**Surface acoustic wave NO2 sensors utilizing colloidal SnS quantum dot thin films**

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**ABSTRACT:** Colloidal quantum dots (CQDs) have shown their advantages in gas-sensing applications due to their extremely small particle size and facile solution based processes. In this study, a high sensitivity of surface acoustic wave (SAW) NO2 sensor was demonstrated using SnS CQDs as the sensing layer. The delay line based SAW device with a resonant frequency of 200 MHz were fabricated on ST-cut quartz substrate. The SnS CQDs with average sizes of 5.0 nm were synthesized and deposited onto SAW sensors using a spin-coating method. The fabricated SAW sensor was capable of detecting a low concentration of NO2 gas at room temperature with a good efficiency and selectivity e.g., with a 1.8 kHz decrease of center frequency of the SAW delay line when exposed to 10 ppm NO2 at room temperature.

***Keywords:*** Colloidal quantum dots; Surface acoustic wave; Gas sensor; Nitrogen oxide; Tin sulfide;

**1. Introduction**

Nitrogen dioxide (NO2), a colorless and hazardous gas, is commonly found in [fossil fuels](https://www.sciencedirect.com/topics/chemistry/fossil-fuel) and automobile exhausts [1]. The emissions of NO2 gas results in formation of [ozone](https://www.sciencedirect.com/topics/chemistry/ozone) and [acid rain](https://www.sciencedirect.com/topics/chemistry/acid-precipitation) as well as lung disease, which causes adverse environmental impact and threaten people’s health [2]. Therefore, monitoring low concentrations of NO2 gas is essential for ensuring good air quality and our health.

Over the past few decades, various sensitive materials such as metal oxides [3-7], polymers [8-10], carbon materials [11-14] and colloidal quantum dots (CQDs) [15-17] have been developed to detect NO2 gas. Among them, CQDs have shown exciting prospects for uses in gas sensor devices. Firstly, large surface-to-volume ratio can provide plenty of active sites for the absorption of target gas molecules. Secondly, the facile solution processability of CQDs enable convenient fabrication of solids directly from the solution phase, which can be deposited onto any reasonable substrate. Most importantly, CQD-based gas sensors can be operated at room temperature, and this is very attractive because of their low power consumption and reduced operational costs. Lead chalcogenides such as PbS [15] and PbSe [17] have been used in the demonstrations of CQD-based NO2 sensor devices. However, their lead content raises environmental and health concerns that may limit their applications.

Recently, tin chalcogenides have attracted significant attention due to their abundance in nature, low cost, and more importantly nontoxic nature [18-20]. As a narrow bandgap semiconductor, orthorhombic tin (II) sulfide (SnS) is known for its wide spectral absorption range and photosensitivity, and has potential applications in sodium-ion batteries, photovoltaics, as well as thermoelectrics [21-23]. So far, however, only a few efforts have been concentrated on the application of SnS in gas sensors. For example, Yin at al. reported a highly sensitive chemo-resistive gas sensor for room temperature NO2 detection using a single crystal of SnS [24]. Afsar et al. tested acetone and alcohol sensing performance of two-dimensional SnS nanoflake based sensors by a solid state reaction method at 600 oC [25].

Herein, for the first time, we developed a NO2 gas sensor made using a layer of SnS CQDs on a ST-cut quartz SAW device, with advantages of high sensitivity, good selectivity and wireless and passive capabilities [26].The solution-processed SnS CQDs were synthesized via a hot injection method and then intergraded onto the SAW devices using the low-cost of spin-coating method, this has significantly simplified the preparation process with much lower cost compare to the other SAW based NO2 sensors fabricated using methods such as magnetron sputtering [27,28]. Experimental results indicate that the sensor device exhibits highly sensitivity toward low concentrations (1-10 ppm) of NO2 gas at room temperature with full recovery.

**2. Experimental section**

*2.1 Preparation of SnS CQDs*

Synthesis of SnS CQDs were carried out using a vacuum/argon Schlenk line through the reaction of Sn-oleate and thioacetamide (TAA) which has been published in literature [29]. Briefly, SnCl2 (0.38 g, 2.0 mmol), trioctylphosphine (TOP, 6.7 mmol, 3 mL), oleic acid (OA, 14.2 mmol, 4.5 mL) and 1-octadecene (ODE, 15.6 mmol, 5 mL) were mixed in a three-neck flask and heated to 60 oC under a vacuum condition for 1 h. The temperature of the flask was increased to 100 oC and then 1 mmol TAA in 3 mL TOP and 5 mL oleylamine (OLA) were rapidly injected inside. After the injection, the heating mantle was turned off and the flask was then transferred into a cold water bath to let the flask temperature decrease to 60oC. The product was rinsed with ethanol and finally dispersed in octane at a concentration of 20 mg/mL.

*2.2Sensor fabrication*

As shown in Figure 1, the SAW gas sensor utilized in this paper consists of an ST-cut quartz, input (220 finger pairs) and output (95 finger pairs) interdigital transducers (IDTs) with a periodicity of 15.8 μm and a sensitive area of 3.6 mm2. Quartz is adopted as the piezoelectric substrate for its nearly zero temperature coefficients at room temperature. The measured center frequency of the device is 200.02 MHz and the insertion loss is -12.82 dB. For film deposition, the solution of SnS CQDs was dropped onto the sensitive area of the SAW device with a pipette, and then spun at 2000 rpm for 45 s to form a sensing layer. There is no need for any post sintering as the CQDs are highly crystalline.

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Figure 1. The schematic (a) and a photograph (b) of the SAW sensor.

*2.3 Characterization*

X-ray diffraction (XRD) measurement was performed using a diffractometer (MAXima\_XXRD-7000, Shimadzu, Japan) with a scanned range of diffraction angle 2θ from 20° to 80°. The UV-vis absorption spectrum was measured with a PerkinElmer Lambda 950 UV/vis/NIR spectrometer. The shape and size of SnO2 CQDS were investigated using a high-resolution transmission electron microscopy (HR-TEM, JEOL, model JYOL-2100), and a scanning electron microscopy (SEM, FEI model SIRION 200).

*2.4 Gas sensing measurement*

The SAW sensor was put into a chamber with a volume of 1 L and then connected to a network analyzer (Keysight, E5071C) to monitor the dynamic frequency shifts of sensor (Figure 2). A syringe was used to inject the target gases into the testing chamber and the response of the sensor was recorded. The concentrations of the NO2 gas were controlled by adjusting the injecting volumes from the syringe. When the sensor response became stable, the test chamber was opened and the NO2 gas was removed by flowing with fresh air, and thus the response of the SAW sensor device could be recovered to its original value.



Figure 2. Scheme of the experimental setup for NO2 sensing testing.

**3. Results and discussion**

*3.1 Characterizations of SnS CQDs*

Figure 3 shows the XRD pattern of the as-synthesized SnS products. All the sharp diffraction peaks can be indexed to those of the orthorhombic SnS (JCPDS No. 39-0354) and no other crystalline impurities are identified. The HR-TEM images in Figure 4 further indicate the highly crystalline structure of the as-synthesized SnS products, which are spherically shaped dots with an average diameter of about 5 nm. The fringe intervals of 0.31 and 0.29 nm are corresponding well with the d-spacings of (021) and (101) crystal planes of orthorhombic SnS, which agree well with the XRD results. UV-vis absorption spectrum of the SnS CQDs was obtained and the corresponding typical result is shown in Figure 5. To identify the optical band gap energies of the prepared SnS CQDs, the Schuster-Kubelka-Munk absorption function is plotted against the photon energy according to the following relational expression:

(1)

where *α*, *h*, *v* and *E*g are the absorption coefficient, Planck's constant, frequency of vibration (nm) and band gap energy (eV), respectively. A is a proportionality constant and n is dependent on the optical transition characteristics of the semiconductor (direct: n = 1/2, indirect transition: n = 2). As shown in the inset in Figure 5, the SnS CQDs exhibit an direct bandgap. By extrapolating the linear portion of the plot (*αhv*)2 versus *hv* to zero, the optical band gap energy of the sample can be estimated to be 1.58 eV, which showed a widened band gap compared to that of material (1.30 eV). This clearly confirms the quantum confinement feature of the as-synthesized SnS CQDs. These observations suggest that the experimental process parameters are perfect to synthesize the SnS CQDs with small sizes.



Figure 3. XRD pattern of the as-prepared SnS CQDs.



Figure 4. (a) TEM image of the as-prepared SnS CQDs. (b) HRTEM image of the as-prepared SnS CQDs.

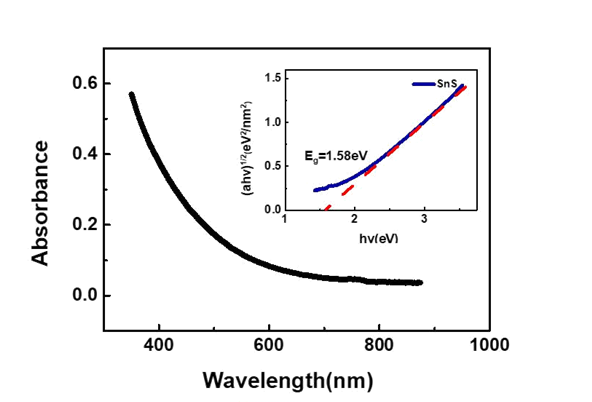


Figure 5.UV–vis absorption spectrum of the as-prepared SnS CQDs.

*3.2 Gas sensing properties*

A layer of SnS CQD thin solid films was spun-coated onto the SAW device at room temperature in air. Transmission coefficient (S21) responses of the SAW devices before and after coated with SnS CQDs were measured prior to the gas sensing experiments. Figure 6 indicates that there are apparent attenuation produced by forming a SnS layer on the SAW device, e.g., about 0.53 dB decrease in the insertion loss; a frequency shift in the phase response of 240 kHz due to the mass loading effect. The low insertion loss produced by the SnS CQDs might be attributed to the flat and compact morphology of the CQD film, which can be seen in the top-view SEM image (Figure 7). This is favorable for the gas sensing measurement because the lower the insertion losses are, the lower the noise level during the measurement.

Figure 8 shows the response curves of the SAW sensor coated with the SnS CQD layer for exposure to 10 ppm NO2 at room temperature. The SAW frequency was decreased upon exposure to NO2 gas, and a frequency shift of ~1.8 kHz was observed after 180 s. Replacement of the NO2 gas in the chamber with fresh air make the frequency back to the initial level within a recovery time around 446 s. The frequency shift during the process was highly repeatable, and a standard deviation of ~37 Hz was found for three successive exposures.



Figure 6.Measured S21 for the SAW device with and without SnS films.



Figure 7. SEM images of the SnS CQD film coated on SAW sensor.



Figure 8.The repeatability testing of the sensor to 10 ppm NO2 at room temperature.

Generally, both the mass-loading effect and acousto-electric interaction effect can result in the decrease of the SAW frequency upon exposure to target gases [30]. It should be noted that the SAW gas sensor in the present work was fabricated on the ST-cut quartz substrate, which has a low value of electro-mechanical coupling coefficient, e.g., k2 = 0.11% [27]. Therefore, mass loading effect could be the dominant mechanism due to adsorption of target NO2 gas on the surface of SnS CQD sensing layer for the SnS/quartz SAW gas sensor. At room temperature, the NO2 molecules are physically absorbed on the surface of the SnS CQD layer and thus induce an increase in the mass, which hinders the acoustic wave propagation and thus results in a decrease in the center frequency. When the NO2 gas was released and dry air was supplied, the sensor frequency was recovered to its original value. The full recovery at room temperature might be explained that the binding energy of NO2 on SnS CQD surfaces was just moderately higher than oxygen [15], and thus it could be desorbed easily after pumping. Besides, SnS gas sensor has a good short-term stability against oxidation at room temperature, which is consistent with the full recoverability once NO2 gas was released [31].

Figure 9a shows the typical dynamic responses of the SnS CQD based SAW sensor towards different concentrations of NO2 gas operated at room temperature. Results showed that as the NO2 concentration is changed from 1 ppm to 10 ppm, the sensor response is increased from 0.5 kHz to 1.8 kHz. Moreover, as shown in Figure 9b, the sensor response shows an almost linear trend with the concentrations of NO2 gas, suggesting its advantages in low-concentration NO2 detection.



Figure 9.(a)The response curves of the SAW sensor to different NO2 concentrations.

(b) The dependence of the response on NO2 concentration.

The theoretical detection limit of the sensor was estimated according to the least-squares method of fitting in the linear regime. The slope of the frequency shift in the linear regime in Figure 9b was -122.2 ppm-1 with a fitting quality *R*2= 0.979. We then calculated the noise of the sensor using the variation in the relative frequency shift in the baseline using the root-mean-square deviation (RMSD) [32]. In detail, 200 data points of the initial frequency in Figure 9a (the baseline of the sensor before the NO2 exposure) were averaged and a standard deviation (*D*) was gathered as 30.2. Therefore, the noise of the sensor was calculated to be 2.12 according to the Eq. (2) and the theoretical detection limit (*DL*) of the sensor is around ~52 ppb according to Eq. (3).

(2)

(3)

Selectivity is another important factor to evaluate the performances of gas sensors. Fig. 10 shows frequency shifts of the SnS CQD based SAW gas sensor when exposed to various detected gases. It can be seen that the frequency shift when exposed to 10 ppm of NO2 gas is 1.8 kHz; whereas the frequency shifts are 0.08 kHz, 0.10 kHz, 0.05 kHz, and 0.03 kHz for 50 ppm of ammonia (NH3), sulfur dioxide (SO2), nitric oxide (CO) and hydrogen (H2), respectively. Therefore, it can be concluded that the sensor based on SnS CQD thin film exhibits a superior NO2 selectivity compared to those for NH3, SO2, CO and H2 gases. The superior selectivity of the sensor to NO2 gas is attributed to the largest adsorption energy of NO2molecules on SnS surface compared to those interference gases [33].

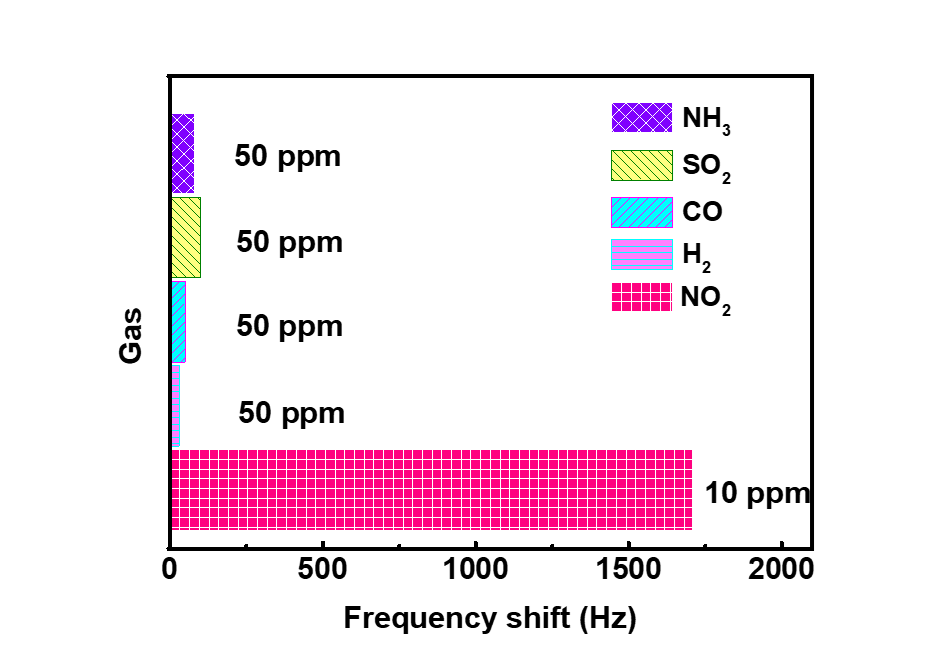


Figure 10. The selectivity of the sensor.

**4. Conclusions**

In this work, we for the first time fabricate a solution-processed SnS CQDs layer and manage to integrate it into a SAW delay lines for detection of NO2 gas at room temperature. Upon exposure to 10 ppm NO2 gas, the center frequency of the sensor device decreased by 1.8 kHz with the response and recovery times of 180 s and 466 s, respectively. The sensor showed a linear dependence of frequency shift upon NO2 concentrations in the range of 1-10 ppm and the theoretical detection limit was calculated to be around 52 ppb. The origin of frequency shift toward the lower value side might attributed to the efficient adsorption of NO2 gas molecules on the surface of SnS CQD film and subsequently leading to the mass loading effect.

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