Synthesis and Characterization of Metal Oxide Nanocomposite Films and their Applications as Opto-electronic Humidity Sensors

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SUMMARY

Over the past few decades, humidity is one of the most frequently measured quantities. Recent achievements in sensor technology have resulted in today in humidity sensors for a wide range of applications ranging from nuclear power reactors to residential air-conditioners. Humidity refers to the water in gaseous form whereas moisture refers to the water in liquid form. Therefore, humidity obeys gas laws. Humid air being less dense is one of the fundamental biotic factors that decide the habitat of a particular plant or animal. Humidity measurement is required for climate monitoring and weather forecasts. The humidity is not only itself a climate variable but also interacts with other climate variables. On one hand, humidity is affected by winds and rainfall, whereas on the other hand, it affects the energy budget and so impacts on temperature. Also, water vapour is the most abundant of all greenhouse gases and acts like a double-edged sword. It absorbs red and infrared energy radiated upward by the earth's surface whereas blocks the harmful UV rays and thus makes life possible on Earth. In the field of Structural Health Monitoring, the early detection of moisture can prevent man-made structures from severe damage and would allow the proper action to be taken and hence proved cost saving. Also, monitoring of moisture level during the food processing is quite necessary. In the field of agriculture, soil moisture content sensor may be proved to be a useful tool for farmers to achieve optimum condition for the satisfactory growth of crop while saving their time and money. In the medical field also, humidity sensor plays a vital role. At high humidity if the surrounding is warmer than the body temperature, the blood brought to the skin cannot dissipate heat by conduction to the air, and results to the condition of hyperthermia. Also certain respiratory disorders and the health of a person can be predicted and detected based on the breathing air flow technique. Also, moisture sensing of the fuel is important as the variation of moisture gives rise to an uncertainty in its energy content. Also in the field of aerospace, humidity sensors have an important role since the water content of soil of any planet significantly influences the chemical and physical properties of the environment and hence the biological processes. At last but not the

least humidity measurement is important for human comfort as in air condition monitoring and for achieving hygienic conditions.

Humidity sensors are classified in mainly as four physical principles: electrical mechanical, optical and integrated sensors. Electrical sensors are limited by distance (capacitive type) and long-term stability (resistive type). Mechanical sensors are very selective. Integrated sensors contain a lot of circuit system. The humidity sensors based on optical methods are the best as they are highly sensitive, can work at remote places and are electromagnetic disturbance-free. There is, therefore, an ever increasing need to develop economical, robust, and highly sensitive opto-electronic humidity sensors.

Chapter 1 contains the introductory part of humidity sensors, their types and the role of nanoscience and nanotechnology in this field. It also focuses on the detailed description of the humidity sensing mechanism and its attribute. Fig. 1 describes some common methods of measuring humidity. Surface morphology with the large surface area and more active sites or interstitial site is an important aspect of humidity sensing. Adsorption and desorption are the two phenomena which are responsible for whole sensing mechanism. For this, semiconducting metal oxides are the best suited. However, obtaining high sensitivity, good reproducibility, negligible ageing effect with fast response and recovery time at room temperature are the major concern which is not yet reported in the literature. The modulation of optical properties of the sensing layer with humidity is expected to improve the response characteristics to a great extent, which can be easily attained using thin film technology. Metal oxide nanomaterials symbolize an increasing asset in numerous productions, particularly due to their sharp chemical, physical, and electronic properties and have versatile applications like environmental monitoring, health and personal care, energy, water treatment. Also, as the crystallite size of these materials reduces, great enhancement in their properties is observed. This diverted the research community towards the low dimensional metal oxides, while ensuring that their properties remain stable even at high operating temperature. Specifically, transition and rare earth metal oxides are of great interest because of their vacant d- and f-orbitals respectively. They can be easily oxidized as well as reduced and hence has potential application in gas/humidity sensing. Their exclusive electronic configuration has conveyed them with excellent magnetic, electrical, optical, and catalytic properties.

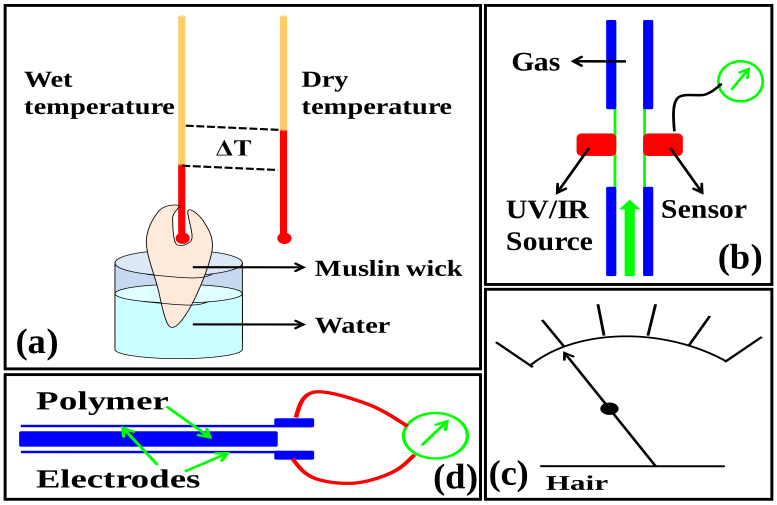

Figure 1: Some commonly used methods of measuring humidity which shows (a) Sling Psychrometer, (b) Hygroscopic Hygrometer, (c) Polymer Sensor and (d) Infrared Hygrometer.

In present investigations synthesis and characterization of nanocomposites of transition and rare earth metal oxides like Yttrium oxide-Zirconium dioxide (Y_2O_3-ZrO_2), Manganese dioxide-Cobalt oxide (MnO_2-CoO), Cerium oxide-Gadolinium oxide-Cobalt oxide (CeO_2-Gd_2O_3-CoO) and Lanthanum oxide-Chromium oxide-Strontium oxide (La_2O_3-Cr_2O_3-SrO) have been carried out. Thin films of various thicknesses and borosilicate glass substrates have been fabricated using a spin coating technique. Further, they were employed as opto-electronic humidity sensor.

Chapter 2 reports the study of modulation in light transmitted through the thin films of nanostructured yttria stabilized zirconia (YSZ) with the exposure of moisture at room temperature. For this purpose, the precursor of YSZ (Y_2O_3-ZrO_2) was prepared and used for the deposition of multilayered thin films on various substrates of borosilicate. The film was then investigated using SEM, XRD and UV-visible absorption techniques. Refractometer investigated the refractive index of the film as 1.448829. SEM showed the macroporous nature of the film and XRD revealed the minimum crystallite size as 5 nm which was further confirmed using TEM and Particle Size Analyser. Energy band-gaps of one-, two-, three- and four-layered films were estimated as 3.927, 3.919, 3.873 and 3.830 eV respectively by UV-vis spectrophotometer. These films were employed as transmission based opto-electronic humidity sensor. Maximum sensitivity was found as ~ 1.937, 1.642, 1.393 and 1.143 μW/%RH for one layered, two-layered, three-layered and four layered films. Response and recovery times were found as 28 s and 30 s respectively. Experiments were repeated from time to time and found that the sensor gave ~94% reproducible results. Thus the investigated opto-electronic sensor has excellent potential to replace its electrical counterpart. The graphical abstract for this chapter is shown in Fig.2.

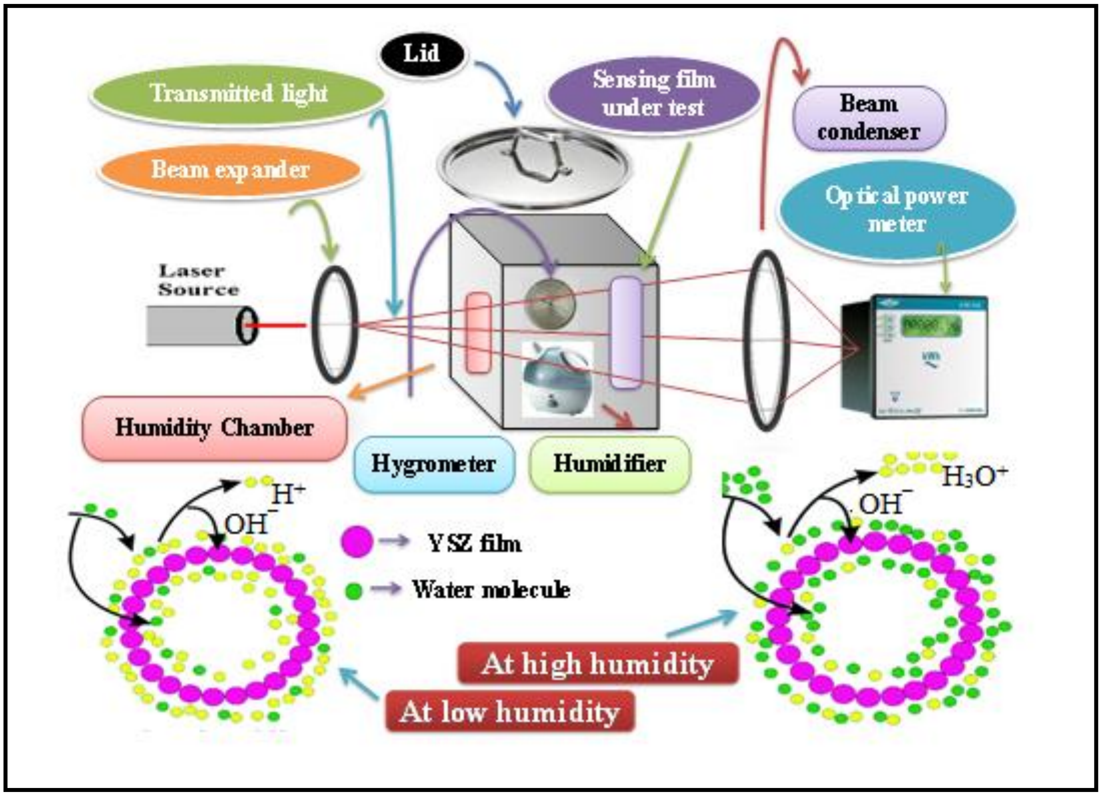

Figure 2: The graphical abstract for Chapter 2.

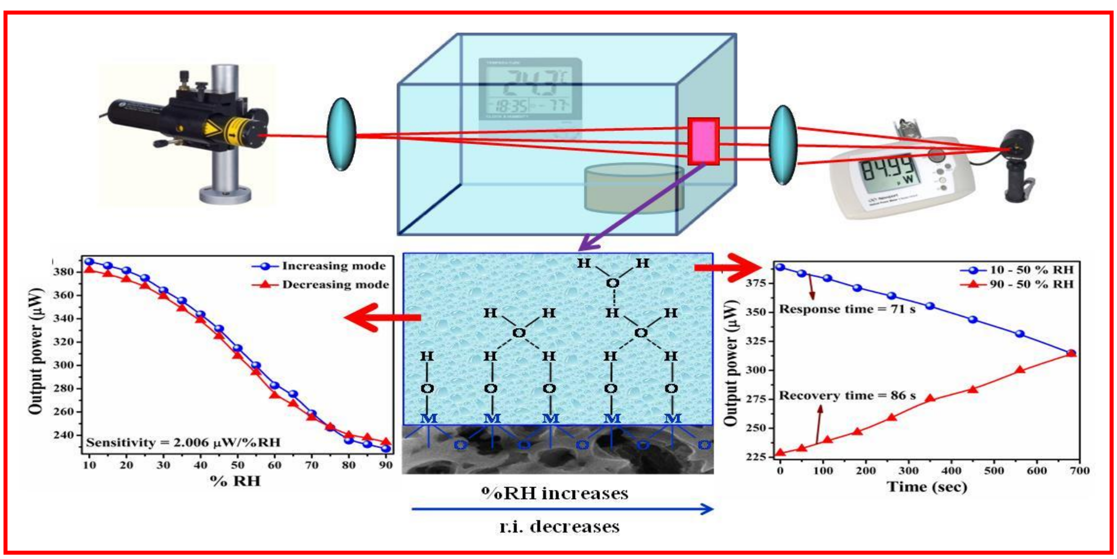

Figure 3: The graphical abstract for Chapter 3.

Chapter 3 describes the synthesis and fabrication of CeO_2-Gd_2O_3-CoO nanocomposite films using metal carboxylates as precursors. The refractive index of the material was found as 1.445538. The crystallinity was confirmed from XRD and the minimum crystallite size calculated was 9 nm. TEM analysis showed the hexagonal crystals where the minimum dimension was obtained as 6 nm. The dimensionality was further confirmed by Particle Size Analyser. The film was then investigated using SEM which revealed highly porous morphology. By UV-Vis spectrophotometer, it was found that the absorption of the film takes place in UV-region and optical band-gap was observed as 3.987 eV from the Tauc plot. The film was employed as transmission based opto-electronic humidity sensor. Maximum sensitivity was found as ~2.006 µW/%RH. Response and recovery times were found as 71 s and 86 s respectively. Experiments were repeated from time to time and the results were found ~ 90% reproducible. The graphical abstract for this chapter is shown in Fig.3.

In **Chapter 4** the synthesis of MnO_2-CoO using metal carboxylates as precursors and effect of humidity on the transmitted power through its thin film at room temperature are reported. The material was investigated through various characterization techniques. The refractive index of the material was found as 1.445930. TEM reported the minimum grain size of ~5.7 nm and SAED confirmed the crystalline nature of the material which was confirmed by XRD. The particle size was further confirmed from Particle Size Analyser. The film was then investigated using SEM which exhibited macroporous morphology. By UV-Vis spectrophotometer, it was found that the absorption of the film takes place in UV-region and optical band-gap was observed as 3.849 eV from the Tauc plot. The film was employed as transmission based opto-electronic humidity sensor. Average sensitivity was found as ~2.225 µW/%RH with the response and recovery times of 47 s and 59 s respectively. Experiments were repeated from time to time and found that reproducibility of the result is ~ 89%. Also in this chapter, an effort has been made to theoretically verify the experimental results from the computational method. The graphical abstract for this chapter is shown in Fig.4.

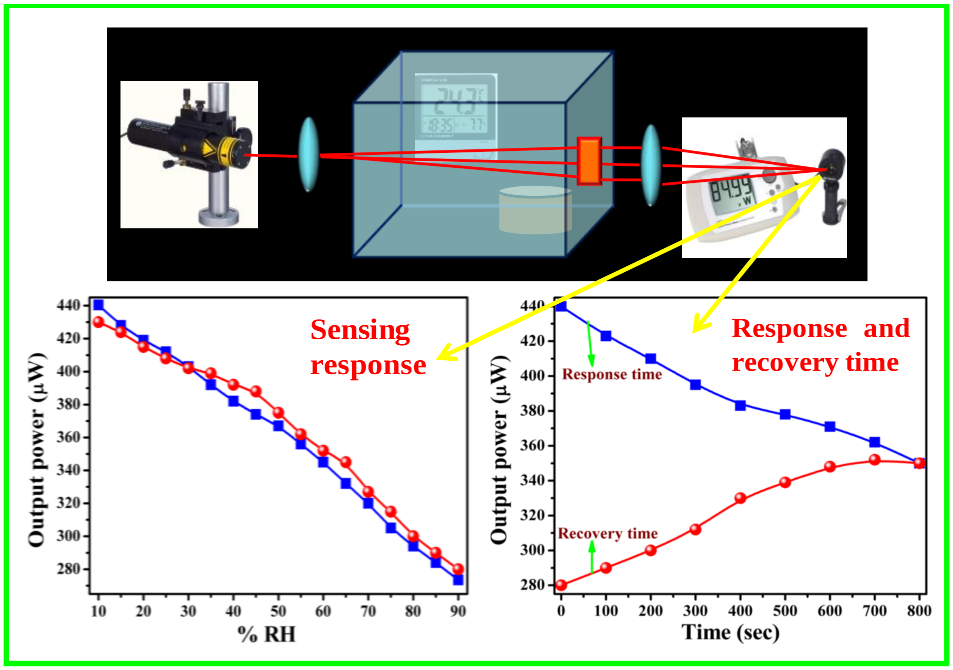

Figure 4: The graphical abstract for Chapter 4.

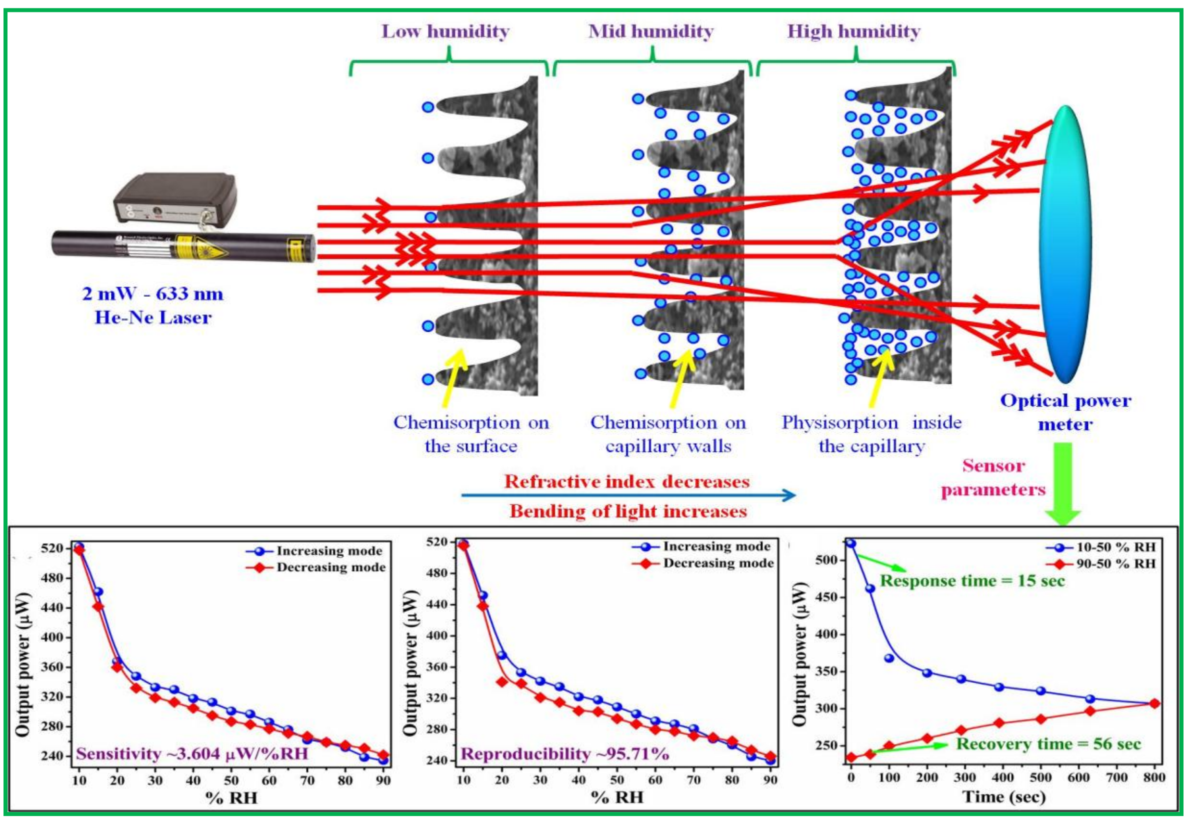

Figure 5: The graphical abstract for Chapter 5.

Chapter 5 reports the synthesis of nanocomposite La_2O_3-Cr_2O_3-SrO by pyrolysis of metal carboxylates and the effect of humidity on the transmitted power through its thin films at room temperature. The refractive index of the material was found as 1.442302. The film was investigated using SEM, XRD and UV-Vis spectrophotometer. SEM revealed the uniform mesoporous nature and XRD showed the crystalline nature of the material with a minimum crystallite size of 9 nm. TEM gave the minimum grain diameter of ~ 2.7 nm. Particle Size Analyzer reported that all the particles in the solution are in the range ~2-50 nm. From UV-Vis spectrum, it was found that the absorption through the material takes place in UV-region and the energy band-gaps of the one-, two-, three- and four-layered films were found to be 3.842, 3.837, 3.802 and 3.789 eV respectively. The dilute solution of the material was investigated through TEM and it was found that the minimum grain diameter was ~2.71 nm. The film was then employed as electrical humidity sensor from which the sensitivities were observed as 9.369, 6.006, 3.699 and 1.995 MΩ/%RH respectively for one-, two-, three- and four-layered film. Electrical sensors have certain drawbacks, therefore, the films were further employed as transmission based opto-electronic humidity sensor. In this case, the sensitivity for one-, two-, three- and four- layered films were found to be 3.604, 3.444, 3.250 and 3.063 µW/%RH respectively. Response and recovery times for one-layered sensing element were found as 15 s and 56 s whereas the reproducibility of the results was ~ 95.71%. Experiments were repeated from time to time and found that ageing effect on the sensor is very less. The investigated sensor being optical in nature has the capability of multiplexing the information with the signal. The graphical abstract for this chapter is shown in Fig.5.

Chapter 6 summarizes the entire work of the thesis with concluding remarks and future scope of bimetallic and multimetallic oxide nanocomposites based humidity sensors. A chapter wise sketch of the Thesis including the sensing materials, their optical energy bandgap, crystallite size, sensitivity, reproducibility, response and recovery times is depicted in Table 1. From this table, it can be inferred that nanostructured composite of La_2O_3-Cr_2O_3-SrO is an excellent material for humidity sensing application at room temperature and using this material a commercialized model of humidity sensor applicable for both indoor and outdoor detection may be designed.

Table 1: A chapter wise sketch of the Thesis

Chapter No.	**Sensing Material**	**Crystallite Size (nm)**	**Optical Energy Band-gap**	**Sensitivity (μW/%RH)**	**Reproducibility (%)**	**Response Time (s)**	**Recovery Time (s)**
Ch. 1	**Introduction**						
Ch. 2	Y_2O_3-ZrO_2	~5	3.927	1.937	94	28	56
Ch. 3	CeO_2-Gd_2O_3-CoO	~6	**3.987**	2.006	91	71	59
Ch. 4	MnO_2-CoO	~4	3.849	2.125	89	47	86
Ch. 5	La_2O_3-Cr_2O_3-SrO	**~2.7**	3.842	**3.604**	**96**	**15**	**30**
Ch. 6	**Conclusion**						

The scope of Further Research Work

In future, research works for productive and efficient devices, the understanding about the role of nanodimensional metal oxides in low-temperature applications would be desirable. This includes incorporation of the recovery aspects onto the surface of sensing element.

- In the present work, a nanostructured composite of La_2O_3-Cr_2O_3-SrO has been optimized to improve the sensing parameters, while there is scope for integration of modifiers like of similar radius or some nanocomposite or CNT's for reducing the response and recovery times of humidity sensor.
- An effort may also be made for the synthesis of nanostructured composites of bimetallic and multimetallic oxides using other methods and their versatile sensing applications.
- The dielectric behaviour of these optical nanocomposites is an interesting area. So attention may be focused in this area.
- The effects of higher annealing temperature on the thin films, substrate, doping, substitution, magnetic field, etc. may be investigated in future.

- Detailed analysis of the evolution of the surface reactions with respect to temperature needs to be carried out, in order to exactly understand the reaction products from the surface interaction.
- Humidity sensing carried out with metal oxide nanocomposites using transmission technique discussed in the thesis should be further investigated using Prism and U-shaped configurations for different coating lengths and coating thicknesses.
- Spectroscopic evaluation of the sensing mechanism using simulations has been important in future research.

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