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A Chemical Synthesis of $CdSnO_3$ Nanoparticles and their Performance in Hydrogen Sensing

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Abstract: ***$CdSnO_3$ nanoparticles were successfully synthesized without any templates by simple co-precipitation synthesis route. Further characterized by using X-ray diffraction (XRD) measurements, field emission scanning electron microscopy (FESEM) and Fourier transform infrared (FTIR) spectroscopy and their hydrogen sensing properties were investigated. The $CdSnO_3$ nanoparticles exhibited outstanding gas sensing characteristics such as, higher gas response, extremely rapid response, fast recovery, excellent repeatability, good selectivity and at ambient operating temperature (~ 30℃). Furthermore, the $CdSnO_3$ nanoparticles can detect up to 5 ppm for hydrogen with reasonable sensitivity at an ambient operating temperature.***
Keywords: Cadmium stannate, Hydrogen sensing, XRD, FTIR, TEM

I. INTRODUCTION

In last two decades zero- and one-dimensional metal nanostructures, such as ZnO, TiO_2 and SnO_2, have attracted enormous interest due to their unique properties and potential use in various applications such as photo catalysis, solar cells and gas sensors [1–5]. However, with keen research in nanotechnology, there is a demanding requirement for specially designed metal oxides to better match the properties of emerging materials. This has led to transformed interesting ternary metal oxides of the form ABO_3 such as cadmium stannate, zinc stannate and some metal titanate. Amongst these all metal, titanate is widely studied due to its piezoelectric nature while study of metal stannate less as compared to metal titanate. Because of this metal stannate having high electron mobility [6], high electrical conductivity, are chemically more stable than binary metal oxides and attractive optical properties that makes it suitable for a wide range of applications in solar cells, sensors for the detection of humidity and gases, negative electrode material for battery and as a photo catalyst[7-9]. The sizes and shapes of nano structures are crucial as it may affect their overall properties. Therefore, synthesis of nanostructures has considerably progressed over the last decade, to achieve different variety of shapes of nano materials. However, the synthesis of complex or ternary structures still remains a challenge for researchers. $CdSnO_3$ is *n*-type semiconductors with a band gap of 2–3 eV, which, due to a high concentration of native defects, are characterized by a rather high conductivity among all metal stannate [10]. The aim of present article is synthesizing$CdSnO_3$ nanoparticles by co-precipitation method. Further, characterize by various characterization techniques and evaluate the hydrogen sensing performance of $CdSnO_3$ thin film. As per our knowledge hitherto no body studied the hydrogen sensing properties of $CdSnO_3$ thin film.

II. EXPERIMENTAL

A. Materials

All chemicals were of analytical grade. The cadmium acetate, stannous chloride and sodium hydroxide were purchased from E-Merck (India) and were used without further purification.

B. Synthesis of the $CdSnO_3$ nanoparticles

In this work, the $CdSnO_3$ nanoparticles were synthesized without any templates by using cadmium chloride, stannous chloride and ammonia as starting materials through a simple and low cost co-precipitation synthesis route. The cadmium chloride was used as the source of Cd^{2+}, the stannous chloride was used as the source of Sn^{2+} and ammonia was used as the precipitating agent to release hydroxyl ions slowly during the reaction. In a typical experiment, the aqueous solution containing 0.2 M Cadmium chloride, 06 M stannous chloride and 15 ml ammonia (30%) dissolved in 15 ml distilled water was prepared and added drop wise in the mixture of cadmium chloride and tin chloride to maintain the pH of the solution ~ 7 during the reaction and continuously stirred for 1 hour at room temperature 30 °C to obtain white coloured precipitate. The resulting cadmium hydroxyl stannate powder was washed with double distilled water and alcohol several times to remove impurities and by products present in the product.

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The precipitate thus formed was dried at 80 °C in hot air oven for 12 h and grounded into a fine powder, which was then calcinated in air at different calcinating temperature at 400 °C for 2 h to obtain the end product.

C. Hydrogen Sensing Properties

The cadmium stannate nanoparticles powder was spin coated on the alumina substrate and the ohmic contacts were made with the help of silver paste to form gas sensing element. For the preparation of spin coated cadmium stannate thin films the cadmium stannate powder was dissolved in mixture of acetyl-acetone and ethanol in the ratio 8:2 to form a suspension in which 0.1 gm of p-hydroxy benzoic acid was added and the suspension was sonicated for one hour. The mixed suspension of cadmium stannate was spin coated using spin coater (SPN2000, Milman Thin Film Systems, Pvt. Ltd., Pune, India) forms the paste then the paste was coated on the alumina electrode and heated at 800°C to remove water from the film for the hydrogen sensing study. The electrical contact leads were fixed 0.7 cm apart with the help of silver paste on the surface of the film. The electrical resistance of the film was measured as a function of gas response by using a simple two probe configuration with a sensitive digital multimeter (2000 Digital multimeter, Keithley) controlled by a personal computer. The continuous variation in resistivity in the present of hydrogen gas was achieved in a simple experimental set-up fabricated in our laboratory in order to investigate the hydrogen sensing properties.

III. RESULTS AND DISCUSSION

A. Characterizations

The sensor was fabricated to study the different gas sensing properties of $CdSnO_3$. The XRD pattern of as-prepared product annealed at temperature 400°C is depicted in Fig.1.All the diffraction peaks in the XRD pattern shown is indexed to cadmium stannate(JCPDS No.: 34-0885), indicating the formation of orthorhombic crystal structure (space group: Pb nm (62), a=5.4578, b=5.5773, c=7.8741) of distorted perovskite type structure. No other peaks were observed, indicating that no impurities were present and confirming that the adopted synthesis method gives pure $CdSnO_3$nanoparticles. The average crystallite size was calculated by fitting the [2 0 0] diffraction peak ($2\theta = 32.9°$) with a Gaussian function and using the values of the diffraction angle and peak full line width at half of maximum (FWHM) in the Debye-Scherrer formula –

$$D = \frac{k\lambda}{B\cos\theta} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where D is the average size of the crystallite, assuming that the grains are spherical, k is constant and it is ~ 0.9, λ is the wavelength of the X-ray radiation, B is the peak FWHM in radian and θ is the diffraction peak position. X-ray diffraction (XRD) analysis was performed with a Bruker diffractometer (D8, Advance, Bruker AXS model) with CuKα radiation (λ=1.5406 nm) operating at 40 kV and 40 mA. The average crystallite size of the $CdSnO_3$ nanoparticles was found to be in the range of 3.65 nm at 400°C. Wang et al and Meena et al shows the synthesis of $CdSnO_3$ nanoparticles and its crystal fitted by JCPDF data card, PDF#34-0758 which is rhombohedral, Hexagonal, R-3(148). They reported particle size of CdSnO3nanoparticles ~40-50 nm. Whereas Jia et al reported orthorhombic, β-$CdSnO_3$ nanoparticles has crystallite size 50 nm.

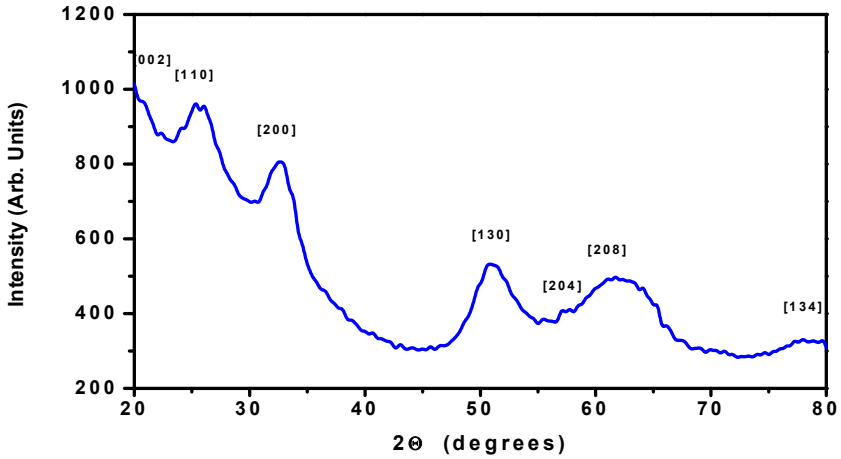


Fig. 1: XRD pattern of $CdSnO_3$ powder (calcinated ~400°C) prepared using Co-precipitation synthesis method.

The FTIR spectrum of the nanostructured CdSnO3 heated at 400°C are shown in Fig.2. The FTIR spectrum for calcinated perovskite $CdSnO_3$sample at 400°C is exhibits a broad band of which is mixing of three non-significant maxima of absorption between 630 and 690 cm^{-1}, the first peak at 638 cm^{-1} (Sn–O bond stretching along the *b* axis), the second at 661 cm^{-1} (Sn–O bond stretching along the *a* axis, the 654 cm^{-1} peak is very small), and the third at 687 cm^{-1}(Sn–O bond stretching along the *a+c* direction). Mainly, the wide band at 440 cm^{-1} is due to Sn–O–Sn scissoring. The peaks at 800–1400 $cm^{-1}$are assigned to CdO. The bands in the region of 520–670 cm^{-1}can be ascribed to the stretching vibration of Sn-O. An upward shift in the frequency range ~ 528–551cm^{-1} is due to the presence of Sn in Cd–O lattice [11-12,19]. The FTIR spectroscopy analysis was performed with a Nicolet FTIR spectrometer (IMPACT 420 DSP) by the conventional KBr method in the spectral range 4000-400cm^{-1}

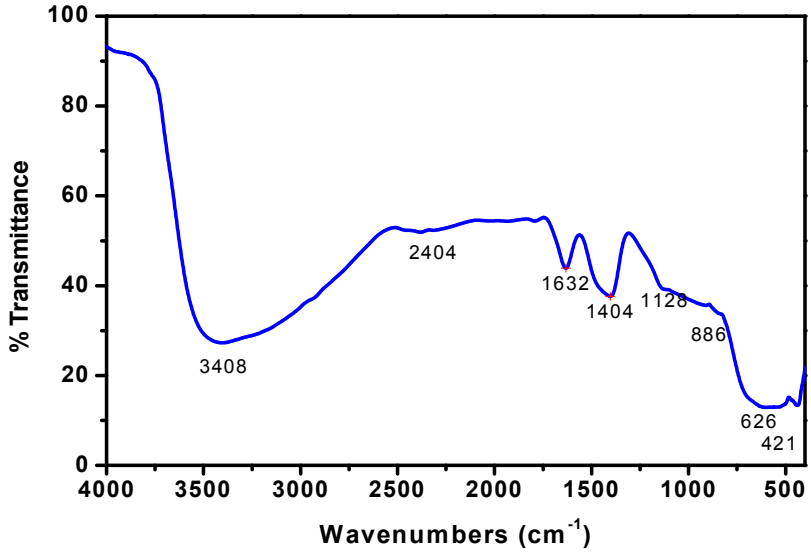


Fig. 2: FTIR spectrum of $CdSnO_3$ nanoparticles calcinated at 400^0C

The TEM image of as-prepared product [Fig.3(a)] exhibits a non-uniform shaped, narrow sized distributed and agglomerated the nanoparticles at 400°C. The average grain size of the $CdSnO_3$nanoparticles is estimated to be around 5-6 nm, which is nearly matches to XRD crystallite size. Fig. 3 (b) shows the high resolution TEM (HRTEM) image. This HRTEM image shows non-uniform fringes and drastic variation in intensity region-wise. This study suggests that nanoparticles are non-uniformed sizes and oriented in particular direction like a single crystalline structure. The surface morphological study was performed by a high-resolution transmission electron microscope (HRTEM, Tecnai G2 20 Twin, FEI, USA)

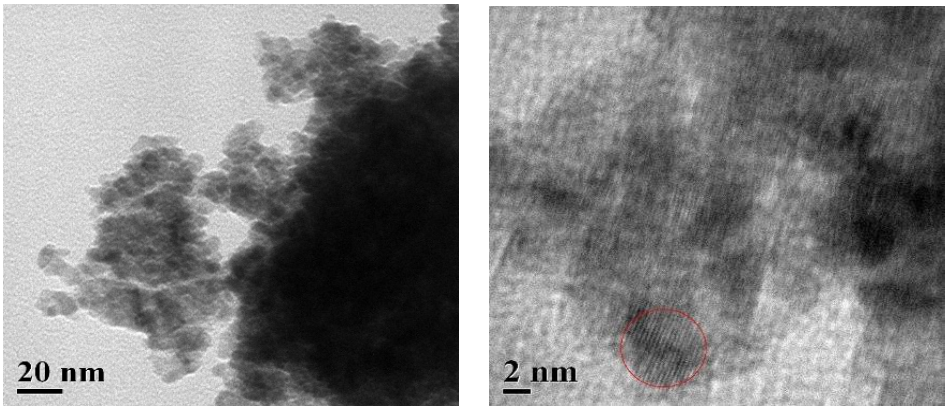


Fig.3: (a) TEM (b) HRTEM image of 400^0C calcinated$CdSnO_3$ nanoparticles

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B. Hydrogen Sensing Characteristics

The H_2 gas sensing experiments were performed at different temperatures in order to find out the optimum operating temperature for H_2 gas detection. Before exposing to the H_2 gas, the sensing element could equilibrate inside the gas chamber at an operating temperature for 1 h. The effect of an operating temperature on the gas response of $CdSnO_2$nanoparticle based sensor to 50 ppm H_2 is shown in Fig.4.

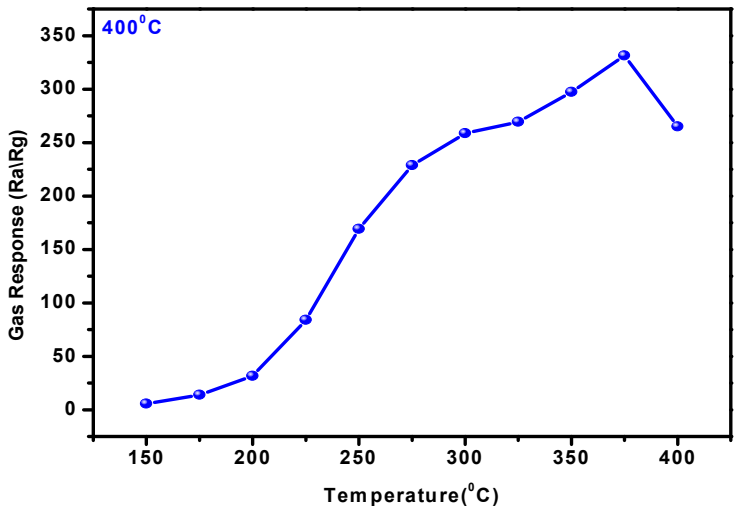


Fig.4: Effect of operating temperature on the gas response of $CdSnO_3$ nanoparticles (calcinatedat 400°C) powder to 50 ppm H_2 gas.

The relationship between the gas response and the operating temperature exhibits a trend of "increase-maximum-decay" behaviour to 50 ppm H_2 gas. To investigate the various H_2 sensing characteristics of this sample such as response and recovery, reproducibility and selectivity, operating temperature is optimized to 300°C. The response and recovery characteristics of the $CdSnO_3$ nanoparticles to 50 ppm H_2 gas at an operating temperature 375°C is shown in Fig. 5. It was observed that the resistance of the sensing element decreases when exposed to the H_2. As can be seen from Fig. 5, the sensor responds very rapidly after introduction of H_2 and recovers slowly when it is exposed to air. The $CdSnO_3$ nanoparticles have response time of ~ 2-3 s and the recovery time of ~ 15-17 s. The $CdSnO_3$ nanoparticles show good reproducibility and reversibility upon repeated exposure and removal of H_2 under same conditions. This suggests that the $CdSnO_3$ nanoparticles can be used as a reusable sensing material for the detection of H_2.

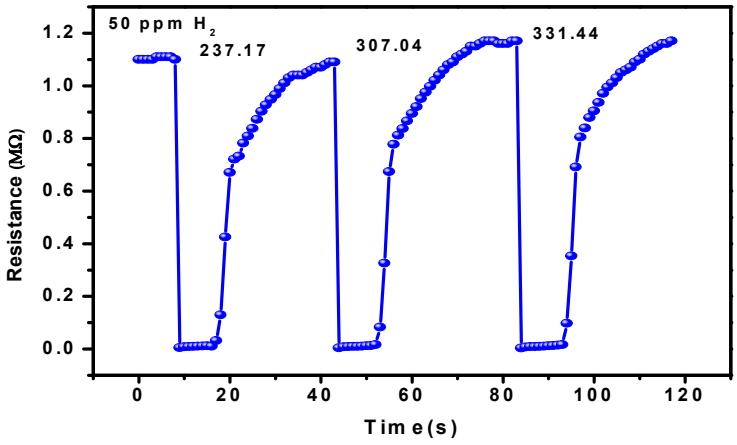


Fig.5: Repetitive response of $CdSnO_3$ nanoparticles to 50 ppm H_2 gas at an operating temperature of 400°C.

The gas response of the $CdSnO_3$ nanoparticles versus H_2 gas concentration at an operating temperature of 375°C is shown in Fig. 6. It was observed that the gas response increases linearly in the range 5-50 ppm H_2 gas. It is found that the response of $CdSnO_3$ nanoparticles can be empirically represented as,

$y = -12.72 + 7.072 * x$, $R_2 = 0.99224$,

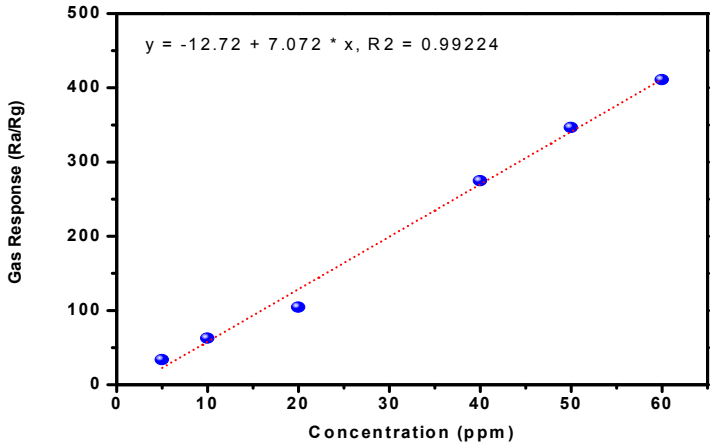


Fig.6: Response of $CdSnO_3$ nanoparticles to 50 ppm H_2 gas at an operating temperature of 400°C.

Where x, y and R_2 represents the H_2 concentration, gas response and correlation coefficient, respectively. The dotted line shows the linear fit to the experimental data, illustrating clearly good quality of the fit. The linear relationship between the gas response and the H_2 concentration at low concentrations (5-50 ppm) may be attributed to the availability of enough sensing sites to act upon the $CdSnO_3$ nanoparticles.

Selectivity is an important parameter of gas sensors and it is the ability of a sensor to respond to a certain gas in presence of other gases. Theoretically, the sensors should have high response to some gases and little or no response to other gases in the same surroundings. To study the selective behaviour of the $CdSnO_3$ nanoparticles to H_2 at an operating temperature of 300 ºC, the gas response towards LPG, CO, $CO_2$and ethanol with concentration 50 ppm each were also measured.

The selectivity property of $CdSnO_3$nanostructured thin film at various pollutant gases is shown in Fig. 7. The $CdSnO_3$ nanoparticles exhibit higher response to H_2 (331), whereas it shows a considerably lower response (<7.62) to LPG, CO, CO_2 and ethanol. The selectivity coefficient (K) of H_2 to another gas is defined as [13, 14]:

$$K = \frac{S_{H2}}{S_B}$$

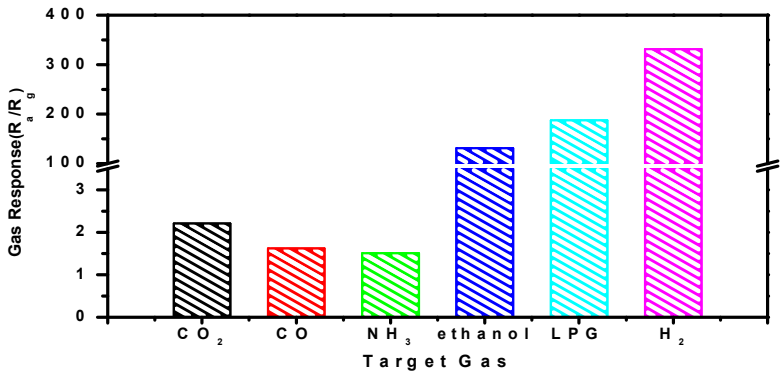


Fig. 7: Bar chart showing the gas response of $CdSnO_3$ nanoparticles for different gases. The gas concentration and operating temperature in all cases were 50 ppm and 400°C, respectively.

Here S_{H2} and S_B are the responses of sensors in H_2 and B gases, respectively. The selectivity coefficients for the $CdSnO_3$ nanoparticles were 187.21 to LPG, 2.21 to CO_2, 1.62 to CO and 131.21 to ethanol. The experimental results indicate that the $CdSnO_3$nanoparticles-basedsensor has a good selectivity to H_2. The reproducibility and stability of the $CdSnO_3$ nanoparticles were measured by repeating the measurement many times.

In literature possible hydrogen sensing mechanism are explained on the basis of adsorption and desorption mechanisms. The probable gas sensing mechanism is upon exposure to H_2 gas, much greater number of trapped electrons are released once the adsorbed surface O species are chemically reduced by the H_2 molecules leading to lowering of the barrier height and increasing the conductivity. Therefore, change in the current as well as the magnitude of the highest current in $CdSnO_3$ nanoparticles is much higher resulting in the enhanced gas response. The Cd involves in Sn-O lattice is already observed in FTIR and XRD result. An increase in the surface-to-volume ratio of the $CdSnO_3$ nanoparticles which would increase number of adsorbed O molecules and an increase in the surface defects, which can influence the chemical as well as electronic properties, the adsorption behaviour. Further these defects also control the carrier concentration via effective near surface electron depletion. Thus, these are main reasons enhancement of H_2 gas response [15-18].

IV. CONCLUSIONS

1) We have successfully synthesized the $CdSnO_3$ nanoparticles at low cost by using a simple co-precipitation method by calcination of $CdSnO_3$ nanoparticles.

2) XRD and FTIR results clearly indicating formation of $CdSnO_3$ nanostructure. TEM study revealed the formation of single crystalline nanostructures.

3) The gas response to 50 ppm of H_2 gas is found to be ~ 331.44. The response time was nearly 3-4 sec and the recovery time was found to be 5-6 sec.

4) The synthesized $CdSnO_3$ nanoparticles are able to detect up to 5 ppm for H_2 with reasonable response at room temperature. Further, it was shown that the $CdSnO_3$nanoparticles can be reliably used to monitor the concentration of H_2 gas over the range (5-60 ppm).

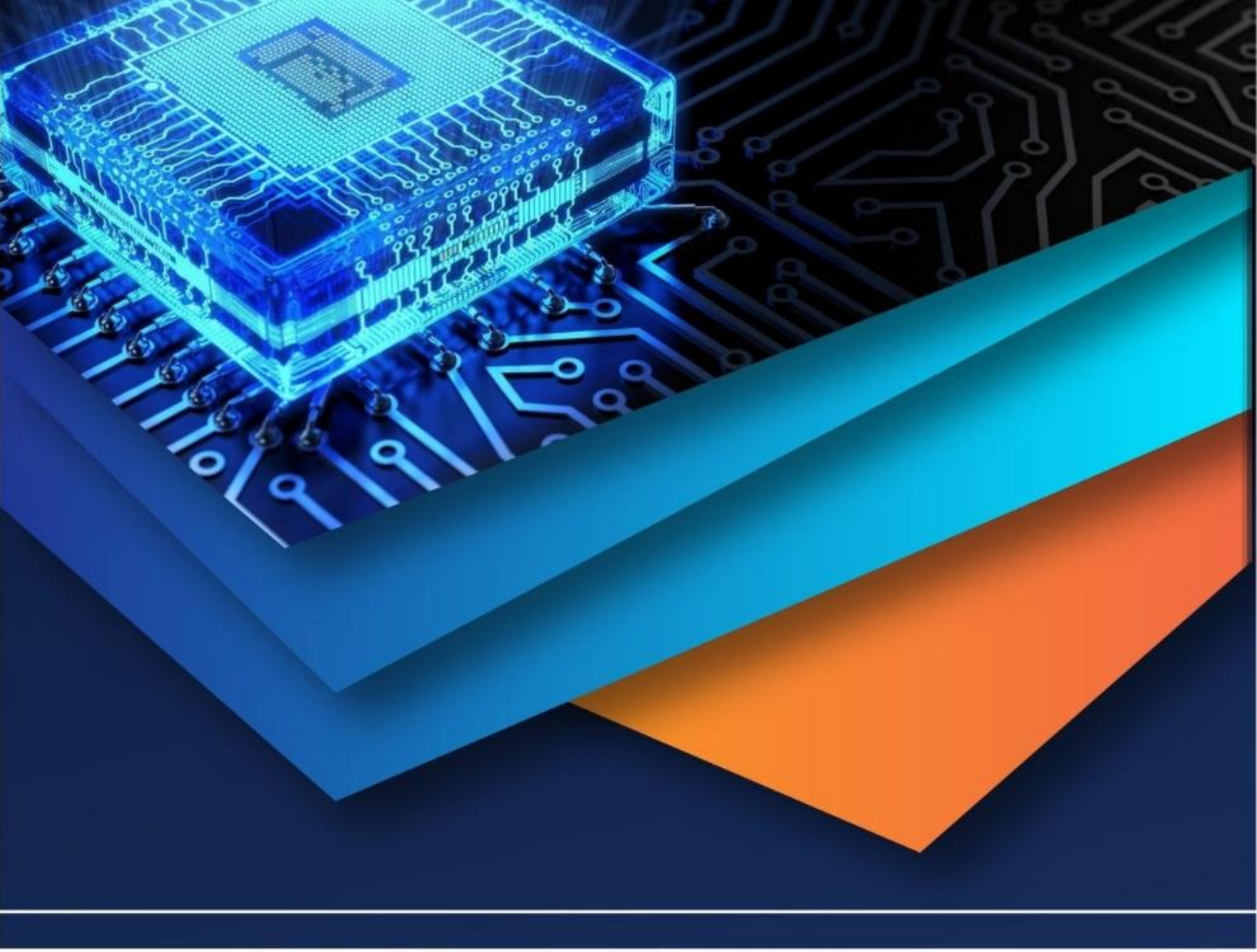
5) These results indicate that the $CdSnO_3$ nanoparticles are indeed very attractive H_2 gas sensing materials.

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