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Synthesis, Optical properties and, amplified spontaneous emission of hybrid Ag-SiO₂-CdTe nanocomposite

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Abstract

In this work, a hybrid nanocomposite of metal-dielectric-semiconductor; Ag-SiO₂-CdTe nanoparticles has been synthesized. Silica shell was used as a spacer to isolate and control the distance between Ag plasmonic and luminescent CdTe QDs. It was found that insertion of silica shell enhances the plasmonic field more than 31%. Accordingly, Ag-SiO₂ plasmonic enhances the luminescence and quantum yield of CdTe QDs by 200% and 55%, respectively. The threshold power of amplified spontaneous emission of CdTe was found to depend on both temperature and excitation wavelength location with respect to Plasmon and exciton absorption. This nanocomposite could be potentially used in light emitting diodes, biological sensing and thermal therapy.

1. Introduction

Interaction of metal-semiconductor nanoparticles (NPs) is an important field in nanophotonic science. Semiconductor NPs are characterized by bandgap tune-ability according to their size and shape [1]. This allows for different technological applications such as, light emitting diodes (LED) [2,3], nanolaser [4], photo-nano sensors [5] and solar cells [6].

Metallic nano-systems are characterized by plasmonic fields which may be classified as volume plasmon, surface plasmon polaritons (SPP) and localized surface plasmons (LSP). The intensity of plasmonic modes depends on electromagnetic (EM) pump frequency. Plasmonic propagation does not emit light but creates a strong local field excite the nearby active molecules (such as dyes or semiconductor NPs). Semiconductor NPs in vicinity of metallic NPs exhibit changes in

their optical and lasing properties depending on their; size and shape, relative concentration ratio and intra-distance [7], in addition to the EM pump frequency [8]. A temporally coherent high-intensity field can be generated by Plasmonics on metallic-nanosystem surface known as SPASER [8,9] (Surface Plasmon Amplification by Stimulated Emission of Radiation). This differs from traditional laser in which photons amplification is obtained by amplified spontaneous emission (ASE), the important parameter in lasing process.

Surface plasmons (SPs), a near-field phenomenon of surface electron oscillations is created by interactions of incident electromagnetic wave (EMW) with free electrons in nano metals. Resonant excitation of metallic NPs forms high LSP of strong EM field and good spatial confinement in the sub-wavelength scale. Thus their propagation avoids the limitation imposed by diffraction [9]. Nano size metal-dielectric interface assists some special EM modes, such as LSP or SPP, to propagate along their interface, where incident EMW induces oscillation of metallic electrons at the dielectric boundary. The energy transfer from the gain medium to SPs excite SP resonance mostly with the same mode yielding more plasmonic modes available for lasing radiation [9].

CdTe quantum dots (QDs) is an important II-VI semiconductor [10] of high luminescence quantum yield. It has wide applications in many fields. The present work studied the optical properties of CdTe QDs for lasing process in presence and absence of metal-dielectric plasmon. Firstly, we devoted to syntheses methods and the optical properties of CdTe QDs, Ag NPs, Ag-SiO₂ core-shell NPs and hybrid Ag-SiO₂-CdTe nanocomposite. Secondly, we studied the effect of metal-dielectric Ag-SiO₂ core-shell on lasing properties (luminescence power dependence and relaxation dynamics) of CdTe QDs.

2. Experimental

2.1 Materials and Reagents. The chemicals used for synthesis were: silver nitrate (AgNO₃, 99.8%, SIGMA-ALDRICH), Sodium citrate (Na₃C₆H₅O₇·7H₂O, MOISTURE), Ethanol (C₂H₅OH, ADWIC), Ammonia (NH₃, 33 %, ADWIC), Tetraethylorthosilicate (TEOS, 98%, FLUCKA), Sodium borohydride (NaBH₄, 97%, ALPHA), Tellurium powder (Te, 99.8%, ALDRICH), Cadmium chloride (CdCl₂·2.5H₂O, 99%, WINLAB), 3-mercaptopropionic acid

(MPA, 98%, MERCK), 3aminopropyltriethoxysilane (APS, 99% , SIGMA-ALDRICH), Hydroxide sodium (NaOH,98% , RIEDEL-DE HAEN). All the used chemicals were of the highest purity available. For aqueous solution, double distilled water was used.

2.2 Experimental procedures

2.2.1 Synthesis of Ag NPs

Colloidal Ag NPs solution was synthesized by reducing AgNO_3 with sodium citrate in an aqueous solution [11]. In details, 1.7 mM of AgNO_3 was dissolved in 200 ml of water with stirring and solution was heated to boiling point then 10ml of 1% tri-sodium citrate was added drop wise with stirring until it became pale yellow where left to cool with stirring for one hour.

2.2.2 Preparation of Ag-SiO₂ core shell NPs

Ag-SiO₂ core shell was prepared following Stöber method [12] where 1.67mM of colloidal Ag NPs solution was stirred with 0.718 M of ethanol and 24 mM of ammonia. Ethanol and ammonia were added to activate the surface of Ag NPs. TEOS was then added and the mixture was stirred for several days. The formed thickness of silica shell depends on relative concentration of TEOS, ammonia and Ag NPs.

The transparent dielectric Silica shell around NPs has the potential to modify and stabilize the plasmonic properties [13] with many possibilities for surface modification. Coating with silica in a seeded growth process decreased the poly-dispersity of NPs and reduces Van Der Waals attraction, so the colloid stability and ability to form colloidal crystals enhances. It also increases the mechanical stability, makes possible transfer into organic solvents, provides a capping layer on the semiconductor nanocrystals and protects metal particles against oxidation.

2.2.3 Preparation of CdTe QDs

Aqueous colloidal solution of CdTe QDs was prepared in the reaction between Cd^{+2} and NaHTe solution in presence of MPA as a stabilizer [14]. NaHTe was first prepared as a precursor of Te by heating a mixture of Te powder (0.3 M) and NaBH_4 (0.6 M) with deionised water (10 mL) at 60° C in a water path for 30 minutes. Hydrogen was released through a small outlet of the tube where white precipitation of sodium tetra-borate was formed. The color of NaHTe solution was violet and NaHTe was separated using a filter paper. The Cd^{+2} precursor was prepared by mixing

$\text{CdCl}_2 \cdot \text{H}_2\text{O}$ solution with 3-mercaptopropionic acid (MPA) at adjusted pH of 7.5 using 1M of NaOH. Then, Cd^{+2} /MPA and NaHTe precursors were mixed at molar ratio 1:2.4:1. Equal volumes of this mixture were exposed to microwaves at different powers. Finally, CdTe QDs were mixed with isopropanol and centrifuge at 5000 rpm to precipitate.

2.2.4 Preparation of hybrid Ag-SiO₂-CdTe nanocomposite

To connect CdTe NPs with Ag-SiO₂ NPs, the surface of silica shell was firstly activated. This was done by modifying the surface of silica shell with a layer of APS to decrease their distance to CdTe QDs (the effective parameter in energy transfer). This layer was formed by adding 25 μl of APS (1 mM) to Ag-SiO₂ NPs (1mM) with stirring for 15 minutes. CdTe NPs was then added to Ag-SiO₂-APS at molar ratio 50:1 and the mixture was stirred at 500 rpm for 12 hours. Silanol group (Si-OH) of APS was connected to silica layer so the surface of Ag-SiO₂ core-shell becomes active with amino group. Amino group of modified surface attracted to COOH of CdTe QDs, so CdTe QDs were self-assembled on the core-shell surface forming hybrid Ag-SiO₂-CdTe nanocomposite.

2.3 Physical characterization

2.3.1 X-ray diffraction measurements

Crystalline structure of the samples was investigated using XRD powder diffractometer (XPRT-PRO-PAN analytical-Netherland) operating with Cu anode. The samples were precipitated using 2iso-propanol and washed with water then translated on a glass substrate for investigation.

2.3.2 High Resolution Transmission Electron Microscopy (HR-TEM) and Energy Dispersive X-Ray Spectroscopy (EDX)

HR-TEM, (Tecnai G20, FEI, Netherland) and EDX were used for the purpose of imaging, crystal structure revelation and elemental analysis. Two different modes of imaging were employed; the bright field at electron accelerating voltage 200 kV, using lanthanum hexaboride (LaB₆) electron source gun, and the diffraction pattern imaging. Eagle CCD camera with (4k*4k) image resolution was used to acquire and collect transmitted electron images. TEM Imaging & Analysis (TIA) software was used for spectrum acquisition and analysis of EDX peaks. A drop of a very

dilute sample solution was deposited on an amorphous carbon-copper grid and left to evaporate at room temperature.

2.3.3 Laser Induced Fluorescence (LIF) with temperature control

LIF technique was applied to measure the fluorescence of the prepared NPs samples. Argon laser of 488nm was used to excite the samples at low temperatures (50K) and fluorescence was detected using a spectrometer. A schematic diagram of LIF experimental setup is shown in Figure 1.

2.3.4 Nanosecond relaxation dynamics

Lifetime measurement of excited levels in fluorescent medium is important for lasing properties. In the present measurements, the excitation source was pulsed N₂ laser (337 nm wavelength) of 800 ps pulse duration and maximum pulse energy of 2 mJ. Laser was focused as a line on the samples by a cylindrical lens. Emission was collected by a lens on a fast photo-tube connected to an oscilloscope (300 MHz) of 1 nsec resolution time.

Curve fitting of the measured time evolution data were carried out using *Orgin Lab vr 8*, applying either first or second order exponential decay with offset fitting equations:

$$y = A_1 e^{\frac{-t}{\tau_1}} + y_0 \quad \text{Equation 1}$$

$$y = A_1 e^{\frac{-t}{\tau_1}} + A_2 e^{\frac{-t}{\tau_2}} + y_0 \quad \text{Equation 2}$$

3. Results and discussion

The formation and structure of the prepared NPs were first investigated using TEM and XRD techniques. Figures 2a, 2b, 2c and 2d presented TEM images of Ag NPs, Ag-SiO₂ NPs core-shell, CdTe QDs and hybrid Ag-SiO₂-CdTe nanocomposite, respectively. Figure 2a shows Ag NPs with average diameter of nearly 30 nm. In Figure 2b, the coated thin layer of silica around Ag NPs appeared to be of an average thickness of 8 nm. Figure 2c shows the prepared CdTe

QDs with average diameter of 3 nm. Figure 2d shows TEM image of hybrid Ag-SiO₂-CdTe nanocomposite where CdTe QDs are self-assembled around the surface of Ag-SiO₂ core-shell.

Figure 3 shows X-ray diffraction at various formation stages of hybrid Ag-SiO₂-CdTe nanocomposite. XRD revealed cubic crystal structure of Ag NPs. It characterized by four distinct features at $2\theta = 38.1^\circ$, 44.2° , 64.4° and 77.4° which attributed to reflection at planes (111), (200), (220) and (311), respectively [15]. Silica shell revealed α -quartz hexagonal structure characterized by distinct feature at $2\theta = 26.7^\circ$ due to (011) reflection [16]. CdTe QDs displays a Zinc blende cubic structure characterized by a feature at $2\theta = 25^\circ$, due to (111) reflection, two broad features at $2\theta = 40^\circ$ and 47° due to (220) and (311) reflections, respectively, and a shoulder at $2\theta = 30^\circ$ due to plane (200) [17]. XRD of Ag-SiO₂-CdTe hybrid nanocomposite displays combined features of all Ag NPs, silica shell and CdTe QDs.

Figure 4a compares the absorption spectrum of Ag-SiO₂ core-shell to that of Ag NPs, at 1mM of Ag NPs. Ag-SiO₂ core-shell shows higher absorption intensity more than 31% with broadening of 11 nm in full width at half maximum (FWHM). These enhancements were attributed to the presence of dielectric silica shell layer which has the potential to modify the plasmonic properties [13].

Figure 4b compares the absorption and photoluminescence (PL) of hybrid Ag-SiO₂-CdTe nanocomposite to those of CdTe QDs at constant concentration of the active fluorescent (3 mM of CdTe QDs). Comparisons was carried out in reference to the standard diluted fluorescein dye (0.1 M NaOH(aq)) which displays high quantum yield (0.925 ± 0.015) [18] in the same spectral region (curves 1 and 2 in Figure 4b). The figure showed ~31% enhancement in the absorption of hybrid Ag-SiO₂-CdTe nanocomposite compared to that of CdTe QDs. This enhancement in the absorption rate of CdTe NPs was attributed to propagation of LSP on the surface of Ag-SiO₂ core-shell [19,20]. Also, an enhancement by ~200% was reported for the luminescence of hybrid Ag-SiO₂-CdTe nanocomposite compared to that of CdTe QDs. This luminescence enhancement was attributed to the presence of silica shell which allow for energy transfer from Ag NPs core to

CdTe QDs and prevents electron transfer from CdTe QDs to Ag NPs core [19,20]. Also, fluorescence spectra displayed Stokes shift of the order of 21 nm associated with 3.6 nm narrowing in FWHM. This was also attributed to coupling between QDs and local SPs of metal (strong dipole-dipole interaction between plasmon and exciton coupling).

The quantum yield was calculated according to the following equation

$$Q_{NC} = \frac{I_{NC}}{I_{Dye}} \times \frac{OD_{Dye}}{OD_{NC}} \times Q_{Dye} \quad \text{Equation 3}$$

Where, I is the integrated emission spectrum and OD is the optical density for the dye and nanocrystals (NC). The quantum yield of both CdTe QDs and hybrid Ag-SiO₂-CdTe nanocomposite were estimated to be 47% and 73%, respectively. The enhancement in quantum yield value can be interpreted in reference to Figure 5 as in the next paragraph.

Figure 5 presents a schematic energy diagram for the physical processes; absorption, radiative and non-radiative decays and plasmon transfer between luminescent CdTe and Ag NPs in presence of silica shell barrier. The diagram shows an incident radiation at wavelength λ_{ex} excites CdTe QDs at excitation decay rate γ_{ex} and absorbed by Ag NPs at absorption rate γ_{abs} . The latter generates plasmon modes propagated through silica shell barrier towards CdTe QDs. In case of in-resonance, plasmon field enhances γ_{ex} which in-turn enhances the radiative decay rate γ_r (fluorescence) [19,20]. It is worth mention that, excitation decay rate (γ_{ex}) depends on excitation wavelength λ_{ex} , nano-metal size and shape, and on plasmon strength which is a function of distance between Ag NPs and CdTe QDs [20]. The figure also shows the emitted photons from CdTe QDs, at wavelength λ_{em} , which transfer to Ag NPs through silica barrier leading to Plasmon enhancement. In addition, CdTe quantum yield changed according to; the change in radiative decay rate (γ_r), intrinsic non radiative decay (γ_{nr}) and AgNPs absorption rate (γ_{abs}). γ_r increased with plasmon field strength but as γ_{abs} depends on λ_{em} therefore re-absorption is maximized if λ_{em} is in-resonance with the peak band of Ag NPs (422 nm) [20]. This induced a reduction in γ_{nr} and increases the γ_r which in-turn enhances the net quantum yield according to the formulae;

$$Q = \frac{\gamma_r}{\gamma_r + \gamma_{nr}} \quad \text{Equation 4}$$

Stimulated emission measurements were carried out for both CdTe QDs and hybrid Ag-SiO₂-CdTe nanocomposite samples at constant concentration of CdTe QDs. The samples were embedded in PVA polymeric films to carry out measurements at low temperature (50 K) without changing the transparency of the samples. Measurements were performed at 50K to reduce thermal effects such as; auger process, surface trapping, and phonons contributions. The samples were excited by 488 nm of Ar laser at different powers (0.1mW to 50 mW). This wavelength located within the absorption spectral range of both CdTe QDs and Ag-SiO₂ NPs (in-resonance). The measured PL-power dependent data for both CdTe QDs and hybrid Ag-SiO₂-CdTe nanocomposite are presented in Figure 6a and 6b, respectively. The figures showed a fast increase in PL intensity upon pumping by powers >20 mW for CdTe QDs and >10 mW for hybrid Ag-SiO₂-CdTe nano-composite. The increase in intensity accompanied with a narrowing of FWHM from 38 nm to 27 nm for CdTe QDs and from 47 nm to 33 nm for hybrid Ag-SiO₂-CdTe nanocomposite. The fast increase in PL indicates the onset of ASE. The reported reduction in ASE threshold power of hybrid Ag-SiO₂-CdTe nano-composite than that of CdTe QDs was attributed to presence of Ag-SiO₂ core-shell surface plasmon.

Figures 7a, 7b and 7c show normalized PL of hybrid Ag-SiO₂-CdTe nanocomposite embedded in PVA polymeric films. PL was detected at 50 K on excitation by Ar laser at 488 nm. Figure 7a compares PL due to excitation at a low pump power; 1 mW (red curve) with a little higher excitation power; ~11 mW (black curve), very close to ASE threshold power. PL at 1 mW (red curve) shows a single symmetric peak, from exciton state only. PL at ~11 mW (black curve) shows a red shifted non-symmetrical peak with broadening in FWHM which attributed to emission from both exciton (X) and biexciton (XX) associated to 1Se -1Sh transition.

At low pump powers, the formation probability of X (e-hole pair) is higher than that of multi-exciton (more than one exciton per dot). As pump power increases, formation of multi-exciton became more probable [21].

Figure 7b compares PL due to excitation at ~11mW (ASE threshold power) to that at a higher power ~25 mW (red and black curves, respectively). PL at the higher excitation is red shifted by

~ 4 nm (~ 17 meV) with a narrowing in FWHM. PL of XX is red shifted about PL of X by 4 nm (17 meV) in relation with binding energy of XX [22,23]. The accompanied reduction in FWHM (~ 11 nm) attributed to overlapping between ASE from XX state with the residual spontaneous emission of X state [22]. This overlapping was examined in Figure 7c by fitting the PL data using Pseudo-Voigt function. The fitting shows two symmetric peaks due to exciton (X) (red curve) and biexciton (XX) (green curve) where intensity due to XX is 4 times higher than that of residual spontaneous emission from X state.

The ASE measurements at 50K were repeated for both CdTe QDs and Ag-SiO₂-CdTe nanocomposite using a diode laser of 405nm. The results are presented in Figures 8a and 8b, respectively. This excitation wavelength located in-resonance with the absorption peak of Ag-SiO₂ core-shell but off-resonance with absorption 1S_h-1S_e state of CdTe QDs. The measurements yielded ASE threshold powers of 26 mW for CdTe QDs (inset of Figure 8a) and 36 mW for hybrid Ag-SiO₂-CdTe nanocomposite (inset of Figure 8b). These threshold powers are higher than those obtained on excitation by 488nm (20 mW and 10 mW, respectively). The increment in threshold powers was nearly 30% for CdTe QDs and 260% for hybrid Ag-SiO₂-CdTe nanocomposite. These increments were attributed to the following; in case CdTe QDs, excitation was to higher states (2S, 2P, ...) which decayed non-radiatively to 1S state, while in case of hybrid nano-composite, excitation wavelength mismatches the resonance of exciton-plasmon interaction, so most absorbed energy by Ag-SiO₂ is dissipated non-radiatively.

ASE of QDs is related to population inversion which achieved when the density of electron-hole pair is more than 1.4 [24]. This means that for above threshold power, biexciton state formed in the majority of the particles. The life-times of X and XX in CdTe QDs and hybrid Ag-SiO₂-CdTe nanocomposite are studied in Figure 9. The fluorescence decay lifetimes were measured using pulsed N₂ laser (337 nm, pulse duration 800 ps). As the lifetime depends on formation of exciton and biexciton, therefore excitation was obtained at two different pump energies; 0.1mJ and 0.7 mJ. At lower pump energy, probability of forming a single X is higher than that of XX; therefore lifetime is related to exciton formation only, so equation 1 was applied.

At higher pump energy, the probability of biexciton formation increased, therefore fluorescence lifetime is due to formation of both exciton and biexciton. Figure 9 presents the experimentally

measured lifetimes at low and high pump energies (symbols) together with their fitted curves using 1st and 2nd order exponential decays equations, respectively. For both CdTe QDs and hybrid Ag-SiO₂-CdTe nanocomposite, lifetimes were calculated to be typically 13 nsec for exciton and 2 nsec for biexciton.

Conclusion

In this work, we synthesized hybrid Ag-SiO₂-CdTe nanocomposite to optimize the ASE of CdTe QDs. Synthesis was carried out via three stages. Firstly, CdTe QDs was prepared by microwave-assisted organic aqueous method. Secondly, Ag NPs was synthesized by reducing AgNO₃ with sodium citrate in an aqueous solution then coated by a thin layer of dielectric silica to modify and stabilize the plasmonic properties. Silica shell was formed by hydrolysis and condensation of TEOS with ammonia. The surface of Ag-SiO₂ core-shell was treated with APS to decrease the distance to CdTe QDs and to enhance energy transfer. CdTe QDs were self-assembled on Ag-SiO₂ core-shell surface, yielding the hybrid Ag-SiO₂-CdTe nanocomposite.

Characterizations of structural and optical properties were obtained by XRD, TEM image, absorption and emission spectra. The results confirmed the formation NPs at different stages of hybrid nanocomposite synthesis. The transparent silica layer around Ag NPs enhances the LSP and prevents electron transfer from CdTe QDs to Ag NPs. This led to enhance luminescence and quantum yield of CdTe QDs. In addition, LSP decreased the ASE threshold power of CdTe QDs gain medium by 50% upon excitation by a wavelength in-resonance with absorbance of plasmon-exciton coupling. Otherwise, on pumping by an off-resonance wavelength, LSP increases the ASE threshold power of CdTe QDs gain medium. In addition, on pumping by an above threshold power, reduction was observed in FWHM of both CdTe QDs and hybrid Ag-SiO₂-CdTe nanocomposite (~10 nm and ~14 nm, respectively). Lifetime measurements of exciton and biexciton yielded 13 nsec and 2 nsec, respectively.

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Figures Caption

Figure 1	setup of laser induced florescence (LIF)
Figure 2	TEM at scale bars 20 nm of a) Ag NPs, b) Ag-SiO ₂ NPs core-shell silica shell thickness is 8 nm. c) CdTe QDs with average nano-size 3 nm. d) hybrid Ag-SiO ₂ -CdTe nanocomposite.
Figure 3	XRD of: Ag NPs, Ag-SiO ₂ core- shell NPs, CdTe QDs and hybrid Ag-SiO ₂ -CdTe nanocomposite.
Figure 4	a) Absorbance spectra of Ag NPs (black line) and Ag-SiO ₂ core-shell NPs (red line) at same molarity of Ag NPs. b) Absorbance and fluorescence spectra of; fluorescein dye (curves 1 and 2), CdTe QDs (curves 3 and 4), and hybrid Ag-SiO ₂ -CdTe nanocomposite (curves 5 and 6). Excitation wavelength is 405 nm
Figure 5	Schematic energy diagram for physical process: excitation (blue arrow); radiative decay (red arrow); non-radiative decay and plasmon transfer (axial arrow) between Ag NPs and CdTe QDs; excitation wavelength (λ_{ex}) excited electrons of Ag NPs to oscillate generating electric fields (plasmon modes) which propagate and transport in silica layer to CdTe QDs causes enhancement in radiative decay rate (γ_r) of CdTe QDs and absorption rate (γ_{abs}) and decreased the non-radiative decay rate (γ_{nr}). Silica shell prevent electron transfer from CdTe QDs to Ag NPs.
Figure 6	Low temperature (50 K) PL for a) CdTe QDs and b) hybrid Ag-SiO ₂ -CdTe nanocomposite, embedded in PVA polymeric films. Inset: power dependence curve of PL intensity and FWHM. Excitation wavelength was 488 nm.
Figure 7	Normalized PL of hybrid Ag-SiO ₂ -CdTe nanocomposite embedded in PVA polymeric film and detected at 50 K on excitation by 488 nm at powers: a) 1 mW and at ASE threshold power (~11 mW), b) ~11 mW and above threshold power of ASE (~25 mW) c) Fitting of PL for (~25 mW) using Pseudo-Voigt function.
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