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Encapsulation of photoresistive structures based on colloidal HgTe quantum dots by atomic layer deposition

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Colloidal quantum dots (CQDs) are a promising material for low-cost infrared photodetectors operating at room temperature. However, the formation of CQD based photosensitive layers is accompanied by the formation of surface defects and oxidation, which leads to the degradation of the devices' performance. In this study, we propose using atomic layer deposition (ALD) to efficiently encapsulate a mercury telluride (HgTe) CQD layer by depositing thin films of hafnium oxide (HfO₂) for the first time. The feasibility of applying the HfO₂ encapsulating layer is confirmed by studying the photoelectric characteristics of the manufactured photoresistors. It is shown that the protective coating reduce the dark current while maintaining the photoresistor's photoresponse under 1550 nm laser irradiation. The obtained results demonstrate the potential of ALD for creating stable and high-performance next-generation IR photodetectors.

Keywords: photosensor, mid-wave IR range, colloidal quantum dots, atomic layer deposition, encapsulation, hafnium oxide.

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Introduction

Infrared photodetectors based on colloidal quantum dots (CQDs) offer several advantages over traditional detectors (e.g., those based on InGaAs or MCT), due to the low cost and simplicity of CQD synthesis using wet chemistry methods, compatibility with CMOS technology, the potential for room-temperature operation, and the mechanical flexibility of the fabricated photosensitive layers on substrates [1–5]. The ability to tune the spectral characteristics of HgTe CQDs through the quantum confinement effect allows their absorption wavelength to be adjusted from the near-infrared to the far-infrared range. This is achieved by controlling the nanocrystal

size during synthesis, making this material promising for the development of IR photodetectors [6]. However, despite the relative stability of HgTe CQDs, the process of fabricating photosensitive layers can lead to the formation of undesirable surface states and/or the slow degradation of the surface layer due to oxidation upon exposure to humid air [7–9]. Furthermore, changes in stoichiometry during post-synthetic treatment of the nanocrystals and device fabrication also lead to a sharp increase in trap states, deteriorating sensitivity and response speed.

One well-developed and effective method for producing thin films is atomic layer deposition (ALD), which involves sequential,

self-limiting chemical reactions [10–13]. During the ALD process, precursors alternately react with the surface multiple times, resulting in the reproducible growth of a thin film, one monolayer per cycle, even on substrates with complex geometries. The self-limiting nature of the surface reactions enables control at the atomic level. The ALD process was developed due to the need for high-quality dielectric and luminescent films to be deposited on large-area substrates [14]. ALD is used to deposit various types of thin films, including oxide (Al_2O_3 , TiO_2 , SnO_2 , ZnO , HfO_2), nitride (TiN , TaN , WN , NbN), metal (Ru , Ir , Pt), and sulfide (ZnS) films for diverse applications in semiconductor microelectronics manufacturing [15–20], photovoltaic devices [21–25], energy storage devices [26–28], catalysts [29, 30], semiconductor gas sensors [31–34], optics and photonics [35–37], and many others [38]. However, the ALD method is relatively expensive, both in terms of the complexity of the equipment required and the high purity and quality demands for the precursors and substrates used.

In the context of infrared photodetectors based on colloidal quantum dots, ALD is used to deposit a passivating aluminum oxide film to improve stability during fabrication steps performed in air. For example, it is used to optimize the photosensitivity and stability parameters of detectors based on PbS and PbSe [39, 40] and HgTe [41].

This paper proposes using the ALD method to form a protective hafnium oxide (HfO_2) coating on an array of HgTe CQDs. The development of the ALD method has made the ability to deposit films at relatively low temperatures (below 150°C) a competitive advantage, ensuring the stability of the surface layer of sensitive materials, including biological and organic samples. The encapsulation efficiency was monitored by measuring the photosensitive characteristics of the photoresistor: the dark current and the magnitude of the photoresponse under illumination by a 1550 nm light-emitting diode.

Experimental section

Synthesis of HgTe CQDs

HgTe CQDs were synthesized via the hot-injection method according to a previously

described protocol [41] at a reaction temperature of 80°C and a reaction time of 30 minutes to obtain CQDs of the desired size.

Fabrication method for the photoresistive structure

The CQDs were deposited onto a quartz substrate with interdigitated Au electrode patterns, employing intermediate layer-by-layer activation with a solution of ethanedithiol in hydrochloric acid and isopropanol ($\text{HCl}:\text{EDT}:\text{IPA} = 1:1:20$ by vol.) and rinsing with isopropyl alcohol.

Encapsulation method for the photoresistive structure

The HfO_2 film growth was carried out at a reactor chamber temperature of 90°C using $\text{Hf}[\text{N}(\text{CH}_3)(\text{C}_2\text{H}_5)]_4$ (TEMAH) and H_2O as precursors. The TEMAH pulse duration into the reaction chamber was 0.5 s, followed by a 12 s purge. The water vapor pulse duration was 0.1 s, followed by a 12 s purge. The temperatures of the TEMAH and H_2O cylinders were maintained stable throughout the entire deposition process at 100°C and 20°C , respectively. The protective coating thickness (27 nm) and growth rate ($\sim 1.36\text{ \AA/cycle}$) were determined using an ellipsometer at a wavelength of 632 nm on a monitor sample of silicon with native oxide.

FTIR spectra were recorded using a Spectrum 100 FTIR spectrometer (PerkinElmer, USA) equipped with an attenuated total reflectance (ATR) accessory.

The current values of the photoresistor under pulsed illumination were recorded using a Keithley 2636B sourcemeter at a constant bias voltage $U = 0.1\text{ V}$. For pulsed illumination, a laser diode with fiber-coupled output at a wavelength of 1550 nm (power 8 mW) was used. The laser diode operated in a square-wave modulation mode (period 4 s, pulse duration 2 s) set by a FeelTech FeelElec FY6900 60M function generator. The radiation from the laser diode passed through a collimator and was then focused by a collecting silicon lens into a light spot approximately $300\text{ }\mu\text{m}$ in diameter onto the photosensitive area of the sample under study. All measurements were performed under ambient conditions in air.

Discussion of the results

The synthesis of HgTe CQDs was carried out using the hot-injection method via the reaction of mercury chloride with a solution of tellurium in trioctylphosphine. The absorption peak maximum of the CQDs synthesized at 80 °C with a nanocrystal growth time of 30 minutes, as determined by FTIR spectroscopy, was 2.83 μm with a full width at half maximum (FWHM) of 0.4 μm (Fig. 1), indicating a relatively narrow size distribution of the mercury telluride nanocrystals. Varying the synthesis time and the nature of the mercury and chalcogenide precursors allows control over the spectral characteristics of CQDs based on mercury chalcogenides [42–44]. The photoresistor was fabricated by layer-by-layer spin-coating of HgTe CQD films from a dispersion. During this process, the layer surface was treated with an ethanedithiol solution to replace the long-chain oleylamine ligands on the CQD surface, which is a recognized approach for tuning the spectral characteristics of CQDs and reducing the number of surface traps [45, 46].

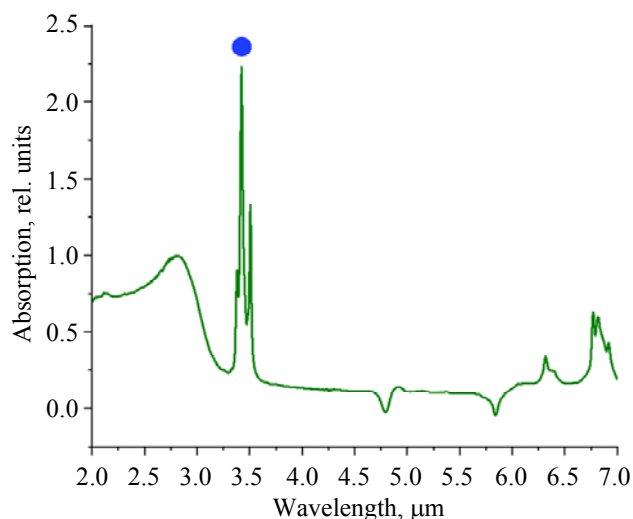


Fig. 1. IR spectrum of HgTe CQDs obtained at a synthesis temperature of 80 °C and time of 30 min. The dot marks the position of the peak corresponding to C-H stretching vibrations of the ligand shell

Storage of the photoresistor structure in air leads to changes in the photosensing characteristics. For instance, exposure for 15 days results in an increase in dark current at contacts with an interelectrode distance of 10 μm from 1.62×10^{-4} to 3.63×10^{-4} A, while measurements at

contacts with an interelectrode distance of 2120 μm show the dark current increasing from 5.22×10^{-8} to 1.09×10^{-7} A. The photoresponse amplitude, calculated as the difference between the current under 1550 nm laser illumination and the dark current, increased from 2.88×10^{-7} to 2.18×10^{-6} A upon storage for 15 days at contacts with a short interelectrode distance, and decreased from 5.79×10^{-10} to 3.47×10^{-10} A when measured at contacts with a long interelectrode distance. This is apparently related to the possible formation of surface traps caused by adsorption and chemical interaction with oxygen and water. The observed changes in characteristics demonstrate the relatively low stability of the sample. Due to the change in photosensitive characteristics, encapsulation of the photoresistor structure with a thin hafnium oxide film is proposed.

Encapsulation of the photoresistor with a hafnium oxide film was carried out at a relatively low reactor chamber temperature of 90° [47]. The protective coating thickness was 27 nm. The effectiveness of this approach was investigated by comparative analysis of the dark current values, the shape of the photosignal rise and decay, and the photosignal amplitude before and after applying the hafnium oxide protective coating under 1550 nm laser irradiation. Encapsulation leads to a reduction in the dark current to 5.47×10^{-5} A and 1.38×10^{-8} A when measured at contacts with interelectrode distances of 10 μm and 2120 μm , respectively. At the same time, the photoresponse amplitude decreased slightly to 9.69×10^{-7} A and 2.10×10^{-10} A, respectively.

Fig. 2 shows the photoresponse waveforms upon illumination of the structure with a 1550 nm laser after fabrication, after 15 days of exposure to air, and after encapsulation with a thin hafnium oxide film. It should be noted that the measurement results are presented without background subtraction. Encapsulation of the structure leads to a sharp reduction in dark current drift, while a rapid rise of the photosignal to a steady-state value is observed (Fig. 2c), whereas the sample before encapsulation was characterized by a strong dark current drift (Fig. 2a, b) and a slow equilibration of the photosignal. Furthermore, the absence of a slow signal decay region after encapsulation should be

noted, compared to the freshly prepared structure and the structure stored in air for 15 days.

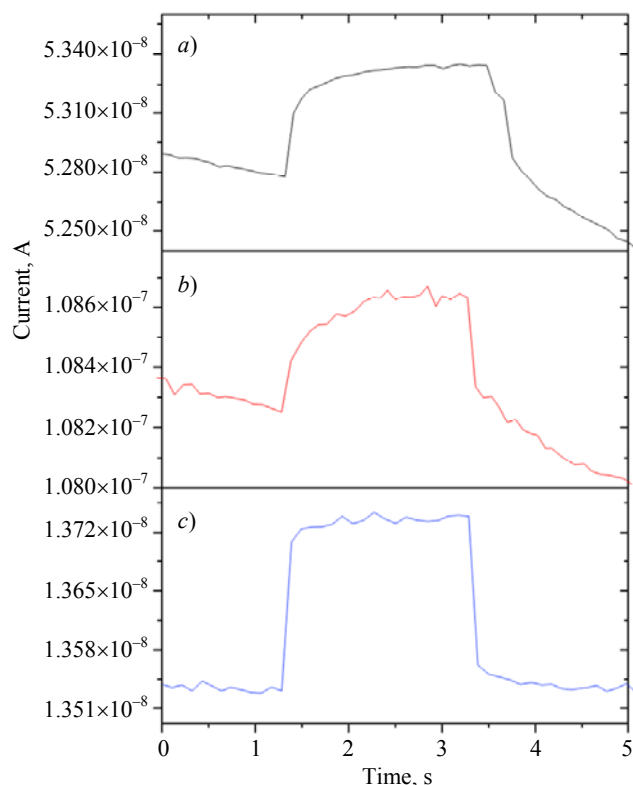


Fig. 2. Photoresponse of the photoresistive structure upon illumination with a 1550 nm laser diode: freshly prepared structure (a), after storage in air for 15 days (b), and after deposition of a hafnium oxide film (c)

Despite the need for additional, more in-depth studies, these preliminary results demonstrate the effectiveness of encapsulating CQD photoresistive structures with a thin

hafnium oxide film deposited by ALD at a relatively low temperature. This opens up broad possibilities for reducing dark current and improving stability in the fabrication of photosensitive layers for IR photodetectors based on CQDs.

Conclusion

HgTe colloidal quantum dots with an absorption peak maximum at 2.83 μm were obtained via the hot-injection method. A photoresistive structure based on these dots was fabricated on gold interdigitated electrodes. The effectiveness of encapsulating the obtained structure with a thin protective hafnium oxide film deposited by ALD at a relatively low temperature of 90 $^{\circ}\text{C}$ is demonstrated. The deposition of the hafnium oxide film leads to a decrease in dark current values, preservation of the photoresponse magnitude, reduction of dark current drift, and acceleration of signal decay upon illumination of the structure with a 1550 nm laser diode. The obtained data indicate the potential of using low-temperature ALD for producing thin encapsulating hafnium oxide films in IR photodetectors based on colloidal quantum dots of various metal chalcogenides.

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