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Cu_2O - TiO_2 Nanocomposite: A Novel Material for Humidity Sensing

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Abstract

Cu_2O - TiO_2 nanocomposites have been prepared through solid-state reaction route. Pellet consisting of samples of 2, 5, 10, 15, and 20 weight % of Cu_2O in TiO_2 are prepared. After annealing the samples at temperatures 200ºC–500ºC they were exposed to humidity in a chamber. Resistance of the pellets continuously decreased when relative humidity in the chamber was increased from 10% to 90%. The sample with 20% of Cu_2O in TiO_2 showed best results with sensitivity of 4.78MΩ/%RH for annealing temperature of 400ºC. This sample manifests high reproducibility, less effect of aging and lower hysteresis for annealing temperature 400ºC. The response and recovery time of this sample is found to be 76 and 296 s, respectively.

Keywords: Sensors, Annealing, Cu_2O, humidity sensor, nanocomposite, TiO_2.

Introduction

Humidity is defined as the concentration of water molecules in the atmosphere. The measurement of humidity has been proved as a critical task in comparison to other types of environmental parameters like temperature. So lot of research is going on for fabrication of an efficient and sensitive Humidity sensor. Humidity Sensors are widely used in the quality control of production processes and products in a wide range of industries, such as the production of electronic devices, precision instruments, textiles and foodstuffs, and also in many domestic applications, such as intelligent control of the living environment in buildings, where humidity sensors are used to maintain a comfortable humidity level and for cooling. In addition, research labs, clean rooms, and nuclear reactors are all environments that are highly affected by moisture levels and require constant monitoring. Research has been going on to find suitable materials that show good sensitivity over a large range of relative humidity (RH), low hysteresis, and properties that are stable. Ceramic humidity sensors[1-2] based on porous and sintered oxides have attracted much attention due to their chemical and physical stability [3-4]. Resistive or impedance type humidity sensors [5-6] have becoming more prevalent due to better quality and low cost than capacitance [7], field-effect transistor [8], and fiber-optic [9-10] sensors. Furthermore, the humidity sensors of thin film or pellet type having nanosize grains and nonporous structures have drawn much interest because of the high surface exposure for adsorption of water molecules. TiO_2 is a very versatile material having a wide-band gap. It is a useful oxide because of its typical properties, e.g., resistivity control over the range , direct band gap, transparency in the visible range, absence of toxicity, abundance in nature, high electrochemical stability[11]. TiO_2 is an important technological material used in electronic [12], electrical [13-14], optical [15–17] and catalytic devices [18-19] due to of its high electric permittivity, large birefringence, chemical stability and high photocatalytic activity. Physical properties of TiO_2 have been the subject of various investigations in terms of its suitability for various applications. TiO_2 occurs in three crystalline polymorphs: rutile, anatase and brookite; rutile being the most stable phase. Addition with impurities such as In, Cr, Cd, Fe and Co can change the magnetic and semiconducting properties of TiO_2. Many authors studied the synthesis, structure, and humidity sensitive electrical conduction of material containing TiO_2 [18-29] Yawale *et al.* have doped semiconducting materials SnO_2 and ZnO with TiO_2 screen printed them in the form of a film. DC-electrical resistance of the films has been measured in presence of humidity. They have found these materials to be good sensing materials for humidity. Rutile and hexagonal structures of TiO_2 and and their

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nanometer grain sizes have been found to be responsible for formation of nanometer sized pores, which ultimately adsorbs water. The adsorption of water (physisorbed water) on a hydroxylated surface causes electron injection [30].Steele *et al.* have fabricated capacitive humidity sensors using countersunk interdigitated electrodes coated with amorphous nanostructured TiO_2 , SiO_2 and Al_2O_3 thin films grown by glancing angle deposition. The sensor utilizing TiO_2 exhibited the largest change in capacitance, increasing exponentially from 1 nF to 1 muF for an increase in relative humidity from 2% to 92% [31].

Cuprous oxide is a natural p-type semiconductor with a direct optical band gap of energy values between 2.1-2.6 eV depending on the fabrication method and stoichiometry [32]. Cu_2O has been chosen for addition to TiO_2 because of its behavior to act as an acceptor impurity or p-type semiconductor**[33]** with TiO_2 that acts as n-type semiconductor and has a significant effect on the electrical and optical properties of TiO_2. It is a semiconductor oxide, which is known for its n-type conduction [34]because of the presence of oxygen vacancies.Cuprous Oxide (Cu_2O) was the first substance known to behave as a semiconductor. Rectifier diodes based on this material were used industrially as early as 1924 and most of the theory of semiconductors was developed using the data on Cu_2O based devices [35]. Cu_2O forms a cubic structure with a lattice parameter of 4.27 Å .It is cheap to produce, nontoxic, and environmentally friendly [36]. Its component elements are readily available in nature. Cuprous oxide is a potentially attractive material for applications in solar energy converting devices, and gas and humidity sensors [37]. Ming et al [38] first used the copper (II) oxide as humidity sensor during his studies of thick film. However, there have been a number of works on humidity sensing structures containing copper oxides in their construction such as the CuO/ZnO thin film [39-40]. These investigations indicate that oxides of copper might be useful for fabrication of humidity sensors and thus their properties need further study .Hence, present paper reports the effect of Cu_2O additive on the humidity sensing characteristics of TiO_2

Sample Preparation

A. Preparation of Cuprous Oxide

Benedict‟s reagent was prepared by dissolving 173 gm of sodium citrate and 90 gm of anhydrous sodium carbonate in 500 ml of deionised distilled water. The contents were heated slightly to dissolve. The solution was now filtered and its volume was made to 850 ml. 17.3 gm of $CuSO_4$.5H2O was dissolved in 150 ml of distilled water separately. This solution was added slowly with stirring to the above solution, the mixed solution was ready for use. Now a mixture was prepared by adding 50 ml glucose solution (0.2g/ml) to 150 ml Benedict‟s reagent and then it was boiled for 5 minutes. The proportion was set to give maximum yield of cuprous oxide particles and nearly full consumption of Benedict‟s reagent and reducing glucose so that we could find highly pure Cu_2O. After cooling the boiled mixture, brick red precipitate of cuprous oxide was obtained which settled down within few minutes. Now filtration of cooled solution gave precipitate of cuprous oxide particles. This precipitate was rinsed with deionised distilled water. The Chemical reactions involved in the process are as following:

$CuSO4 \longrightarrow Cu^{++} + SO4^{--}$ .... (i)
Cu^{++} + Sodium citrate $\longrightarrow$ cupric sodium citrate complex .... (ii)

Reducing glucose $\xrightarrow{Na_2CO_3}$ Enediols forms of reducing glucose .... (iii)
Enediols + Cupric: Sodium citrate complex $\longrightarrow$ Cu^{+} + Mixture of glucose acids .... (iv)
$Cu^{+} + OH^{-} \longrightarrow CuOH$ .... (v)

$2CuOH \xrightarrow{heat} Cu_2O + H_2O$ .... (vi)
The precipitate was dried slowly and brick red fine powder of cuprous oxide was obtained.

B. Experimental Methadology

The TiO_2-Cu_2O nanocomposite samples has been prepared through solid-state reaction route. The starting material is TiO_2 (99.58%, Qualigens). 2 weight % (sample CT-2), 5 weight % (Sample CT-5), 10 weight % (Sample CT-10), 15 weight%(Sample CT-15), and 20 weight%(Sample CT-20) of Cu_2O powder has been added to TiO_2. 10 weight % glass powders has been used as binder. The powders have been mixed uniformly and made fine by grinding in mortar with pestle for 2 h. The resultant powders have been pressed into pellet shape by uniaxially applying pressure of 260MPa in a hydraulic press machine at 27^0C (room temperature). The pellet samples prepared are in disc shape having a diameter of 8 mm and thickness 4 mm. The pressed powder pellets have been annealed in air at temperatures 200ºC–500ºC for 3 h in an electric muffle furnace.

Characterization

A. Scanning Electron Microscope Study

The study of surface morphology of the samples CT-2, CT-5, CT-10, CT-15, and CT-20 has been carried out using scanning electron microscope (SEM, Hitachi S-570). Micrographs show flakes of TiO_2 scattered throughout the whole substrate forming a network of pores and flakes. These pores

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are expected to provide sites for humidity adsorption. SEM micrographs reveal that as the temperature increases the porosity of the material increases forming clusters for Cu in TiO_2. The SEM micrographs show that the porous structure is dependent on the composition. Each composition is characterized by a typical porous structure and small crystallites without inside pores but many inter grain pores. In addition, one can observe that the intergranular pores are linked through the large pores. The pore structures should be regarded as interconnected voids that form a kind of capillary tubes. This structure favors the adsorption and condensation of water vapors. One such\ micrograph for sample CT-20 is shown in Fig. 1. The grain size calculated from SEM micrograph for sensing elements CT-2, CT-5, CT-10, CT-15, and CT-20 are shown in Table I. The grain size is minimum at 104 nm for the sensing element CT-20.

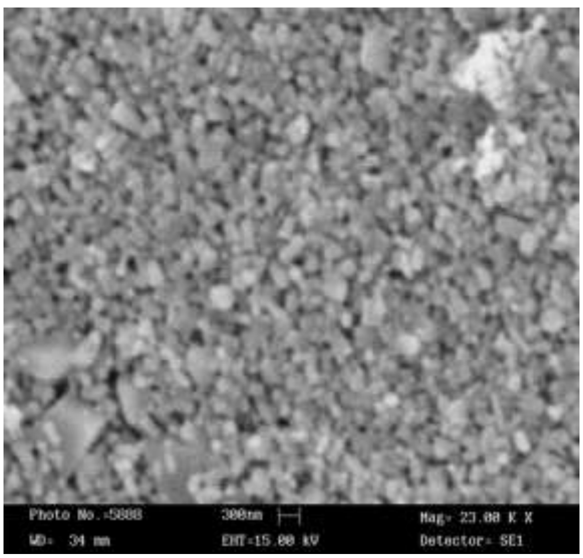

Fig .1 : Scanning Electron Micrograph of the Sample-CT-20

Table 1. Grain Size of the Nanocomposite by SEM (in nm)

CT-2	CT-5	CT-10	CT-15	20
126	133	147	163	104

X-ray diffraction has been studied using (Rigaku D/max-2500). Fig. 2 shows X-ray pattern for the sensing element CT-20. The pattern shows extent of crystallization of the sensing element in the form of powder. The average crystalline size of the samples has been calculated using Scherer's formula

$$D = K\lambda / B\cos\theta.$$

Here, D is the crystallite size, K is a fixed number of 0.9, is the X-ray wavelength, θ is the Bragg angle, and B is the full width at half maximum of the peak. The crystallite size for the sensing elements CT-2, CT-5, CT-10, CT-15, and CT-20 calculated from Scherer's formula are shown in Table II.

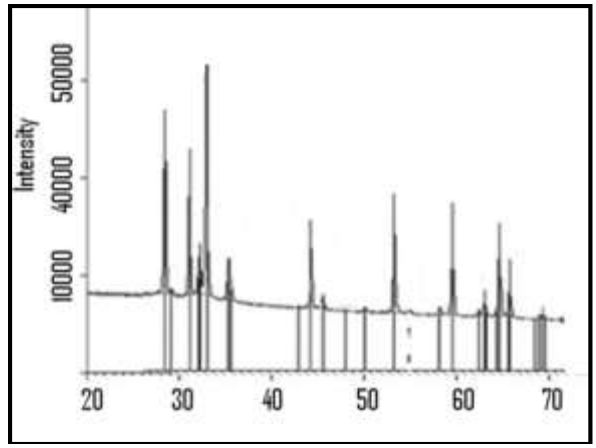

Fig 2: XRD pattern of Cu_2O-TiO_2 nanocomposite

Table II. Crystallite Size by XRD(in nm)

CT-2	CT-5	CT-10	CT-15	CT-20
95	106	102	114	119

Principle Of Operation For Humidity Sensor

In ceramic sensors, the type of conduction mechanism can be ionic or electronic. In the ionic mechanism, the impedance of the sensor decreases with an increase of RH due to physisorption and capillary condensation of water molecules on the surface of the material. In the electronic type, water molecules act as electron donating gas and their chemisorption increases or decreases the electronic conductivity depending on whether the material is nor p-type semiconductor. Electronic conduction depends mainly on the semiconducting nature of ceramic materials and less sensitive to their porous characteristics.[41]In the present scheme of things, since TiO_2 is n-type semiconductor, sensitivity to humidity is a result of electronic conduction.

As semiconducting dry oxide of TiO_2 nanocomposite are brought in contact with humid air, water molecules chemisorb on the available sites of the oxide surface. The adsorption of water molecules on the surface takes place via a dissociative chemisorption process which may be described in a two-step process as given below.

i) Water molecules adsorbed on grain surface react with the lattice A(Ti) as

$$H_2O + O_o + A \rightarrow 2OH - A + V_o + 2e^-$$

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Where O_0 represents the lattice oxygen and is V_0 the vacancy created at the oxygen site according to the reaction

$$O_O \rightarrow O^{2-} + V_O$$

ii) Doubly ionized oxygen, displaced from the lattice, reacts with the H^+ coming from the dissociation of water molecules to form a hydroxyl group as given below

$$H^+ + O^{2-} \rightarrow OH^-$$

TiO_2 has electron vacancies. Hence, because of this reaction, the electrons are accumulated at the TiO_2 surface and, consequently, the resistance of the sensing element decreases with increase in relative humidity. Copper oxide has been chosen for addition to TiO_2 because of the behavior of this as an acceptor impurity in n-type TiO_2 and has a significant effect on the electrical and optical properties of TiO_2 .

Experimental

After annealing, samples have been exposed to humidity in a specially designed humidity control chamber.Fig.3 shows schematic diagram of humidity sensing apparatus. Inside the humidity chamber, a thermometer C and standard hygrometer are placed for the purpose of calibration. Variation in resistance has been recorded with change in relative humidity. Relative humidity has been measured using the standard hygrometer. Variation in resistance of the pellets has been recorded using a resistance meter. Copper electrode has been used to measure the resistance of the pellet. The resistance of the pellet has been measured normal to the cylindrical surface of the pellet. The electrical resistance at different relative humidity levels of the sensing elements in the form of pellets has been determined by a two-probe method,**[42]** as the present work is to measure the changes in surface conductivity as a function of applied field. The electrical contacts have been made on the surface of pellet by means of two thin copper sheets. Given the high resistivity of the materials under consideration, the potential inaccuracy due to contact resistance is assumed negligible. After studying humidity sensing properties, sensing elements have been kept in laboratory environment and their humidity sensing characteristics regularly monitored. To see the effect of aging, the sensing properties of these elements have been examined again in the humidity control chamber after six months. The stability of the sensing element has been checked by keeping the sensing element at fixed values of % RH in the chamber and the values of resistance recorded as a function of time. The values have been found to be stable within 3% of the measured values.

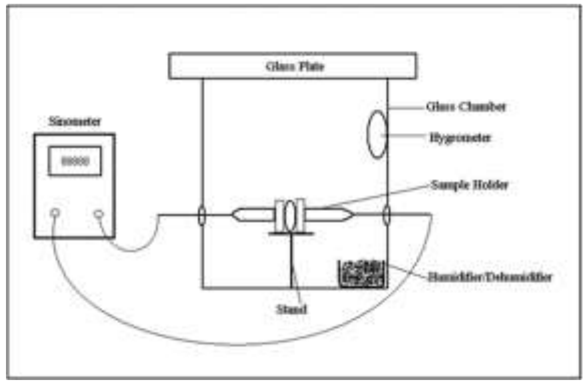

Fig 3: Schematic Diagram of Humidity Sensing Apparatus

Results and Discussion

A. Humidification Graphs

Variation in resistance with the change in relative humidity has been recorded for the sensing elements of Cu_2O- TiO_2 nanocomposite for annealing temperatures 200ºC–500ºC. All the results have been found to be repeatable. Variation in resistance with the change in relative humidity for all the sensing elements CT-2, CT-5, CT-10, CT-15, and CT-20 for annealing temperature 400ºC has been shown in Fig. 4. There is continuous decrease in the value of resistance with increase in the % RH for samples CT-2, CT-5, CT-10, CT-15, and CT-20 for all the annealing temperatures. Sensing elements CT-15 and CT-20 are showing higher values of resistance and sharp decline in the values of resistance as compared to sensing element CT-2, CT-5, and CT-10 up to relative humidity 40% RH. After 40% RH, the sensing elements CT-15 and CT-20 also show the similar trend. It is understood that the increase in the conductivity of the sample with relative humidity in the lower range (40% RH) is due to the adsorption of the water molecules on the pellet surface within capillary nanopores. Higher porosity increases surface to volume ratio of the materials and enhances diffusion rate of water into or out-of the porous structure; and thus, helps in getting good sensitivity. At high relative humidity (40% RH), liquid water condenses in the capillary like pores, forming a liquid like layer.

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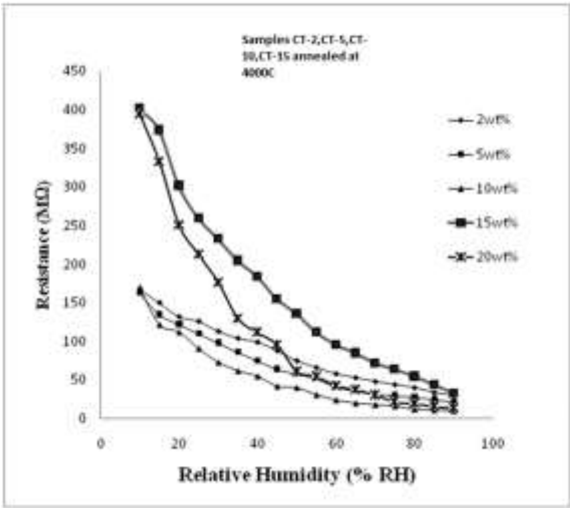

Fig 4: Variation of resistance with change in % RH for samples CT-2,CT-5,CT-10,CT-15,CT-20 for annealing temperature of 400 C

B.Humidification and Desiccation Graph: Hysteresis

An important factor related to sensing elements of metal oxide materials is the hysteresis effect. The phenomenon of hysteresis may be attributed to the initial chemisorptions on the surface of the sensing elements. This chemisorbed layer, once formed is not further affected by exposure to or removal of humidity, it can be thermally desorbed only. Hence, in the decreasing cycle of % RH, the initially adsorbed water is not removed fully leading to hysteresis. To determine the hysteresis effect in the sensing elements annealed at 200ºC–500ºC, the humidity in the chamber has been increased from 10%–90% RH and then cycled down to 10% RH. It has been observed that all sensing elements have an acceptable hysteresis value; sample CT-20 has the lowest hysteresis value with 2.30% for annealing temperature of 400ºC and 3.78% for annealing temperature of 500ºC in the value of sensitivity. The data sheets of some commercially available humidity sensors have indicated hysteresis values in the range from 1.20%–5.00% [43-44]. Fig. 5 shows the hysteresis graph for the sensing element CT-20. This figure shows the graphs both for the increasing and decreasing cycles of relative humidity.

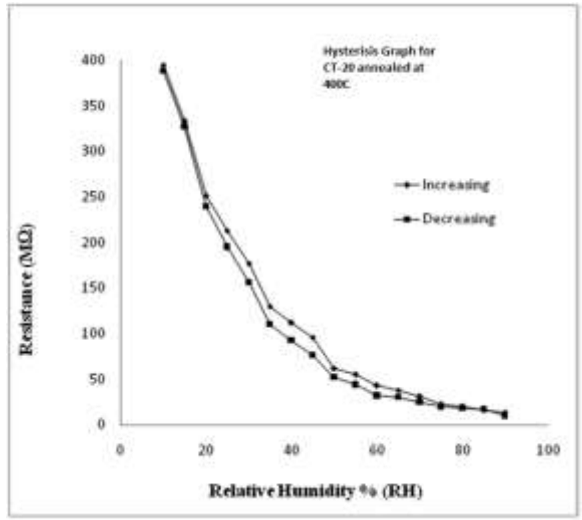

Fig 5: Variation of resistance with change in % RH for samples CT-20 for annealing temperature 400 C: a. increasing cycle and b. decreasing cycle

A. *Problem of Aging in Humidity Sensors*

Metal oxide sensors suffer from the problem of aging. Aging mechanisms in humidity sensors may be due either to prolonged exposure of surface to high humidity, adsorption of contaminants preferentially on the cation sites, loss of surface cations due to vaporization, solubility and diffusion, or annealing to a less reactive structure, migration of cations away from the surface due to thermal diffusion. Generally, the more sensitive a material is to humidity, the more it tends to be susceptible to aging. This is a significant problem in sensing devices [45]. To appreciate the effect of aging and reproducibility, the sensing elements have again been then exposed to humidity after six months. The results have been found to be generally reproducible over different cycles of operation. Fig. 6 shows the repeatability graph for the sensing element CT-20 annealed at 400ºC. In this figure, both an initial increasing cycle and another increasing cycle after six months have been shown together.

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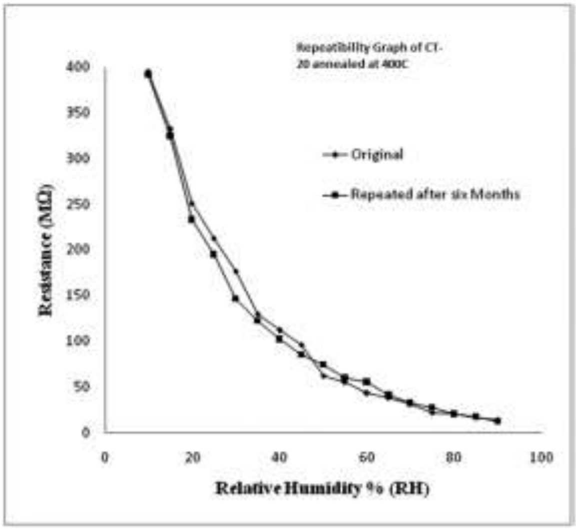

Fig 6: Variation of resistance with change in % RH for CT-20 for annealing temperature 400 C after six months: a. increasing cycle and b. another increasing

A. Sensitivity

Sensitivity of the humidity sensor is defined as the change in resistance of the sensing element per unit change in relative humidity (RH %)

$$S = \Delta R/(\Delta\%RH).$$

For calculation of sensitivity, the humidity from 10% to 90% RH has been divided in equal intervals of 5% RH each. Difference in the value of the resistance for each of this interval has been calculated and then divided by 5. The average has been taken for all these calculated values. Sensitivity values for all the sensing elements for all annealing temperatures have been calculated. All the sensing elements CT-2, CT-5, CT-10, CT-15, and CT-20 generally show increase in sensitivity with increase in percentage of Cu_2O in TiO_2. When annealing temperature is increased from 200ºC to 400ºC, for all doping percentage, the sensitivity increases, showing maximum sensitivity for annealing temperature of 400ºC; the sensitivity then decreases for annealing temperature of 500ºC. The sample CT-20 shows results with maximum sensitivity of 4.78MΩ/%RH among all samples when annealed at 400ºC and 4.23MΩ/%RH when annealed at 500ºC as compared to the sensitivity values of TiO2-Sb2O5 or TiO2-SnO2 nanostructure Humidity sensors [46-47]. The sensitivity is highest among samples because grain size of this sample is minimum which increases surface to volume ratio which, in turn, provides more sites for water vapor to be adsorbed on the surface of the sensing element.

These results have been depicted in Fig. 7. With increase in the annealing temperature the grain size generally decreases. This leads to increase in surface to volume ratio. Thus, more of the surface area is exposed for adsorption, leading to higher sensitivity. For our experiment, this seems to be happening up to annealing temperature of 400ºC. Beyond this the sensitivity decreases at 500ºC.The plausible explanation for this could be in terms of the clustering tendency of Cu in the composite. Perhaps the clustering tendency of Cu in the sample increases at higher annealing temperature leading to increase in the grain size which, in turn, decreases surface to volume ratio leading to decrease in sensitivity. For sensing element CT-20 annealed at 400ºC, the value of sensitivity is found to be repeatable within 2.3% in decreasing cycle of relative humidity and within 0.6% over increasing cycle of relative humidity after six months. For this sensing element CT-20 annealed at 500ºC, value of sensitivity is found to be repeatable within 3.78% in decreasing cycle of relative humidity and 7.92% in increasing cycle of relative humidity after six months. Over repeatable cycles of operation, sensing element CT-20 annealed at 400ºC is showing more faithful results in terms of higher sensitivity, lower hysteresis, and higher reproducibility as compared to the annealing temperature of 500ºC.

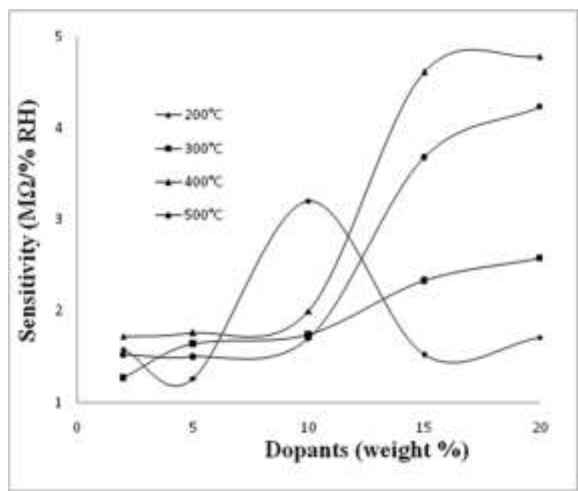

Fig. 7. Variation of sensitivity with weight % of Cu_2O in TiO_2.

A. Response and Recovery Time

For calculation of response time for all the sensing elements annealed from 200ºC to 500ºC, the humidity in the chamber has been switched over from 10% RH (low humidity) to 90% RH (high humidity) and for calculation of recovery time the humidity in the chamber has been brought down from 90% RH (high humidity) to 10% RH (low humidity). The response and recovery time of sensing element CT-20

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is found to be 76 and 296 s, respectively. Adsorption and desorption of the water molecules take place at different energy levels. Adsorption is an exothermic process, where as desorption needs external energy for water molecules to depart from the metal oxide surface. Since desorption is an endothermic process, it takes a longer time to desorb the water vapor; therefore, the recovery time is always greater than the response time [48-50]

Conclusion

Sensing element CT-20 annealed at 400ºC proves to be the best sensing element with sensitivity of 4.78 .Sensing element CT-20 manifests lower hysteresis, less effect of aging and higher reproducibility for annealing temperature 400 C. As calculated from Scherer's formula
the crystallite size for this sensing elements is 119 nm and according to SEM micrograph grain size is 104 nm.

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