap and hot-carrier states has been observed in CdTe/Au core–shell NWs with respect to bare CdTe NWs [28].

As mentioned in the Introduction, to show the importance of having LSPRs with energies close to that of the intrinsic electronic states of the semiconductor, in order to observe the differences, we also studied the optical properties of ZnSe NWs decorated with Au NPs. In this case the LSPR is not resonant with the band gap of ZnSe NWs [20]. Figure 8 shows the 2D transient map of the $\Delta A$ intensity versus time delay and probe energy for Au-decorated ZnSe NWs excited spectra [27], in which the contributions from dipole and quadrupole are clearly distinguished.
with a pump of 3.02 eV at an excitation intensity of 260 μJ cm⁻². In these Au-decorated NWs, beyond the bleaching signal at the NBE energy, we can clearly see contributions of the Au LSPR to the TA signal at energies around 2.3 eV. This feature is favored by a lower absorption of the ZnSe NWs in the Au LSPR region than at the energy of the Ag LSPR. As a result, one of the positive wings of the plasmonic signal is clearly visible at 2.5 eV. At lower energy, the TA signals of the Au LSPR and that of the ZnSe defects mix with each other (figure 8(b)). The most important point, however, is that the TA dynamics of the NBE signal in Au-decorated samples does not differ from that observed in the pristine samples. The decay time in the case of the Au-decorated NWs is \( \tau_d = 3.69 \pm 0.21 \) ps, and the rise time is \( \tau_r = 150 \pm 90 \) fs. If we use a pump energy resonant with the Au plasmon (2.34 eV, 530 nm), which is smaller than the ZnSe band gap, we observe very weak bleaching of the NBE absorption in all samples, pristine or metal-decorated, because of the partial absorption of the pump by defect states in ZnSe [22] that excites electrons in the conduction band. The bleaching signal appears very similar in all samples.

3.2. Photoluminescence

The PL spectra of ZnSe at 10 K typically show three emission bands corresponding to the NBE region, to shallow centers, and to the Y-line at low energies [21, 28]. With ZnSe being a polar crystal, because of the strong electron–phonon Fröhlich interaction, phonon replicas often appear in the PL spectra from phonon-assisted emission of free and bound excitons as well as donor–acceptor pairs (DAPs) [29].

Figure 9 shows the PL spectra at 10 K of our ZnSe NWs, with and without the decoration of Ag and Au NPs. The intensity of the NBE PL in the decorated samples is reduced with respect to that of the pristine samples because of absorption and scattering of the pump and of the PL by the metal NPs. The reduction is of about the same magnitude in the Ag- and Au-decorated samples. In the Ag-decorated NWs, the relative intensity of the Y-line decreases with respect to the NBE emission, while the emission of the energy region immediately below the NBE increases.

In particular, the spectra of the pristine sample (red lines in parts (a), (b), and (c) of figure 9) are characterized by a broad NBE emission peaking at about 2.8 eV, followed by a broader emission at low energies (the Y-line; see also [21]). Depending on the substrate used (GaAs, quartz, or sapphire), some differences can be observed from sample to sample in NBE width and relative intensity with respect to the broader low-energy emission. In particular, for the sample grown on sapphire, the broad impurity-related emission is much stronger than the NBE emission, probably because of contamination from the substrate. Independently of the relative intensity of defect and NBE signals, in all samples the deposition of Ag...
NPs dramatically changes the line shape of the PL spectra (black lines in parts (a), (b), and (c) of figure 9). The broad emission at around 2.76 eV splits into several narrower peaks. We distinguish the NBE at 2.81 eV, while in the energy range between 2.6 and 2.76 eV, four or five (depending on the sample) equidistant peaks are observed with energy separations equal to the longitudinal optical (LO) phonon energy of ZnSe (31.5 meV).

We attribute the equally separated peaks observed in the Ag-decorated samples to no-phonon (at the highest energy) and phonon replicas of the impurity-related recombination (bound exciton and/or DAPs) [29–32]. The formation of Ag NPs on the ZnSe NW sidewalls gives rise to a strong enhancement and a narrowing of the emissions relative to the impurity-related recombination, also allowing the observation of distinct phonon replicas.

We point out that Au NPs deposited on the ZnSe sidewalls do not cause any significant change to the PL line shape (green lines in figures 9(a) and (b)), giving rise only to a decrease of the NBE PL intensity due to bare absorption and scattering of both excitation and emission light because of the presence of the light-absorbing Au NPs. The defect-related emission in the energy range 2.6–2.75 eV is slightly reduced relative to the NBE emission, while the Y-line is slightly enhanced.

Time-resolved measurements of the NBE luminescence in all samples (not shown) do not indicate any significant change in the recombination lifetime (about 0.5 ns) between decorated and pristine samples, in agreement with the invariance of the PL intensity.
4. Discussion

Electronic interaction between a semiconductor and a plasmonic nanostructure mainly occurs through two pathways. These pathways depend upon the spatial proximity and the relative energy position of the LSPR of the plasmonic nanoparticles and the semiconductor electronic states. As mentioned in the Introduction, these two mechanisms, namely hot electron injection and resonance energy transfer (RET) [4, 33], benefit from the proximity between the materials involved. But for RET only, a further necessary condition is a spectral overlap between the LSPR and the absorption band of the semiconductor [4]. In our system of Ag-decorated ZnSe NWs, there are spectral overlaps (see figure 2) and physical contact between the metal and semiconductor NW. This could make possible both RET and hot electron transfer. In the case of Au-decorated ZnSe NWs, there is physical contact between the metal and semiconductor, but no spectral overlap between Au LSPR and ZnSe band gap absorption. Hence, only hot charge carrier transfer could be observed in our experiment.

The decay of the NP LSPR produces hot electrons in both Ag and Au NPs, whose energy distribution will reflect the density of states in the two metals. Because of the band alignment between the metals and ZnSe [34, 35], part of the hot electrons will have energy above the ZnSe conduction band. It is worth pointing out that the electronic density of states at the Fermi level is small for both metals. In both cases, indeed, the Fermi levels of the metals lie well above the center of the relative electronic d band, at 2.58 eV for Au [36] and 3.93 eV for Ag [37]. Given the presence of a Schottky barrier between the metals and ZnSe [35], in both metals only those electrons close to the Fermi level will have sufficient excess energy to overcome it. Then, for the electronic conditions, we have very close similarities between the Ag/ZnSe NW and Au/ZnSe NW interfaces. Therefore, we argue that if the changes in the optical properties of the Ag-decorated NWs were due to hot electron transfer from the metal NP to the semiconductor, we should observe similar modifications to our TA and PL spectra with both metals, which is not the case. Hence, we suggest that a resonant mechanism underlies the modifications observed in the TA and PL spectra after the formation of the Ag NPs on the NW sidewalls. In particular, the increase in the rise time of the band gap bleaching in the Ag-decorated ZnSe NWs may indicate the presence of a RET mechanism, where the Ag NPs act as donors and the ZnSe NPs as the acceptors [4, 33]. The increase in rise time could be explained as being due to the excitation of e-h pairs in ZnSe at a later time than for direct photoexcitation, as the result of the energy transfer from the NPs toward the NWs.

On the other hand, a decrease in the decay time of the absorption bleaching is observed over the whole energy range, from NBE down to the defect band. The absorption spectrum of the Ag NPs is very broad, due to the presence of both dipole and quadrupole excitation of the plasmon and of the shape distribution of the NPs [20, 24, 27, 38, 39]. At low energies, free-carrier absorption also contributes to their absorbance spectrum. The broad width may allow the resonant interaction with the defect band, with effects similar to those observed for the NBE. The more efficient coupling of the carriers to phonons suggested by the low-temperature PL of the Ag-decorated NWs (see below) may also cause carriers to lose their initial energy towards electronic states at lower energy. An increased carrier-phonon scattering rate would explain the faster decay time in the TA signal of the Ag-decorated samples. Furthermore, the experimental observation of the plasmonic bleaching in the Au/ZnSe NW transient absorbance spectrum but not in the Ag/ZnSe NW case could indicate that the energy transfer in the latter system is very efficient, i.e., the fact that the Ag NPs are not significantly heated suggests that the energy absorbed by the plasmon resonance is efficiently transferred to the ZnSe NWs. Of course, we cannot rule out that the Ag LSPR bleaching could be small with respect to the ZnSe NBE bleaching, but we notice that the bleaching of the Ag LSPR in a similar 3D array of Ag NPs on silica NWs has an intensity of about 30 mOD [27], which is of the same order as the ZnSe NBE bleaching observed here. Therefore, the absence of a clear contribution by the Ag LSPR to the TA signal of the Ag/ZnSe NW system points towards a very efficient resonant energy transfer, as suggested above.

The decision not to refer explicitly to FRET when discussing our TA results is due to the fact that it is difficult to invoke the Förster interaction to explain the changes of the PL line shape in the Ag-decorated ZnSe NWs and, in particular, the enhancement and narrowing of the impurity-related recombination with its phonon replicas. Nevertheless, a resonant mechanism between the NW and NP electronic states must lie at the origin of the observed features, in particular favoring the carrier–phonon coupling. Indeed, we can rule out a simple passivation mechanism due to the presence of the Ag NPs on the NW sidewalls, because not even the slightest line shape change of the same type is observed with Au-decorated NWs. Noticing that the NP coverage of the NWs is very similar for the two cases of Ag and Au, we can also exclude that the differences are due merely to different degrees of surface passivation. The impurity-related recombination occurs only at low temperatures because of the involvement of shallow donors and acceptors [40], and related effects cannot be observed at room temperature, at which we perform our FTAS measurements. This prevents us from making a direct comparison between the low-temperature PL and the room temperature FTAS. Nevertheless, both measurements point towards a more efficient carrier–phonon interaction rate. On the other hand, Raman measurements performed at room temperature (not reported) do not show any relevant difference between the spectra of bare and Ag NWs. A reduction of the overall intensity of the Raman peaks of the decorated NWs is the only observed feature, due to excitation light absorption and scattering by the metal NPs.

5. Conclusion

We have reported the changes of transient absorption and luminescence of ZnSe nanowires induced by the fabrication of Ag nanoparticles on the nanowire sidewalls. In particular,
we have observed the change of rise and decay times characteristic of the dynamics of absorption bleaching in the nanowires and the dramatic changes in the low-temperature luminescence spectra, where a strong enhancement of the phonon-mediated impurity-related recombination was observed. On the other hand, the coverage of the ZnSe NWs with Au nanoparticles does not lead to any significant change in transient absorption and luminescence of ZnSe. We have attributed the observed features to resonant electronic interactions between ZnSe NWs and Ag NPs, which are favored in the Ag-ZnSe system with respect to that of the Au-ZnSe system because of the near energy resonance of Ag localized surface plasmon resonance and the ZnSe band gap. We hope that our experimental results will stimulate further investigation, including theoretical calculations.

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