IJCRT.ORG ISSN : 2320-2882

INTERNATIONAL JOURNAL OF CREATIVE RESEARCH THOUGHTS (IJCRT)
An International Open Access, Peer-reviewed, Refereed Journal

Synthesis Methods in the Context of Obtaining Materials for a Hydrogen Sensor Include Different Approaches.

T. Kalimulla[1*], N. Umamaheswara Reddy[1], B V Chakravarthi[2]

[1*, 2] T. Kalimulla, Department of Physics, Government Degree College, Kalyandurg, AP, India.

[1]N. Umamaheswara Reddy, Department of Physics, Government Degree, College, Banaganapalle, AP, India.
Corresponding author: kalimullaforu@gmail.com

Abstract

Researchers studying safety in industries including metallurgy, pharmaceutics, power generation, and the chemical industry are very interested in developing hydrogen sensors. This interest is heightened by the advent of hydrogen as an automotive fuel. For the purpose of detecting hydrogen, various types of sensors have been created, such as conductometric, optical, surface acoustic wave, and resistive sensors. The characteristics of the material used in the sensor's sensitive region are crucial in determining how well it works. In addition to the material's nature, the synthesis process and factors employed during the synthesis have a significant impact on the final qualities of the material. This paper presents the latest findings in hydrogen detection, which were acquired by four popular synthesis and deposition techniques: sol-gel and spin-coating.

Keywords: surface acoustic wave; sensors; hydrogen; sensor; sol-gel; co-precipitation; Spin-coating; pulsed laser deposition

1. Introduction

The scientific community is currently interested in hydrogen. It is becoming the source of a new generation of fuel because of its energy potential. It is a clean, efficient, renewable, and environmentally beneficial energy source [1]. Energy produced by hydrogen is meant to take the place of fossil fuels in many contemporary environmental issues. Hydrogen has been employed as a rocket fuel in aerospace projects up to this point. Vehicles powered by hydrogen and based on hydrogen fuel cells have already been released onto the market by BMW, Honda, Toyota, and Hyundai [2]. Because of its ability to reduce, hydrogen is also utilized in the chemical, aerospace, pharmaceutical, metal reduction, petroleum extraction, and synthetic fuels industries (Figure 1). Consequently, it's becoming more common.

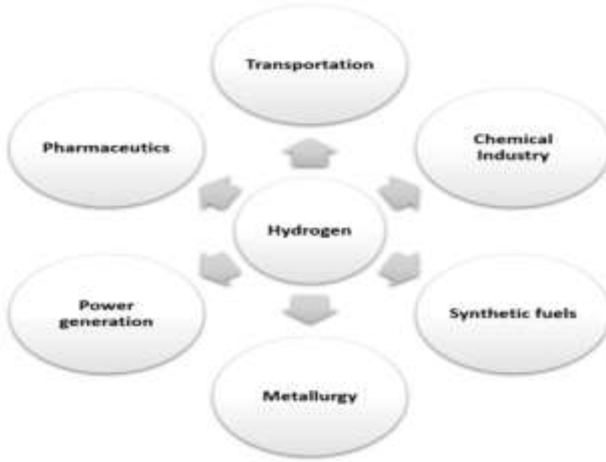

Figure 1. Areas of applicability of hydrogen.

Hydrogen poses no risk of flammability or explosion up to a concentration of 4%. The risk of inflammation rises at this threshold, and at much higher doses, it explodes. It should be remembered that hydrogen is a relatively small molecule that has a high air diffusion coefficient (0.6 cm2/s at 0 °C), making it able to penetrate a wide range of materials [3]. This feature makes it possible for uncontrolled accumulations to occur in some areas, which encourages the creation of explosions. Moreover, elevated hydrogen concentrations cause a reduction in oxygen levels, which in turn causes asphyxia, apoptosis of the neurons, and other problems of the neurons. Because the human senses cannot directly detect hydrogen through smell or colour, and because of, its short atomic radius [4] this detection must be carried out using specialized sensors. Current research aims to link hydrogen's lack of color and odor to these features. This is especially important in locations where its release and accumulation are impending, to make detection easier, including by humans.

The following are the performance requirements for a hydrogen sensor: selectivity to other reducing gases, like NO, CO, and H_2S; high sensitivity, high accuracy, quick response and recovery times; appropriate operating temperature, preferably room temperature; stability to environmental factors, like temperature and humidity; repeatability; long-term stability; and low cost [3]. The literature on hydrogen detection examines a few of the sensor types that fit these requirements, including resistive, conductometric, optical, surface acoustic wave, and catalytic sensors. While the working principles of each of these sensors vary, they are all similar in that they use a sensitive substance to detect the presence of an analyse. As a result, Researchers have a strong interest in the creation of sensitive material because it plays a significant role in the functioning of the sensors. The study will cover a few topics pertaining to the sensors' sensitive material. To get the best possible outcomes, the following generally applicable properties of a material must be considered during the design process: composition, structure, microstructure, and morphology. The synthesis method and the conditions selected for the process have an impact on these properties. Metals, polymers, composite materials, and semiconductor metal oxides [4] are the material types most frequently utilised in hydrogen sensor technology. The benefits and drawbacks of each of these materials can change according on the kind of sensor being used for detection, as well as the settings under which the sensors are evaluated. Another critical element is that the synthesis processes are unique to each material group. Sol-gel [5], evaporation [1], RF magnetron sputtering [6], DC magnetron sputtering [7], precipitation [8], electro spinning [9], pulsed laser deposition (PLD) [10], thermal oxidation [11], hydrothermal [12], and in situ self-assembling [13] are among the most commonly utilized synthesis processes.

The goal of this study is to present the most recent research on the production of new materials using simple and reasonably easy processes, thereby encouraging the development of hydrogen sensors. Sol-gel and spin coating are the approaches investigated here.

2. Synthesis Methods

The development of novel materials with new qualities that improve performance in today's technology is a major research priority. The synthesis of new materials or materials with novel properties inevitably leads to advancements in synthesis processes. New or better material qualities have been obtained by unconventional synthesis methods, resulting in technological advancement. Small particle sizes (below 100 nm), different types of morphology, the ability to control doping concentrations or complex compositions, obtaining materials with multiple phases, or the synthesis of 2D materials are the main characteristics of materials obtained by such methods that have led to advancements in the field [14].

Several methods of synthesis and deposition have been utilized most frequently in the domain of sensors, particularly for hydrogen sensors: sol-gel [5], hydrothermal [12], thermal evaporation [14], PLD [10], magnetron sputtering [6], co-precipitation [15], and spin coating [16].

Given that hydrogen is a relatively small molecule with considerable permeability through many materials and that selective detection is difficult to obtain, the synthesis of sensitive materials for higher performance sensors remains a challenge [12].

To accurately select a synthesis technique for a material, it is vital to understand the processes involved as well as the primary elements influencing the final properties of the materials. In this manner, the synthesis parameters can be modified to attain the qualities required for a certain application.

This paper will describe two approaches for synthesis of materials and thin films for hydrogen sensors: sol-gel [5] and spin coating [16]. This method was chosen because of simple to implement and have previously shown promising outcomes in the field of sensors.

2.1. Sol-Gel

The sol-gel synthesis process is an unusual synthesis route noted for its ability to regulate the final properties of materials. It enables control from both a compositional and morphological standpoint by altering the synthesis condition.

The sol-gel approach is based on two types of reactions: hydrolysis and poly-condensation [5]. In practice, a solution is turned into a gel, which subsequently undergoes a heating process to reach the powder stage. The hydrolysis reaction begins with an alkoxide that has been dissolved in a solvent. It then proceeds to poly-condensation, which occurs following the addition of a little amount of water and results in the development of a polymer chain and hence the gel-type material. After that, a thermal procedure, most commonly in steps, the liquid phase of the gel is removed, and a powder is formed. Depending on the type of deposition to be made, there are many processing methods available after getting the sol. As a result, the sol can be placed on the substrate, generating a xerogel that, after heat treatment, transforms into a dense film [17]. Another method of treating the sol, as shown in Figure 2, is gelation, evaporation (to create a xerogel or an aerogel), followed by a heat treatment at high temperatures to obtain a material as dense as feasible. There are also processing methods that avoid the gel phase, such as precipitating the sol or electro spinning fibers.

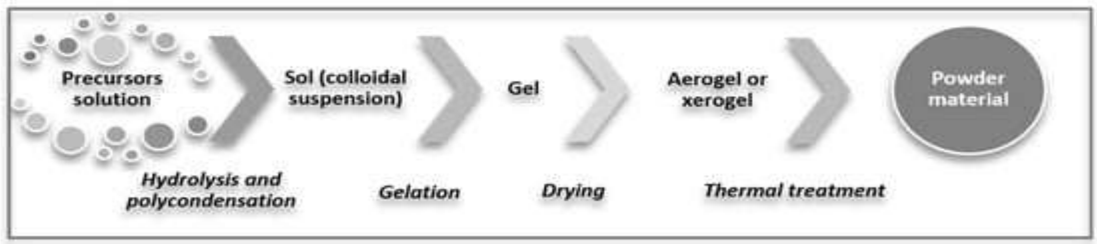

Figure 2. Synthesis scheme for sol-gel method.

Figure 2 depicts the benefits and drawbacks of the sol-gel synthesis method. Regardless of the sol-gel synthesis method used, among the benefits of this technology are the ability to control the reactions involved in the synthesis and create materials with homogeneity, even in systems with a large number of components. Another significant advantage of this approach is that it does not require any special synthesis conditions, such as maintaining a specific pressure or atmosphere [5].

The sol-gel process to create Pd-WO_3 multilayer composite films. Using Pluronic F127 as a template, the porosity of the films was improved, resulting in a greater specific surface area, which is advantageous for gas detection. The presence of Pd resulted in a sensitivity that was 346.5 times greater than in the absence of Pd. the instance of just WO_3. The development of p-n hetero-junctions also enhanced this characteristic.

By combining the sol-gel and spin coating techniques, researchers were able to generate ZnO films under various synthesis circumstances. They discovered that the morphology of the sensitive material in hydrogen sensors greatly affects how well they work after analyzing the various morphologies that were produced as a result of changing the synthesis conditions. The impact of a sol-gel SnO_2 film on the sensitivity of a thin layer Ga_2O_3 hydrogen sensor was examined. The tests conducted at various temperatures showed a notable improvement in sensor performance, and they produced impressive results at room temperature, confirming that the employment of the sol-gel synthesis method is a facilitator for the enhancement of sensors for hydrogen [14]. The way the sensors behave at different temperatures and how they react at room temperature differ significantly. It is crucial that the sensors react at room temperature because one goal in the development of sensors is for them to be able to function at that temperature. The sensor tested at room temperature recorded values that were not too dissimilar from those obtained at high temperatures in terms of response and recovery durations.

Advantages	Disadvantages
• Control of the reactions involved in the synthesis • Homogeneity • The possibility to obtain materials in a wide range of oxide compositions • Control of the composition, structure, microstructure, porosity of the material • Easy to ensure the synthesis conditions (without special or expensive equipment) • The possibility to be combined with other deposition methods	• Relatively high cost of precursors • In multicomponent compositions, there is the possibility of preferential precipitation of one of the components • Difficult to avoid or eliminate residual porosity or OH groups • Material shrinkage

2.2 Spin Coating

Surface modification through thin film deposition or functionalization has become a popular study topic, contributing significantly to the advancement of fields like as medicine, sensors, and various areas of technology. One method for achieving this alteration is to apply thin films to the surfaces of various materials.

Spin coating is one of the most used processes for producing thin films. It is a technique for producing uniform thin films with thicknesses in the micrometer and nanometer range on flat-shaped substrates. It is capable of depositing both organic and inorganic compounds. This approach employs centrifugal force to distribute the material solution equally throughout the full surface of the substrate. [18].

A typical spin coating deposition procedure (Figure 2) starts with preparing the deposition material in a somewhat volatile solvent, which must have a specified viscosity to allow uniform deposition throughout the entire substrate. The substrate is secured by suction in the spin coater on a chuck that rotates the sample. A portion of the material solution is placed into the center of the substrate, which is then accelerated (up to 8000 rpm) according to a predetermined program. The substance is spread across the surface of the substrate by centrifugal force, and the thin film is formed when the solvent evaporates. This method can be replicated for multilayer architectures [19].

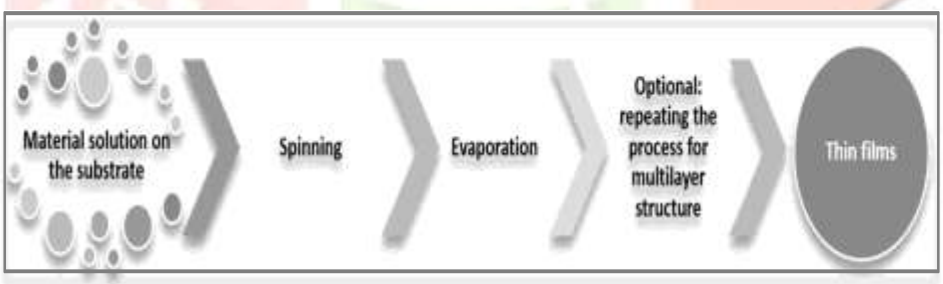

Figure 3. Synthesis scheme of spin-coating method.

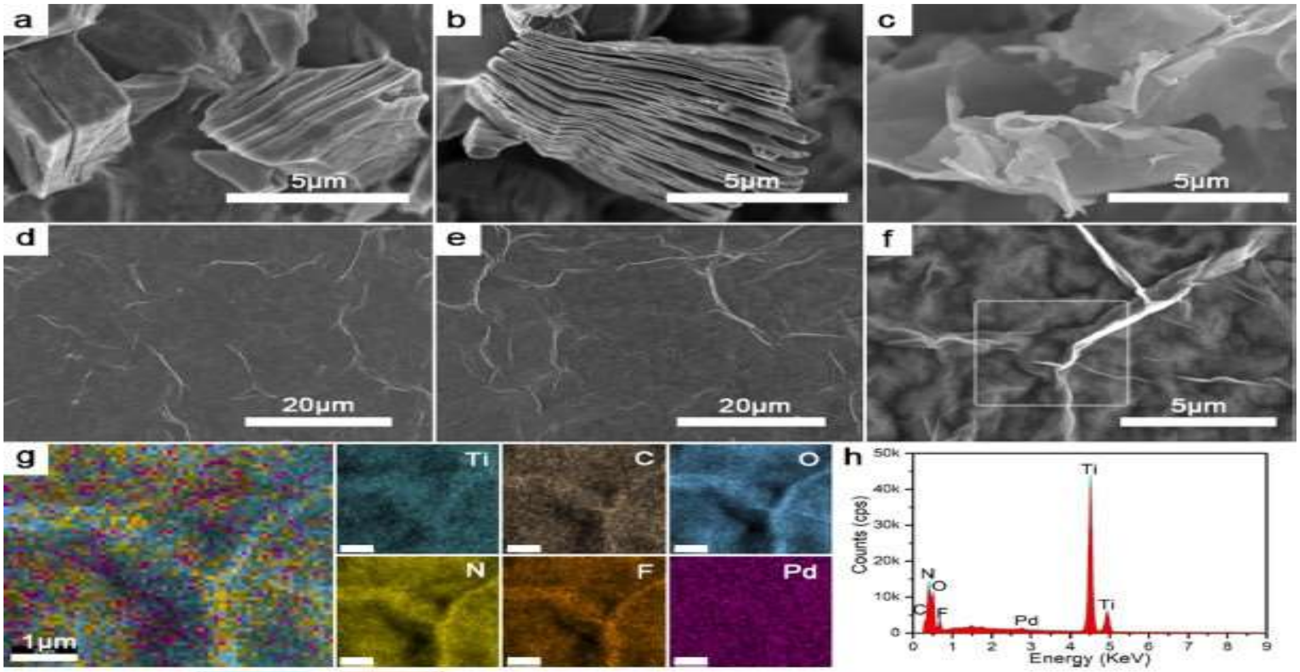

Figure 4. SEM and EDX images of the material surface morphology. (**a**) SEM image of bulk Ti_3AlC_2; (**b**) morphology of the commercial $Ti_3C_2T_X$; (**c**) SEM image of d-$Ti_3C_2T_X$ after intercalation;(**d**) morphology of/ d-$Ti_3C_2T_X$ film; (**e**–**h**) Pd (II)@ alkyne-PVA/d-$Ti_3C_2T_X$ film surfaces morphology, element mapping (1 µm scale bar), and EDX spectrum.

The viscous force and surface tension are two significant factors that contribute to the creation of a thin film. Other parameters, such as material solution properties, substrate properties, spin speed, and acceleration, have a substantial influence on the ultimate properties of the films. One of the qualities that may be regulated in this process is the thickness of the film, which is influenced by a few parameters. Equation (1) [20] indicates how various parameters influence and govern film thickness, where h—thickness, A—density of volatile liquid, —viscosity of solution, m—rate of evaporation, and —angular speed.

Advantages	Disadvantages
• Simple and inexpensive technique • Low energy consumption • Easily adaptable for use with other synthetic methods • Usable for both organic and inorganic materials • The possibility to realize multilayer films	• Small deposition area • Low efficiency related to the amount of material deposited • Possible impurities following incomplete evaporation of the solvent

Conclusions

One of the greenest energy sources is hydrogen, which is already used in a number of industries including metallurgy, electricity generation, and transportation. However, at concentrations greater than 4% in a closed space, hydrogen poses a risk of explosion and irritation. Controlling the quantity of hydrogen in a particular environment is therefore crucial, and creating sensors with high sensitivity and selectivity is currently a highly sought-after objective.

The creation of the materials and their properties for this purpose is the initial stage in the development of such sensors. The production of nanomaterials using the sol-gel and co-precipitation processes allows for the detection of hydrogen at the lowest feasible quantities because of their big surface area specifically.

Because of the heat treatment that is applied, the sol-gel method of material synthesis guarantees the production of some materials with complicated compositions and regulated morphologies. Precursors for the sol-gel process are often expensive. Rather, co-precipitation synthesis uses less expensive ones, which is a benefit of this synthesis technique. The synthesis of thin films on the surface of the sensor devices can be achieved bythe simple spin coating method. As shown, it allows the synthesis of high-quality films, requiring low costs. Furthermore, the co-precipitation is unique in that it is a straightforward process that yet permits the production of doped materials or materials with intricate compositions.

References

1. Xu, H.; Liu, Y.; Liu, H.; Dong, S.; Wu, Y.; Wang, Z.; Wang, Y.; Wu, M.; Han, Z.; Hao, L. Pd-decorated 2D SnSe ultrathin film on SiO2/Si for room-temperature hydrogen detection with ultrahigh response. *J. Alloys Compd.* **2021**, *851*, 156844.
2. Yue, M.; Lambert, H.; Pahon, E.; Roche, R.; Jemei, S.; Hissel, D. Hydrogen energy systems: A critical review of technologies, applications, trends and challenges. *Renew. Sustain. Energy Rev.* **2021**, *146*, 111180.
3. Yang, T.; Zhangn, Y.; Li, C. Large scale production of spherical WO_3 powder with ultrasonic spray pyrolysis assisted by sol–gel method for hydrogen detection. *Ceram. Int.* **2014**, *40*, 1765–1769.
4. Mirzaei, A.; Yousefi, H.R.; Falsafi, F.; Bonyani, M.; Lee, J.-H.; Kim, J.-H.; Kim, H.W.; Kim, S.S. An overview on how Pd on resistive- based nanomaterial gas sensors can enhance response toward hydrogen gas. *Int. J. Hydrogen Energy* **2019**, *44*, 20552–20571.
5. Noh, H.J.; Kim, H.-J.; Park, Y.M.; Park, J.-S.; Lee, H.-N. Complex behavior of hydrogen sensor using nanoporous palladium film prepared by evaporation. *Appl. Surf. Sci.* **2019**, *480*, 52–56.
6. Jaballah, S.; Dahman, H.; Ghiloufi, I.; Neri, G.; El Mir, L. Facile synthesis of Al-Mg co-doped ZnO nanoparticles and their high hydrogen sensing performances. *Int. J. Hydrogen Energy* **2020**, *45*, 34268–34280.
7. Nguyen, T.D.T.; Van Dao, D.; Kim, D.-S.; Lee, H.-J.; Oh, S.-Y.; Lee, I.-H.; Yu, Y.-T. Effect of core and surface area toward hydrogen gas sensing performance using Pd@ZnO core-shell nanoparticles. *J. Colloid Interface Sci.* **2021**, *587*, 252–259.
8. Chen, K.; Yuan, D.; Zhao, Y. Review of optical hydrogen sensors based on metal hydrides: Recent developments and challenges. *Opt. Laser Technol.* **2021**, *137*, 106808.
9. Del Orbe Henriquez, D.; Cho, I.; Yang, H.; Choi, J.; Kang, M.; Chang, K.S.; Jeong Bae, C.; Han, S.W.; Park, I. Pt Nanostructures Fabricated By Local Hydrothermal Synthesis For Low-Power Catalytic-Combustion Hydrogen Sensors. *ACS Appl. Nano Mater.* **2021**, *4*, 7–12.
10. Liu, W.; Zuo, H.; Wang, J.; Xue, Q.; Ren, B.; Yang, F. The production and application of hydrogen in steel industry. *Int. J. Hydrogen Energy* **2021**, *46*, 10548–10569.
11. Okolie, J.A.; Patra, B.R.; Mukherjee, A.; Nanda, S.; Dalai, A.K.; Kozinski, J.A. Futuristic applications of hydrogen in energy, biorefining, aerospace, pharmaceuticals and metallurgy. *Int. J. Hydrogen Energy* **2021**, *46*, 8885–8905.

12. Li, X.; Gao, Z.; Li, B.; Zhang, X.; Li, Y.; Sun, J. Self-healing superhydrophobic conductive coatings for self-cleaning and humidity-insensitive hydrogen sensors. *Chem. Eng. J.* **2021**, *410*, 128353.
13. Moon, J.; Hedman, H.P.; Kemell, M.; Tuominen, A.; Punkkinen, R. Hydrogen sensor of Pd-decorated tubular TiO_2 layer prepared by anodization with patterned electrodes on SiO2/Si substrate. *Sens. Actuator B-Chem.* **2016**, *222*, 190–197.
14. Kabcum, S.; Channei, D.; Tuantranont, A.; Wisitsoraat, A.; Liewhiran, C.; Phanichphant, S. Ultra-responsive hydrogen gas sensors based on PdO nanoparticle-decorated WO3 nanorods synthesized by precipitation and impregnation methods. *Sens. Actuator B-Chem.* **2016**, *226*, 76–89.
15. Kim, J.-H.; Mirzaei, A.; Kim, H.W.; Kim, S.S. Combination of Pd loading and electron beam irradiation for superior hydrogen sensing of electrospun ZnO nanofibers. *Sens. Actuator B-Chem.* **2019**, *284*, 628–637.
16. Gautam, Y.K.; Kumar, A.; Ambedkar, A.K.; Kumar, V.; Pal Singh, B. Hydrogen induced resistance and optical transmittance of pulsed laser deposited Pd/Mg thin films. *Appl. Innov. Res.* **2019**, *1*, 96–100.
17. Zhang, Y.; Peng, H.; Zhou, T.; Zhang, L.; Zhang, Y.; Zhao, Y. Hydrogen sensor based on high-birefringence fiber loop mirror with sol-gel Pd/WO_3 coating. *Sens. Actuator B-Chem.* **2017**, *248*, 71–76.
18. Han, Z.; Ren, J.; Zhou, J.; Zhang, S.; Zhang, Z.; Yang, L.; Yin, C. Multilayer porous Pd-WO_3 composite thin films prepared by sol-gel process for hydrogen sensing. *Int. J. Hydrogen Energy* **2020**, *45*, 7223–7233.
19. Ling, C.; Xue, Q.; Han, Z.; Lu, H.; Xia, F.; Yan, Z.; Deng, L. Room temperature hydrogen sensor with ultrahigh-responsive characteristics based on Pd/SnO_2/SiO_2/Si heterojunctions. *Sens. Actuator B-Chem.* **2016**, *227*, 438–447.
20. Tonezzer, M.; Iannotta, S. H_2 sensing properties of two-dimensional zinc oxide nanostructures. *Talanta* **2014**, *122*, 201–208.