

# PbS and PbSe in Room-Temperature Infrared Photodetectors

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Infrared photodetectors have received much attention for several decades due to their broad applications in the military, science, and daily life. However, for achieving an ideal signal-to-noise ratio and a very fast response, cooling is necessary in those devices, which makes them bulky and costly. The earliest information about lead-based semiconductor materials comes from a patent published in 1904 by Bose, who found and utilized the photovoltaic effect of a crystal of galena. Subsequently, Case carried out his research on thin films of thallous sulfide ( $\text{Ti}_2\text{S}$ ) in 1917 and 1920. Due to the military needs of infrared information in World War II, Germany developed lead salt (PbS, PbSe and lead telluride (PbTe)) materials vigorously in the 1930s. During that period, different methods for preparing lead salt thin films developed rapidly. Gudden and Kutzscher prepared lead salt films by evaporation and chemical deposition, respectively. Shortly after German scientists firstly studied it, the United States scientists also conducted research on it. Cashman of Northwestern University began work on  $\text{Ti}_2\text{S}$  in 1941 and later turned his full attention to the preparation of thin films of PbS, PbSe and PbTe by vacuum evaporation. Among the three typical lead salts used in infrared detectors, PbS and PbSe have been developed and produced to some extent, but PbTe has not been adapted for production and has been gradually phased out.

Keywords: infrared photodetectors ; PbS ; PbSe

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## 1. Introduction

In nature, every object above absolute zero can emit infrared photons with unique fingerprints of different temperature information, though they cannot be captured by human eyes. Infrared detectors, however, can convert infrared radiation into a measurable electronic signal, which is significant in infrared technology <sup>[1]</sup>. The technical progress of infrared technology is mainly related to the development of narrow-band semiconductor infrared photodetectors, which have both an ideal signal-to-noise ratio and a very fast response. However, to achieve this goal, the photodetectors need to be cooled at low temperatures to avoid thermal-activated carriers. As a result, the system is bulky, costly and inconvenient to use. As a result, developing room-temperature infrared photodetectors has become an important research direction <sup>[1]</sup>.

Driven by Moore's Law, the feature size of highly integrated electronics has already been reduced to a few nanometers <sup>[2]</sup>. Consequently, in infrared technology, one major focus is how to reduce pixel scale and increase format array for a highly integrated photoelectric detection system with high performance. Traditional infrared photodetectors based on narrow-band semiconductors, such as indium–gallium arsenide (InGaAs) and mercury–cadmium telluride (HgCdTe), need the flip-up bonding technique. This causes great difficulties in coupling with silicon electronics, limiting the development of the focal plane array (FPA). Low-dimensional nanostructured materials are appealing in the field of photoelectric detection because they can be integrated with traditional silicon electronics and even with flexible large-area substrates by liquid phase processing, such as spin-coating, spraying, or stratified deposition <sup>[3]</sup>. In addition, they have great potential in subwavelength pixel, large array and multicolor devices <sup>[4]</sup>. However, there are still many challenges in material growth, device fabrication and coupling with circuits. The development of low-dimensional material infrared photodetectors started 40 years ago. In recent times, most of the reported infrared detectors, such as those based on mercury telluride (HgTe) colloidal quantum dots (CQDs), are still single-pixel devices <sup>[5]</sup>.

Low-dimensional materials can be classified into zero-dimensional (0D), one-dimensional (1D) and two-dimensional (2D) materials.

CQDs, which are typically 2–20 nm in diameter, are important representatives of 0D materials <sup>[6]</sup>. Benefiting from quantum confinement, the band gap of CQDs is strongly dependent on nanocrystal (NC) size, which makes it possible to design a band gap independently. This means that, by controlling the size of CQDs, rather than their chemical composition, a wide spectrum can be covered <sup>[7]</sup>. For example, the spectra of HgTe CQD can cover short-wave infrared (SWIR), mid-wave

infrared (MWIR), long-wave infrared (LWIR) and even Tera Hertz (THz) [8]. Due to the rapid development of material systems, it is now even possible to achieve this wide spectral range using non-toxic and heavy metal-free quantum dots [9]. In addition, the preparation process of CQDs has significant advantages. CQDs are typically made from II-IV, III-V and IV-VI semiconductors through inexpensive and scalable wet chemical synthesis [6]. This enables cheap large-scale manufacturing of optoelectronic equipment at room temperature by several techniques, such as roll-to-roll processing [7]. The flexibility of CQDs is also reflected in the fact that the modern solution of ligand exchange protocols can change the properties of CQDs prior to the deposition process. For example, long-chain organic ligands on the surface of CQDs can be replaced by short-chain organic compounds and even by inorganic ions such as bromides or iodides for better carrier transport [10]. In fact, CQDs have already achieved great success in high-performance infrared detection and in multi-functions such as those in SWIR/MWIR dual band photodetectors [11] and polarization infrared detectors [12] due to unique structure-related photoelectric properties.

In recent years, 1D and 2D nanomaterials have become the focus of nanotechnology research. At present, 1D nanomaterials can be synthesized from a single crystal form, whose chemical composition, shape, doping state, length, diameter and other key parameters are controllable [13]. Now, near-infrared (NIR) photodetectors based on MBE-grown InAs NWs show high-performance optoelectronic properties. Single InAs NW photodetectors have an impressive  $I_{on}/I_{off}$  ratio of  $10^5$  with a maximum field-effect mobility of  $\sim 2000 \text{ cm}^2/\text{V}\cdot\text{s}$  [14]. For 2D materials, graphene is one main research direction in infrared detectors. A waveguide-integrated graphene photodetector that simultaneously exhibits high responsivity, high speed and broad spectral band width can achieve a photoresponsivity exceeding  $0.1 \text{ AW}^{-1}$  together with a nearly uniform response between 1450 and 1590 nm [15]. Another 2D material of interest is black phosphorus. Black phosphorus MWIR detectors have been demonstrated at  $3.39 \mu\text{m}$  with high internal gain, resulting in an external responsivity of  $82 \text{ A/W}$  [16]. In addition, there is a lot of research on the physicochemical properties of 2D electron gas [17] [18] [19].

Excellent light response and signal-to-noise ratios are indispensable for obtaining an excellent room-temperature detector. For PC devices,  $1/f$  noise and shot noise are inevitable because of bias voltage. For PV devices,  $1/f$  noise and Johnson noise are the main interference signals when there is no bias voltage, whereas shot noise has a greater effect than Johnson noise when bias voltage is applied.

## 2. PbS

### 2.1. Physical Properties of PbS

The study on the optical properties of PbS mainly focuses on single or polycrystalline films in the early stage. Many studies have shown differences in light scattering and absorption in nanostructures compared to bulk materials. Therefore, the particle size effect on the optical properties of semiconductors has attracted much attention.

Exciton, formed by direct light excitation or by the combination of free carriers [20], is an important concept in understanding nanoparticle properties. The delocalization region of excitons may be much larger than the semiconductor lattice constant, which may even be several or tens of nanometers. Although nanoparticle sizes are comparable to exciton radii, their physical properties are greatly changed [21]. In the case of PbS nanoparticles, the band gap in the electron spectrum increases from 0.41 to 1.92 eV when the particle diameter is reduced to 5 nm, and the band gap continues increasing when the diameter is further reduced [22]. This indicates that the band gap increases with the decrease in semiconductor particle size, which means a blue shift of the absorption band. There are several reports on optical transmission properties of PbS nanoparticle films.

### 2.2. Fabrication Methods

There have been many mature methods on the preparation of PbS CQDs. For PbS CQDs, there are two major organic synthesis methods [23].

One way is to react lead oleate and bis(trimethylsilyl) sulfide (TMS) in octadecene (ODE). They produce monodisperse CQDs that are 2.6 to 7.2 nanometers in size with corresponding absorption peaks ranging from 825 to 1750 nanometers. However, not all sizes of CQDs made in this way are air-stable [24]. The following are the detailed operation steps: First, the lead oxide (PbO) in oleic acid (OA) is heated under argon or vacuum to prepare lead oleate. After that, a solution of TMS in ODE is injected into the solution until the ratio of lead to sulfur is 2:1. It is worth mentioning that trioctylphosphine (TOP) can also be used as a solvent for TMS and has no material effect on the reaction results. The reaction temperature is controlled according to the desired particle size. The NCs are then precipitated with a polar solvent such as methanol or acetone and are subsequently redispersed into an organic solvent such as chloroform or toluene. Precipitation and

redissolution are repeated to ensure the removal of the reaction solvents. Finally, the aqueous NC dispersion is centrifuged to remove any remaining impurities [23].

The second method is to react lead chloride ( $\text{PbCl}_2$ ) and elemental sulfur in oleylamine (OAm) under nitrogen. PbS NCs were prepared by thermal injection, similar to the first method. The size range (4.2–6.4 nm) of CQDs prepared by this method is much smaller than that of the first method. In addition, the corresponding absorption peak range is from 1200 to 1600 nanometers. The CQD films prepared by this method show good optical stability [25]. In addition, there are many preparation methods of PbS CQDs, most of which are based on the improvement of the above two methods.

Preparing PbS CQD solids is further mentioned in many articles, and different researchers differ in method details. Here, a method is introduced to offered by McDonald et al. It is necessary to carry out ligand exchange first, and this method is an improvement upon Hines's method [23]. They precipitated the OA-coated NCs with methanol, dried them and dispersed them in excess octylamine. The solution was heated, and the octylamine-coated NCs were precipitated with N, N-dimethylformamide and then redispersed into chloroform. The NCs were then mixed with 2-methoxy-5-(2'-ethylhexyloxy-p-phenylenevinylene) (MEH-PPV). The P-phenylenevinylene (PPV) hole transport layer was rotated onto an indium tin oxide (ITO)-coated glass sheet and annealed in vacuum to allow polymerization. A mixture of MEH-PPV and NCs was dissolved in chloroform and spun onto the PPV layer to create a thin film. Finally, the upper contact was prepared by vacuum evaporation [26].

### 2.3. Devices

PbS NC films have been used in many devices, including infrared LEDs [27], mid- and long-wave infrared detectors [28], upconversion photodetectors [29] and field effect transistors (FET) [30]. Here are some typical concrete examples.

A hybrid graphene PbS QD phototransistor with ultra-high gain has been reported. Using the strong light absorption of QDs and the high mobility of graphene, the materials were mixed into a system to make it have high sensitivity for light detection. The ultra-high gain of graphene phototransistors was realized for the first time by using the charge transfer of the two materials [31]. Coupling with graphene has always been a research direction for the improvement of the performance of detectors.

Here is another PbS QD photodetector coupled with CVD-grown graphene. It shows amazing responsivity of  $1 \times 10^7 \text{ AW}^{-1}$  at a power of 30 pW [32]. Graphene was also used in PbS QD field effect phototransistors as electrodes. PbS QDs are used as channels, and field-effect transistors show good responsivity of  $4.2 \times 10^2 \text{ A/W}$  [33]. By comparing specific the detectivity of these PbS-graphene devices, the device reported by Konstantatos et al. shows the best property which reaches a specific detectivity of  $7 \times 10^{13}$  Jones.

Besides graphene, many new materials are candidates for coupling materials of PbS devices. A PbS QD-sensitized InGaZnO photoinverter for NIR detection is reported in 2016. This hybrid photoelectric device has good light-response performance of high specific detectivity of  $10^{13}$  Jones [34], which is similar to that of the best performing optoelectronic devices based on PbS-graphene, as mentioned before.

In addition, in the research of lead sulfide detectors, there are many novel methods to treat thin films. Single-step fabrication of FET using CQD ink was studied also reported the importance of removing ligands after deposition. They showed that this superficial coating method with blades can prepare high-quality thin films, and the device has a good response [30]. It is also the work of many researchers to make devices by coupling other materials with PbS to expand spectral range. A hybrid PbS CQD transition metal dichalcogenides photodetector with high sensitivity was reported in 2019.

Perovskite is a hot material that has been used with 3D-printing technology in recent years. As a result, IR photodetectors made of PbS nanotubes with perovskite ligands were studied and reported in 2019. This all-printed device shows a cut-off frequency of over 3 kHz and a high detectivity of  $10^{12}$  Jones [35], which shows the same order of magnitude as the hybrid PbS CQD transition metal dichalcogenides photodetector, as mentioned before.

The fabrication potential of flexible devices is recognized as a great advantage of CQD materials. A flexible broadband photodetector based on the heterostructure of PbS/ZnO nanoparticles was reported in 2019, and it shows the widest detectable spectral range (UV-Vis to NIR) of devices [36]. This detector shows a high detectivity of  $3.98 \times 10^{12}$  Jones.

Upconversion device is an exciting field because of its unique properties. A QD-based solution-treated upconversion photodetector was studied and reported. The photodetector has a low dark current, a high detectivity of  $6.4 \times 10^{12}$  Jones,

a millisecond response time and compatibility with flexible substrates [29].

### 3. PbSe

PbSe thin films are widely used for NIR and MWIR range applications due to their unique physical properties. PbSe polycrystalline thin films are widely used for infrared detectors [37]. Considering that the photosensitivity of thin films is very sensitive to crystallite size, a research group prepared thin films with a thickness of 1.2  $\mu\text{m}$  and different crystallite sizes. After post-processing, the detectivity of the photodetector achieved  $2.8 \times 10^{10}$  Jones at room temperature [38]. PbSe has many forms, such as polycrystal, monocrystal and QDs, which have been studied deeply and are widely used in infrared detectors. In order to reduce costs and improve performance, there has been a lot of research on nanotechnology in the past few decades [39]. Photonic applications of CQDs involving lead chalcogenides are mainly associated with PbS and PbSe.

#### 3.1. Properties

As lead chalcogenides, PbS and PbSe have many similarities in optical properties. PbSe is a typical direct band-gap semiconductor with a narrow band gap of 0.27 eV at room temperature [40]. The narrow band property of bulk PbSe makes it ideal for MWIR detection. The small electron effective mass in PbSe causes a large Bohr radius of 46 nm, which makes the material ideal for studying quantum size effects observed only in large particles with a small surface-to-volume ratio [41]. Combining these properties makes it possible to precisely alter the band gap and the spectral range of optical photoresponsivity [39].

Using sodium selenite sulfate as the selenium source and lead acetate as the lead source, Begum et al. prepared nanocrystalline PbSe films on glass substrate by the chemical bath deposition (CBD) method. The results show that the optical absorption of PbSe films increases with increases in deposition temperature. This may be due to an increase in grain size and a decrease in defects [42]. The  $(\alpha h\nu)^2$  vs  $(h\nu)$  plots of PbSe thin films are linear over a wide spectral range. This indicates that there is a direct optical band gap in the PbSe films [43]. Zhu et al. used pulse the sonoelectrochemical synthesis method to prepare PbSe nanoparticles and estimated the band gap of the materials by optical diffuse reflectance spectroscopy [44].

The absorption spectra of PbSe CQDs with different particle sizes were given by Gao et al. [45]. They used PbSe CQDs with an average radius of 2.8–3.5 nm and a corresponding band gap of 0.60–0.76 eV, judging from the position of the band edge peak in the absorption spectrum. Thambidurai et al. developed a high-performance infrared photoelectric detector up to 2.8  $\mu\text{m}$  based on PbSe CQDs [46]. They gave the photocurrent and voltage characteristics of photodetectors based on PbSe CQDs with different thicknesses (500, 900 and 1400 nm) in the wavelength range of 1.5–2.8  $\mu\text{m}$  [46].

#### 3.2. Fabrication Methods

Photodetectors based on PbSe can be divided into two categories depending on the target wavelength of photosensitivity. In the early days, PbSe intrinsic semiconductors were widely used in MWIR detectors. Bulk PbSe has an optical band gap of 0.27 eV and a sensitive wavelength of 4.4  $\mu\text{m}$ . In recent decades, however, there has been much more discussion about PbSe low-dimensional materials. Scholars have turned their attention to exploiting the quantum confinement effect, referring to the phenomenon that the energy quantization of microscopic particles becomes more obvious with decreases in its space motion limitation size, and the energy level changes from a continuous energy band to a discrete energy level, especially when the ground state energy level moves up and blue shift occurs. A common idea is to reduce the grain size below the Bohr radius. The sensitivity wavelength can be as short as 690 nm, and the band gap can reach 0.18 eV by adjusting the grain size of PbSe [39]. PbSe-based infrared detectors have the potential to span the MWIR, SWIR, NIR and even visible wavelengths.

Massive PbSe semiconductor films prepared by chemical water baths and progress in preparation of PbSe NCs are described here.

##### 3.2.1. Chemical Bath Deposition

CBD is a simple and effective method to synthesize high-quality semiconductor thin films without expensive and complex equipment [47]. However, it should be noted that film properties with CBD are greatly affected by precursor fluid composition, bath time, bath temperature and PH value. CBD films are often considered to be deposited on silicon, glass

or gallium arsenide (GaAs) substrates with a thickness between 0.2 and 2  $\mu\text{m}$  [38][39][46]. Some scholars believe that a rough substrate surface leads to better deposition [49].

Regarding CBD, there are many different combinations of precursors. In 2003, Hancare et al. prepared PbSe films using lead nitrate and selenosulphate as precursors and tartaric acid as the complexing agent. PbSe thin films were deposited onto cleaned, spectroscopic-grade glass substrates [50]. In 2010, Kassim et al. prepared PbSe films with lead nitrate solution as the lead source, sodium selenate as the selenium source and tartaric acid as the complexing agent [51]. In 2013, Qiu et al. carried out research on the room-temperature PbSe photodetector, mixing sodium hydroxide, lead acetate and selenosulfate into a precursor solution in a ratio of 12:1:1 [38].

In addition, there are few references about the influence of the timing of the chemical water bath deposition process. Anuar et al. found that the duration of the water bath affects grain size, film thickness and surface roughness. They demonstrated in reliable experiments that grain size, film thickness and surface roughness increase when the deposition time increases from 20 to 150 min. The atomic force microscopy (AFM) images of different samples indicate that the film obtained by 60 min of deposition was uniform and that the substrate surface was covered with good spherical particles [52]. Hone et al. prepared three different samples with deposition times of 30, 45 and 60 min. According to X-ray diffraction analysis (XRD) patterns, they found that the peak intensities and preferred orientations of the crystals were affected by different deposition times. When choosing deposition times of 30 and 60 min, the crystals preferred orientations along the (111) plane, whereas for a deposition time of 40 min, the crystals preferred an orientation along the (200) plane [53].

The influence of temperature and PH value on CBD has been widely reported. Deposition temperature has always been considered the most important parameter affecting film quality. It is believed that, with increases in deposition temperature, grain size increases, and dislocation density and microstrain decrease. Reductions in dislocation density and microstrain indicate reductions in lattice defects, i.e., the improvement of film quality. Additionally, deposition temperature has a significant effect on preferred orientation and film thickness [54].

CBD polycrystalline films have good properties when sensitized with oxygen and iodine, which is necessary to activate PbSe as an MWIR detector. However, the specific mechanism of sensitization has not been clearly defined. The sensitization process of PbSe varies from reference to reference, but it usually involves two thermal steps: oxidation and iodization. In order to obtain better performance for PbSe infrared detectors, it is very important to study the mechanism behind the sensitization process, but there are still many doubts.

CBD also shows good prospects in the preparation of PbSe NCs. The chemical characteristics of PbSe thin films are strongly influenced by growth conditions such as ion concentration, PH value and deposition time. Studies have shown that the average size of PbSe nanoparticles increases from 23 nm to 51 nm as the deposition time passes from 1 h to 16 h [55]. In addition, increases in temperature also make the grain size larger [56].

In addition to chemical water bath deposition, PbSe films can also be synthesized by a variety of deposition techniques, such as co-evaporation [57], pulse acoustic electrochemical [44], thermal evaporation [58] and pulse laser deposition [59].

### 3.2.2. Fabrication of PbSe NCs

Monodispersion is required for photodetectors based on PbSe CQDs. A rapid nucleation followed by a slow growth process is considered the key [60]. Nucleation is affected by temperature, degree of supersaturation in solution, interfacial tension, etc. [61]. There are two ways to stop nucleation. One way is to lower the concentration of the solution below a certain level. The other is to rapidly inject precursors into a high-temperature mixed solution, which has achieved the purpose of rapid cooling, commonly known as thermal injection [62]. Thermal injection is considered to be the most widely used method for synthesizing CQDs.

Murray et al. reported a method using lead oleate as the lead source and trioctylphosphine selenide as the selenium source, and the two are dissolved in trioctylphosphine. The above-room-temperature solution is quickly injected into a fast-stirring solution containing diphenylether at 150 °C. The growth rate of NC can be accelerated by increasing the solution temperature, and NC with the larger size can be prepared at a higher temperature. Solution temperatures of 90–220 °C correspond to NC diameters of 3.5–15 nm. When the grains reach the target size, the dispersion is cooled, short-chain alcohols are added to flocculate the NCs, and it is then separated from the solution by centrifugation [63].

### 3.3. Devices

Research on PbSe photodetectors is focused on improving efficiency, making large imaging FPAs, manufacturing thermoelectric cooling imaging systems, and making more compact and low-cost systems [39]. There are many types of

photodetectors based on PbSe, such as photoconductor [64], phototransistor [65] and photodiode [65].

Regarding the most advanced PbSe photodetector equipment, there are mainly the following kinds: broadband photodetectors using PbSe QD [66], PbSe-based photodiode detectors [67], tandem photodiode detectors [68] and PbSe-based FET detectors [69].

Jiang et al. reported an ultra-sensitive tandem CQD photodetector, which shows maximum detectivities of  $8.1 \times 10^{13}$  Jones at 1100 nm and 100 K [67].

Many research groups have tried to prepare IR photodetectors by coupling graphene with PbSe CQD. A heterojunction phototransistor based on PbSe CQD–graphene hybrids, which shows the highest responsivity of  $10^6$  A/W, was reported in 2015 [70]. In addition, the preparation of graphene electrodes on PbSe CQD vertical phototransistors has also been tried. The phototransistor exhibits an excellent responsivity of  $1.1 \times 10^4$  A W<sup>-1</sup>, a detectivity of  $1.3 \times 10^{10}$  Jones, and an external quantum efficiency of  $1.7 \times 10^6\%$  [69]. Besides graphene, coupling between other materials and PbSe materials has also been studied and published. A hybrid photodetector based on Bi<sub>2</sub>O<sub>2</sub>Se nanosheets sensitized by PbSe CQDs was reported. Compared to pure Bi<sub>2</sub>O<sub>2</sub>Se or PbSe CQDs, the interfacial band offset between the two materials enhances the device's responsivity and the response time. This PbSe CQDs–Bi<sub>2</sub>O<sub>2</sub>Se photodetector can render an infrared response above  $10^3$  A/W at 2  $\mu$ m under external field effects [71].

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