Preparation of Nanostructured HgI₂ Nanotubes/Si Photodetector by Laser Ablation in Liquid

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Abstract

In this study, we present a high-performance HgI₂ nanotubes/Si heterojunction photodetector by a simple one-step pulsed laser ablation in ethanol at a laser fluence of 12.7 J/cm². The structural and optical properties of HgI₂ nanotubes (NTs) are studied. The X-ray diffraction (XRD) measurement shows the presence of the mixture of tetragonal and orthorhombic phases of HgI₂. The optical properties results show that the optical energy gap of HgI₂ NTs was 2.98 eV measured at room temperature. The photoluminescence (PL) spectrum of HgI₂ displays a single emission centered at 535 nm. Scanning electron microscope (SEM) image show the formation of nanotubes and spherical nanoparticles with an average particle size of 23 nm. The transmission electron microscope (TEM) image of HgI₂ confirms the formation of nanotubes morphology with an average diameter of 80 nm and an average length of 1.6 μm. The current–voltage characteristics of HgI₂ NTs/p-Si heterojunction were measured at dark and illumination. The HgI₂ nanotubes/Si photodetector is sensitive to the spectral region ranging from visible to near infrared region. This photodetector presents a responsivity of up-to 1.09 A/W at 450 nm under an external bias voltage of 5 V. The specific detectivity (D*) and external quantum efficiency (EQE) of the photodetector are 3.6 × 10¹² Jones and 3 × 10²% at 400 nm, respectively, without using post annealing. The fabricated photodetector has good linearity characteristics with a large linear dynamic region, and the saturation in photocurrent is observed at 180 mW/cm². The figures of merit of the fabricated photodetector are compared with those of some heterojunction-based silicon photodetectors.

Keywords HgI₂ · Laser ablation · Nanotubes · Photodetector

1 Introduction

Mercury iodide is a semiconducting material with an energy gap as large as 2.13 eV at room temperature. As reported, there are three elemental phases for stoichiometric HgI₂, namely, tetragonal α-HgI₂, super tetragonal, and orthorhombic β-HgI₂ with red, orange, and yellow colors, respectively, and the most stable phase is red tetragonal [1]. HgI₂ nanostructures were prepared with different morphologies such as nanoparticles and nanorods [2]. Due to the excellent physical and chemical properties of nanostructured HgI₂, it is used in various technological and industrial potential applications, for example, photodetector, solar cells, ionization radiation detectors, and nanocatalyst [3–5]. Its tunable energy gap is one of the most important properties that enables HgI₂ to be used significantly for optoelectronic devices. This manipulation of the energy gap can be obtained via changing the particle size. The properties of nanosized HgI₂ depend on the preparation route and experimental conditions. Many techniques were employed to prepare the nanostructured HgI₂ for instance, rapid co-precipitation, spray pyrolysis, laser deposition, and hydrothermal [6–8]. Actually, a few papers were reported on the preparation and characterization of nanostructured HgI₂. Nanostructured semiconducting materials are very attractive and promising for high-performance photodetectors and solar cells since they can simply be integrated with silicon electronics [9–11].
based silicon photodetectors exhibit many superior characteristics such as high responsivity, long wavelength detection, low noise, no high temperature processing needed, high voltage bias operating, large dynamic region, and cost-effective [12–14]. Recently, a high responsivity HgI₂ nanorods/Si heterojunction photodetector was fabricated using a pulsed laser deposition technique [8]. To the best of our knowledge no studies reported on synthesis of HgI₂ nanotubes are found in literatures. Here, we report on the synthesis of HgI₂ nanotubes for the first time via pulsed laser ablation in liquid. The fabrication and characterization of an HgI₂ NTs/Si heterojunction photodetector without the use of a buffer layer were performed.

2 Experimental

2.1 2–1. Synthesis of HgI₂ Nanotubes

Pulsed laser ablation was used to synthesize the HgI₂ NTs in ethanol. High purity HgI₂ powder purchased from Riedel–De Haen AG Seelze-Hannover company was pressed first using a hydraulic compressor in order to prepare a pellet with an area of 2.5 cm² and thickness of 1 cm. A Q-switched Nd:YAG laser pulses with λ = 1064 nm, pulse repetition frequency of 1 Hz, and pulse duration of 7 ns were used for ablation of HgI₂. A conversion lens with a 10 cm focal length was used so as to focus the laser beam on the HgI₂ pellet located in a glass vessel filled with ethanol with a height of 2 ml above the HgI₂ target. The ablation time was 20 min and the actual laser fluence was 12.7 J/cm² after taking into account the transmission of ethanol at 1064 nm.

2.2 2–2. Characterization of HgI₂ NTs

The structure of the film was studied using an X-ray diffractometer (XRD-6000, Shimadzu). To measure the optical absorption of HgI₂ NTs. To measure the optical absorption of the colloidal HgI₂ NTs, a UV–Vis double-beam spectrophotometer (Shimadzu -1800) was used. The stability of the colloids was investigated by Zeta potential (Brookhaven com.). To study the chemical bond assignment and IR-absorption of HgI₂ NTs, FT-IR (Shimadzu 8400S) was used. The morphology and particle size of HgI₂ were studied using a transmission electron microscope TEM (EM208, Philips) and a scanning electron microscope SEM (Zeiss).

2.3 2–3. HgI₂/Si Heterojunction Synthesis

The HgI₂/Si heterojunction photodetector was fabricated by drop casting of HgI₂ film on a 1 cm² mirror-like p-type (111)-oriented single crystalline silicon having an electrical resistivity of 3–5 Ωcm and a thickness of 300 µm. As shown in Fig. 1, In and Al films were deposited on HgI₂ and the back side of the Si substrate, respectively, by thermal evaporation system to create ohmic contacts. The dark and illuminated current–voltage characteristics of HgI₂/p-Si were measured at room temperature a using digital DC power supply, electrometer and lamp source. A Silicon power meter was used to measure the light intensity. In order to measure the responsivity of the photodetector, Jobin Yvon monochromator was used after making power calibration.

3 Results and Discussion

Figure 2 shows the XRD patterns of the HgI₂ powder and nanoparticles. The XRD pattern of bulk HgI₂ exhibits eleven peaks at 2θ = 14.5⁰, 21.7⁰, 25⁰, 29.7⁰, 32.4⁰, 35.4⁰, 41.2⁰, 43.8⁰, 47.1⁰, 48.8⁰, and 59.9⁰, which correspond to (002), (101), (102), (103), (112), (104), (114), (006), (211), (106), and (008) planes, respectively. All these observed peaks are related to the tetrahedral structure of HgI₂ planes according to JCPDs #73–0455. The XRD patter
of HgI₂ NTs shows the presence of five peaks positioned at 2θ = 13°, 21.7°, 25, 39.1°, and 53.1°, corresponding to (002), (101), (102), (123), and (214) planes, respectively. The (002), (123), and (214) plans belong to orthorhombic β-HgI₂ according to JCPDS-ICDD #15–0034, while (101) and (102) planes are indexed to tetragonal α-HgI₂ [7, 15], indicating that the synthesized HgI₂ NTs are a mixture of two phases, α-HgI₂ and β-HgI₂. As shown, the highest peak intensity for HgI₂ NTs was observed for the (002) plane, confirming that the grains grow in a direction perpendicular to the substrate (c-axis). The average crystallite size (D) was determined from XRD data and using Debye–Scherrer equation along the (002) plane

\[ D = \frac{0.9\lambda}{\beta \cos \theta} \]  

Fig. 3 Show the optical absorbance of HgI₂ colloids. Inset is the freshly prepared HgI₂ suspension
where $\lambda$ is the wavelength of the X-ray source and the $\beta$ is the full width at the half maximum in units of radians.

Our calculation revealed that the average crystallite size of HgI$_2$, dislocation density, and the strain were 14 nm, $5.32 \times 10^{-3}$ nm$^{-2}$, and $25.3 \times 10^{-3}$, respectively. The lattice constants of the HgI$_2$ was calculated from XRD data and found to be $a = 0.41$, $b = 0.77$, and $c = 1.2$ nm. The optical absorbance of HgI$_2$ colloids is shown in Fig. 3. As shown, the optical absorbance of HgI$_2$ was flat in the range 250–350 nm and sharply decreased after 347 nm and saturated after 400 nm.

The optical energy band gap was calculated using Tauc plot. As shown in Fig. 4, plotting of $(\alpha h\nu)^2$ vs ($h\nu$) and extrapolating the linear part of the second region to the photon energy gives the optical energy gap, which also indicates that HgI$_2$ is a direct band gap [16]. The direct optical band gap of HgI$_2$ NTs was found to be around 2.9 eV.

Figure 5 shows the zeta potential (ZP) plot of HgI$_2$ colloids, which is approximately 22 mV confirming the synthesized HgI$_2$ NTs have a high degree of stability and high despiration with a low tendency for aggregation and agglomeration [17, 18].

Figure 6 demonstrates the PL spectrum of HgI$_2$ colloids exited with a laser source of 300 nm wavelength. The spectrum shows the existence of only a strong PL emission centered at 535 nm (2.31 eV). This value is smaller than the optical energy gap determined from UV–Vis results due to the effect of trapping defects inside the band gap of HgI$_2$ [19]. The existence of a small peak at 540 nm is due to the free excitons and bound excitons emission [20].

Figure 7 shows the IR vibration spectrum of HgI$_2$ colloids. Two IR assignments were observed at 612 and 1102 cm$^{-1}$ that belonged to Hg-I and the C-O stretch, respectively [21].

The EDX spectrum of HgI$_2$ NTs deposited on the silicon substrate is depicted in Fig. 8, which shows the existence of mercury and iodide elements, and we attributed the presence peaks of carbon and silicon to the organic environment and the substrate, respectively [20]. The Hg/I weight ratio was 1.23 indicating the synthesized
HgI$_2$ NTs were small-off stoichiometric (see the inset of Fig. 8).

Figure 9 illustrates the SEM image of HgI$_2$ with two magnifications. Spherical nanoparticles mixed with nanotubes were observed (Fig. 9-a). Fig. 9-b shows the SEM image of the tetragonal bi-pyramids, or truncated pyramids structure, of HgI$_2$. The particle size distribution given in Fig. 9-c confirmed that the mean particle size of HgI$_2$ was 23 nm. We have observed the presence of only monodispersed nanoparticles, which is in good agreement with the results of the Zeta potential result. Figure 10 shows the TEM image of HgI$_2$ which confirmed the formation of monodispersed nanotubes with an average diameter of 80 nm and an average length of 1.6μm as well as the existence of spherical nanoparticles. As shown in
TEM image, some of the nanoparticles are found to be attached to the nanotubes.

Figure 11 shows the current–voltage properties of the HgI₂ NTs/Si heterojunction measured at room temperature. It is clear that the forward current consists of two regions: in the first region, the current increased slightly with bias voltage, which indicates that the recombination current is larger than the generation current, while at large bias, the current increases exponentially with voltage due to the domination of diffusion current [22–24]. The reverse current was shown to be increased slightly at low voltage and after bias with 2 V, it increased significantly due to the surface leakage current flowing through the edges of the heterojunction.

The ideality factor of the heterojunction was calculated using the diode equation and its value was 4 for first region and 12 for the second region (recombination region). When the ideality factor > 1 indicates that there are structural defects and surface states in the interface region [25] as well as due to the mismatch in lattice constants between HgI₂ and silicon. The mismatch in lattice constant between HgI₂ and the silicon substrate was calculated and found to be 23%, where the lattice constants of the silicon and HgI₂ are 0.543 and 0.41 nm, respectively. This large value of mismatch exerts mechanical stress and leads to the formation of structural defects. As the photodetector illuminated with white light, the current of the photodetector increases due to the production of e–h pairs in the depletion region. Figure 12 depicts the illuminated I-V characteristics of the HgI₂/p-Si heterojunction under white light illumination. The photocurrent increases with bias voltage as a result of the widening of the depletion region. The photocurrent increased with light intensity; this increase was attributed to more photon absorption and the generation of electron–hole pairs in the depletion layer [26]. The linear dynamic range (LDR) of the photodetector was determined from the variation of photocurrent with the light intensity plot shown in Fig. 13. The saturation in photocurrent started at a light intensity of 180 mW/cm².

The most important figure of merit of the silicon photodetector is the responsivity, which is defined as the ratio of the generated photocurrent to the power of incident light. Figure 14 illustrates the responsivity versus wavelength of the HgI₂/p-Si HJ photodetector measured at 5 V bias. We can see that the maximum responsivity was 1.09 AW⁻¹ at 450 nm, which is higher and comparable to some heterojunction-based silicon photodetectors as shown in Table 1. We attributed the
improvement in the responsivity in the visible region to the large surface area of \textit{HgI}_2 and light trapping that comes from nanotubes morphology \cite{27}.

The responsivity of the photodetector at 450 nm is due to the absorption edge of \textit{HgI}_2 NTs, which matched with result of band gap. Figure 15 shows the detectivity and quantum efficiency spectra of \textit{HgI}_2/Si HJ photodetector. The maximum detectivity was found to be $3.6 \times 10^{12}$ Jones at 400 nm wavelength. The \textit{HgI}_2/Si HJ photodetector show high quantum efficiency of about $3 \times 10^2\%$ at 450 nm. This high quantum efficiency gives an indication of the high value of carrier collection efficiency \cite{32–35}.

\textbf{Fig. 9} SEM image of (a) \textit{HgI}_2 NTs and spherical NPs, (b) tetragonal \textit{HgI}_2, and (c) histogram distribution of \textit{HgI}_2 film

\textbf{Fig. 10} TEM image of \textit{HgI}_2 NTs
HgI$_2$ nanotubes have been prepared by means of laser ablation in liquid. The XRD pattern shows the observed peaks belong to orthorhombic and tetragonal structure. The optical energy gap of HgI$_2$ NTs was 2.98 eV. PL result showed a single emission centered at 535 nm wavelength. The FT-IR observed small bonds at 612 and 1102 cm$^{-1}$ attributed to the existence of Hg-I and the C-O stretch, respectively. The SEM image shows the formation of nanotubes with some spherical nanoparticles. The mean particle size was 23 nm and the average diameter of nanotubes was 80 nm with an average length of 1.6 μm. A high-performance HgI$_2$/p-Si heterojunction photodetector was fabricated and characterized. The current–voltage of the heterojunction exhibited rectification properties, and the ideality factor at low voltage was 4. The photodetector exhibited broadband operating in the spectral region of 400–1000 nm. The responsivity of the photodetector was 1.09 A/W at 450 nm. The maximum specific detectivity and quantum efficiency of the photodetector were $3.6 \times 10^{12}$ Jones and $3 \times 10^2\%$, respectively. Our work demonstrates a simple and inexpensive route towards fabricating high-performance heterojunction-based silicon photodetectors operating in the visible and near infrared regions, which can be significantly used for the detection of low power signal applications.

4 Conclusion

HgI$_2$ nanotubes have been prepared by means of laser ablation in liquid. The XRD pattern shows the observed peaks belong to orthorhombic and tetragonal structure. The optical energy gap of HgI$_2$ NTs was 2.98 eV. PL result showed a single emission centered at 535 nm wavelength. The FT-IR observed small bonds at 612 and 1102 cm$^{-1}$ attributed to the existence of Hg-I and the C-O stretch, respectively. The SEM image shows the formation of nanotubes with some spherical nanoparticles. The mean particle size was 23 nm and the average diameter of nanotubes was 80 nm with an average length of 1.6 μm. A high-performance HgI$_2$/p-Si heterojunction photodetector was fabricated and characterized. The current–voltage of the heterojunction exhibited rectification properties, and the ideality factor at low voltage was 4. The photodetector exhibited broadband operating in the spectral region of 400–1000 nm. The responsivity of the photodetector was 1.09 A/W at 450 nm. The maximum specific detectivity and quantum efficiency of the photodetector were $3.6 \times 10^{12}$ Jones and $3 \times 10^2\%$, respectively. Our work demonstrates a simple and inexpensive route towards fabricating high-performance heterojunction-based silicon photodetectors operating in the visible and near infrared regions, which can be significantly used for the detection of low power signal applications.
Fig. 13 Photocurrent versus light intensity plot of the photodetector at bias voltage of 5 V

![Photocurrent vs Light Intensity Plot](image)

**Saturation**

LDR: 21dB

Fig. 14 Spectral responsivity plot of the HgI₂/Si HJ photodetector at bias voltage of 5 V

![Spectral Responsivity Plot](image)

**Table 1** Figures of merit of fabricated photodetector compared with some other silicon heterojunction-based photodetectors

| Photodetector type     | Responsivity (A/W) | Detectivity (Jones) | External Quantum efficiency% |
|------------------------|--------------------|---------------------|-----------------------------|
| HgI₂ NTs/Si [present]  | 1.09 at 450 nm     | 3.6 × 10¹² at 400 nm| 3 × 10⁵ at 400               |
| CsPbBr₃/n-Si [28]      | 0.6 at 520 nm      | 9.5 × 10¹¹ at 520 nm| 1.4 × 10² at 520 nm          |
| Graphene/ZnO/silicon [29]| 0.26 at 400 nm   | 3.9 × 10¹³ at 400 nm| 80 at 400 nm                 |
| Sb₂S₃/Si [30]          | 0.29 at 500 nm     | 1.6 × 10¹¹ at 500 nm| 72 at 500 nm                 |
| CdO/Si [31]            | 0.5 at 600 nm      | 7 × 10¹¹ at 600 nm  | 62 at 600 nm                 |
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Author contributions Amnah and Raid conceived of the presented idea.
Raid and Mudhafar supervised the finding of this work.
All authors discussed the results and contributed equally to the final manuscript.
Mudhafar and Amnah conducted the experiments.
All authors provided critical feedback and helped shape the research, analysis and manuscript.

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Declarations

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Fig. 15 Specific detectivity and quantum efficiency as a function of wavelength of the Hgl₂/Si photodetector
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