

Hydrogen Gas Sensor Based on CuO and Pd-doped CuO Thin Films Grown by sol-gel Spin Coating Method

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Abstract

In this work, hydrogen sensors were demonstrated based on copper oxide (CuO) and palladium-doped copper oxide (CuO:Pd) thin films grown by sol-gel spin coating technique. The sensing properties of CuO layer surfaces were analyzed for hydrogen concentrations varying from 1000-15000 ppm in a temperature range between 110 and 180°C. For undoped CuO-based sensor, operating temperatures higher than 110°C were required for low H₂ concentrations below 4000 ppm. Whereas higher temperatures above 140°C led to better response and more reliable results. At fixed temperature of 160°C, the highest response achieved by CuO: Pd films reached 0.45 which was four times those of undoped CuO counterpart (~ 0.1). In contrast to CuO, sensor based on CuO:Pd even with minimal doping of only 2% enabled the detection process of H₂ reducing gas with low concentration of about 5000 ppm at reduced operating temperatures as low as 60°C. By introducing Pd as dopant, the sensing properties of H₂ gas significantly improved, especially for higher concentrations. This is attributed to a synergy of morphological, compositional and electrical effects. Our findings confirm the potential of CuO-based thin films fabricated by sol-gel spin coating technique as an efficient and simple method for hydrogen sensing applications at operational environment.

Keywords: Metal Oxide Thin Films; Copper Oxide; Doping; Sol-Gel Technique; Hydrogen Gas Sensor

Introduction

Nowadays, hydrogen is applied in transport, manufacturing and residential sectors at different temperature and pressure conditions [1-3]. For concentrations above 4% (40,000 ppm) in air, hydrogen becomes explosive [4,5]. Therefore, it is an imperative specification for hydrogen sensors to detect below this lowest explosion limit (LEL) of 40,000 ppm in air [6]. As a typical reducing gas, H₂ can be detected by pure or doped metal oxides sensors at high operating temperatures [7,8]. Nevertheless, it is still risky to operate H₂ at high temperatures due to its combustible character. Thus, novel functional materials are always sought for sensitive H₂ detection at low operating temperature [9]. A wide variety of gas sensitive materials were exploited, such as metal oxide semiconductors [7,10-12], conducting polymers [13,14], hybrids nonmaterial [15] and grapheme [16]. In particular, resistive gas sensors which are based on metal-oxide semiconducting thin films are widely studied for the detection of virulent and explosive gases due to their low cost, simple fabrication and system integration compatibility [17]. The most widely used gas sensing materials are SnO₂ [17-19] and ZnO [20]. TiO₂ [21], WO₃ [22], In₂O₃ [23] and Fe₂O₃ [24] which exhibit n-type semi-conductivity.

Different methods have been used to improve their sensing properties including doping with noble metals [25,26] or other metal oxides [27]. In general, to enhance their gas sensing properties, the changes in metal oxides may occur either on the surface or within the lattice with different materials such as Ag, Au, Pd, and Rh which are known by being the effective sensitizers or promoters for gas sensors [23]. In contrast, p-type metal-oxide semiconducting films, such as CuO, NiO [28] and Co₃O₄ have recently attracted attention as sensing materials to further improve some critical parameters such as sensitivity, stability, selectivity, response and recovery time [29-31].

However, p-type metal-oxide semiconducting films gas sensors, have not really been scrutinized and deeper analytical researches are still required for the design, and the exploitation of sensitive and safe p-type gas detector [25].

In our previous work [28,32] we demonstrated effi-

cient hydrogen sensors based on p-type NiO and NiO:Li thin films grown by simple sol-gel technique. One of the most promising materials is p-type CuO semiconductor with a band gap in the range of 1.2 - 3.5 eV and excellent inherent materials properties and cost-effective manufacturing [19,23,26,27,33,34].

For instance, it was reported that CuO films deposited by sputtering and hydrothermal methods enabled the detection of H₂ gas with a concentration of 1000 ppm, but at high operating temperatures between 200°C and 300°C. Also, doping of CuO with Pd results in enhanced sensitivity although the requirements in terms of operation temperature (300°C) and concentration (1000 ppm) remained high [32]. Interestingly, we demonstrated in a previous paper improved electrical conductivity of CuO thin films by means of Pd doping. This could be a boost for hydrogen sensing capabilities [34].

In this work, we studied hydrogen gas sensors operating at low temperatures and H₂ concentrations based on undoped and Pd-doped CuO thin films by simple and cost-effective sol-gel spin coating technique. According to the literature survey, this is the first work to employ Pd-doped CuO films prepared by the sol gel method in resistive hydrogen sensors. First, CuO thin films were tested as sensor for hydrogen concentrations ranging from 1000 to 15000 ppm in synthetic air. Then, the films were doped with 0%, 2%, and 6% of Pd to be systematically characterized with regard to hydrogen gas sensing response as a function of the operating temperature and hydrogen concentrations. The results showed that the Pd-doped CuO sensor is able to detect extremely low concentration of hydrogen (10000 ppm) at moderate operating temperature (60°C).

Experimental Details

Films Coating

For the preparation of undoped and Pd-doped CuO layers, the precursor solutions from copper (II) acetate hydrate [(CH₃COO)₂Cu xH₂O] (98 %, Sigma Aldrich) and [Pd(CH₃COO)₂] (98%, Sigma Aldrich) were used. Then, copper (II) acetate was put into mixture of 2-methoxyethanol (C₃H₈O₂) (99.8%, Sigma Aldrich) and diethanolamine (MEA) (99%, Merck). We control the

ratio of MEA to copper (II) acetate at 1:0, and the concentration of copper (II) acetate to be 0.5 M. The resultant Cu solution was stirred at 80°C for 2h to produce a visibly clear and homogeneous solution. A dissolution of palladium (II) acetate (as a dopant precursor) with 2-methoxyethanol was performed to prepare a 0.5 M solution. Next, we mixed both solutions with different volume fractions to form stoichiometric, transparent and stable copper acetate (II) solutions with different Pd contents. The molar ratio of Pd in the solution [Pd / (Pd+ Cu)] was 2% and 6%. Prior coating, a solvent cleaning of glass substrate based on a sequence of acetone, ethanol and deionized water in ultrasonic bath was done. Then, undoped and Pd-doped CuO thin films were deposited on glass substrates by spin coating at room temperature, in rotation rate and time conditions of 4000 rpm for 30 s. To remove organic residues, the produced films were heated up at 300 °C in air for 15 min on hot plate, following each spin-coating process. Based on previous optimization work, this cycle of coating and drying process (one layer) was repeated to obtain a thickness of 220 nm corresponding to 4 layers. Finally, all the films were annealed, in air at 550 °C for 1 h [34].

Characterization Methods

To analyse the crystallographic structure of different CuO-based films, we employed X-ray diffraction (XRD), using a Bruker D 8 advance X-ray diffractometer with Cu K_{α} ($\lambda_{Cu K_{\alpha}} = 1.5418\text{\AA}$) radiation, considering 2θ values between 20° and 70°. The surface morphology and the grains size of the prepared films was investigated by means

of Scanning Electron Microscopy (SEM) in Hitachi S4800 system. The electrical resistivity of CuO-based layers was assessed by four-point probe technique.

Gas Sensing

A home-built sensing setup was used for hydrogen sensing tests [35]. Following the evacuation of aluminum vacuum chamber at a pressure of 1 Pa, a filling with synthetic air at atmospheric pressure was performed. Hydrogen gas was then introduced into the chamber by means of a mass flow controller (Bronkhorst) monitored by a computer. Using the partial pressures measured by the Baratron gauge, the hydrogen concentration in air was then determined [33]. The undoped and Pd-doped CuO thin films were tested for hydrogen concentrations of 15000, 12000, 10 000, 7000, 5000, 3000, 2000 and 1000 ppm in air. The prepared films were placed on hot plate temperature equipped with Ni-Cr thermocouple for temperature measurement. The temperature conditions were 110, 120, 140, 160 and 180°C for the undoped CuO films and at 160°C and 60°C for Pd-doped CuO films. Under a constant bias voltage of 1 V, the electric current flowing through the films was measured using 485 Keithley picoampere meter with two copper electrodes pressed mechanically on the film surface. Any change in the conductivity of the sensor upon the reaction with H₂ gas resulted in a current change through the electrical circuit, then tracked in real time and visualized on a digital display. For various hydrogen concentrations and operating temperature, the sensor response (S) was calculated as follows [36]:

$$S = \frac{R_g - R_0}{R_0} \quad (1)$$

With R_g , the electrical resistance of the sample in the presence of the hydrogen gas and R_0 the resistance of the sample in synthetic air.

Results and Discussions

Thin Film Material Properties

The X-ray diffraction patterns of pure CuO and CuO: Pd films with palladium concentrations of 2% and 6%

are shown in Figure 1 (a). The patterns of CuO are related to monoclinic CuO (JCPDSNo. 01-089-2529) showing well-defined diffraction peaks at $2\theta = 32.52, 35.55, 38.73, 48.74, 58.30, 61.56, 66.25$ and 68.13° assigned to (110), (002), (111), (-202), (202), (-113), (-311) and (220) planes of monoclinic CuO, respectively. A closer look at the specific regions (35 - 40°) of XRD patterns (Figure 1(b)) reveals some differences. Once comparing the Pd-doped CuO patterns with the pure CuO, it was noticed that (-111) and (111) peaks were a little shifted towards lower angles, with the increase

of Pd dopant concentration from CuO: Pd 2% to CuO: Pd 6%. This implies a change of lattice constant after Pd doping. It is worthy to mention that the ionic radius of Pd²⁺ (0.87 Å) is larger than that of Cu²⁺ (0.74 Å) [37,38]. Therefore, the substitution of Cu²⁺ by Pd²⁺ ions induces lattice expansion causing such peak shifting to lower angles. Importantly this is a clear indication that Pd²⁺ ions were successfully incorporated into the CuO matrix. Furthermore, it can be noticed that for the CuO: Pd films, only the (110) and (002)

planes appear, but with a large decrease in their corresponding intensity. Such peak decrease indicates a crystallinity degradation of the doped films after the incorporation of the Pd ions that may be explained by Pd²⁺ ions prevention of CuO nanocrystals aggregation [39].

Another observation is that Pd doping of CuO enlarges the peak FWHM. The average crystallite size of the film grains can be calculated from the (111) peak by using Scherrer's Formula [40].

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (2)$$

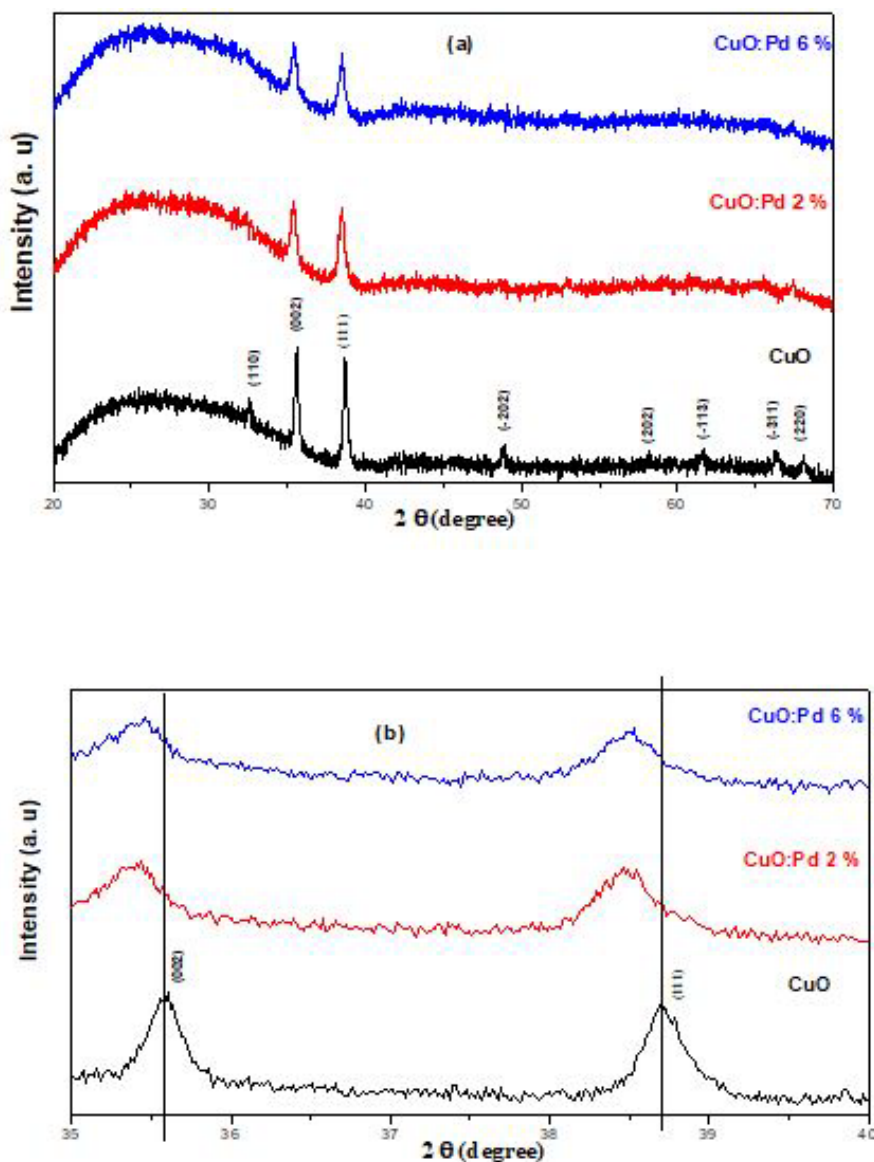


Figure 1: (a) (b) X-ray diffraction patterns for CuO, CuO: Pd 2%, and CuO: Pd 6% thin films

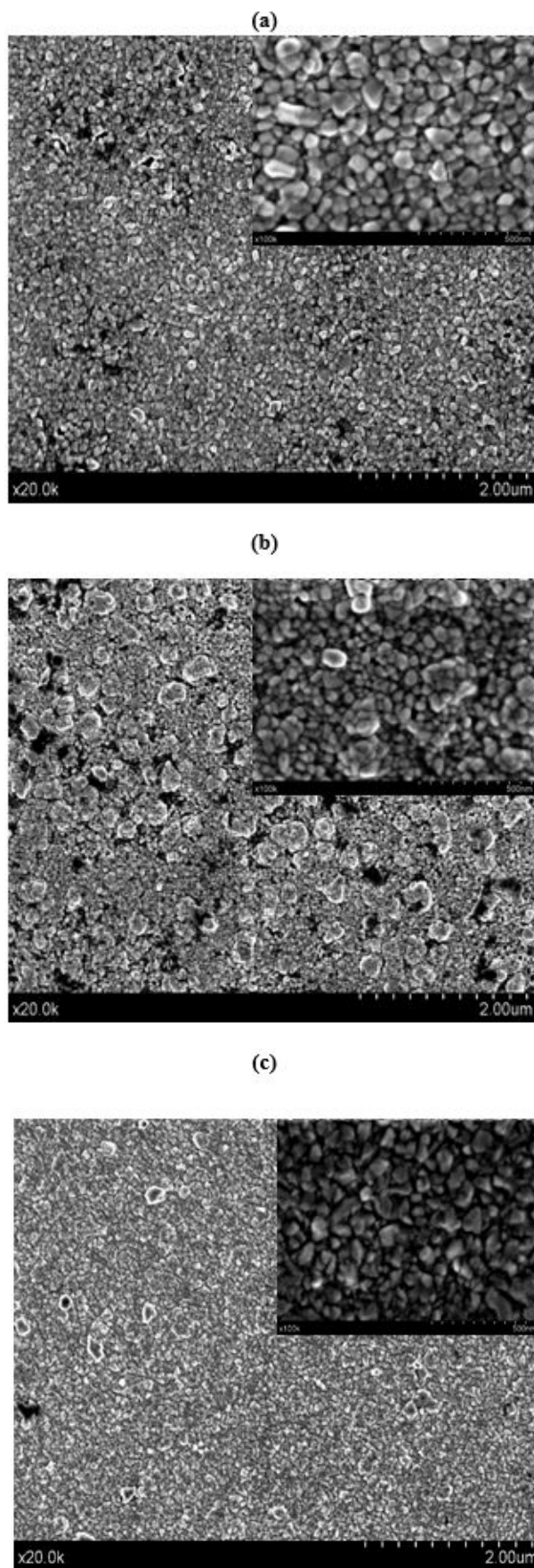


Figure 2: SEM images of CuO and Pd-doped CuO thin films as a function of Pd doping: (a) 0%, (b) 2% and (c) 6%

where D is the crystallite size, λ is the X-ray wavelength used for the measurements (1.5418 Å in the case of $\text{Cu}_{K\alpha}$), β the full width at half maximum of the peak, θ is Bragg's angle of the XRD peak and k is a constant ~ 0.9 . The results showed that the crystallite size decreases with the increasing Pd concentration and were found to be 35 nm, 24 nm and 20 nm for pure CuO, CuO: Pd 2% and CuO: Pd 6%, respectively. The decrease in crystallite size with the increment of dopant concentration can be attributed to the replacement of larger radius Cu ions by smaller radius Pd ions [8,41].

The morphology and the grain size of sensing material is an important factor for the gas detection performance. Fig. 2 shows SEM images of CuO, 2%, and 6% Pd-doped and pure CuO thin films at low magnification ($\times 20\text{K}$). High-magnification SEM images ($\times 100\text{K}$) in the inset of Fig. 2 (a), (b) and (c) show that the films consist of a uniform, monodisperse, porous and nugget-like structure with aggregates of individual nanoparticles. This type of nanostructure increases the gas diffusion as well as the surface reaction efficiently and enhances the response and the recovery of the gas sensor [8,41].

In order to estimate the average grain sizes of the undoped and Pd-doped CuO films, SEM figures are comput-

ed with the Image J software and the statistical distributions were determined. The determined grain sizes are 32 nm, 22.6 nm and 17.5 nm for CuO, CuO: Pd 2% and CuO: Pd 6% samples, respectively. These results are consistent with the extracted size values from the XRD studies. Interestingly, such a decrease of grain size improves the sensor response thanks to larger effective surface area in direct reaction with the agent gas [8,34,35,41,42].

Response to Hydrogen for Undoped CuO

In the following section, undoped CuO thin film grown by sol-gel technique on glass substrate was tested as a hydrogen sensor under operating temperature ranges from 110 to 180 °C and for a gas concentration range from 1000 to 15000 ppm. The response obtained of undoped CuO sample to hydrogen at an operating temperature of 180 °C for the gas concentration range between 1000 and 15 000 ppm is shown in Figure 3. It is clearly noticed that by augmenting the gas concentration, the sensing response gradually increased. The sensitivity drops by about 60% from 0.25 for the highest concentration of 15 000 ppm to 0.1 for the lowest concentration of 1000 ppm. However, the detection capability at low hydrogen concentration points out the suitability and the functionality of CuO-based resistive hydrogen sensor.

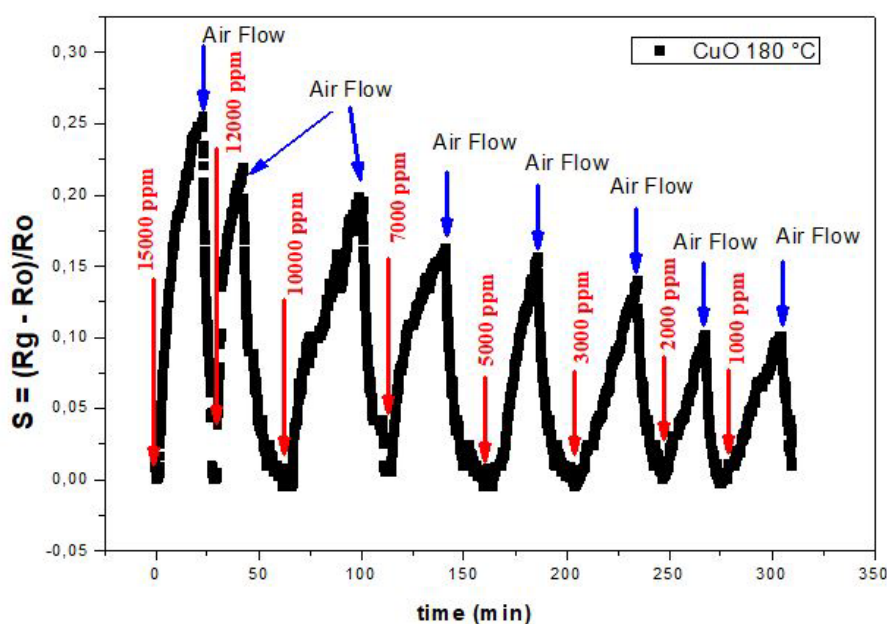


Figure 3: Response of the undoped CuO sensor to hydrogen at 180 °C operating temperature, for the hydrogen concentration range 15 000 – 1000 ppm.

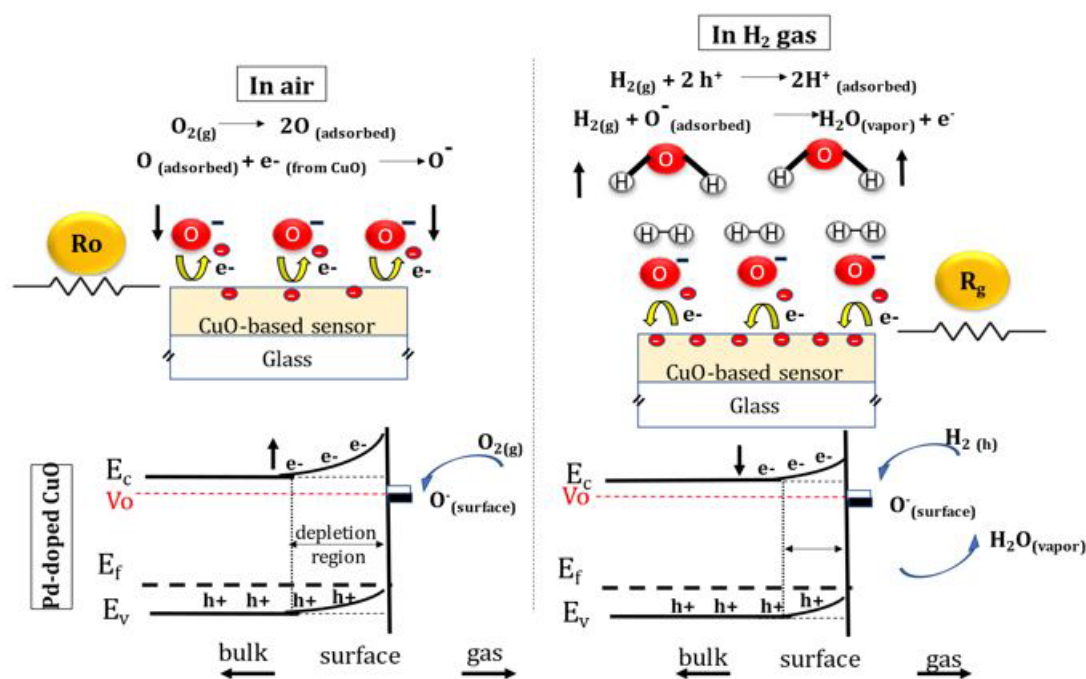


Figure 4: Illustration of hydrogen sensing mechanism of by CuO-based sensor and relevant energy band diagram

The mechanism of hydrogen sensing by CuO-based sensor and the relevant energy band diagram are illustrated in figure 4. Upon the exposure to reducing hydrogen gas, CuO is considered as surface sensitive gas sensor materials [32]. This implies a sensitivity controlled by change in conductivity due to band bending.

Depending on the equilibrium with the gas phase, the concentration of the charged oxygen species is altered. By introducing hydrogen reducing gas, the reaction with pre-adsorbed oxygen species reduces their concentrations leading to increased conductivity of gas sensor film [8].

On the surface of p-type CuO, large density of oxygen vacancies and dangling bonds are formed. By introducing synthetic air, the adsorption of negatively charged oxygen species on CuO film leads to the formation of oxygen ions after the capture of electrons from the conduction band due to high affinity and the creation of more holes. This induces a large upward band bending with wide depletion region on CuO surface. The electrons extraction mechanism results in a diminution of charge carriers in the conduction band and therefore a drop in conductivity. Depending on the equilibrium with the gas phase, the concentration of the charged oxygen species is altered. By introducing hydrogen reducing gas, the reactions with pre-adsorbed oxy-

gen species reduce their concentrations leading to increased conductivity of gas sensor film [8].

Upon the detection of hydrogen at operating temperature, a reaction between reducing hydrogen gas and oxygen ions on adsorbed on the surface of CuO films is manifested leading to the formation of free electrons that are released back to the conduction band of p-type CuO thin film. Then, a recombination between holes charge carriers and free electrons within p-type CuO surface takes place, resulting in a narrowing of depletion region depth. Therefore, the minimization of hole concentration due to recombination mechanism with released free electrons induces an increase in the electrical resistance [43].

Figure 5 illustrates the CuO sensor response to hydrogen concentrations in air basically varied from 1000 to 15000 ppm, at different operating temperatures between 110 °C and 180 °C. It is clearly seen that the response values gradually rise with an increasing H₂ gas concentration for all temperature. This enhancement of sensitivity is more pronounced for higher temperatures due to several factors. At higher temperatures, the kinetic energy of hydrogen molecules and the metal oxide surface increases, leading to faster adsorption and desorption of hydrogen atoms onto the sensor surface [10,32]. This enhances the speed of the

chemical reactions involved in the sensor's detection process [8]. The rate of the surface reactions such as hydrogen dissociation or interaction with oxygen vacancies becomes faster as temperature rises, improving the overall sensor response [36,37]. Moreover, thanks to thermal excitation, charge carrier activity and concentrations increase, especially holes for p-type CuO, improving the electrical conductivity, consequently, the sensor's sensitivity. As temperature increases, the density of surface oxygen vacancies as active sites for hydrogen adsorption in CuO tends to increase, facilitating the interaction between hydrogen and thin film surface and promoting the response of the sensor [17]. At lower temperatures, the activation energy required for hydrogen interaction with CuO surface might not be fulfilled, which resulting in lower sensor activity [1,11].

It is noteworthy that undoped CuO film in our experiment was not responsive to H₂ sensing for concentra-

tions below 4000 ppm at 120°C and even for 10000 ppm at 110°C.

Response to hydrogen for Pd-doped CuO

In p-type CuO, the energy levels of Cu²⁺ cations as acceptors are located just above the energy valence band, while the hole charge carries (h⁺) are characterized by an energy level below the top of the valence band. Upon Pd doping, the incorporation of Pd²⁺ dopants into CuO lattice induces additional h⁺ sites deeper in the valence band and Pd²⁺ acceptors above the valence band resulting in a shift of valence band energy and Fermi level [32]. Based on the process reactions including the absorption of negatively charged oxygen species and hydrogen reducing atoms, the augmentation of holes promotes their trapping in a reduced accumulation region changing CuO surface towards metallic Cu surface. This favors the sensitivity to hydrogen reducing gas detection [31].

Table 1: Overview about of different morphological, electrical and H₂ sensing properties of CuO-based sensors

Sensor	Cristallite size(nm)	Grain size(nm)	Resistivity(10 ²)	Gas ResponseS H ₂ concentration(1000 ppm)	Gas ResponseS H ₂ concentration(15000 ppm)
undoped CuO	35	32	1.22	0.23	1.40
CuO :Pd(2 %)	24	23	1.11	0.53	2.10
CuO :Pd (6 %)	20	17	1.04	0.35	4.10

Table 2: Comparison of hydrogen gas sensor performance with reported data $S = \frac{R_g - R_0}{R_0}$; $S^\# = \frac{R_g}{R_0}$

Samples	Synthesis	Operating	Concentration	Gas Response	References
	method	temperature	(ppm)	S	
CuO	Hydrothermal	200 °C	1000	1.6	[22]
CuO	Sputtering	300 °C	1000	3 [#]	[32]
Mesoporous CuO film	ink solution	300 °C	1000	25 [#]	[44]
CuO	Hollow Spheres	400 °C	1000	1.65 [#]	[44]
CuO	Sol-gel	140 °C	1000	4.4	This work
CuO : Pd	Sol-gel	60 °C	5000	0.09	This work

Then, we examine the hydrogen sensing capabilities of Pd-doped CuO films in comparison to undoped counterpart under different temperature and gas concentration conditions. In p-type CuO, the energy levels of Cu^{2+} cations as acceptors are located just above the energy valence band, while the hole charge carriers (h^+) are characterized by an energy level below the top of the valence band. Upon Pd doping, the incorporation of Pd^{2+} dopants into CuO lattice induces additional h^+ sites deeper in the valence band and Pd^{2+} acceptors above the valence band resulting in a shift of

valence band energy and Fermi level [34]. Based on the process reactions including the absorption of negatively charged oxygen species and hydrogen reducing atoms, the augmentation of holes promotes their trapping in a reduced accumulation region changing CuO surface towards metallic Cu surface. This favors the sensitivity to hydrogen reducing gas detection [33]. Moreover, Pd doping of CuO improves the electrical conductivity [34] and may affect the conduction process of charge carriers through the triangular barrier caused by band bending at the surface of gas sensor.

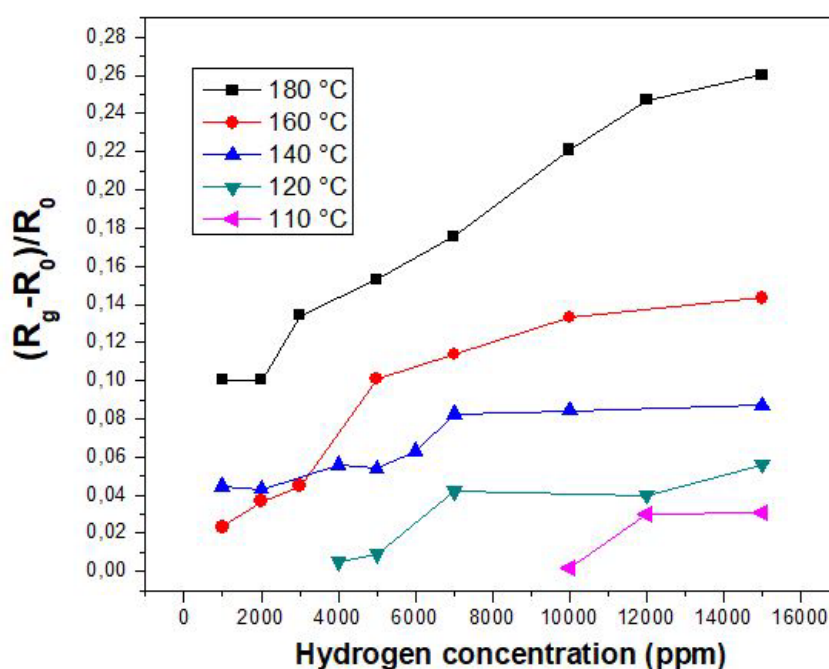


Figure 5: Sensor response of undoped CuO with respect to hydrogen concentration for various operating temperatures

Figure 7 depicts the sensor response of Pd-doped CuO at temperature as low as 60°C. The response gradually rises with increasing gas concentrations for both doped 2% and 6% samples. The responses of CuO: Pd 2% are comparable to CuO: Pd 6% for all gas concentrations. It is noteworthy that gas sensors based on undoped CuO is not responsive in such low temperature condition. The results confirm that the incorporation of Pd even with small amount en-

ables the detection of H_2 at operating temperature down to 60°C. In table 2, we present a benchmarking for H_2 gas sensors based on CuO thin films. In the state-of-art, several CuO-based sensors with and without Pd doping operate at high temperature between 200°C and 400 °C for hydrogen concentration of 1000 ppm [2,3,5]. In our current work, hydrogen gas sensors based on CuO: Pd enable efficient detection of H_2 concentration (5000 ppm) at low temperature (60°C).

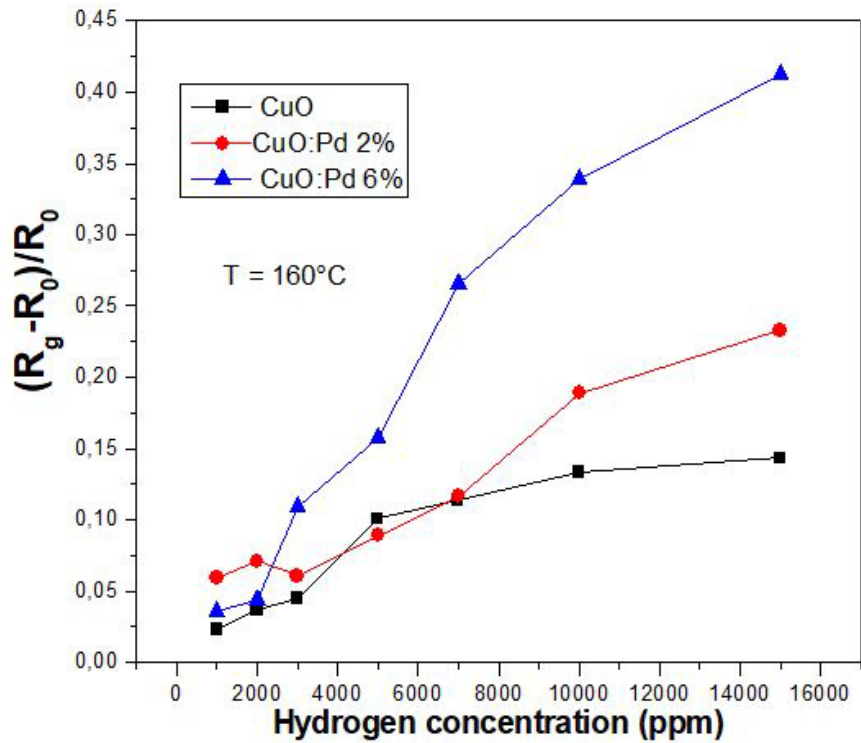


Figure 6: Maximum response of CuO, CuO: Pd 2% and CuO: Pd 6% sensors to different hydrogen concentrations for an operating temperature of 160 °C

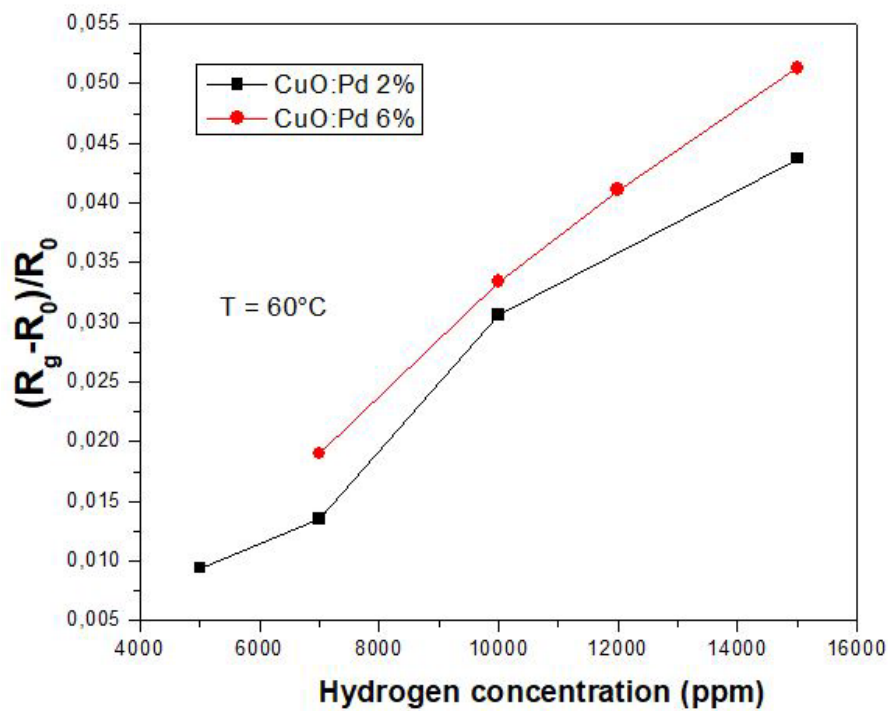


Figure 7: Comparison of the response of the sensors CuO: Pd 2% and CuO: Pd 6% to different hydrogen concentrations at 60°C

Conclusion

In this work, we demonstrated resistive hydrogen sensors using undoped and Pd-doped CuO thin films grown by sol-gel spin coating technique. Structural and morphological characterizations confirmed the polycrystalline nature and monoclinic phase of CuO-based films irrespective of Pd doping content. The investigation of different sensor devices revealed a clear increase of response outcome with both higher working temperature and hydrogen concentration conditions. As temperature increases, the reactivity, charge carrier mobility, surface defect density, and overall electrical conductivity of p-type CuO improve, which enhances the hydrogen sensor's response and sensitivity. However, undoped CuO-based sensor was not responsive at applied temperature of about 110 °C when H₂ concentration is below 10000 ppm. To overcome these limitations, Pd doping can lead to not only improved response in common working conditions of undoped CuO film in terms of temperature and gas concentrations, but also extend the detection capabilities to further lower temperatures around 60°C. Such improvement of gas sensing upon Pd doping of CuO films is assumed to be due to synergetic effects of different

morphological and electronic mechanisms such as larger effective surface area with smaller grain sizes and more appropriate electronic structure and lower electrical resistance. Our results pave the route for the simple and efficient application of p-type CuO films in hydrogen detection within practical conditions and large-scale systems.

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Declaration of Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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