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Synthesis and study of structural properties of MoS_2@MoO_3 hybrid structure for highly enhanced H_2S gas response

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Abstract:
In this work, we synthesized MoS_2@MoO_3 hybrid structure by a two-step method. In the first step, the thin films of MoS_2 was obtained by chemical bath deposition (CBD) method. During the synthesis, the interaction was between ammonium molybdate ($(NH_4)_6Mo_7O_{24}.4H_2O$) and sodium sulfide(Na_2S) solution, a products contain a mixture between amorphous molybdenum disulfides and oxide is obtained. In the second step MoS_2 thin films were annealed in an air ambient at temperature of 100℃ , 200 ℃ and 300℃. As-deposited MoS_2 thin film and annealed thin films were characterized by X-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), atomic force microscopy (AFM) and energy –dispersive X-ray analysis(EDX). The analysis of XRD showed that the as-deposited thin film and annealed thin film at 150℃ was amorphous while annealed thin films at 300℃ for 2hours directly lead to partial oxidation and the formation of α-MoO_3.The atomic force microscopy analyzes revealed a change in the surface morphology of as-deposited film from mount like to column –like structure formations with small protrusions, when the temperature is varied between RT and 300℃.The analyzes of FEMES revealed the annealed thin film at 300℃ have spherical-like shapes and stacked of plate –like morphology. EDX analysis was performed to confirms co-existence of Molybdenum, sulfur and oxygen in as-deposited thin film and annealed thin films. The gas – sensing characteristic of sensor were investigated using reducing gas (H_2S) at working temperatures ranging from RT to 200℃. The MoS_2@MoO_3 gas sensor display an N-type like response towards H_2S with high sensitivity of 92% to 100 ppm H_2S at 300 ℃.
Key words: chemical bath deposition, molybdenum disulfides, gas sensor, Hydrogen sulfide

1-Introduction

Gas sensing is an electronic device important in our society, that quickly identifies the presence and /or concentration of a toxic gases and organic vapours and has been widely used in many fields such as agricultural production and environmental monitoring medical diagnosis etc[1]. A continuous exposure to different gases such as CO, NH_3, SO_2, NO, H_2S and NO_2 on human body may be inflict serious health problem like cough, lung cancers and asthma even at an extremely low concentration[2]. Among them, Hydrogen sulfide gas(H_2S) is colorless, extremely toxic, hazardous, and flammable with a strong odour of rotten eggs[3]. it is produced naturally crude oil, coal mines, volcanic gas, natural gas and decaying of vegetation and marine micro-organisms. [4,5]. H_2S gas is responsible for many accidents caused by exposure to toxins during work,

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especially in the coke ovens, sewage treatment, natural gas and petroleum industry. The health effects of H_2S gas depend on its concentration and the duration of exposure. H_2S gas is immediately fatal when inhalation of high concentrations are over 500-1000 ppm [6]. when H_2S gas is inhaled, It is toxic because when it enters the bloodstream , it combines with the hemoglobin in red blood cells, preventing the absorption of oxygen into the blood, which can lead to asphyxiation [4]. In this frame, we have decided to study the influence of hybrid structure of MoS_2 @ MoO_3 thin films on the detection of hydrogen sulfide (H_2S). Fabrication and synthesis of hybrid 2D heterostructure by sulfurizing α-MoO3 and/or oxidizing MoS_2 layered crystals have been attracting many researchers interest in recent years [7]. Many studies have indicated that crystalline MoO_2 could present in material conversions from MoO_3 to MoS_2 upon thermal vapor sulfurization (TVS) but this rarely happens in the reverse conversions, i.e. from MoS_2 to MoO_3 upon oxidation[8] Molybdenum disulfide (MoS_2), is one of the transition metal semiconductors, and belong to the class of dichalcogenides [9]. It has a unique layered structure of hexagonally structured network of molybdenum (Mo) and sulphure (S) atoms , in which layer of Mo atoms are sandwiched between two layers of sulfur atoms and held together by covalent bond , whereas adjacent layers of (S-Mo-S) are formed by week van-der-waals interaction[10]. These van der waals interactions allow gas molecules to adsorption and diffuse freely within these layers, MoS_2 has character a enormous active sites, larger surface to volume ratio, higher adsorption efficiency, high mobility at room temperature and more crystal defects, which prerequisite for sensor gas. As a result, the resistance of MoS_2 can a significant change when the adsorption and diffusion of gas molecules between the sensing layers[11]. The chemical property of MoS_2 is very stable , thus it is insoluble in dilute acid, organic solvent and water, but can react with aqua regaia, hot nitric acid and hot sulfuric acid [12]. Other properties of MoS_2 are : the ability to adhere strongly to the bearing surface , the tendency to prevent contamination or erosion, being chemically inert, stability against radioactive agents ,and its non-dependency on the adsorbed vapors for lubrication[10].

Besides Transition Metal Dichalcogenids (TMDs), another layered molybdenum-based material is orthorhombic α-MoO3, which is the thermodynamically most stable phase has a unique layered atomic structure can also be exfoliated to generated isolated 2D layers that can be used in different fields such as photochrmic device, solar cell , electrodes in supercapacitors[9].

For the synthesis (MoS_2 and MoO_3), numerous approaches have been investigated , like hydrothermal [13] reactions between solutions[14], chemical vapor depositions [15], decomposition and annealing of precursors [16] and many other method. In this work used the CBD method because of its advantages including very simple, low cost and can be used for large area deposition.

2-Experiential Work

The MoS_2@MoO_3 hybrid structures was synthesis by using two –step. In First MoS_2 thin films are prepared onto glass substrates by chemical bath deposition. The glass substrates washed by acetone , ethanol and then distilled water. Thin films of MoS_2 was deposited on the glass substrates by CBD in aqueous solution containing 50ml of 0.05M ammonium molybdate ($(NH_4)_6Mo_7O_{24}.4H_2O$) was used as source material of molybdenum in 100ml chemical cell. then, 0.5ml of 1M sulphuric acid (H_2So_4) were mixed with the above solution. Later, 20ml aqueous solution of 0.2M sodium sulfide(Na_2S) was added to reactive solution as source material of

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sulphur . After stirring for several minutes, the solution color became brown and homogenous ,then cleaned substrates are immersed vertically in the reactive solution. The deposition process of MoS_2 thin films was carried out in thermal bath at 40°C for 24 hours, without stirring. After the MoS_2 deposition, the glass substrate was removed from the reactive bath and washed by deionized water and air dried. In the second step MoS_2 films were annealed in a furnace at two different temperature (150°C and 300°C) for 2 hours.

The surface morphology of $MoS_2@MoO_x$ hybrid structure were carried out field emission scanning electron microscopy(SEM, EBSD-ZESS SIGMA VP Germany)and atomic force microscopy (AFM, AA 3000 scanning probe microscope. tib NSC35/AIBS).Structural studies of the thin films was carried out using an X-ray diffraction (XRD, Bruker D2 PHASER) with Cu-Ka radiation having wavelength λ= 0.15406nm at 40 Kv, 30mA, the X-ray scans were performed between two angle 20° and 80°. Elemental composition of $MoS_2@MoO_x$ was carried out using energy –dispersive X-ray analysis(EDAX). The sensing response (S%) of the gas sensor to H_2S gas is defined as[17].

$$(S\%) = \frac{R_a - R_g}{R_a} \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (1)$$

Where, R_a, R_g is sensor resistance in the air and the target gas respectively.

3-Results and Discussion

1- X-ray diffraction

The X-ray diffraction (XRD) for as-deposited thin film and annealed at 100°C , 200°C and 300°C as shown in figure(1).The result of XRD Patterns characterized by no peak ascribable to the crystal phase like MoS_2 and MoO_3, as well as the existence of the typical amorphous halo, that confirms prepared material is poor crystallinity and were almost amorphous as shown in fig.1(a, b), which suggests a large extent of destacking in the synthesized MoS_2[18], whereas annealed film at 200°C is characterized by the presence of minor peak at 2θ = 23.5 . The elemental analysis of the thin film confirms that the films seem to be a mixture of molybdenum oxides and molybdenum sulfides (table 2) as Afanasiev et al. showed that aqueous solution of ammonium heptamolybdate reaction conditions might yield different compounds such as binary sulfides of MoS_6, $MoS_{5.6}$ and MoS_3 as well as binary oxides of MoO_3 and MoO_2 [19].Heat treatment of MoS_2 at 350°C is product MoO_3 [20]. Figure (1,C)shows the XRD pattern of annealed film at 300°C for 2hours directly lead to the formation of MoO_3. The peaks at 2θ = 23.57, 25.79 and 27.46 belong to the orthorhombic $\alpha - MoO_3$(JCSPS# 050508)[9] , which are indexed to the (110), (040) and (021)respectively , and other peaks at 2θ =33.88 and 39.0815 corresponding to reflection from (100)and (103) plane are in agreement for MoS_2 (JCSDS #37-1492)[21]. The crystallite size shows in table(1)of $MoS_2@MoO_3$ hybrid structures synthesized at annealed temperature of 300°C was calculated using the Scherer formula[22].

$$D = \frac{0.9\lambda}{\beta cos\theta} \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (2)$$

Where, D is crystallite size , λ is the wavelength of the X-ray used , which is 0.15406μm, β is full width at half maximum(in radians) , and θ is Bragg angle of the XRD Peaks.

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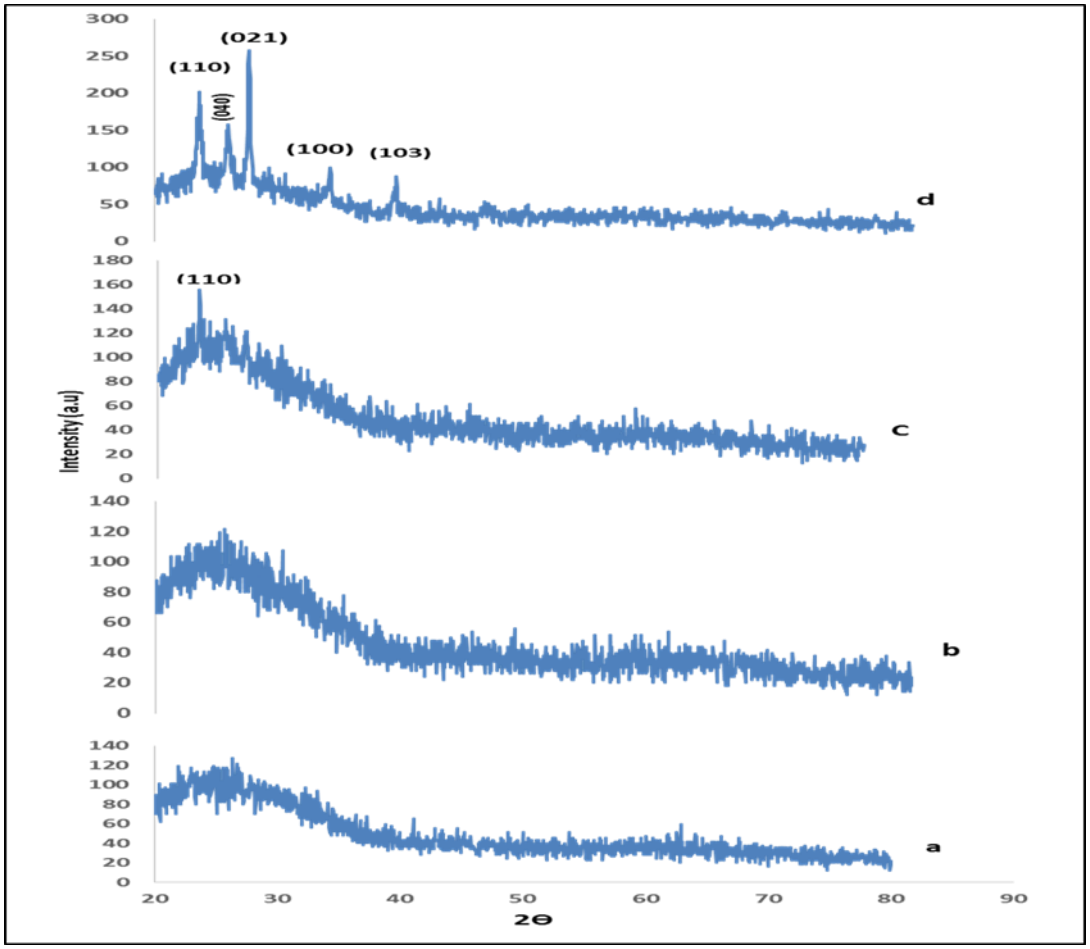

Figure 1 XRD pattern of thin films for (a) As-deposited MoS_2 and different temperature (b)100℃, (c)200℃, (d)300℃,

2-Surface morphological study

Fig.2 (a- d) shows FE-SEM images that the levels of evolution for thin film were prepared on glass substrates. Fig. 2(a, b, c) shows the surface morphologies of the as-deposited film and annealed film at 100℃ temperatures can be seen in the samples (spherical-like shapes and plate-like shapes), characteristic of different proportion of MoS_2@MoO_x.The average particle size obtained from FE-SEM images are 42.43-228.4nm and 31.26-97.95.5nm of as-deposited film and the film annealed at 100℃ respectively. At 300℃ annealed temperature thin film grain boundaries are prominent and homogeneous morphology characterized by spherical-like shapes and stacked of plate –like morphology, with the average particle size obtained from FESEM images is 58.06-155.1nm. From the FE-SEM images and XRD spectra, we conclude that the films contain MoS_2 nanostructure and MoO_3 nanostructure.

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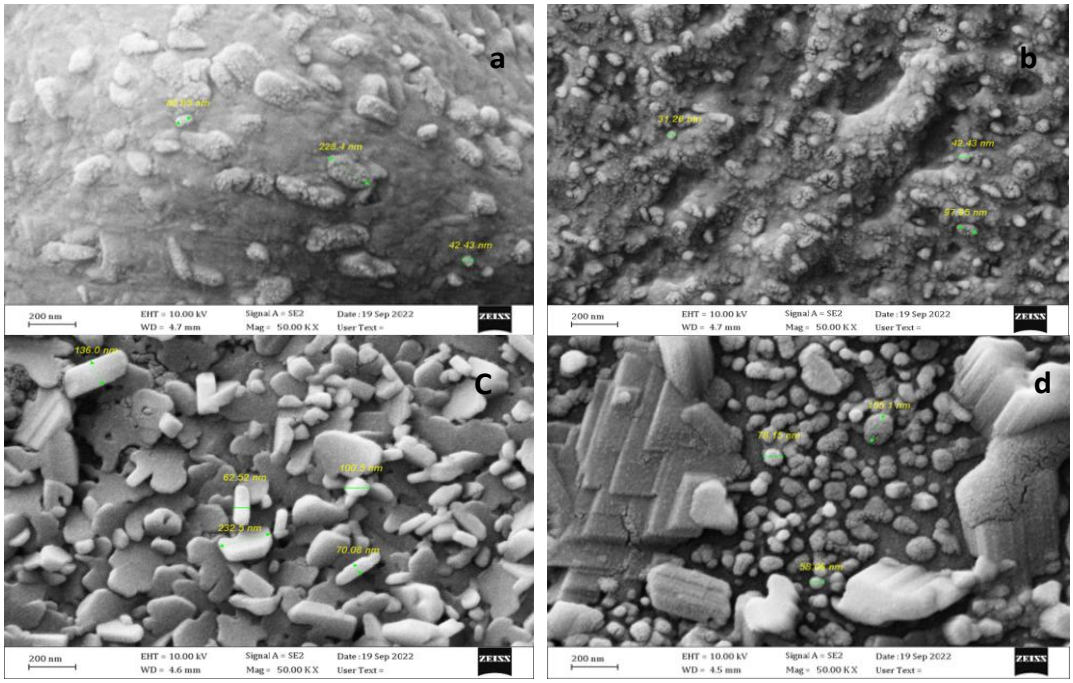

Figure 2. FE-SEM of thin films for (a) As-deposited MoS_2 and different temperature (b)100℃, (c)200℃, (d)300℃, .

3-AFM characterization

Fig.3 (a- d)shows the atomic force microscopy (AFM) images of the surface of all prepared thin films, the impact of annealing temperature on the grain size and surface morphology of the thin films. as clearly seen from figure that the surface continuity of the film is very good, and over the complete substrate is covered by the $MoS_2@MoO_x$ film without voids and pinholes. Three-dimensional AFM image reveals that the surface of as-deposited film is rough with few mount like structures, but after annealed column –like structure formations with small protrusions as compared to the unannealed film, which can be attributed to the crystalized nature of $MoS_2@MoO_x$ thin film. This is in agreement with predictions of fig.2(d) and XRD results. AFM gives Rq and Ra morphology that is the root mean square average and arithmetic average of height deviation taken from the mean image data plane. The as-deposited sample contain many contrast particle sizes which lead to high surface roughness, Rq=108.1nm, but at an annealing temperature of 100℃ and 300℃ could lead to coalescence of multiple growth clusters and systematic reduction in the surface roughness from 15.91 to 3.343nm respectively as shows in table (1).

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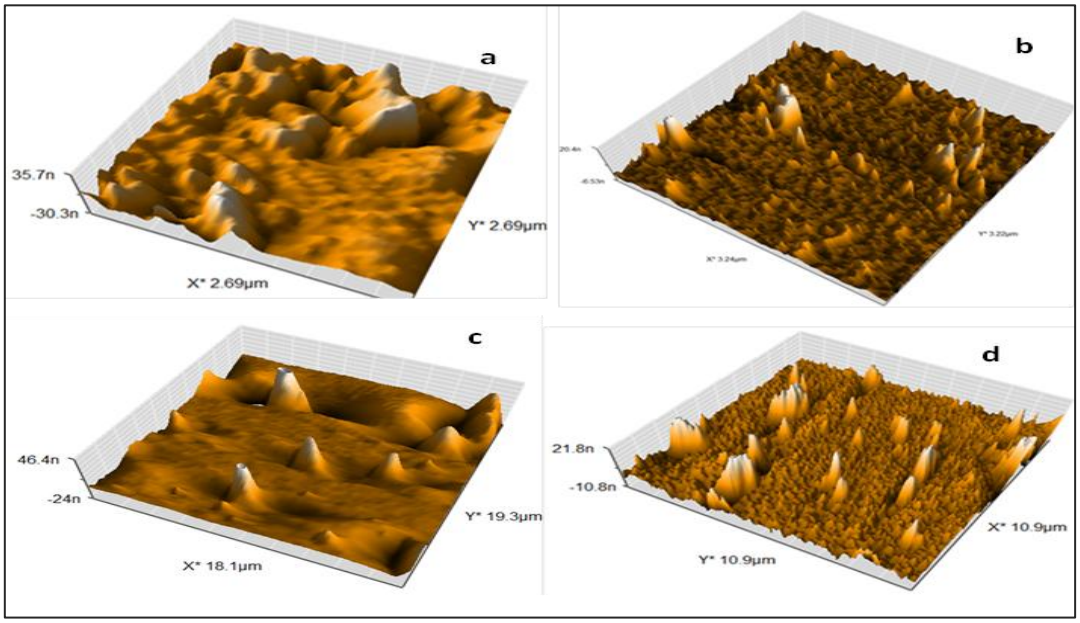

Figure 4. AFM analysis of MoS_2 films at (a)as-deposited and annealed at (b)100℃, (c)200℃, (d)300℃

Table (1) Variation of roughness , root mean square and grain size of as-deposited thin film and annealed at 100℃ , 200℃ and 300℃ from AFM, FESEM and XRD.

Annealing temperature℃	Grain size (nm)AFM	Grain size (nm)FESEM	Grain size (nm) XRD	Rq (nm)	Ra (nm)
As-deposited	127.5	~42.43-228.4	-	108.1	85.97
100	83.75	~31.26-97.95	-	15.91	12.76
200	356.9	~62.52-232.5		5.122	2.455
300	88.87	~58.06-155.1	56.862	3.343	1.965

4- Energy-dispersive X-ray.

Compositional analysis of films are carried out utilizing the EDX technique to measure the proportion of elements existence in the prepared film. The result of EDX analysis clearly showed the presence of Molybdenum (Mo), sulfur (S) and oxygen (O) as major elements of film. Additionally, the peaks of Na and Si elements appear from the substrate. The energy of Mo and S elements ranging from 2kev to 2.6 Kev which is similar to previously results were obtained by other researcher[14,21]. The weight percentage obtained from EDX analysis as shown in table (2) of Molybdenum is evaluated as (58.6)whereas sulfur is found to be (4.2) and oxygen (29.0) at room temperature, while at annealed temperature of 300℃, the intensity of Oxygen peak is quite greater than Molybdenum indicating that the amount of oxygen increases with the deficient in sulfur content, this confirming formation MoO_3, which is consistent the XRD results.

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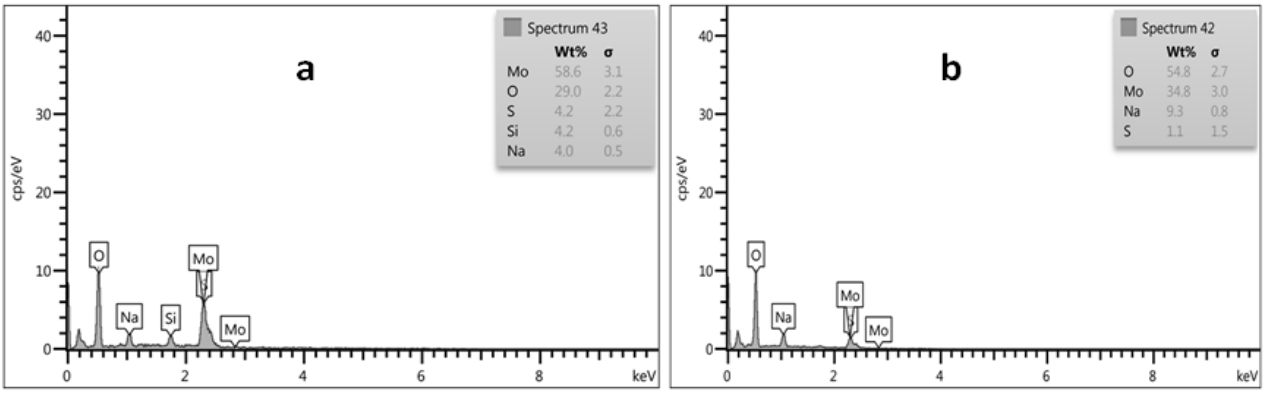

Fig.3 EDX spectra of MoS_2 thin film of as-deposited and annealed at 300 °C

Table 2 . Elemental concentration determined by DEX of as-deposited film and annealed at 300 °C

Annealing temperatures (°C)	Elements					Total
	MO	O	S	Si	Na	
	Weight%					
As-deposited	58.6	29.0	4.2	4.2	4.0	100%
300 °C	34.8	54.8	1.1	-	9.3	100%

5-Gas-sensing performance

Sensing response characteristics of the MoS_2@MoO_3 hybrid structure (as-deposited thin film and annealed at 100 °C, 200 °C and 300 °C) were investigated over a temperature range of RT-200 °C for 100 ppm of H_2S is shown in Fig. (4). It note from the table that the sensitivity decreases with increasing working temperature for as-deposited film and annealed at 100 °C , while annealed films at 200 °C and 300 °C increase with increasing working temperature, this may be attributed to the roughness of the surface of the film shown by AFM . The presence of high surface roughness for as-deposited film and annealed at 100 °C seems to be advantageous for obtaining the enhanced response due to large surface area available in sensing layer for adsorption of gas molecules[23], faster charge transport in sensing layer, impact of modulating favourable adsorption sites by morphology[mos_2 revio]. The gas sensor clearly_exhibit the highest performance at room temperature within the measured temperature range, which can be attributed to large various active sites to enhance the gas molecules adsorption at RT [2]. Namely, the number of defected such as vacancies was play a crucial role in the sensor at RT. These defects act as adsorbing sites for the target gas, thereby increasing gas response at RT[19]. When the working temperature of sensor was increased, the number of defects decreased, which reduced the number of sensing sites on the film surface. Thus, the gas response decreased at higher temperature . The sensor based on the MoS_2@MoO_3 annealing temperature of 300 °C was expected to have the highest sensitivity (92%).

The response /recovery times at higher sensitivity to 100 ppm H_2S were about 26s/35s , 20s/18s, 35s/33 and 26s/37s for as-deposited and annealed thin film at 100 °C, 200 °C and 300 °C respectively.

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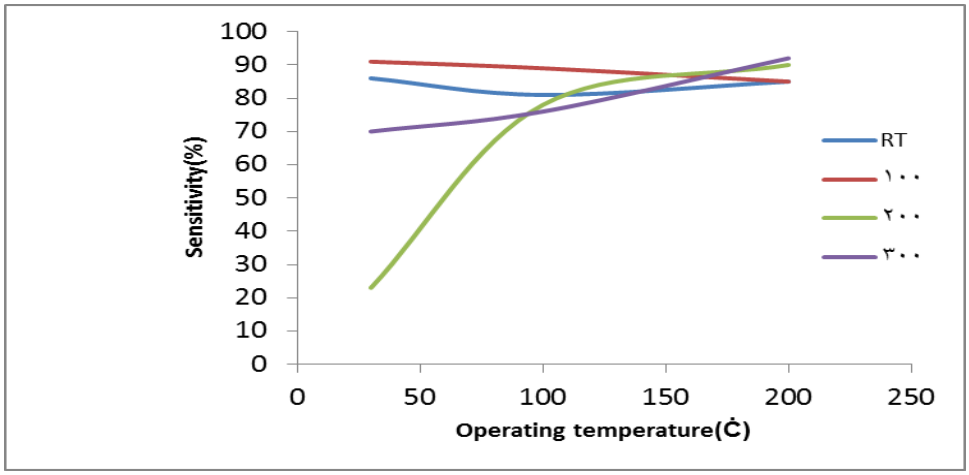

Fig.4 the variation sensitivity with the operating temperature of as-deposited MoS_2 and annealed at 100 °C 200 °C and 300 °C.

To test the selectivity of the sensor, the gas response of the sensor toward another four gases (NO_2, Ethanol, oxygen and propane), measurement were performed at the operating temperature of RT and 200 °C for as-deposited film and annealed at 300 °C respectively. The sensor exhibited maximum response towards H_2S as compared to gases for as-deposited at operating temperature of RT as shown in figure(5).

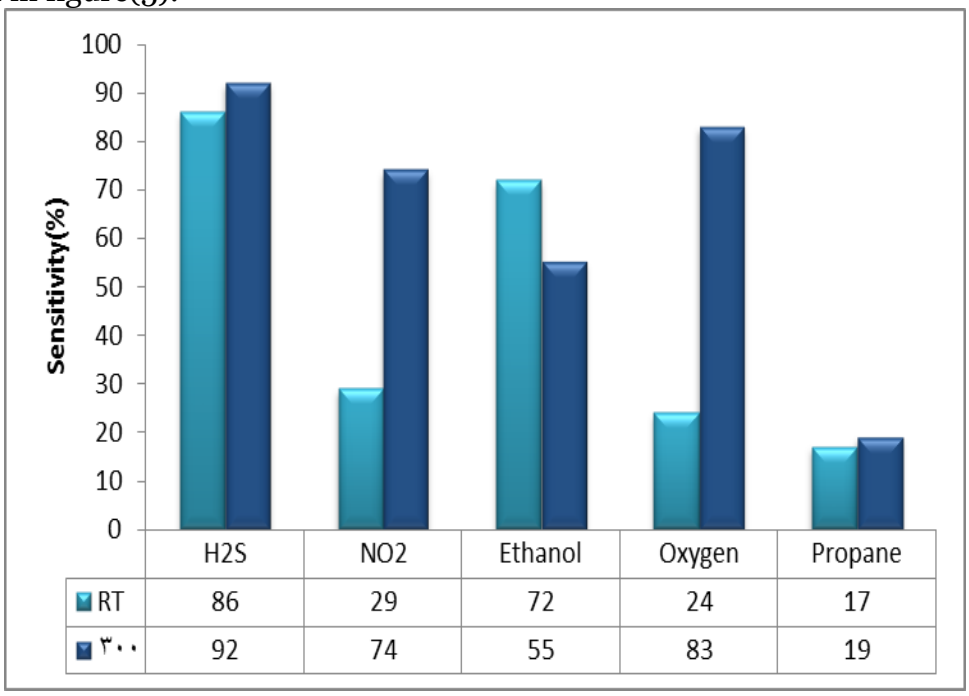

Fig. 5 Selectivity of sensor towards different gases

Table 3 the value of sensitivity for as-deposited MoS_2 thin film and annealed at 150 °C and 300 °C.

Operation temperature	Sensitivity(S%)

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	Temperature annealing			
	RT	100 °C	200 °C	300 °C
RT	86	91	23	70
100	81	89	78	76
200	85	85	90	92

5-Gas-sensing mechanism

The electrical resistance of sensor will vary after being exposed to various reducing or oxidizing gases. The H_2S sensing mechanism is based on the amount of O_2 molecules adsorb on the sensor surface that leads surface adsorbed O_2 species (O_2^-, O^-, O^{2-}) by extracts free e^- from conduction band of the sensor material . the reaction are define as in Equations[21].

$$O_2(gas) \rightarrow O_2(ads) \quad \ldots\ldots (3)$$
$$O_2(ads) + e^- \rightarrow O_2^- \quad \ldots\ldots (4)$$
$$O_2(ads) + 2e^- \rightarrow 2O^- \quad \ldots\ldots (5)$$
$$O_2(ads) + 4e^- \rightarrow 2O^{2-} \quad \ldots\ldots (6)$$

Both MoS_2 and α-MoO_3 are typical N-type semiconductors to the prevalence of anion vacancies in their pristine structure due to their low formatting energy[26]. In the case of n-type semiconductors , the presence of chemically adsorbed oxygen on the material surface could cause decreased in the number of electrons available (electron depletion) in the sensing materials and building up of Schottky surface barrier; consequently, the electrical conductance of sensor decreased to a minimum . Upon exposure to the H_2S gas, the sensor resistance decreased (negative sensitivity) as shown in figure(6). This means that the H_2S gas act as electron donors, transferring electrons to the conduction band of MoS_2[27].The response to H_2S can be explained as a reaction of gas with to $O_2(ads)^-$ [28]

$$3H_2S_{(g)} + 3O_2(ads)^- \rightarrow 2H_2O(g) + 2So_2(g) + 3e^- \quad \ldots\ldots (7)$$

Thus, this leads to increased electron concentration and conductivity , and the resulting n-doping brings the Fermi level closer to the conduction band edge.

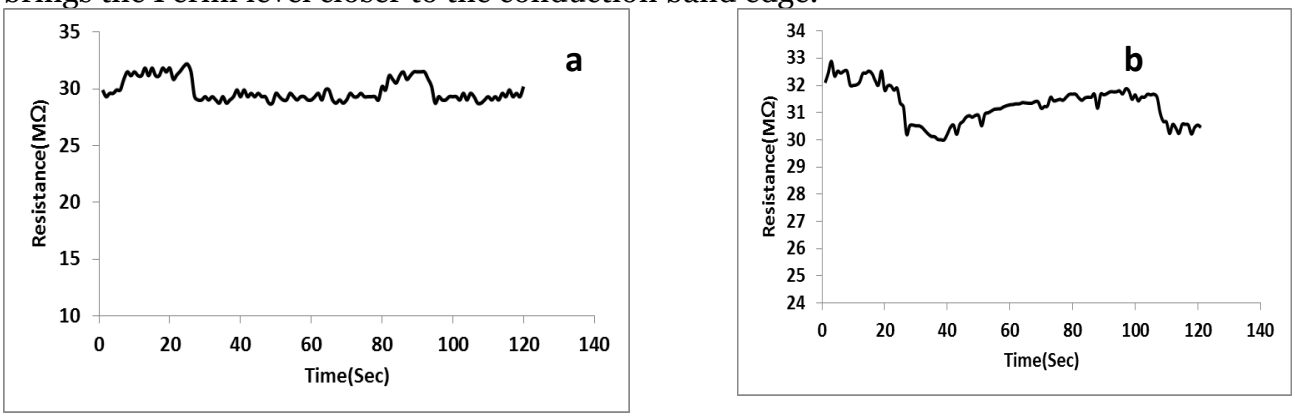

Fig. 6 variation of resistance MoS_2 @ α-MoO_3 as function of time for as- deposited and annealed at 300 °C

Conclusion

MoS_2@MoO_3 hybrid structure have successfully been synthesized by oxidation of MoS_2 on glass substrate. Gas sensors fabricated with oxidation MoS_2 as sensing layers MoS_2@MoO_3 display

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a N-type response towards H_2S with highest sensitivity of 92% To 100 ppm H2S at the optimal working temperature at RT. The gas- sensing mechanism of H_2S was discussed in detail. The spherical-like shapes and stacked of plate –like morphology of $MoS_2@MoO_3$ hybrid structure were observed in FESEM study. As a result, an amount of oxygen in annealed thin film react with molybdenum and became molybdenum oxide .In additionally, that the concentration of molybdenum decrease with the increase in the amount of the oxygen, which was confirmed the formation of this hybrid.

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