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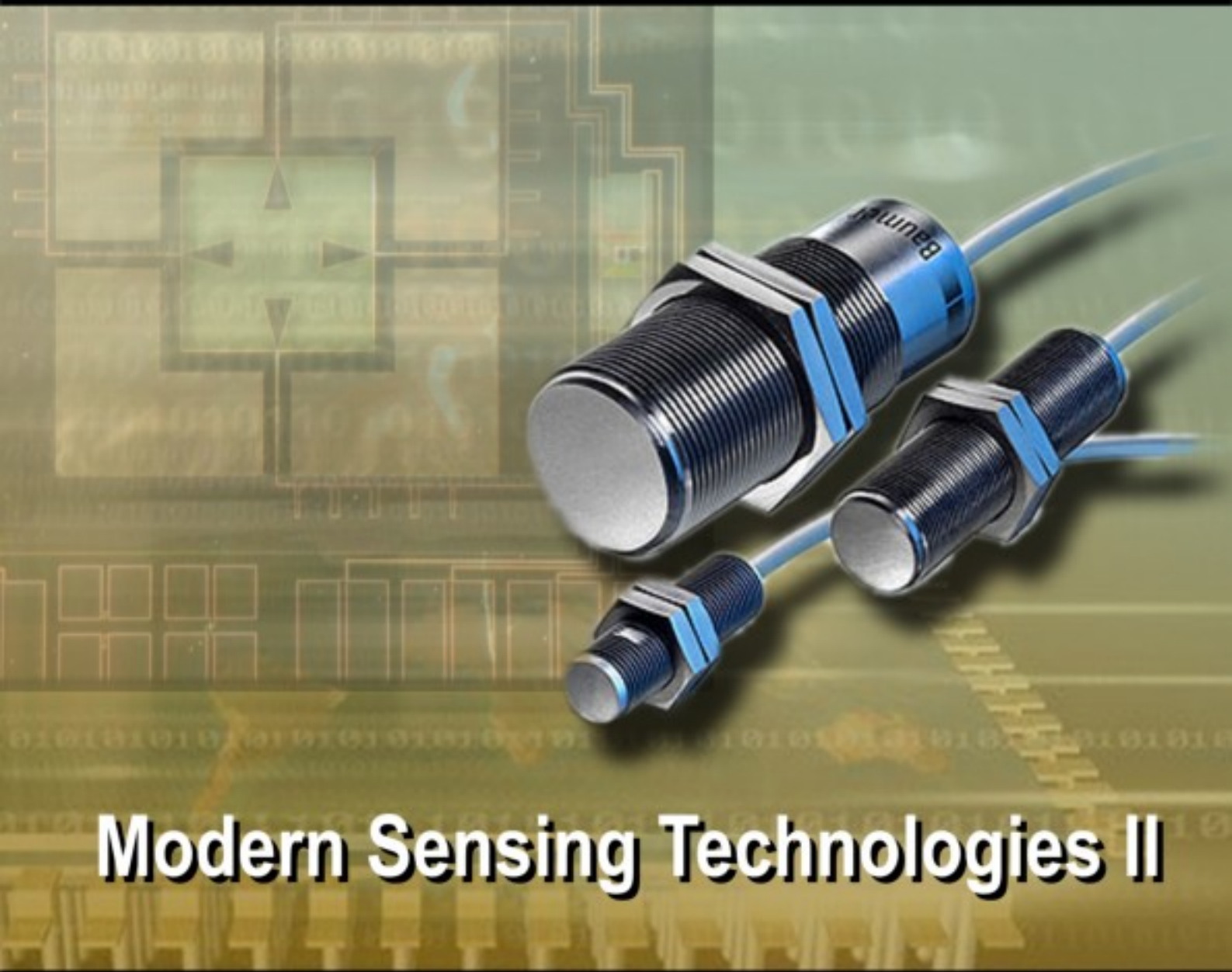

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# Studies on Gas Sensing Performance of Pure and Surface Modified $SrTiO_3$ Thick Film Resistors

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**Abstract:** Strontium Titanate ($SrTiO_3$ (ST)) was prepared mechanochemically from $Sr(OH)_2$ and $TiO_2$. XRD confirms the Perovskite phase of material. Thick films of ST were prepared by screen-printing technique. The gas sensing performances of thick films were tested for various gases. It showed maximum sensitivity to CO gas at 350°C for 100 ppm gas concentration. To improve the sensitivity and selectivity of the film towards a particular gas, ST thick films were surface modified by dipping them in a solution of nano copper for different intervals of time. These surface modified ST films showed larger sensitivity to $H_2S$ gas (100 ppm) at 300°C than pure ST film. A systematic study, of sensing performance of the sensor, indicates the key role-played by the nano copper species on the surface .The sensitivity, selectivity, response and recovery time of the sensor were measured and presented. 

**Keywords:** Strontium titanate ($SrTiO_3$, ST), Thick films, CO gas sensor, $H_2S$ gas sensor, Sensitivity, Selectivity

## 1. Introduction

In recent years, the global climate change has emerged as one of the most immediate challenges of the mankind; as it is well known, this phenomenon is mainly caused by the emission of CO, $CO_2$, and $H_2S$

to the atmosphere by combustion engines and industrial processes. It is inevitable to have a continuous control of these hazardous gases in atmosphere. The gases such as CO, $H_2$, $C_2H_5OH$, $CO_2$, $NO_x$, $O_2$, $CH_4$, $NH_3$, $H_2S$, $C_2H_2$, $C_2H_4$, $C_3H_6$, LPG etc. have to be controlled for the betterment of living beings.

Fuels are widely consumed for transport services all over the world. During combustion, various polluting and toxic gases are released resulting in environmental pollution which can cause serious health hazards. Upon burning, toxic hydrogen sulfide gas is oxidized to sulfur dioxide by atomic oxygen, molecular oxygen or ozone [1]. Combustions of petroleum and coal are the predominant sources of the gases containing sulfur [2]. The gases containing sulfur can result in undesirable disastrous deformations such as infection to respiratory track and lung cancer [2, 3]. Thus $H_2S$ is harmful to human body and the environment. According to the safety standards established by American conference of Government Industrial Hygienists, the threshold limit value (TLV) for $H_2S$ is 10 ppm. Even at low concentration its effect on the nervous system is severe [4].

The emission of $H_2S$ and release of sulfur needs effective monitoring which can allow one to control its free release to environment. Using suitable gas sensors often does Suxh monitoring. Currently there are a number of materials being used for such sensoric applications but these are often not so effective and suffer from various drawbacks, such as low sensitivity, poor selectivity, slow response and recovery times. Therefore there is a need for low cost and more effective $H_2S$ sensor operable in sub-ppm range. In addition, concentration of $H_2S$ varied from the types of oil or natural gas used. The concentration of $H_2S$ thus is different for different source of oil or natural gases mines. Therefore, the detection and monitoring of $H_2S$ should be considered of high importance for both resource exploitations and human health. Similarly, carbon mono oxide is a deadly gas that is released due to combustion of the fuels in automobiles. The principal source of CO is the exhaust products from motor vehicles in common busy route, CO comprises for as much as 80 % of all automobile emissions. Drivers are most affected peoples. CO is toxic because it forms a complex with hemoglobin in blood, and this complex is more stable than oxy-hemoglobin. This prevents the hemoglobin in red blood corpuscles from carrying oxygen round the body. This causes an oxygen deficiency leading to unconsciousness and then death. Hence to monitor CO is very important.

In the recent years, a number of semiconductor sensors have been found to be suitable for $H_2S$ gas e.g. $SnO_2$, $WO_3$, $In_2O_3$, $ZnO_2$ and a few perovskite-type materials like $NdFeO_3$ and $NiFeO_4$ [5-12]. Generally these sensors are fabricated by thick film technology or by deposition of a film by chemical bath deposition method etc.

A different approach to make selective metal-oxide gas sensor is by using metals or additives that enhance the chemisorptions of specific gases. It is well known that perovskite oxides are used as gas-sensing materials because they possess good properties such as chemical and thermal stability, environmental adaptability and wide range of working temperature. The perovskite oxide ceramics promoted interest in chemical sensors over the last decade. Furthermore, some composite systems such as (Ba, Sr)$TiO_3$ and (Ba, Pb)$TiO_3$ ceramics have been studied to broaden the application of $BaTiO_3$-based thermistors for wider temperature range [13,14]. Recently, $SrTiO_3$ and $BaTiO_3$-based sensors have received much attention because of their multisensing properties, such as humidity, thermal and photosensitivities [15].

Strontium titanate $SrTiO_3$ is a non stoichiometric compound where the extent of non-stoichiometry of in $SrTiO_{3-\delta}$ may assume very high values ($\delta$ =0.28). Strontium titanate is an important ternary oxide, which has a cubic perovskite structure with a lattic parameter and 0.3905 nm at 27 $^oC$. It has been studided extensively because of its electrical properties [16-19]. The main applications include grain boundary layer capacitors, gas sensors and $SrTiO_3$ bifunctional elements. These applications all depends on the defect structure of polycrystalline $SrTiO_3$.

In the present studies, surface modified $SrTiO_3$ thick film resistors were fabricated and tested for a range of gases and these are discussed in this article.

## 2. Experimental

### 2.1. Preparation of $SrTiO_3$ Powder (ST)

The AR grade powders of $Sr(OH)_2$ and $TiO_2$ were milled for 2h using planetary ball mill to obtain fine grained powder. Then hot water is added with constant stirring, followed by slow heating up to dryness. The powder was calcined at 1000 $^oC$ for 6 h. The XRD pattern of so-prepared powder confirmed the sub-microcrystalline perovskite phase. The sub micron size powder was then used to formulate the paste for screen-printing of thick films on glass substrate in the desired pattern [20, 21].

### 2.2. Preparation of Thick Films

The thixotropic paste was formulated by mixing the fine powder of ST with the solution of ethyl cellulose (a temporary binder) in a mixture of organic solvents such as butyl cellulose, butyl carbitol acetate and terpineol etc. the ratio of the inorganic part to organic part was kept at 75:25 in formulating the paste. The thickness of films was measured by using Taylor-Hobson (Talystep, UK) system. The thicknesses of the films were observed in the range from 65-75 μm. The reproducibility in the thickness of the films was possible by maintaining proper rheology and thixotropy of the paste. The film these films were fired at 550 $^oC$ for 30 min.

### 2.3. Surface Modification of Thick Films

The nano Cu activated $SrTiO_3$ thick films were obtained by dipping them in a 0.02 % aqueous solution of nano copper at different intervals of dipping time: 5, 10, 20 and 30 min as explained elsewhere [22]. These films were dried at 80 $^oC$, followed by firing at 550 $^oC$ for 30min. The films so prepared are termed as 'surface modified ST' films. The silver contacts were made for electrical measurements.

### 2.4. Characterization

The structural properties of the powder were studied using a Rigaku Model DMAX-2500 X-ray diffractogram (XRD) with Cu Kα radiation, having $\lambda = 1.5406$ Å. The micro structure and chemical composition of the films were analyzed using a scanning electron microscope (SEM, JEOL JED 6300) coupled with an energy dispersive spectrometer (EDS, JEOL JED2300LA). The thicknesses of the thick films were measured using a Taylor-Hobson (Talystep, UK) system. The sensing performance of the sensor was examined using a 'static gas sensing system' as explained elsewhere [22].

## 3. Results and Discussion

### 3.1. Structural Analysis

Fig. 1 shows XRD spectrum of ST thick film. The XRD revealed that the material is in sub-microcrystalline perovskite phase. The observed peaks are well matched with ASTM reported data of SrTiO3 material and are found to be microcrystalline in nature. The average grain size was determined by using Scherrer formula and was estimated to be 29 nm.

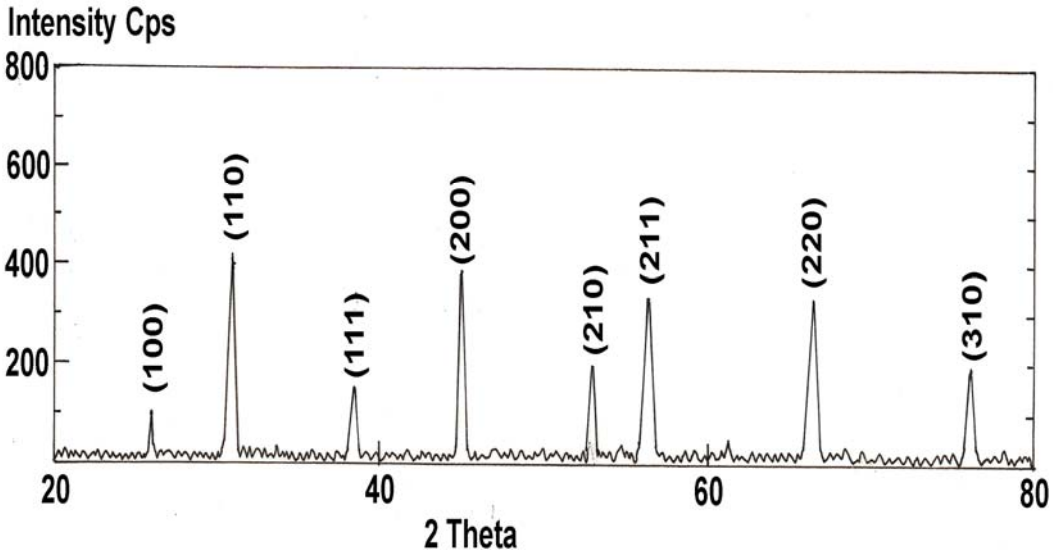
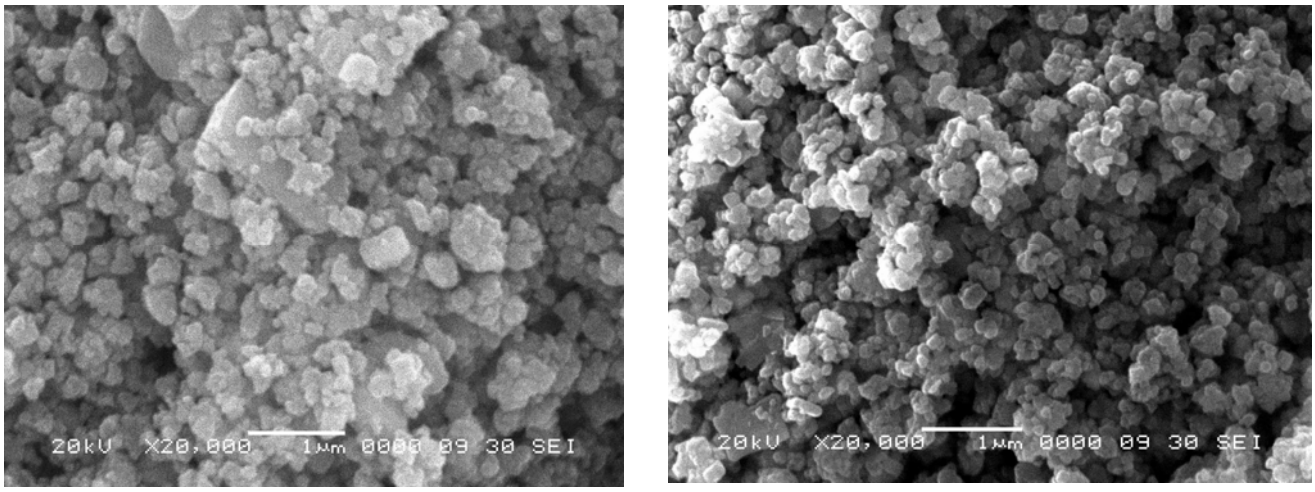



**Fig. 1.** X-ray diffractogram of the ST thick films.

### 3.2. Microstructural Analysis

Fig. 2(a, b) depicts the SEM images of unmodified and surface modified ST thick films (20 min) fired at 550$^{o}$C. The unmodified film consists of large number of grains ranging from 0.1 μm to 1 μm in size, leading to high porosity and large effective surface area available for the adsorption of oxygen species. While the surface modified ST film shows a number of small particles distributed uniformly between the larger grains around the ST, which may be attributed to the presence of nano copper. The grain size range of ST was observed to be from 0.1 μm to 0.3 μm.




(a) (b)

**Fig. 2.** SEM images: (a) unmodified and (b) surface modified ST thick film (20 min).

### 3.3. Quantitative Elemental Analysis of Unmodified and Surface Modified Film

The quantitative elemental compositions of surface modified films are presented in Table 1.

**Table 1.** Quantitative elemental analysis.

| Elements (wt %) | Dipping Time (min) | | | | | |
|---|---|---|---|---|---|---|
| | **0** | **5** | **10** | **20** | **25** | **30** |
| (Sr + Ti) | 68.86 | 67.69 | 66.94 | 66.08 | 66.38 | 66.83 |
| Cu | 0.00 | 0.63 | 1.15 | 2.70 | 1.30 | 0.68 |
| O | 31.14 | 30.98 | 30.96 | 29.92 | 30.88 | 32.29 |
| ST | 100.00 | 99.30 | 99.05 | 98.70 | 98.56 | 99.80 |

Stochiometrically (theoretically) expected wt % of cations ( Sr, Ti) and anions (O) are 67.28 and 32.72 respectively. The wt % of constituent cations and anions in as prepared ST and nano Cu surface activated ST were not as per the stoichiometric proportion and all samples were observed to be oxygen deficient, leading to semiconducting nature of ST. It is clear from Table 1 that the weight percentage of nano copper went on increasing with dipping time. The film with dipping time of 20min is observed to be more oxygen deficient (29.92 wt %). This oxygen deficiency would promote the adsorption of relatively larger amount of oxygen species favorable for higher gas response.

### 3.4. Electrical Properties

Fig. 3 shows the dependence of conductivity of unmodified and surface modified ST films in air ambience. The conductivity of these films goes on increasing with increase in temperature, indicating negative temperature coefficient (NTC) of resistance. This shows the semiconducting nature of the films.

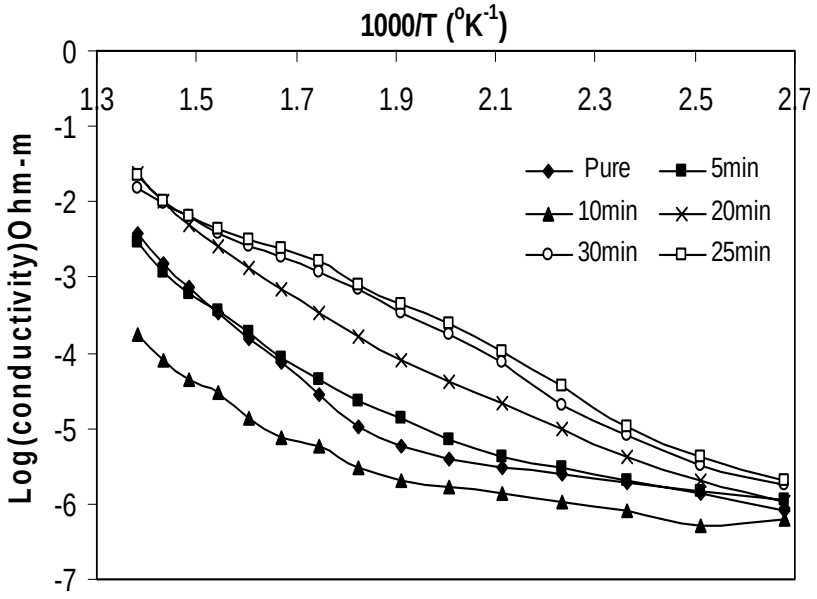



**Fig. 3.** Electrical profile of unmodified and surface modified ST thick films.

# 4. Gas Sensing Performance

## 4.1. Unmodified ST Films

### 4.1.1. Sensitivity with Temperature

Fig. 4 presents the variation in the sensitivity to CO gas (100 ppm) with operating temperatures ranging from 100 $^{o}$C to 450 $^{o}$C. It is noted from the graph that response increases with increasing temperature, attains a maximum at 350$^{o}$C, and decreases with further increase in operating temperature.

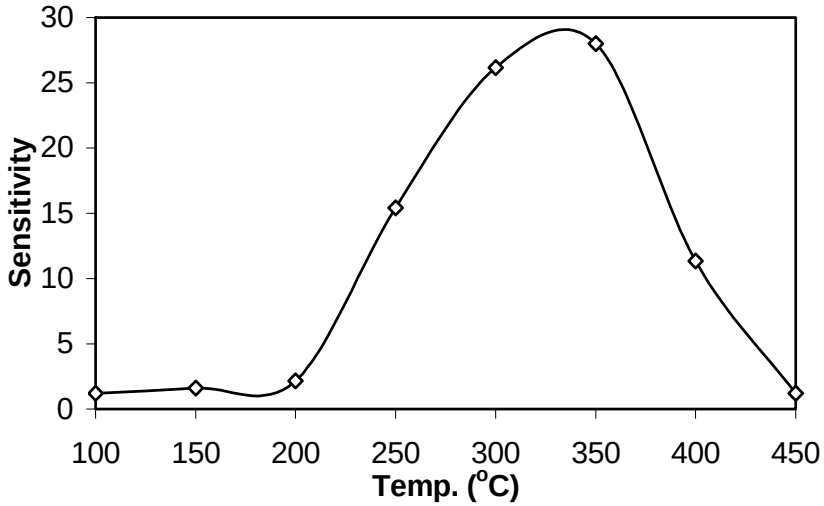



**Fig. 4.** Variation of sensitivity with temperature.

### 4.1.2. Selectivity of Pure ST Film

Fig. 5 presents the bar diagram indicating selectivity of unmodified ST film at 350 $^{o}$C to CO gas against the other gases. The sensor is the most selective to CO gas against the other gases except $NH_3$.

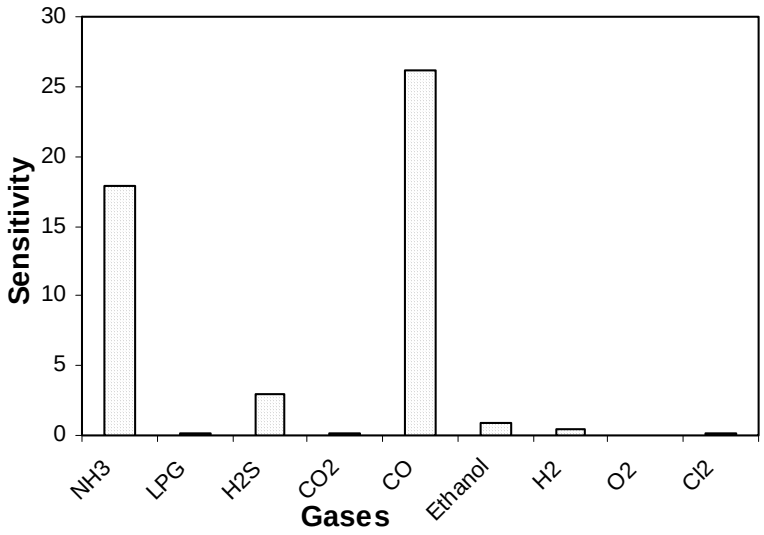



**Fig. 5.** Selectivity of unmodified ST film.

### 4.1.3. Response and Recovery Time of Pure ST Film

Response and recovery times are two important parameters of a gas sensor since their values determine the applicability of the sensor. In gas detection, response time is usually defined as the time taken to achieve 90% of the final change in conductance of the sensor in a given gas concentration [23].

Recovery time is the time taken to return from the conductance in a given gas concentration to 90 % of initial conductance of the sensor [23]. However, figures are often quoted to 50 % or 70 % of the changes, particularly for semiconductor sensors. This is because the shape of the response curve is such that the lower figures may be a better indication of the response [24]. The response and recovery time of unmodified ST film was 6 s and 20 s respectively. This response and recovery profile is represented in Fig. 8.

## 4.2. Surface Modified ST Films

### 4.2.1. Sensitivity with Operating Temperature

Fig. 6 presents the variation in the sensitivity of unmodified and surface modified ST films to $H_2S$ gas (100 ppm) with operating temperatures ranging from 100 $^oC$ to 450 $^oC$. It is noted from the graph that sensing response increases with dipping time and attains maximum sensitivity for dipping time of 20min and subsequently decreases on further increase in dipping time. The pure film showed highest response to CO, while modified films shows to $H_2S$ gas.

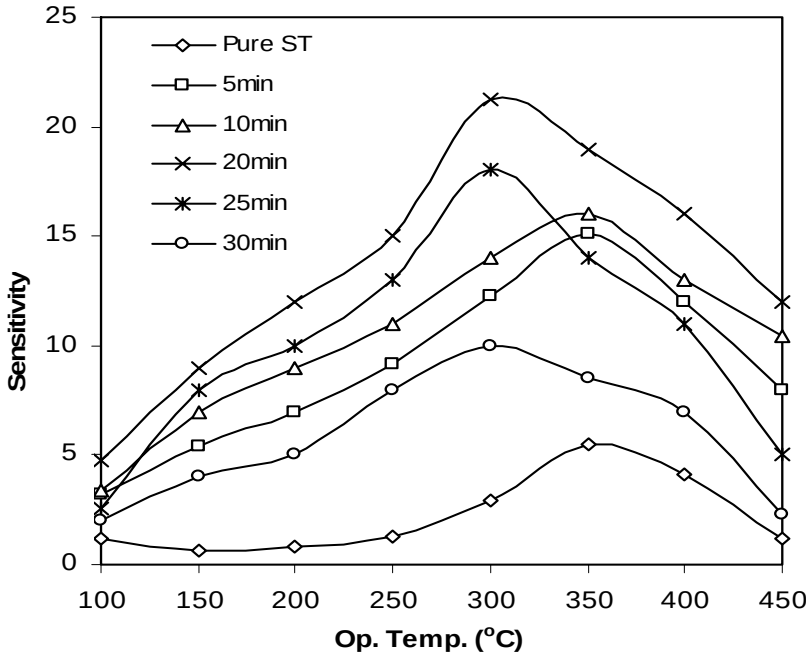
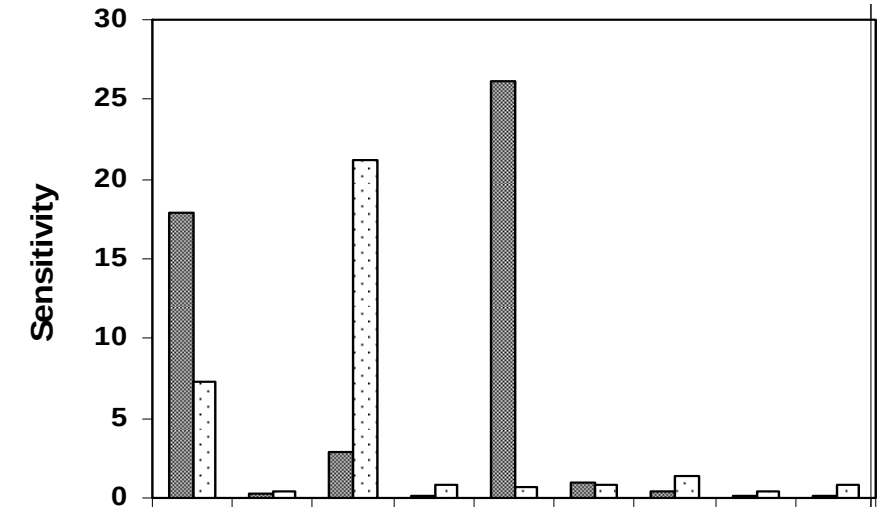



**Fig. 6.** Variation of sensitivity with temperature.

### 4.2.2. Selectivity of Unmodified and Surface Modified ST Films

Fig. 7 presents the bar diagram indicating selectivity of surface modified ST film dipped for 20 min at 300 $^oC$ to $H_2S$ gas against the other gases. The sensor is the most selective to $H_2S$ gas against the other gases except $NH_3$.




| | NH3 | LPG | H2S | CO2 | CO | Ethanol | H2 | O2 | Cl2 |
|---|---|---|---|---|---|---|---|---|---|
| PureST | 17.9 | 0.21 | 2.95 | 0.14 | 26.2 | 0.93 | 0.37 | 0.07 | 0.19 |
| 20min | 7.24 | 0.4 | 21.2 | 0.86 | 0.65 | 0.82 | 1.41 | 0.45 | 0.87 |

**Fig. 7.** Selectivity of pure and modified ST films.

### 4.2.3. Response and Recovery Time of Surface Modified ST Film

Fig. 8 represents the response and recovery time of surface modified ST film (20 min). It has been found that the response and recovery time was 6s and 28 s respectively. The recovery time was found to be larger compared to unmodified ST film.

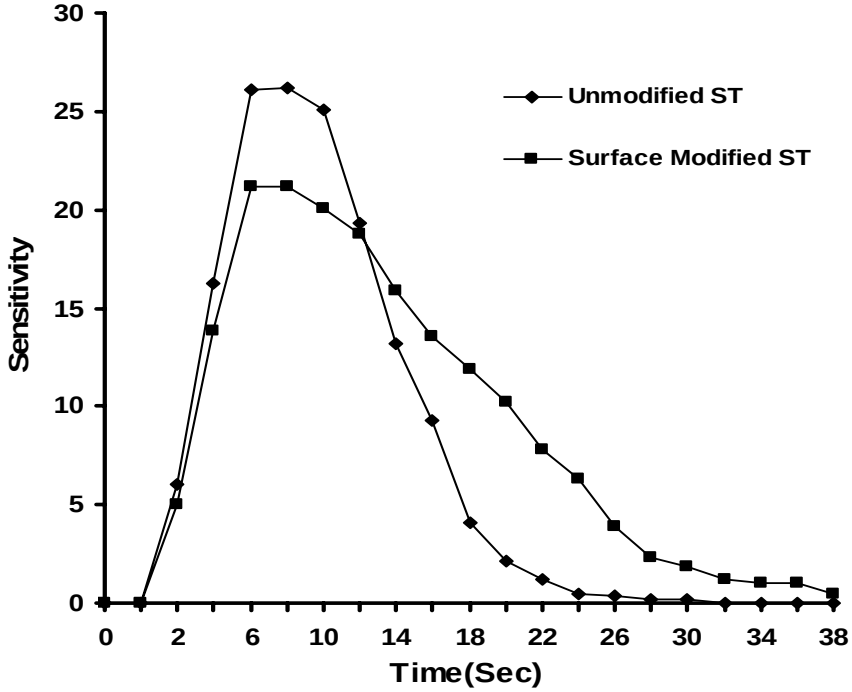



**Fig. 8.** Response and recovery of unmodified and surface modified ST films.

# 5. Discussion

## 5.1. Unmodified ST Film as CO Gas Sensor

On the surface of the ST film there are two kinds of oxygen viz; the adsorption oxygen and the lattice oxygen. These reactions of oxygen with CO based on the reported literature could be of the following types [25 -28]:
According to reaction of CO:

$$CO+1/2O_2 \rightarrow CO_2 \quad (1)$$

for the oxygen adsorption:

$$1/2CO2+2e^- \rightarrow O^{2-}_{ads} \quad (2)$$

$$O + e^- \rightarrow O^-_{ads} \quad (3)$$

for the CO adsorption:

$$CO_g \rightarrow CO^+_{ads} + e^- \quad (4)$$

$$CO_g \rightarrow CO_{ads} \quad (5)$$

reaction of adsorbed species:

$$CO_{ads}+O^- \rightarrow CO_2 + e^- \quad (6)$$

forming a surface donor complex:

$$CO_g \rightarrow [OCOV_o]^+ + e^- \quad (7)$$

forming a surface vacancy

$$CO_g + O^x_o \rightarrow CO_2 + e^- + V^+_o \quad (8)$$

reaction with a surface complex:

$$1/2O_2 + [OCOV_o]^+ + e^- \rightarrow CO_2 + O^x_o \quad (9)$$

reaction with a surface vacancy:

$$CO+1/2O_2 + V^x_o + e^- \rightarrow CO_2 + O^x_o \quad (10)$$

Equations (7)-(10) include the lattice oxygen reaction and surface complex and it has been described that in order for oxygen to react with CO, the lattice oxygen vacancies must be continuously diffused to the surface, which occurs at higher temperature. For the vacancies to migrate from the bulk to the surface, as shown in eqs. (7)-(10) temperature higher than 300 $^oC$ is normally required [28]. Since, we have used the operating temperature range between 100-450 $^oC$, therefore, the eqs. (3)-(6) are appropriate to consider to explain the phenomenon.

Since, the oxidation of CO is a process of thermal activation, and the desorption of oxygen is enhanced with the increase in temperature. Thus oxidation CO needs a suitable temperature range. In the present

study, the optimized temperature was found to be 350 $^{o}$C. The films used for CO detection can be completely recovered by heating to a temperature of over 450 $^{o}$C. The results support the view that the catalytic properties of the pervoskite phase are a reversible process.

### 5.2. Surface Modified ST Film as $H_2S$ Gas Sensor

Fig. 9 illustrates the effect of dispersion of surface additive on the film conductivity. Uniform and optimum dispersion of an additive dominates the depletion of electrons from semiconductor. Oxygen adsorbing on additive (misfits) removes electrons from the nearby surface region of the semiconductor and could control the conductivity.

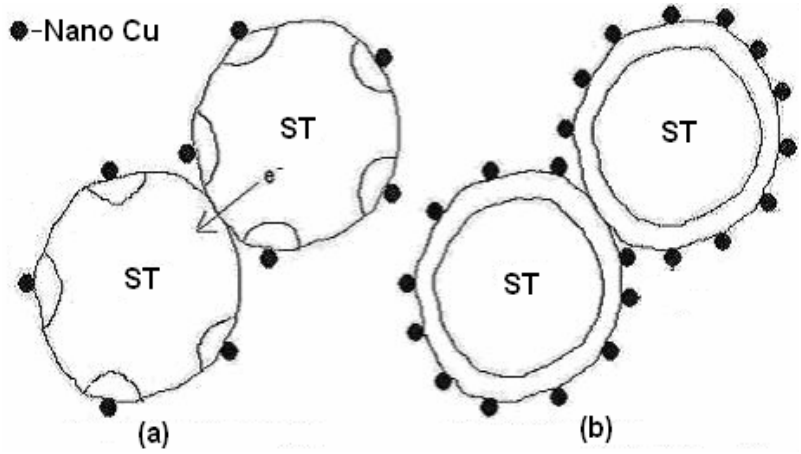



**Fig. 9.** Catalyst dispersion (a): poor, (b): adequate.

For optimum dipping time (20min), the number of nano Cu misfits would be optimum and would disperse uniformly covering the complete film surface (Fig. 9(b)). Adequate dispersion of nano Cu misfits (20 min) on film surface would produce depletion region on the grain surfaces and conductivity could be monitored systematically. The film conductivity would be very low in air and very high on exposure of hydrogen sulfide gas (due to conversion of Cu into CuS) and therefore, the sensitivity would be largest [29].

For the dipping time smaller than the optimum, the number of nano Cu misfits would be smaller, their dispersion would be poor and the depletion regions would be discontinuous and there would be the paths to pass electrons from one grain to another (Fig. 9(a)). Due to this, the initial conductance (air) would be relatively larger and in turn, sensitivity would be smaller.

The introduction of nano Cu atoms on the surface of ST films by dipping technique increases the adsorption capability of the films. The nano Cu increases the surface to volume ratio of the film. The nano Cu particles on the surface control the grain boundary [30]. The different chemical identities on the surface of the film would alter the adsorption-desorption kinetics and gives unusual physical and chemical properties.

## 6. Summary

The unmodified ST film showed highest response to CO gas (100 ppm) at 350 $^{o}$C. The ST films were modified by nano copper solution by dipping technique. The surface modified films showed maximum

response to $H_2S$ gas (100 ppm). The surface modification shifts the response of film from CO to $H_2S$ gas. Due to introduction of nano copper on the surface would alter the adsorption-desorption relationship if the film. The optimum dipping time was found to be 20 min. The surface modification alters only the surface morphology of the films not the bulk properties.

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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. *Sensors & Transducers Journal* is indexed and abstracted very quickly by Chemical Abstracts, IndexCopernicus Journals Master List, Open J-Gate, Google Scholar, etc.

## Topics Covered

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;
- Frequency (period, duty-cycle)-to-digital converters, ADC;
- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

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