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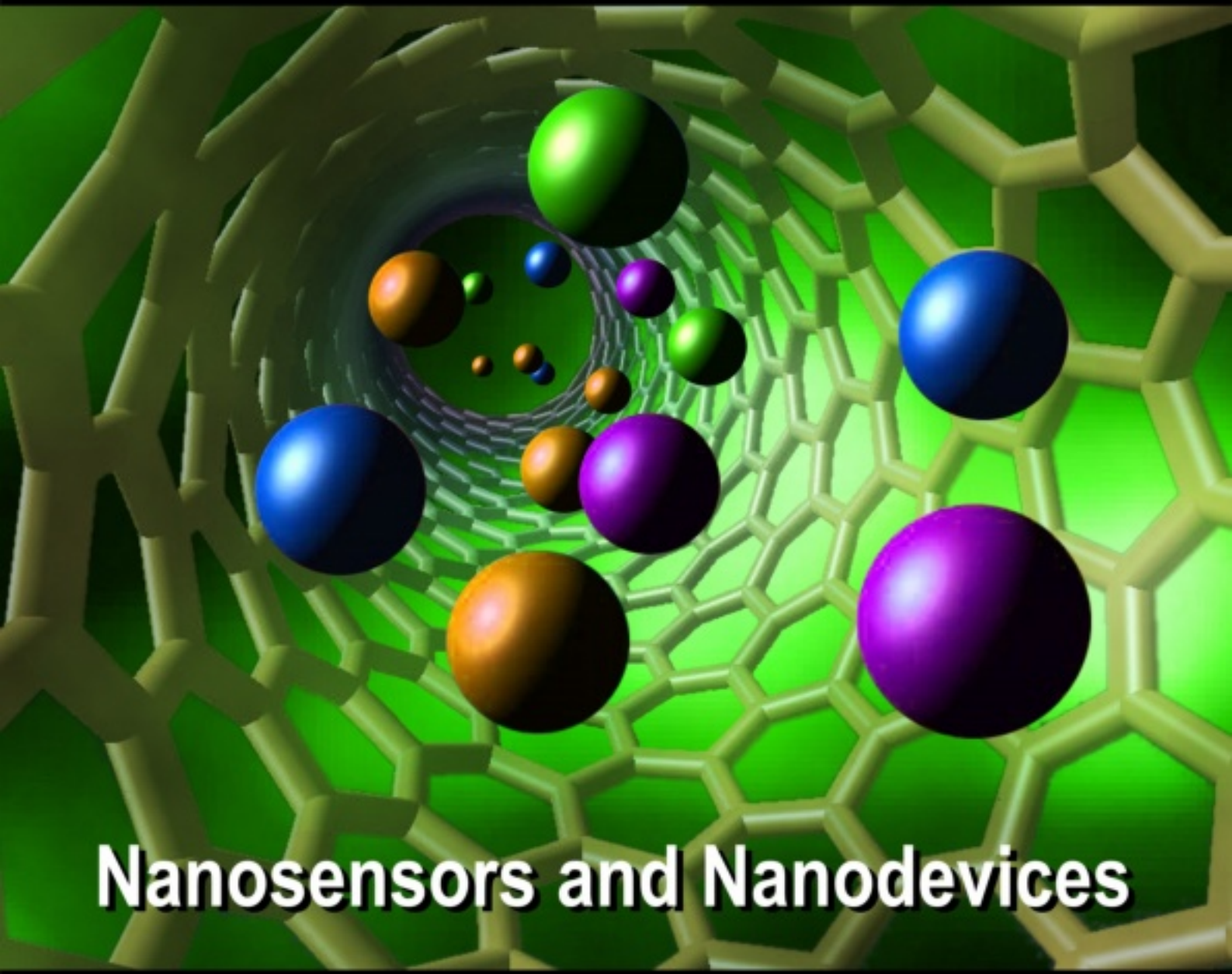

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Detection of Hydrogen Sulphide Gas Sensor Based Nanostructured Ba_2CrMoO_6 Thick Films

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Abstract: Nanocrystalline pure and doped Ba_2CrMoO_6, having an average crystallite size of 40 nm were synthesized by the sol-gel citrate method. Structural and gas-sensing characteristics were performed by using X-ray diffraction (XRD) and sensitivity measurements. The gas sensing properties to reducing gases like Hydrogen sulphide (H_2S), liquefied petroleum gas (LPG), carbon monoxide (CO) and hydrogen gas (H_2) were also discussed. The maximum sensitivity was obtained for 5 wt % Ni doped Ba_2CrMoO_6 at an operating temperature 250^oC for H_2S gas. Pd incorporation over 5 wt% Ni doped Ba_2CrMoO_6 improved the sensitivity, selectivity, response time, and reduced the operating temperature from 250 to 200^oC of the sensor for H_2S gas. This sensor also shows good satiability.

Keywords: Nanoparticles, Ba_2CrMoO_6, Sensitivity, Selectivity, Hydrogen sulphide gas sensor

1. Introduction

Hydrogen sulfide (H_2S) is a colorless toxic and flammable gas, occurring naturally in crude petroleum, natural gas, volcanic gases, and hot springs with smells like rotten eggs. Other sources from industrial activities include food processing, coking ovens, craft paper mills, tanneries, and petroleum refineries. The in situ detection and monitoring of H_2S gas is very important in industrial such as oil and natural gases exploitation plants and coal manufacturing.

With these properties, H_2S has become a recent target of rather extensive research. So far H_2S sensors have mostly been based on semiconducting oxides, such as WO_3 [1, 2] ZnO [3], SnO_2 [4, 5]. Although

these sensors reportedly show fairly good H_2S sensing properties, they have still difficulties in sensitivity and/or response rate to very dilute H_2S. Apart from these, there have been attempts to develop H_2S sensors by using the solid electrolytes of K_2SO_4 [6] and Y_2O_3-stabilized zirconia (YSZ) tube with a sensing oxide layer of WO_3 [7]. The resulting devices can detect H_2S in air, but at high temperature such as 820°C. So, reliable and stable H_2S sensors with high response value, selectivity, fast response and low energy consumption are in high demand for environmental safety and industrial control purposes.

Semiconductor metal oxide (SMO) chemical sensors have shown advantages in commercialization prospect and market potential. These advantages include long lifetime, fast response and recovery time, low cost, simple electronic structure, and low maintenance [8].

There has been much interest in perovskite structured compounds (of general formula ABO_3) because of their unique catalytic action [9] and gas-sensing properties [10-12]. The materials are highly defective and have oxygen deficient structures, in which the valence state of metal ions may be controlled by temperature, oxygen partial pressure and dopants [13]. Ba_2CrMoO_6 was prepared successfully by J. Paul Attfied et.al. [14] having the 6-layered, hexagonal (6H) perovskite structural.

Besides, in order to improve selectivity for particular application, surface modification by proper choice of additives or dopants to the base material is often used. The doping is generally based on the selection of most effective catalysts, which modulate specific chemical reaction on the semiconductor sensor surface. The noble metals, well known as active catalysts, have been confirmed to possess the promoting effects on many semiconductor gas sensors [15–17]. One critical approach is to modify the metal oxide surface by using noble metals (Au, Pt or Pd) [18, 19]. All these results suggested that noble metals played vital roles in sensor response improvement of gas sensors.

The present work was undertaken to investigate the gas sensing behavior of Ba_2CrMoO_6 specimens doped with 5 wt% Ni was prepared by a sol–gel citrate method. The gas sensing properties to H_2S, H_2, CO, and LPG were discussed. The results showed that Ba_2CrMoO_6 doped with 5 wt. % Ni was very promising in detecting H_2S at an operating temperature 250°C due to its high response and selectivity. After Pd incorporation, the operating temperature decreases from 250 to 200°C with increasing sensitivity of H_2S gas.

2. Experimental Details

2.1. Synthesis and Characterization of Material

The Ba_2CrMoO_6 specimens were prepared by using Barium nitrate hexahydrate [$Ba(NO_3)_3.6H_2O$], Chromium nitrate nonahydrate [$Cr(NO_3)_3.9H_2O$], and Ammonium molybdate [$(NH_4)_6Mo_7O_{24}.4H_2O$]. A stoichiometry mixture of nitrates was mixed with citric acid and ethylene glycol. The resultant mixture was stirred magnetically at 80°C for 2 h to get homogeneous mixture. The solution was further heated in pressure vessel at about 130°C for 10 h and subsequently kept at 350 °C for 3 h. The dried powder then calcined in the range of 450-850 °C in order to improve the crystallinity of ceramic. The solution of Nickel nitrate hexahydrate $Ni(NO_3)_3.6H_2O$, was used as dopant in the precursors of Ba_2CrMoO_6. Different noble metals such as Pd, Ag and Au were incorporated in the 5 wt% Ni doped Ba_2CrMoO_6 . Appropriate quantities of corresponding chlorides of Pd, Ag and Au solution were added to the nitrating mixture.

The synthesized samples were characterized for their structure by powder X-ray diffraction using a Siemens D5000 diffractometer .The X-ray diffraction data were recorded by using Cu K_α radiation (1.5406 A°). The intensity data were collected over a 2θ range of 20–75°. The average crystallite size

of the samples was estimated with the help of Scherrer equation using the diffraction intensity of all prominent lines.

2.2. Fabrication of Sensor Device

The nanometer size powders obtained above were mixed with a 2 % PVA (polyvinyl alcohol) as a binder and 5 % ethanol as a solvent; the resulting paste was applied on alumina tube. Then, the paste was packed into an Al_2O_3 ceramic tube on which two electrodes had been installed at each end. The ceramic tube was about 8 mm in length, 2 mm in outer diameter, and 1.7 mm in inner diameter, as shown in Fig. 1. To improve stability and repeatability, the gas sensors were calcined at 600^{o}C for 2 h. The analyte gas was injected into the test chamber through an injection port and the resistance was measured as a function of time till a constant value was attained. Then the chamber was purged with air for about 5 min and the experiments were repeated. The sensor element, with a nichrome heater to provide the necessary temperature and a chromelalumel thermocouple for temperature monitoring is fixed inside the specimen chamber made of aluminum for studying the gas sensing characteristics. The different test gases are injected into the specimen chamber through an inlet. The output voltage across the sensor element at a 10 V input voltage is taken to determine the resistance of the element.

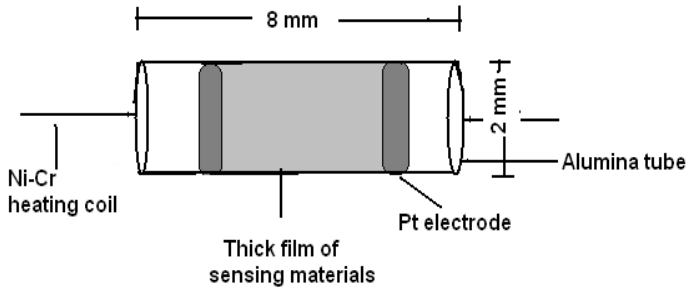

Fig. 1. Schematic drawing of sensor element.

The resistance of a sensor was measured in air and in a sample gas. The gas sensitivity (*S*) is defined as the ratio of the change of resistance in presence of gas (R_g) to that in air (R_a).

$$S = (R_a - R_g)/R_a = \Delta R/R_a \quad (1)$$

The test temperature range is from 50^{o}C to 350^{o}C and the gas concentration tested from 100 -1000 ppm.

3. Results and Discussion

3.1. X-ray Diffraction

The X-ray diffraction pattern of Ba_2CrMoO_6 can be compared with $Ba_3Cr_2MoO_9$ [20]. This has a 6-layered, hexagonal (6H) perovskite structure, as was originally found for one of the polymorphs of $BaTiO_3$ [21]. Fig. 2 (a) shows the XRD pattern of the Ba_2CrMoO_6 synthesized at 550^{o}C for 6 h. A definite line broadening of diffraction peaks gives an indication that the synthesized materials are in nanometer range. The crystallite size was calculated from Scherrer formula applied to the major peaks and was found to be around 40 nm. Fig. 2(b) shows that the 5 wt.% Ni doped Ba_2CrMoO_6 calcined at 550^{o}C for 6 h has also a high degree of crystallinity and complete phase formation. On doping of

5 wt.% Ni, the peak intensity increases indicating the formation of solid solution. No extra peaks are observed due to the addition of Ni in Ba_2CrMoO_6.

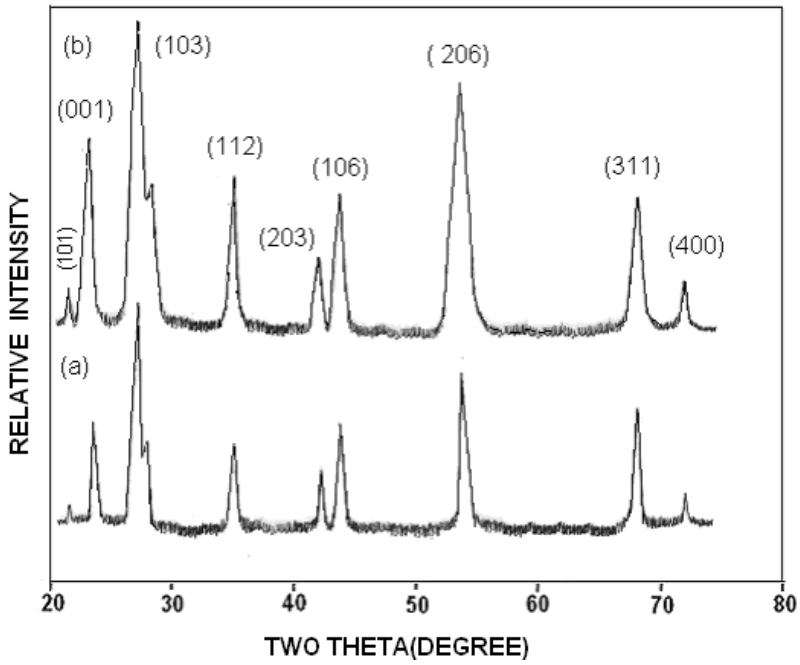

Fig. 2. XRD pattern of (a) Ba_2CrMoO_6 calcined at 550°C and (b) 5 wt. % Ni doped Ba_2CrMoO_6 calcined at 550°C.

3.2. Gas Sensing Characteristics

It is well known that the sensitivity of metal-oxide semiconductor sensors is mainly determined by the interaction of a target gas and the sensor surface. At a low operating temperature the low sensitivity can be expected because the gas molecules do not have enough thermal energy to react with the surface adsorbed oxygen species O^{2-}, i.e. the reaction rate between the target gas and O^{2-} is essentially low [22]. The enhanced sensitivity with increasing the temperature can be attributed to two facts. On one hand, the thermal energy obtained is high enough to overcome the activation energy barrier of the surface reaction; on the other hand, the adsorbed oxygen species convert from O^{2-} to O^{-} at elevated temperatures, and thus an increase in electron concentration results from the sensing reaction of a test gas and O^{-}. Moreover, the reduction in sensitivity after the maximum is due to the difficulty in exothermic gas adsorption at higher temperatures. Therefore, an optimum operating temperature should be considered to obtain a high sensitivity [23].

Fig. 3 shows sensitivity as a function of calcination temperatures in the range of 450-850°C for Ba_2CrMoO_6 based H_2S gas sensor at an operating temperature 250°C. It is observed that the sensitivity increases for the samples calcined from 450-550°C and then decreased above 550°C. This may due to increased in surface area from the sample calcined above 450°C and saturated at 550°C. Ba_2CrMoO_6 powder calcined at 550°C was used for further study.

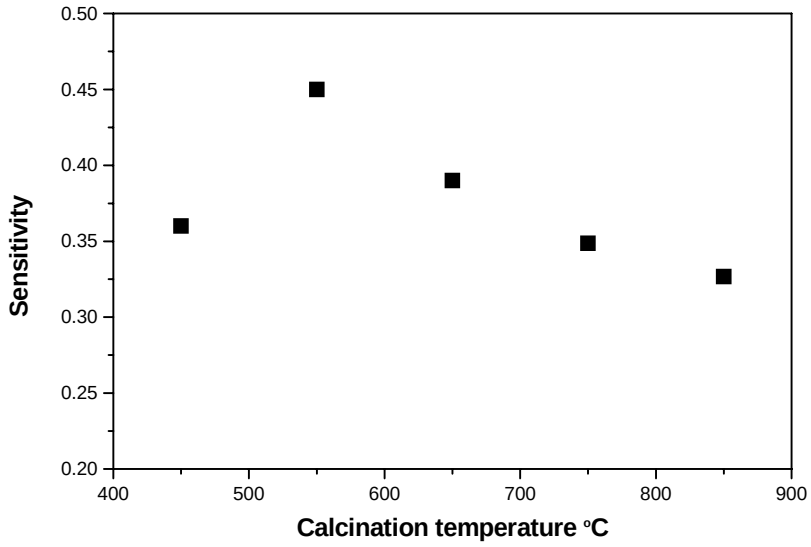

Fig. 3. Gas Sensing characteristics of Ba_2CrMoO_6 at different operating temperatures for H_2S gas calcined at temperature ranges from 450-850°C.

The sensitivity values of pure Ba_2CrMoO_6 was determined at various operating temperatures ranging from 50-350°C. Fig. 4 shows variations in sensitivity to various reducing gases like H_2S, CO, LPG and H_2 gas with operating temperature of the pure Ba_2CrMoO_6 calcined at 550°C. The sensitivity goes on increasing with increase in operating temperature, attains its maximum (at 250°C) and then decreases with further increases in operating temperature. It is clear from the figure that the optimum operating temperature is 250°C.

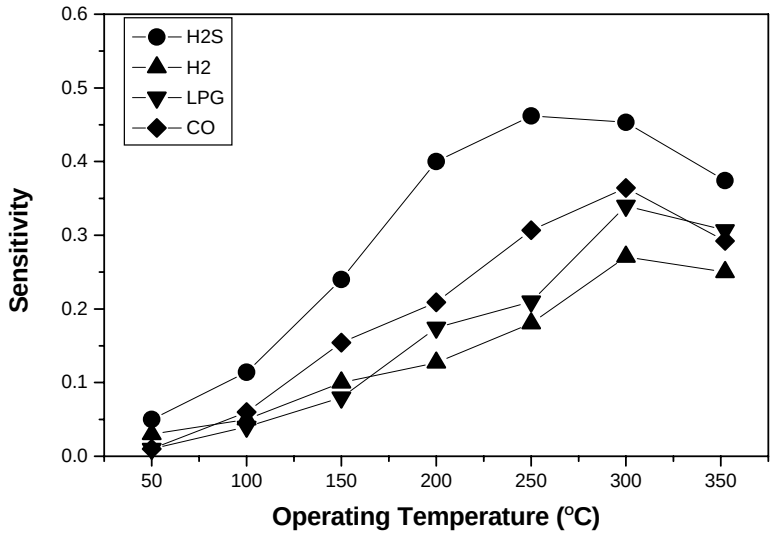

Fig. 4. Gas sensing characteristics of Ba_2CrMoO_6 calcined at 550°C for different operating temperatures for various reducing gases.

With the increasing operating temperature, the state of oxygen adsorbed on the surface of Ba_2CrMoO_6 sensor undergoes the following reaction [24, 25]:

$$O_{2\,(g)} \leftrightarrow O_{2\,(ads)} + e^- \leftrightarrow O_{2\,(ads)}^- + e^- \leftrightarrow 2O_{(ads)}^- + e^- \leftrightarrow O_{2\,(ads)}^- \quad (2)$$

With the introduction of the H_2S gas, it would react with oxygen ions adsorbed on the surface of the sensor. The possible reaction process is as follows [26]:

$$H_2S + O_2^-{}_{(ads)} \leftrightarrow SO_{2\,(g)} + H_2O_{\,(g)} + 6e^- \quad (3)$$

In order to promote gas sensitivity, dopants were shown to effectively influence the semiconductive properties of sensor materials. Ba_2CrMoO_6 based elements doped with transition metal was subjected for measurements of the H_2S gas sensing characteristics. Fig. 5 clearly shows that, 5 wt.% Ni was singularly outstanding in promoting the sensitivity than the other wt.% (3 wt.% & 7 wt.% Ni). Thus, 5 wt.% of Ni was the optimal dopant for Ba_2CrMoO_6 based H_2S sensing element.

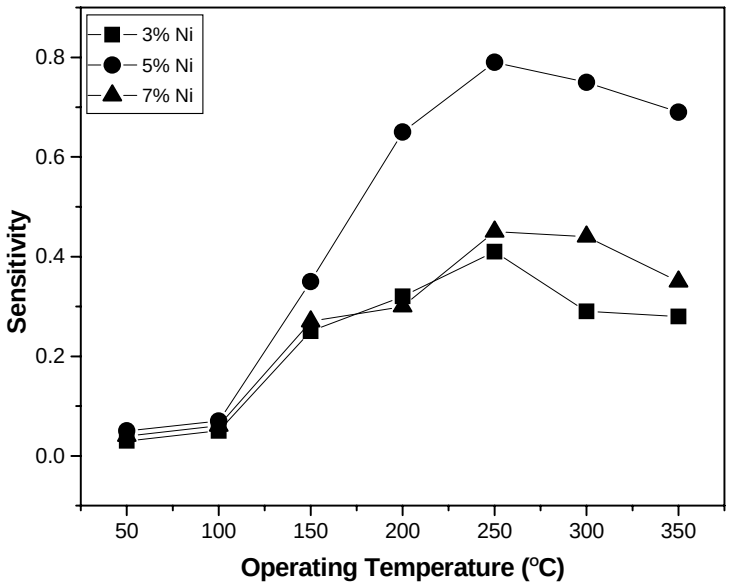

Fig. 5. Sensitivity of Ba_2CrMoO_6 doped with different amount of Ni for H_2S gas sensor calcined at 550°C. (A) 3 wt% Ni, (B) 5 wt% Ni, (C) 7 wt% Ni.

The ability of sensor to respond to a certain gas in the presence of other gases is known as selectivity. Fig. 6 shows the cross sensitivity of 5 wt.% Ni doped Ba_2CrMoO_6 for H_2S, CO, LPG and H_2 gases as a function of operating temperature. It is evident from the figure that the 5 wt.% of Ni doped Ba_2CrMoO_6 sensor was highly selectivity to H_2S gas against CO, LPG and H_2 gases. As expected, the sensitivity increased with an increase in the operating temperature. For H_2S gas, the sensitivity increased and reached saturation values around 250 °C and sensitivity for other gases decreases.

As far as the effect of the noble metals on the improvement of the sensor response, the increase in sensor response of Au and Pd is predominantly used due to the change, which is termed as the electronic sensitization [27,28]. While a reducing gas is oxidized on the noble metal oxide surface, the noble metal oxide is converted to noble metal, which leads to disappearance of the electronic interactions between the noble metal and the semiconductor. Electrons are given back to the semiconductor leading to sensitivity. This change is contributed to the high response of the sensor [29]. The further improvement for H_2S gas sensing properties was observed by addition of small amount of various noble metals such as Pd, Au and Ag with different weight percentage as shown in Fig. 7. The maximum sensitivity for Pd doped 5 wt.% Ni: Ba_2CrMoO_6 sensor element was found to be extraordinary large, indicating that the H_2S gas detection was quite effectively sensitized by the addition of Pd.

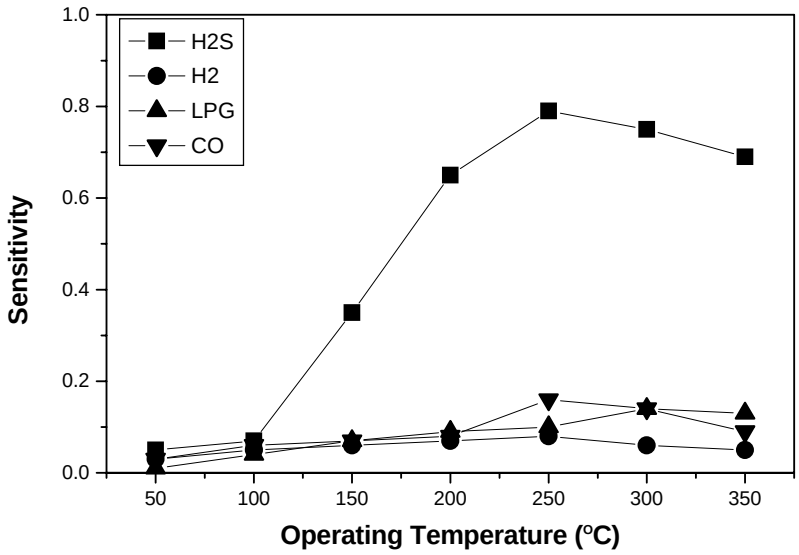

Fig. 6. Cross sensitivity as a function of operating temperature for 5 wt.% Ni doped Ba_2CrMoO_6 calcined at 550°C for various reducing gases.

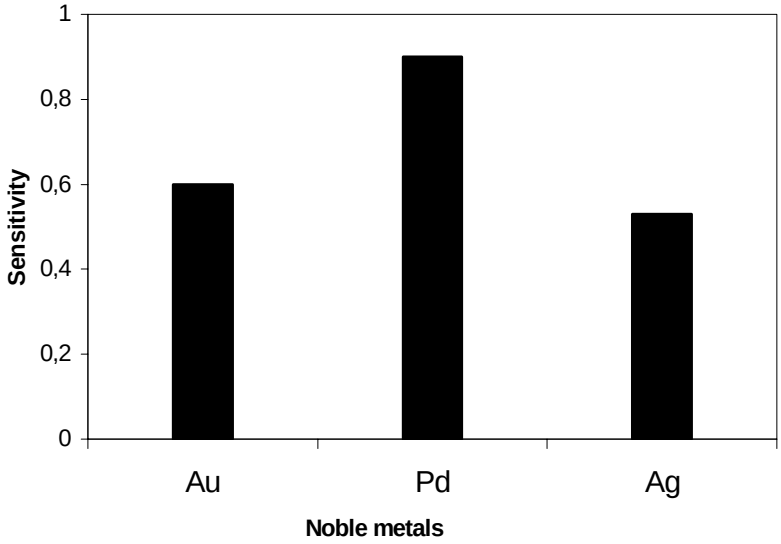

Fig. 7. Sensitivity as a function of different noble metals doped Ba_2CrMoO_6 calcined at 550°C at operating temperature 250°C for H_2S gas.

Fig. 8 shows the sensitivity as a function of the operating temperature for 5 wt.% Ni: Ba_2CrMoO_6 doped with different amount of Pd. The Pd content was varied from 0.2 to 0.6 wt.%. As seen in Fig. 8, the 0.4 wt.% Pd shows maximum sensitivity to H_2S gas at an operating temperature 200°C. When Pd content was increased from 0.2 to 0.4 wt.%, the sensitivity increases and then sensitivity decreases above 0.4 wt.%, suggested the important of dispersion of Pd on the semiconductor materials.

Fig. 9 shows the variation in sensitivity with H_2S gas concentration in air (ppm) for 0.4 wt.% Pd doped 5 wt.% Ni: Ba_2CrMoO_6. As seen from it, the sensitivity initially increases slowly with increasing the concentration and then linearly as the gas concentration increased. It is seen that the 0.4 wt.% Pd doped 5 wt.% Ni: Ba_2CrMoO_6 reaches the saturated sensitivity at 1000 ppm for H_2S gas with reasonable sensitivity at an operating temperature 200°C.

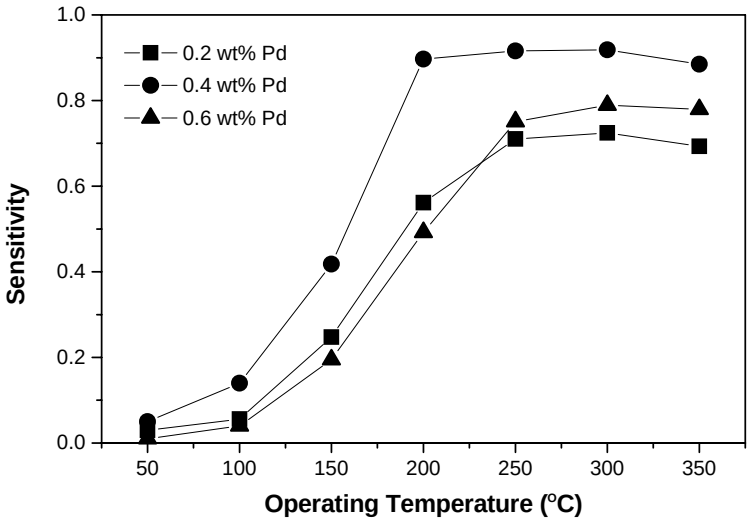

Fig. 8. Sensitivity with different concentration of Pd doped 5 wt.% Ni : Ba_2CrMoO_6 calcined at 550^OC for H_2S gas sensor.

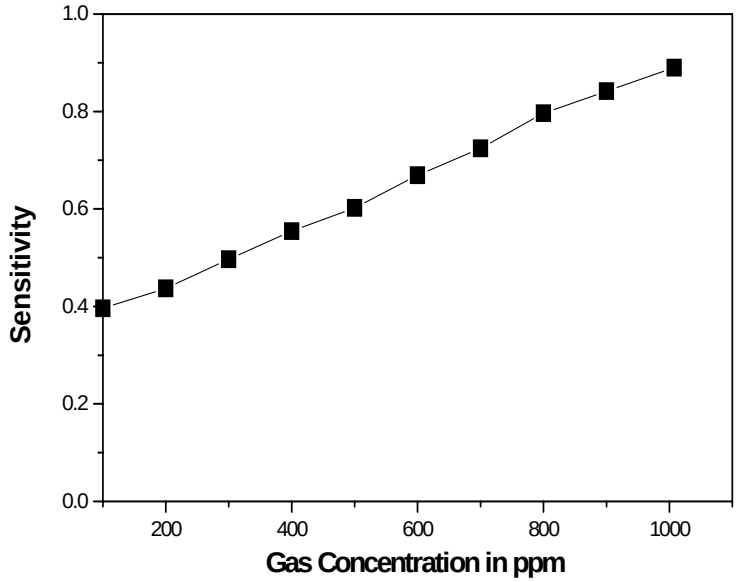

Fig. 9. Sensitivity of 0.4 wt.% Pd doped 5 wt% Ni : Ba_2CrMoO_6 calcined at 550^OC for H_2S gas concentration in ppm.

The response time is an important parameter to characterize a sensor. Fig. 10 clearly shows that the response time of 0.4 wt.% Pd doped 5 wt.% Ni: Ba_2CrMoO_6 is faster than the 5 wt.% Ni doped Ba_2CrMoO_6 . This kind of sensitization has been well explained by an electronic interaction between noble metal and semiconductor oxide [30]. The effect of Pd was thus seen not only increase in sensitivity to H_2S gas considerably but also the increase in rate of response.

Stability is the consistence of the output signal vibration of a sensing element under continuous testing. The response of 0.4 wt.% Pd doped 5 wt.% Ni: Ba_2CrMoO_6. The Fig. 11 shows that the sensitivity rises in the first 10 days and then remains relatively stable, indicating good stability of the sensor. The good stability may be because the materials were prepared under higher temperature calcinations. By the heat processing, the surface energy state and the internal stress of the materials were reduced, and their composition and structure became homogeneous and equilibrated, contributing to the long term stability.

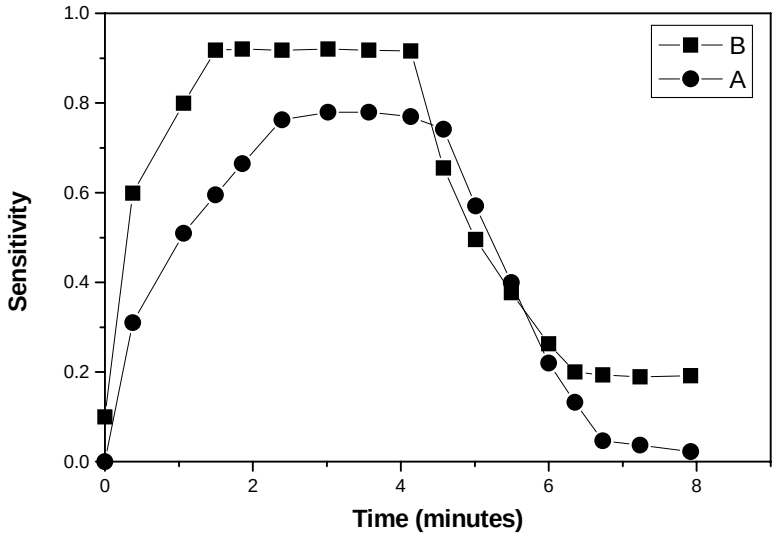

Fig. 10. Response characteristics of (A) 5 wt% Ni doped Ba_2CrMoO_6 (B) 0.4 wt.% Pd doped 5 wt.% Ni : Ba_2CrMoO_6 calcined at 550^{o}C for H_2S gas.

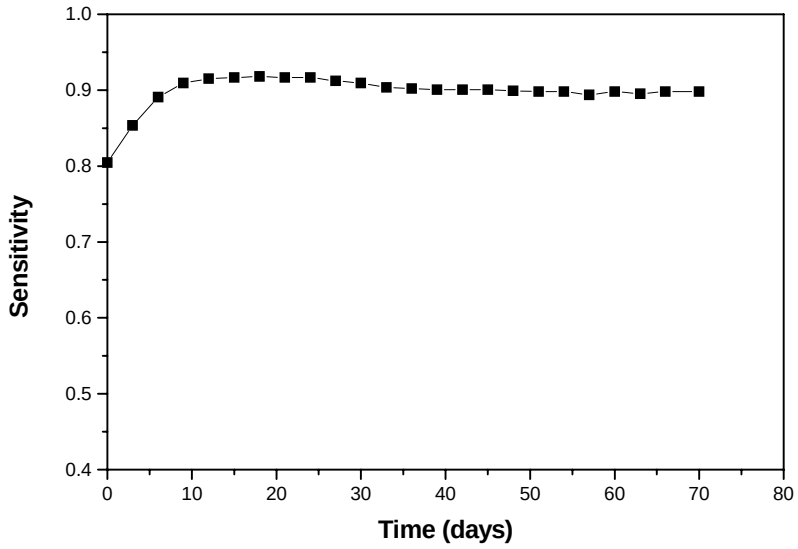

Fig. 11. Stability of 0.4 wt.% Pd doped 5 wt.% Ni : Ba_2CrMoO_6 calcined at 550^{o}C based H_2S gas sensor over time.

4. Conclusions

- 5 wt.% Ni doped Ba_2CrMoO_6 nanoparticles with 40 nm size was synthesized by using sol-gel citrate method.
- Sensitivity studies for reducing gases is carried out to demonstrate that 5 wt.% Ni doped Ba_2CrMoO_6 is highly selective and sensitive to H_2S gas at an operating temperature 250^{o}C.
- The 0.4 wt% Pd incorporation lowers the operating temperature from 250 to 200^{o}C and enhances the H_2S gas sensitivity. The sensor based on this material showed excellent sensor response, good stability and selectivity to H_2S gas at 1000 ppm.

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Guide for Contributors

Aims and Scope

Sensors & Transducers Journal (ISSN 1726- 5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In additional, some special sponsored and conference issues published annually.

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Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
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- Technologies and materials;
- Nanosensors;
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