Ternary alloyed HgCdTe nanocrystals for short-wave and mid-wave infrared region optoelectronic applications

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Abstract
Semiconductor quantum dots (QDs) are emerging as the forefront alternative for the conventional imaging technology, particularly in infrared region from near infrared (0.75–1.4 µm) to long-wave infrared (8–14 µm) region. A handful of materials are explored for mid infrared imaging QDs and they are all invariably binary semiconductor compounds. Ternary alloyed quantum dots in many previous cases have shown properties that are unique and better than parent binary compounds. In this work, we have synthesized ternary alloyed HgCdTe quantum dots and studied their photophysical properties. Previously studied ternary alloyed HgCdTe CQDs absorb and emit in regions limited up to near-infrared region. We have tuned the excitonic absorption of HgCdTe QDs in the range of 2.2–5 µm, where addition of cadmium clearly showed blueshift in excitonic peak as compared to that of HgTe QDs. Structural properties are studied by TEM, XRD & XPS techniques. Electrical behaviour is studied by measuring I-V, I-V-T curves. Photodetectors are fabricated in photoconductive geometry showing promising photo-response under visible (532 nm) and NIR (810 nm, 1550 nm) excitation. Responsivity of the devices is in the order of 1 mA W⁻¹ at 1 V bias and show good linearity over irradiance range of 0.025 and 2.5 W cm⁻². These results pave the way for development of next generation cost-effective short-wave and mid-wave infrared region optoelectronic devices based on narrow bandgap HgCdTe nanocrystals.

1. Introduction
Colloidal semiconductor quantum dots (QDs) have been receiving major research efforts for the past two decades towards reaching a performance that is competitive to their bulk semiconductor counterparts in a range of applications. The optoelectronic devices such as photodetectors, solar cells, field effect transistors, LEDs, thermoelectrics etc enjoy the multitude of advantages offered by the QDs. QDs come with important benefits such as simplified device fabrication and cost reduction.

Despite that silicon rules over the commercial spectroscopy and imaging technology in the visible region, it cannot access wavelengths longer than 1100 nm. For short(SWIR, 1–2.7 µm), mid(MWIR, 3–5 µm), long (LWIR, 8–14 µm) wavelength infrared regions, PbS, PbSe, HgCdTe, InSb and epitaxial superlattices are predominantly employed for their eminent performance [1, 2]. Considering bulk and thin film form, HgCdTe is one of the most important materials in the field of entire infrared (near IR to far IR) photodetectors, due to its widely tunable bandgap from 1.44eV (Bulk CdTe) to −0.3 eV (bulk HgTe). However, focal plane array based IR imaging devices made from these materials suffer from issues like high cost of device-quality material growth, complex steps for integrating these crystals to focal plane arrays by bonding techniques, limitations in pixel resolution, subsequent sacrifice in performance, additional requirements for thermal cooling [1, 3, 4]. These limitations restrict the domain of IR imaging largely to defense and research. Coming up with cheaper
alternative materials that use simpler integration techniques would open up a window of opportunities for a wide range of important applications in everyday life including medicine.

Colloidal quantum dots have shown a host of advantages as they can be readily deposited on a variety of substrates with high packing density by basic deposition methods [3, 6], their spectrum can be tuned broadly [7], devices can be fabricated at low-cost at room temperature, with a huge decrement in complexities [8, 9]. At present the colloidal QDs of HgTe, HgSe, HgS, Ag2Te have been explored for absorption in MWIR region [10–12]. HgTe CQDs was the first material to be reported and subsequently studied widely in MWIR and LWIR region [11, 13, 14]. Improvements with doping has made it reach impressive levels of performance with detectivity approaching that of InSb single crystals [3, 10]. They may be expected to make entry into commercial applications soon.

In this work, we have explored ternary alloyed HgCdTe QDs for the MWIR region. Apart from wide bandgap tunability, the lattice constant values of HgTe and CdTe are very close leading to a very low lattice mismatch at 0.308%. Along with this, a total miscibility of the two materials in solid phase gives way for a high-quality HgCdTe growth [15]. Moreover, alloying HgTe with Cd hardens the lattice structure and could improve the strength and stability of the material [16].

Ternary alloying presents with a new degree of freedom to control the bandgap continuously by varying the composition independent of the crystal size-tuning. It means, for a given QD size, a desired absorption wavelength can be targeted from among a wide spectrum which are sometimes not accessible for parent binary material [17]. Alloying can also be important for manipulating the opto electronic properties by controlling the localised defect states, conduction band offsets, carrier concentration etc [18]. Compositional tuning could often lead to distinctly different properties from binary materials and often enhances the performance of the material [19, 20]. Instances of improved thermoelectric performances [18], higher photovoltaic power conversion efficiency [21] serve as examples for this idea.

So far, there are no ternary alloyed HgCdTe QDs reported in the MWIR region except our another work [22]. Nevertheless, there are multiple works available on HgCdTe nanocrystals for the near IR region. The previous attempts to synthesise HgCdTe are largely intended for biomedical applications and absorption region fall below <2 μm [9]. The attempts to achieve activity in MWIR and LWIR region were unsuccessful possibly due to the aqueous synthesis protocol that is mainly followed. Herein, we have synthesised HgCdTe QDs showing absorption from SWIR to MWIR(2.2–5 μm) by using the strategy of nonaqueous synthesis protocol. The nanocrystals have a cubic zinc blend structure and monodisperse tetrapod shapes analogous to the parent HgTe QDs. Photoconductor devices are fabricated from the QDs and their photocurrent behaviour is investigated.

2. Experimental methods

2.1. Chemicals

Mercury chloride (HgCl2, Thermo Fisher, 97%), Cadmium Chloride (CdCl2, Thermo Fisher, 95%) tellurium pieces (Te, Sigma-Aldrich, 99.999%), trioctylphosphine (TOP, Sigma Aldrich, 90%), oleylamine (Otto Chemie, 90%), dodecanethiol (DDT, Sigma-Aldrich, 98%), 1,2-ethanedithiol (EDT, Fluka, 98.0%), hydrochloric acid (HCl, SDFCL, 25%), ethanol absolute anhydrous (SDFCL, 99.9%), methanol (SDFCL, 99.8%), acetone (SDFCL, 99.8%), toluene (SDFCL, 99.5%). Mercury compounds are highly toxic. Handle them with extreme care.

2.2. HgCdTe QDs synthesis

HgCdTe colloidal quantum dots are synthesized by modifying the method which was introduced by Keuleyan et al. [23] for HgTe nanocrystals. Tellurium is dissolved in TOP solution to prepare a 0.5 M TOP-Te solution. Tellurium pellets are gently stirred in TOP at 60 °C for 6 hrs followed by stirring at room temperature for 18 h under N2 gas flow. A clear yellow solution obtained is centrifuged to settle down any undissolved particles and the upper part of the solution is used. The solution can be kept for a month to use in multiple syntheses. For a typical QD synthesis, 36 mg of HgCl2 and a measured amount of CdCl2 are dissolved in 4 ml of oleylamine under nitrogen gas flow. CdCl2 is added in different molar percents varying from 10% to 40% in each synthesis. The HgCl2 and CdCl2 salts are constantly stirred in oleylamine at a temperature of 120 °C for an hour to ensure complete dissolution. Then the temperature is brought down to 80 °C to inject 0.2 ml of 0.5 M TOP-Te solution mixed in 1 ml of oleylamine. The samples are grown for desired time intervals varying from 10 to 60 min while maintaining at 80 °C. The growth is stopped by quenching per ml of the reaction mixture with a solution of 0.9 ml toluene, 0.1 ml DDT, 40 μl TOP. Later the quantum dots are washed with methanol to remove the unreacted precursors. Thus washed QDs are dispersed in toluene at a concentration of 50 mg ml⁻¹ for further use in making devices.
2.3. Optical & structural characterisation
Fourier-transform infrared spectroscopy (FTIR) and x-ray powder diffraction (XRD) measurements are performed by Perkin Elmer Frontier and Rigaku Smartlab respectively, on the QDs drop-cast as a film. Transmission electron microscopy (TEM) imaging is carried out by FEI Tecnai G2 T20 on samples dispersed on a copper grid. X-ray Photoelectron (XPS) spectra are obtained from Kratos Axis Ultra DLD under the condition of 13 mA emission current and 12 kV as accelerating voltage (156 W). Samples for XPS analysis are prepared by spin coated films on silicon substrate followed by vacuum drying.

2.4. Photocapacitor device fabrication
The QDs present as such are insulating due to their long chain ligands and are to be exchanged with short length EDT. This is a standard step employed in many QDs based devices. The devices are built by drop casting the as-prepared-QD solution on 40 nm thick gold interdigitated electrodes. The electrode pattern contains 50 pairs of finger electrodes that are 2.5 mm wide and has a 10 micron spacing. The drop-casted solution is held on a slanting level to evenly dry out followed by standard EDT ligand exchange procedure. It involves dipping the film in a solution of 2% EDT-ethanol with 1% HCl for 90 s and further rinsing in ethanol for 30 s. After repeating the procedure for five such layers, the resistance of the film dropped down to the order of 25 kΩ. The device is vacuum dried before making ready for further measurements.

2.4.1. Fabrication of interdigitated electrodes
Au interdigitated electrodes are fabricated on SiO2(400 nm thick)/Si wafer by method of standard optical lithography. AZ5214 Photoresist is spin coated on a cleaned SiO2/Si wafer at 2000rpm for 40 s. The photoresist film is baked at 110 °C for 90 s and then exposed to UV light through the photomask for an optimised 2 s.

Another baking at 125 °C for 2 min inverts the photoresist. Then, a 40 s flood exposure is carried out following which the film is developed with AZ726 developer for 32 s. Now 99.999% Au wire is thermally evaporated on the patterned photoresist to 40 nm thickness under a vacuum of 10⁻⁶ torr. Finally, the photoresist film is lifted off by immersing in acetone bath for an hour.

2.5. Electrical & photo-response measurements
The electrical measurements are performed using Keithley Sourcemeter 2450. A lab-built cold finger cryostat housed the sample for low-temperature I–V measurements. Light sources used for different incident wavelengths are a 532 nm laser diode, 810 nm LED (ELJ-810–228B), and a 1550 nm LED (Thorlabs LED1550L).

3. Results and discussions

3.1. FTIR
When a range of light wavelength is incident on the QDs, the onset of light absorption happens when the photon energy is equal to or greater than the band gap. The photon absorption in QDs results in the creation of a bound electron hole pair called an exciton. The excitonic absorption peak position in FTIR spectra is a direct measure of the band gap of the QDs. When a range of light wavelengths is incident on the QDs, the onset of light absorption happens when the photon energy is equal to or greater than the band gap. The photon absorption in QDs results in the creation of bound electron hole pairs, which can be observed as excitonic peaks in the FTIR spectra.

Figure 1(a) shows the absorption spectra of QDs synthesized with different quantities of the Cadmium precursor i.e. 20%, 30%, 40% by molar weight of HgCl₂. Upon progressive addition of CdCl₂ salt into the synthesis mixture, the QDs show an increasing blue shift in the excitonic peak position which indicates the formation of alloyed Hg₁₋ₓCdₓTe QDs [25, 26]. The bandgap of bulk HgTe is ~0.15 eV and that of CdTe is 1.5 eV. According to band structure calculations based on Effective mass Approximation model, when Hg²⁺ is substituted by Cd²⁺ in HgTe, the bandgap energy increases as the conduction band shifts to higher energy and the valence band to lower energy [27]. Correspondingly, the excitonic peak position of Hg₁₋ₓCdₓTe also gradually blue shift towards the shorter wavelengths (or equivalently, higher photon energies) with increasing ‘x’ as higher photon energy would be required to create the exciton.

To empirically express the energy gap Eₓ as a function ‘x’, several equations have been developed the most widely used being that of Hansen et al. [28].
Thus, bandgap tuning is achieved by composition control as well as size control in the synthesized QDs.

3.2. TEM and XRD

X-ray diffraction spectra are studied to understand the crystal structures of the samples. Figure 2 shows the TEM images and XRD spectrum of HgCdTe QDs. The presence of distinct reflection peaks in figure 2(d) indicate the high crystallinity of the QDs. The $2\theta$ positions of the three dominant peaks are $24^\circ$, $39.7^\circ$, $40.7^\circ$ corresponding to $(111)$, $(220)$, $(331)$ lattice planes of cubic zinc blend structure of HgTe and CdTe $[29, 30]$. The characteristic peaks of HgTe and CdTe both lie very close to each other as referred from JCPDS database. This makes it difficult to remark on the alloying of HgTe with CdTe on the basis of the XRD peaks.

From the FWHM of the $(111)$ peak, the mean sizes of the crystals are calculated using the Debye–Scherrer equation $[31]$. The crystals grow from 8.85–11.85 nm with the growth time varying from 2–60 min.

TEM images suggest the shapes of QDs to be uniform and well defined with a good monodispersity of size. The nanocrystals are present as triangles or irregular parallelograms which can be more or less counted as tetrapod shape. A zinc blende material naturally develops into a tetrahedral shape when the different facets are growing at different rates $[23, 32]$.

The d-spacing value of 0.37 nm measured from the HRTEM images agrees well with the spacing of $(111)$ lattice planes of HgTe & CdTe zinc blende structure $[33]$. The apex-to-opposite edge length of the triangle is around 8.66 nm for QDs grown for 2 mins and 11.31 nm for those grown for 60 min. These numbers are consistent with the QD sizes obtained from the Debye–Scherrer equation. Also, QDs of same time period of growth are found to have almost same size despite progressive addition of Cd content. In contrast, their corresponding FTIR excitonic absorption peaks are blue shifted gradually as seen in figure 1(b). This directly verifies that the bandgap of the QDs are increased upon Cd addition due to alloying.

3.3. XPS

Figure 3 shows the wide-scan XPS spectra and the slow-scan of Hg, Cd, Te regions of the synthesized QDs. For the purpose of comparison, QDs are also synthesized without adding CdCl$_2$ and its XPS spectrum is given for reference along with HgCdTe QDs. Characteristic Cd peaks are clearly recorded in HgCdTe QDs at their characteristic binding energy values of 405 eV and 412 eV corresponding to Cd 3d$_{5/2}$ and 3d$_{3/2}$ peaks respectively. HgTe QDs show a marked absence of peaks at this energy position.

Atomic percentage of Hg, Cd present in QDs can be estimated by analysing their corresponding Hg4f, Cd3d peaks. Different batches of HgCdTe QDs synthesised with different starting amounts of Cd precursor salt are studied and their atomic composition is tabulated in table 1. The trend show that the ratio of Cd incorporated into crystal lattice increases with the increasing addition of Cd precursors. Further, the ratio of overall cation to anion stoichiometry is found to be around 70:30 indicating the excess of cations. A previous report also

$$E_g = -0.302 + 1.93x - 0.81x^2 + 0.832x^3 + 5.35(1 - 2x)10^{-4}T$$ (1)
Figure 2. (a), (b), (c) TEM images of the QDs (d) X-Ray diffraction pattern of the QDs.

Figure 3. XPS elemental analysis of HgTe and HgCdTe QDs. Wide scan spectra (a), XPS peaks in the regions of Hg 4f, Cd 3d, Te 3d of HgTe QDs (b), (c), (d) and HgCdTe QDs grown for 10 mins (e), (f), (g).
mentions about excess of cations in the case of HgTe QDs which may occupy the surface and bond with ligand molecules creating surface trap states [34].

In addition to the two main peaks, two more chemically shifted peaks of tellurium are present at 577 eV and 587 eV suggesting the possible formation of tellurium oxides and halides [15, 35]. More careful handling of materials and reaction synthesis under highly inert ambience is essential.

3.4. Photocurrent measurements

The dark current is important as it forms the major noise of the photodetector. The figure 4(a) shows the current density-voltage characteristics of the devices made with the HgCdTe films. HgCdTe films with long chain native ligands of DDT exhibit megaohms of resistance. Hence they are exchanged with shorter EDT through the standard ligand exchange procedure, which reduces the resistance of the films to 25 kΩ marking a good efficiency of exchange. The thiol end groups in EDT interpenetrate each other and cross link the neighbouring QDs thus pulling the QDs closer leading to improved conductivity [36]. The EDT treated films show 3 orders of magnitude higher current density compared to untreated films and show a symmetric, linear current response with applied bias indicating ohmic contacts. This is in contrast to the non-ohmic poor conducting behaviour of the DDT films as given in the inset of figure 4(a). For photodetector applications, reducing the dark current noise is important for device performance to be sensitive to weak light signals. Photovoltaic architecture tend to have an advantage in this aspect because of the presence of injection barrier [3, 37]. Here in case of photoconductor architecture, cooling the system down to low temperatures, the dark current can be reduced [5]. Inset of figure 4(b) shows the dark I–V curves in lower temperatures. It can be seen that the ohmic response is maintained throughout low temperatures upto 100 K accompanied by an exponential decrement in magnitude of current. This transport characteristic is explained by thermally excited charge carriers that hop from one isolated state to the next. The exponential drop I(T) is plotted in the figure 4(b) and an Arrhenius fit at room temperature gives a weak thermal activation energy of 49meV. This value is smaller than half the bandgap energy 240 eV of the material and thus does not correspond to thermal activation of intrinsic charge carriers [38]. Rather, it could be the energy required for the extrinsic carrier population from shallow impurity states.

Table 1. Atomic concentration of HgCdTe QDs.

| Molar % of Cd added in initial reaction mixture | Atomic % incorporated |
|-----------------------------------------------|-----------------------|
| No Cd added                                   | 100% 0                |
| 10%                                           | 93.5% 6.5%            |
| 20%                                           | 89.6% 10.4%           |
| 30%                                           | 78% 22%               |

Figure 4. (a) Dark I–V characteristics of the device (inset: Dark I–V characteristics in linear scale). (b) Dark I–T curve and Arrhenius Fit (inset: Dark I–V w.r.t. temperature.)
On account of the quantum confinement effects, the absorption edge can be tuned over a wide range of wavelengths resulting in a wide spectral response. The spectral sensitivity of the QDs in visible, IR, near IR are tested by measuring the photoresponse of the photoconductor under illumination of wavelengths 532 nm, 810 nm, 1550 nm all at same intensity 100 mW/cm² and applied bias of 1 V. The bandgap of the QDs used in the device is 0.5 eV and absorbs light over a wide range of wavelengths from 2500 nm downwards. Upon illumination, excitons are generated which further gets dissociated by the applied electric bias and the excess charge carriers transit through the device giving rise to photocurrent.

Under light irradiation, the devices maintain their linear I–V behavior but with reduced resistance as given in figure 5(b). Figure 5(a) shows the photocurrents measured in response to the 3 different wavelength LEDs that switch on and off periodically. The photodetector follow the light source and give repeatable and stable response.

The rise time and fall time of the photocurrent enable to get an idea about the speed of response of the device. Fitting biexponential curves over the transient decay of the photocurrent, the time constants of the fast and slow components are calculated as 32 ms and 377 ms. Existence of two components denote two recombination channels for the carriers with recombination happening through traps in the slow component \[34, 39\]. But the instrument has speed limitation and cannot capture response faster than 130 ms. Therefore, the value can be used only to understand that the device responds not slower than that.

Apart from gold, the QD film makes ohmic contact with ITO electrode as its work function is close to that of Au. It led us to fabricate another vertical structure of photoconductor device with ITO coated glass as bottom electrode and ultra-thin Au film (8 nm) as top electrode. A vertical structure has better conductivity compared to an in-planar structure as the separation between the electrodes is as thin as the QD film thickness. As an added advantage, this structure enables the device to be illuminated from either side as the ultra-thin Au film is semi-transparent. Cross-bar electrodes are employed in this case, so that they dont block the active device area with wire contacts. The dark I–V of the device is given in figure 6(a) and their photo-response to 810 nm LED illumination from either side is given in figure 6(b).

External Quantum Efficiency (EQE) is a parameter that characterises the photon-to-charge carrier conversion efficiency of the photodetector. It is the percentage ratio of the number of photo carriers produced in the external circuit to the number of photons incident on the device \[40\].

\[
EQE = \frac{J_{\text{light}} - J_{\text{dark}}}{J_{\text{light}}} \frac{hv}{eP_i}
\]  

(2)

Table 2 shows the EQE values of the photoconductor at different wavelengths under 1 V applied bias. The higher value for longer wavelength 1550 nm promises performance in infra red region.

For a given incident optical power, EQE increases with applied bias as implicitly inferred from the equation. The variation of EQE versus applied bias is shown in the figure 7(a). At lower bias voltages, the photocurrent increases non-linearly and after a particular point, it becomes linear. It could be understood on the basis of the energy required to dissociate the excitons generated by the incident light. Once the applied electric field is large enough to dissociate the exciton, the photocurrent tends to become linear \[41\].
Linearity in photosensitivity over range of excitation light power is another important parameter of a photodetector. The curve shown in figure 7(b) is measured by illuminating 810 nm light from 25 mW cm\(^{-2}\) to 2.5 W cm\(^{-2}\) at 0.5 V bias. The photocurrent density increases linearly over the 2 orders of magnitude of excitation. The corresponding responsivity values are also shown in the same graph and they remain almost unchanged. This is immediately seen from the responsivity relation, \( R \propto \frac{I_{ph}}{P} \). If the photocurrent \( I_{ph} \) depends linearly on light power, then the responsivity will be a constant value over the illuminated range.

In another approach, it is informative to consider the power law followed by the dependence of photocurrent on excitation power \( I_{ph} \propto P^\alpha \). Fitting the \( I_{ph} \) curve w.r.t \( P \) obtains the value of the exponent to be \( \alpha = 1 \) as shown in the inset of figure 7(b). This denotes a first-order or quasi-monomolecular recombination process which is dominant when the density of trapped charges is much more than free carrier density \([42, 43]\). It is in contrast to bimolecular process (bandgap recombination through radiative and non-radiative paths).
which $\chi = 0.5$ predominantly seen in trap-free insulators. A quick background to understand this argument is given by the equation of rate of change of free carrier density $n$ (assuming electrons as majority carriers): [44, 45]

$$ \frac{dn}{dt} = G - (N_t + n)nb $$

(3)

where $G$ is the rate generation of photocarriers, $N_t$ is density of trapped carriers, $b$ is the recombination efficiency factor. At steady-state,

$$ G = (N_t + n)nb $$

(4)

where the term $N_t + n$ is equal to the total density of holes available for recombination for the electrons as there are $n$ number of holes corresponding to the $n$ free electrons and another $N_t$ trapped holes. When $N_t \gg n$,

$$ G \approx N_t nb $$

(5)

Since $n$ is linearly related to current, this results in linear dependence on excitation power. The high density of traps indicated here agrees with the low value of activation energy 49 meV obtained above from the Arrhenius fit in figure 4(b).

4. Conclusion

In this work, ternary alloyed HgCdTe QDs are synthesised with good stability, exhibiting excitonic absorption edges in the mid-IR region. The band gap can be tuned between 2.2–5 μm by controlling size as well as Cd composition. The tetrapod shape of the crystals and stoichiometric excess of cations are analogous to the case of parent HgTe QDs. Ohmic behaviour is obtained with gold and photoconductor devices are fabricated with simple drop casted films on interdigitated gold electrodes. After EDT ligand exchange, the resistance of the devices drop to the order of KΩs and demonstrate promising photo-response under excitation of visible (532 nm), IR (810 nm, 1550 nm) wavelengths. The responsivity value is typically around 1 mA W$^{-1}$ at 1 V bias and within 1–2 V, an exponential increase in value is observed. A consistent linearity in photo-response is also observed over irradiance range of 0.025–2.5 W cm$^{-2}$. Though the basic photo-response behaviour is demonstrated well, there is scope for huge further development. For one thing, presence of shallow traps hinted by Arrhenius plot and responsivity-irradiance curve should be investigated further and passivated. Also a reduction in dark current can lead to great enhancement in properties. These insights on photophysical properties of HgCdTe QDs and further efforts on optimizing film thickness, stoichiometry, device geometry, improving surface passivation, monodispersivity will pave the way for their possible applications for developing next generation of NIR and MWIR optoelectronic devices.

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