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The Performance of V_2O_5:Ag Nanoparticles as Thin Film and Bulk Pellet Sensor for NO_2 and NH_3 Detection

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Abstract

A comparison of gas sensing performance of V_2O_5:Ag nanoparticles as thin film and as bulk pellet toward NO_2 and NH_3 is presented. V_2O_5:Ag nanoparticles thin films were deposited by vacuum thermal evaporation method on glass substrates while the pellets were prepared by powder technology. XRD patterns of thin film and pellet were polycrystalline with an orthorhombic structure. The value of average grain size is about 60 nm. The morphological properties of the samples have been distinguished by atomic force microscopy (AFM) and field effect scanning electron microscopy (FESEM) which indicated that the films showed homogeneous surfaces morphology and contained pores between the grains. Sensing results showed a various response to NO_2 and NH_3 gases. It was found that the sensitivity of thin films sensor is superior to that of the pellets sensor.

Keywords: Ag nanoparticles doped V_2O_5 nanoparticles; Thin Films; Pellets; NO_2 and NH_3 Gas Sensor; XRD; AFM; FESEM.

الأداء الكشفي لمادة V_2O_5:Ag النانويه كغشاء رقيق و عينه كتليه للغازات NO_2 و NH_3

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الخلاصه:

تم اجراء مقارنه بين اداء غشاء رقيق و عينه كتليه على شكل قرص من مادة V_2O_5:Ag النانوية لتحسسية الغازات NO_2 و NH_3 . رسبت الأغشيه الرقيقه على ارضيات زجاجيه بطريقة التبخير الحراري في الفراغ بينما حضرت الأقراص بواسطة تكنلوجيا المسحوق. أظهرت انماط حيود الأشعه السينيه بأن الأغشيه الرقيقه و الأقراص متعددة التبلور و ذات ترتيب هندسي ثلاثي الأبعاد و معدل حجم الحبيبات حوالي 60 nm . درست الخصائص الشكليه للعينات بواسطة مجهر القوه الذريه و المجهر الألكتروني لمسح الأنبعاث الميداني بأن الأغشيه ذات سطوح متجانسه و تحتوي على مسامات بين الحبيبات. أظهرت نتائج التحسسية استجابات مختلفه لكل من غاز NO_2 و NH_3 و كذلك يوجد تباين بالأستجابات للغشاء الرقيق و القرص. لوحظ أن تحسسية الأغشيه الرقيقه أفضل من الأقراص.

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1. Introduction

Nowadays, an increased demand for gas analyzers, required for technological processes control, as well as for gas sensors for the detection of industrial emission of toxic and hazardous substances is observed due to growth of industrial production. Improvement of many of these processes, their fail-safety and environmental safety is determined by reliability and response speed of automatic analytical instruments based on gas sensors, both standalone as well as part of control systems [1]. A gas sensor is an instrument able to detect the existence of several gases in an area and exhibits information concerning the gas concentration by means of electrical or optical signals. Generally, gas sensors are utilized to detect poisonous gases (e.g., H_2S, SO_2, CO, NO_x, etc.) and flammable gases (e.g., CH_4, H_2 and C_2H_5OH, etc.) [2]. NH_3, as one of the model air contaminants, is a type of colourless, volatile, nuisance gas which could irritate human skin, eyes, and respiratory tract even at a concentration lower than 50 ppm [3]. Gas sensors are used for monitoring of toxic gases because of their low-cost, small size and easy operation. These days, several gas sensors based on diverse philosophies, like electrochemistry, surface acoustic wave, quartz crystal microbalance, and cataluminescence (CTL), have been prepared for the sensing of various analyses [4]. Nitrogen oxide NO_x (NO or NO_2) is mostly produced during combustion of fuels. Newly, metal oxide semiconductor NO_2 gas sensors have been discussed widely for their easy manufacture procedures and low cost. Particularly, Vanadium pentoxide (V_2O_5) is one of the perfect materials for NO_2 sensing because of its high sensitivity and perfect selectivity to low concentrations of NO_2 gas [5]. Vanadium oxide (V_2O_5) has obtained a lot of research attention due to its excellent structural flexibility, lower band gap and high energy density. These properties allow V_2O_5 to be used for many applications concerning gas sensing. Nano-sized V_2O_5 with various structures have been manufactured for the electrochemical sensing of ethanol, ammonia and xylene [4]. From a chemical point of view, V_2O_5 is a very good catalyst due to its wealthy and varied chemical configuration. This constitutional variety permits the presence of a broad diversity of various coordination with oxygen ions that supply a significant control on the physical and chemical surface characteristics [6]. Electrochemical researches have revealed that the efficiency of vanadium oxide film is immediately correlated to its crystallinity, morphology, disorder degree, stoichiometry, and additional factors correlated to the techniques and circumstances of preparation [7]. Matters at the nano scale frequently display higher diverse physical, chemical, and biological characteristics than their ordinary sized counterparts [8]. Nano-materials have individual physical characteristics leading to further and further interest of their use as a cathode in rechargeable ion batteries and selective gas sensors like ammonia due to their rise surface area and redox action [9]. New progress in nano materials offer the chance to significantly improve the response of these materials, as their performance depends on exposed surface volume. The new obtainment of several metal oxide materials of high surface-area nano powder, as well as application of recently advanced nanofabrication technologies, provide enormous chances for sensor manufactures [8]. Doping is an active process to utilize implementations of semiconductors. Doped metal oxides show diverse kinds of morphologies producing several characteristics, and thus gaining further applications [10]. Silver has long been known as an efficient catalytic element. Zhang et al. presented that the addition of silver to V_2O_5 importantly reduced the surface acidity and raised the redox capability of the catalyst, causing to enhance transformation of toluene. Although, the catalytic characteristics of Ag nanoparticles are immediately relevant to their size and dispersion, the uniformly dispersed Ag nanoparticles with tiny size are perfect for elevated catalytic efficiency because of their large surface area [11, 12]. Haitao et al. reported that silver vanadium oxide can be used as a gas sensing material for detecting amines [13].

Schneider and Maziarz demonstrated that V_2O_5 thin films show a sensitivity towards NO_2 [14]. The humidity sensing properties of V_2O_5 thin films synthesized by rf sputtering method were investigated by Hassan et al. [15].

The purpose of this research is to offer a promising material for monitoring NO_2 and NH_3 in ambient conditions.

2. Experimental

V_2O_5 and Ag nanoparticles powder were obtained from (Nanoshel, India, 99.9 % pure) and (Hongwu International Group Ltd, China, 99.9% pure), respectively. 0.35% weight of Ag was mixed with V_2O_5 and grinded till uniformity was achieved. Then the powder was sintered at 600°C for 3h and used as material sources for the prepared sensors. Thin films were deposited onto glasses substrates which were cleaned with washing liquid, ultrasonic spirits bath followed by distilled water then dried with warm air to achieve the vaporization of dissolvents and water [2]. The deposition was achieved by thermal evaporation method in high vacuum using coating system (Thermionics Laboratory, Inc. HAYWARD, CA. USA) at a pressure of 10^{-5} Torr. The substrates were placed in a revolved substrate holder to guarantee the best uniformity in the film thickness through the deposition. A glow-discharge was carried out to confirm the cleanness of the substrates previous to the deposition. Some of the films were annealed at different temperatures (100, 150, 200, 250 and 500°C). Bulk sensors were prepared, by compressing V_2O_5:Ag powder under 260 MPa pressure at room temperature, into pellets of 9mm diameter and 2mm thickness. Al grid was deposited through a mask on the front surface of the prepared thin films and pellets which acted as the sensing elements of the gas sensors. In addition, two contacts of fine copper wire and silver paste were attached to the sensing elements.

The structure of the thin films was analyzed by X-Ray diffractometer (Shimadza XRD-600, Japan) utilizing CuK_α irradiation. The accelerating voltage and the used current were 45 kV and 40 mA, respectively. The surface morphological studies for V_2O_5:Ag films were carried out by atomic force microscopy (AFM) (SPM-AA 300, Angstrom Advanced Inc., USA) and field effect scanning electron microscope (FESEM) (FESEM TESCAN MIRA3 FRENCH) with an accelerating voltage 15 kV.

The properties of the prepared sensors, to the oxidizing gas NO_2 and the reducing gas NH_3, were studied using a homemade test chamber of variable temperature. The temperature was adjusted with a heater placed inside the chamber, also a thermocouple (k-type) was installed to measure and control the temperature. Gas detecting properties of the sensors were tested in the temperature scope of 150-300°C. A Pirani gauge with a rotary pump was utilized to regulate the flux of the tested gas in the test chamber. The tested gas of known concentration (ppm, part per million) was introduced into the chamber by calibrated leaks through needle valves. The resistance of the sensing elements (thin film and pellet) was calculated by gauging the current across the elements at a fixed voltage of 1V using a multimeter (Victor 86 B). The sensor response was computed utilizing the relation:

$$S=R_g-R_a /R \quad (1)$$

where R_g and R_a are the electric resistance of the sensing elements in the presence of target gas and in atmospheric air, respectively.

3. Results

Figure1 demonstrates X-ray diffraction(XRD) pattern of V_2O_5: Ag thin film. The pattern displays peaks at 2Θ values of 17.8, 28.5, 32.1 and 55 corresponding to (200), (203), (312) and (104) planes, respectively. Figure 2 shows X-diffraction pattern of V_2O_5: Ag pellet. The pattern peaks at 2Θ values of 21.4, 27.8, 29.5, 32, 34.2, 41, 46.2 and 50.5 correspond to (101), (110), (301), (011), (310), (002), (411) and (112) planes, respectively. X-ray analysis confirmed that all peaks correspond to the V_2O_5 orthorhombic phase. The results are well agreed with those obtained by Zhang et al. [4], Fu et al.]13] and Schneider [16]. The diffraction peaks of the samples manifest that the samples were well crystallized and correspond to the JCPDS Card numbering 41-1426. It is noted that the peaks of the XRD pattern of the thin film are of higher intensity than those of the pellet XRD pattern. The crystallite size of the V_2O_5: Ag nanoparticles, was computed by the Scherrer formula:

$$D=k\lambda/\beta_{hkl}\cos\Theta \quad (2)$$

Where: D is the crystallite size, λ is the wavelength of X-ray radiation (1.5406 Å for Cu k_α), k is the shape factor which is constant equal to (0.9), β_{hkl} is the full width at half maximum (FWHM) at 2Θ of 17.85 and Θ is the Bragg angle in radian.

The average crystallite size was found to be 60 nm. The calculated value is in very good approximation to those reported in the literature [17, 18].

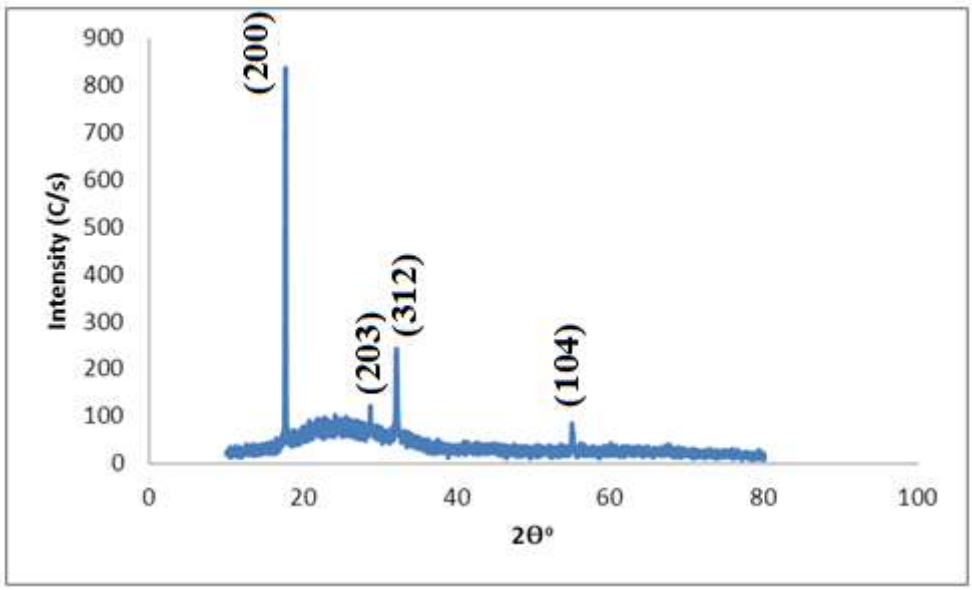

Figure 1: X-ray diffraction pattern of V_2O_5:Ag thin film

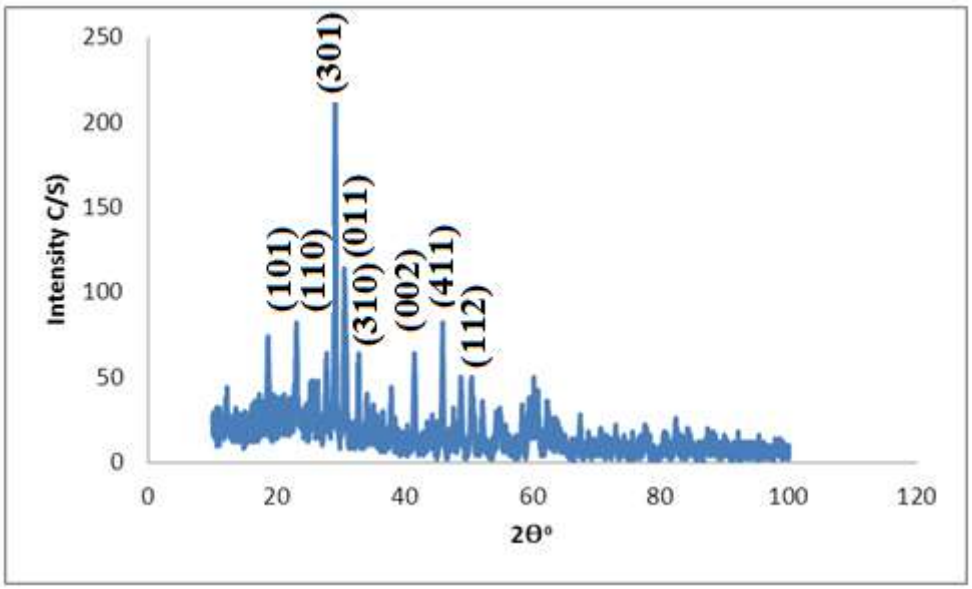

Figure 2: X-ray diffraction pattern of V_2O_5:Ag pellet

Surface topography and roughness were assessed based on AFM measurements. The AFM images show 2D topographies of as-prepared and annealed at (100, 150, 200, 250 and 500 °C)V_2O_5:Ag NPs thin films, as presented in Figure 3(a, b, c, d, e and f), respectively. The annealing causes an essential variation in the physical properties of V_2O_5 thin films that led to improve its performance as a gas sensor [15]. The images showed a polycrystalline surface morphology and columnar microstructure. It can be noticed from the images of the as-prepared samples, that the tiny particles are ordered more compactly. There are much larger particles shaped in the annealed films, in comparison to the as-deposited samples. Figure 3f presents a uniform pattern of pore walls and pore openings in the film, accompanied by clear determined grain borders of film annealed at 500 °C. Furthermore, the rise distribution of grains was symmetrical and therefore showed perfect homogeneity of the surface. Statistical study of the figures was done to gain the root-mean-square surface roughness RMS, lateral grain size of the films and average roughness. The mobility of molecules and ions in the film increases with enhanceing of the annealing temperature, this perhaps motivate crystallization and thus the surface morphology of the film differs [19]. Table 1 shows the values of average roughness, RMS, grain size and grain maximum height.

Table 1: Roughness average, root mean square, grain size, and grain maximum height of V_2O_5:Ag thin films as prepared and annealed at 100, 150, 200, 250, and 500 °C

Temperature °C	Roughness Average nm	Root Mean Square nm	Grain Size nm	Grain Maximum Height nm
25	2	2.52	60.20	12.6
100	2.85	3.6	68.51	21.6
150	4.14	4.87	70.82	18.9
200	3.52	4.11	78.41	15
250	3.69	4.35	82.67	17.3
500	4.22	4.95	95.93	18.9

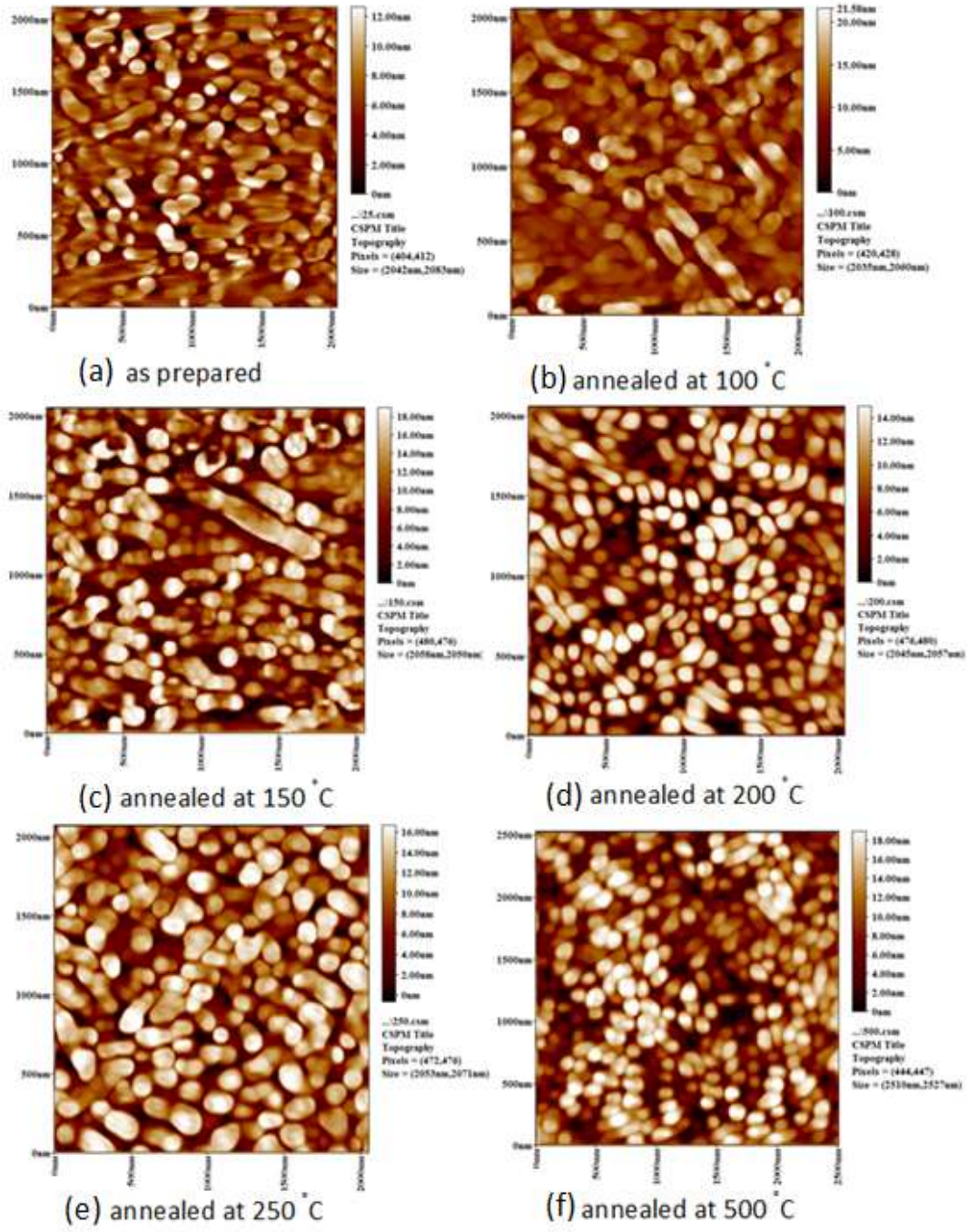

Figure 3: 2D AFM image of V_2O_5:Ag thin film: (a) as prepared, and of the annealed at (b) 100, (c) 150, (d) 200, (e) 250 and (f) 500 °C

The FESEM image of thin film revealed homogeneous particles and quasi-spherical agglomerates. The film contained pores among the grains of the film, which is of importance for the diffusion of the molecules of the tested gas [20, 21]. These pores supply locations for the molecules of the adsorbed gas. Porosity rises surface to volume ratio of matter and for that reason assists in getting good sensitivity [10].

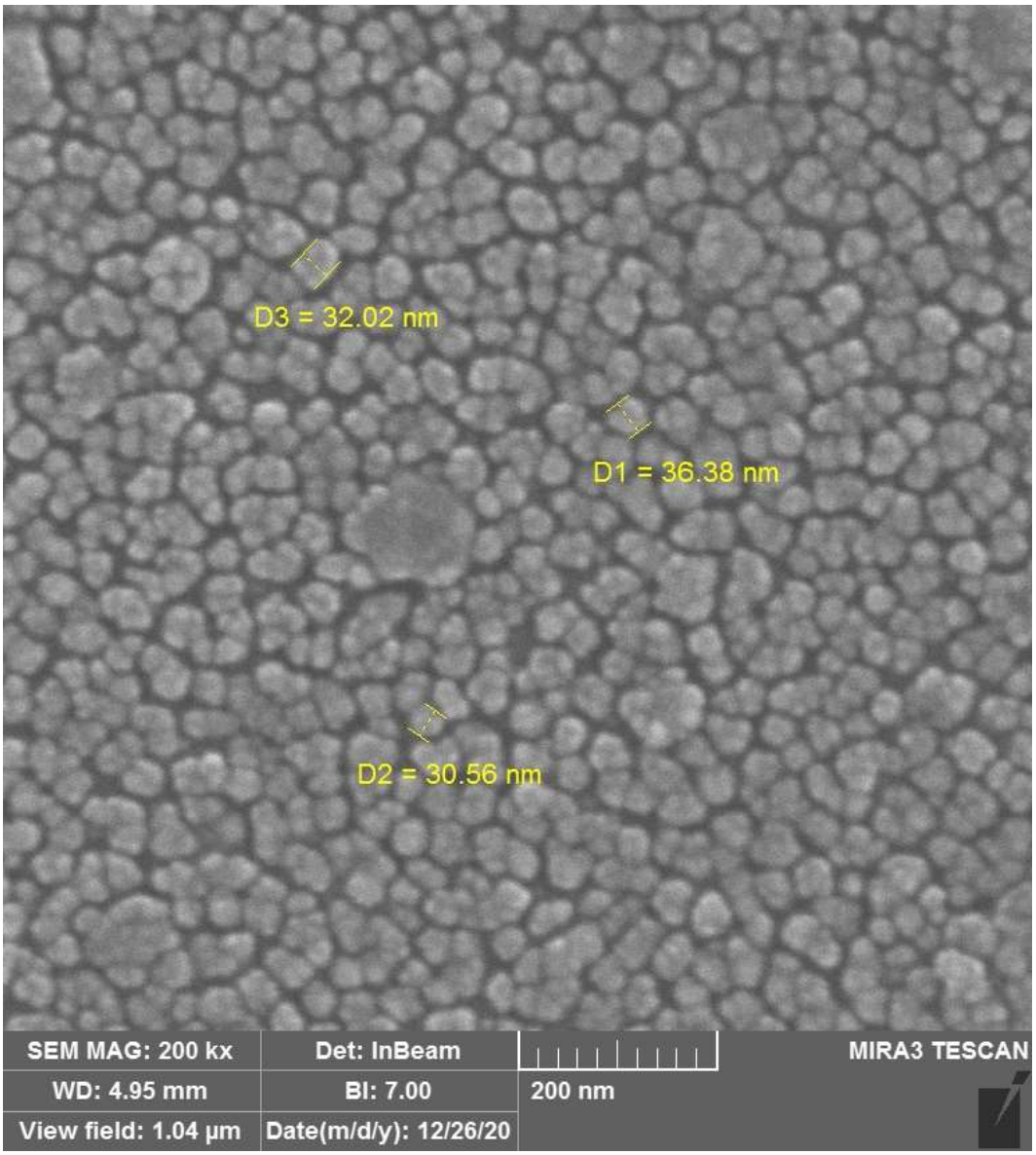

Figure 4: FESEM image of V_2O_5:Ag thin film.

Sensing response properties of the V_2O_5: Ag nanoparticles thin film and bulk sensors to test NO_2 and NH_3 gases were studied. The gas sensing procedure is generally relevant to the surface adsorption and surface interaction of gas molecules on the metal oxide nanoparticle. The operating temperature acts a crucial part in gas detection and has a chief effect to determine the kind of chemisorbed oxygen species [4].

Figure 5 exhibits the sensitivity of thin film and bulk sensors to NO_2 gas at various operating temperatures. The sensitivity of the thin film sensor decreased at temperature higher than 150 ^oC and then increased when the temperature is above 200 ^oC; it showed the highest sensitivity at 265 ^oC and then reduced with further increasing of temperature. Preferably, sensors are expected to reveal elevated sensitivity to some gases and depressed or no sensitivity to others in the same environment. In the V_2O_5 sensor, variation in the oxygen equilibrium of the oxide layer leads to an alteration in its conductance. In the situation of an oxidizing gas (NO_2), interactions immediately occur on the oxide surface. Through the interaction procedure, molecules consume conduction electrons and then enhance the depletion region at the surface and increase the resistivity of the sensor. While temperature

rises, the surface of the sensor will be more effective and causes increase of sensitivity [22]. The noticed increasing and decreasing of sensitivity represents the adsorption and desorption phenomenon of the gases. The dropping of sensitivity for temperatures above the operating temperature, can be attributed to the higher desorption rates at these temperatures. On the other hand, the bulk sensor demonstrated a very low sensitivity. The values of R_a and R_g decide the maximum sensor response [23].

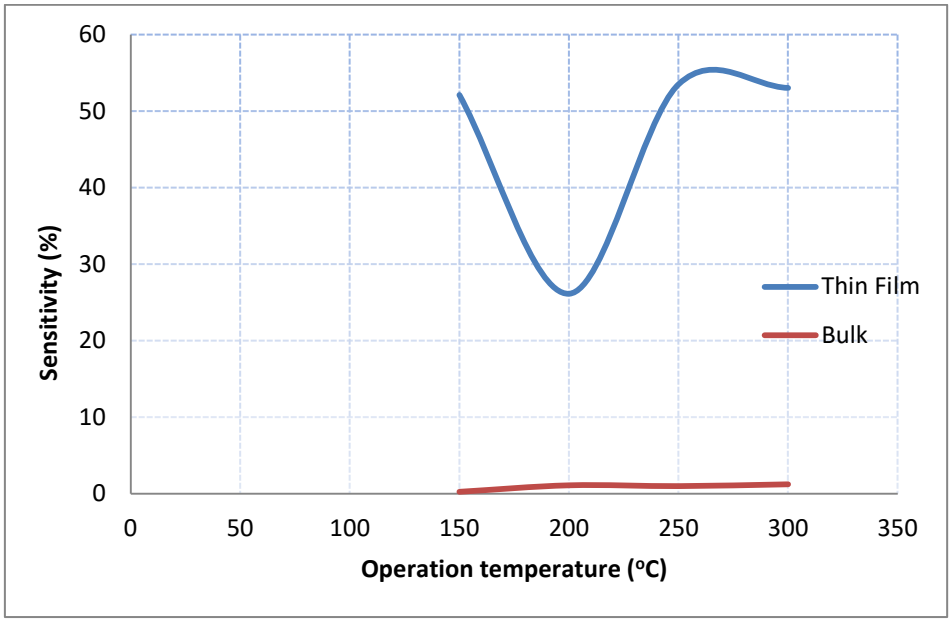

Figure 5: Sensitivity of V_2O_5: Ag thin film and bulk as a function of operating temperature tested with NO_2 gas

Figure 6 displays the sensitivity of thin film and bulk sensors to NH_3 gas at various operating temperatures. sensitivity of the thin film sensor was low at temperatures less than 180 ^oC at which it started to increase sharply reaching maximum sensitivity at 260 ^oC then reduced with further increase in temperature. While in the case of the bulk sensor, the sensitivity increases above 150 ^oC reaching a maximum at 200 ^oC and decrease at temperatures above 200 ^oC. It was observed that the sensitivity sensitivity can be explained as an oxygen adsorption reaction on the sensing element surface, which is highly influenced by the operating temperature [21]. Sensitivity is controlled by two factors : the speed of chemical reaction on the surface of the grains and the speed of the diffusion of gas molecules. At low temperatures the sensitivity is limited by the speed of chemical reactions [24,25]. At high temperatures, the sensitivity is limited by the speed of the diffusion of gas molecules. At moderate temperatures the speed of both processes is the same and the sensitivity attains its maximum value [26]. At the moment that critical temperature is attained, desorption of the adsorption species of reductive gas (NH_3), moreover the re-adsorption of the adsorption species in the air, happens very quickly, and the gas for sensing interacts at the surface of the electrode. This prevents the sensor gas from infiltrating the sensor electrode, thus leading to a dropping of the sensitivity of the sensor.

The response of thin film sensor to test gases was higher than that of bulk sensor , owing to the active surface area of the nanostructure, which can offer more active space for the interaction between V_2O_5: Ag and the tested gases.

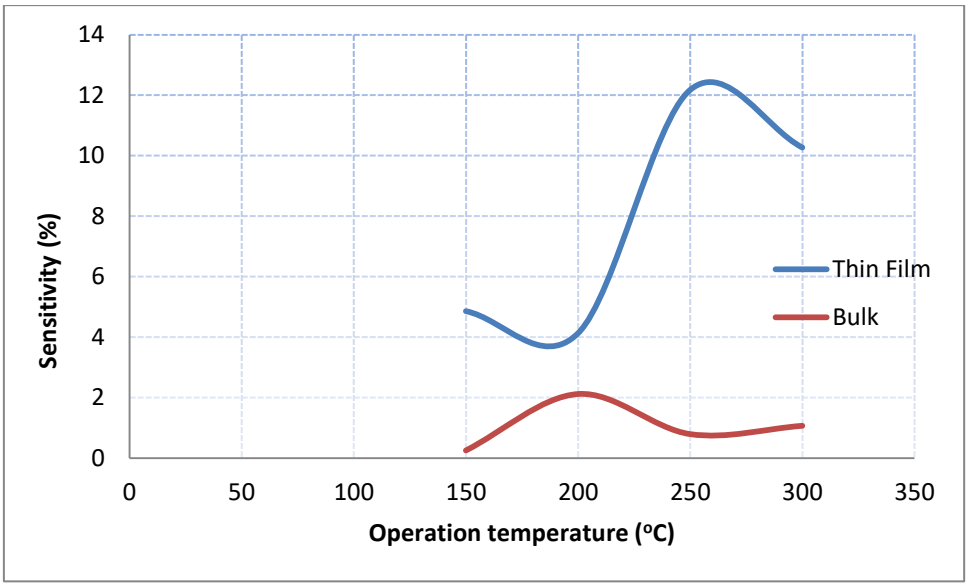

Figure 6: Sensitivity of V_2O_5: Ag thin film and bulk sensors as a function of operating temperature with NH_3 tested gas.

Figure 7 illustrates the change of response time and recovery time with the operating temperature of the V_2O_5 thin film using NO_2 as the tested gas.

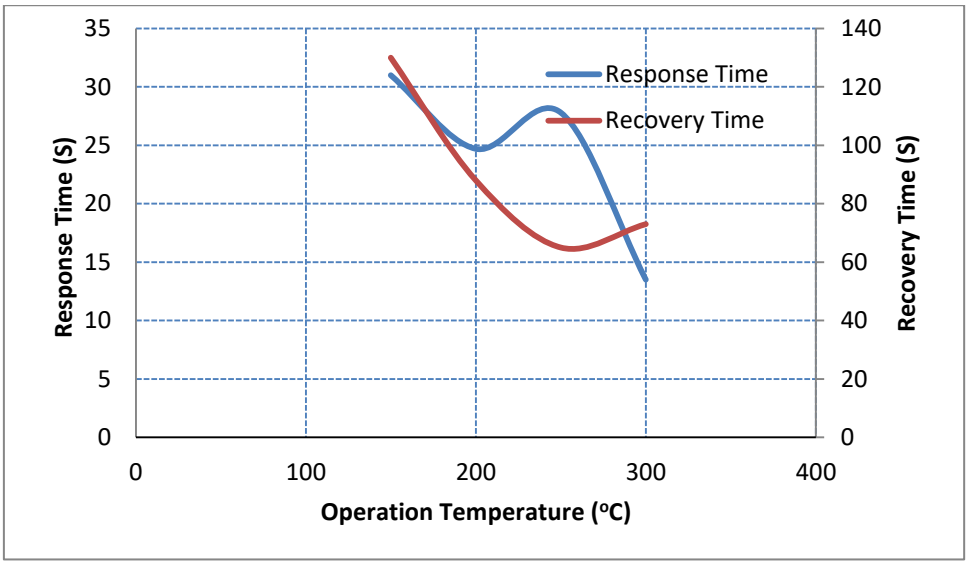

Figure 7: Response time and recovery time of V_2O_5 :Ag thin film sensor versus operation temperature with NO_2 tested gas.

The response time and recovery time are two significant factors to a gas sensor. The response time is known as the time required by the sensor to acquire 90% of the response on exposure to target gas. The recovery time is realized as the time required to attain 90% of recovery when gas is turned off. As can be noted from the figure, the response time of the thin film sensor was decreasing with the increase of temperature till 200 ^oC, when it started to increase up to 250 ^oC and then started to decrease. While, the recovery time was decreasing till 250 ^oC then increased with further increase of temperature. Response and recovery times for thin film sensor with N_2 as the tested gas, at 250^oC, determined from the Figure 7, were 27.8s and 65s, respectively.

Figure 8 shows the response time and recovery time changes with operating temperature of

the V_2O_5:Ag thin film sensor using NH_3 as the tested gas, which were less than their counterparts using NO_2 as the tested gas. The thin film sensor response time increased till nearly 200 ^oC then decreased with temperature up to 250^oC and then started to increase with further increase of temperature. The recovery time decreased till nearly 230^oC then increased slightly with further increase of temperature.

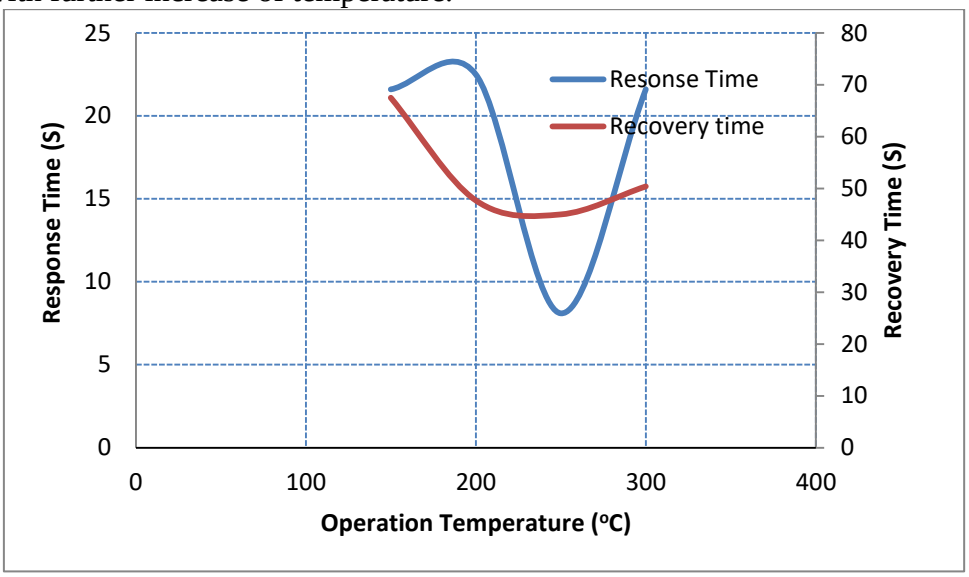

Figure 8: Response time and recovery time of V_2O_5: Ag thin film sensor versus operation temperature with NH_3 as the tested gas

Response and recovery times for thin film sensor with NH_3 as the tested gas ,at 250^oC, as determined from the Figure 8, were 8.1 s and 45 s, respectively.

Figure 9 presents the resistance changes of thin film with respect to time at operating temperatures of 150 ^oC , 200 ^oC, 250 ^oC, and 300 ^oC when the films were exposed to NO_2 gas. The dc electrical resistance decreased with increasing the operating temperatures and its values were (31, 11, 8 and 6 MΩ) at (150, 200, 250 and 300 ^oC), respectively then dramatically increased with further time due to the oxidizing nature of NO_2 gas.

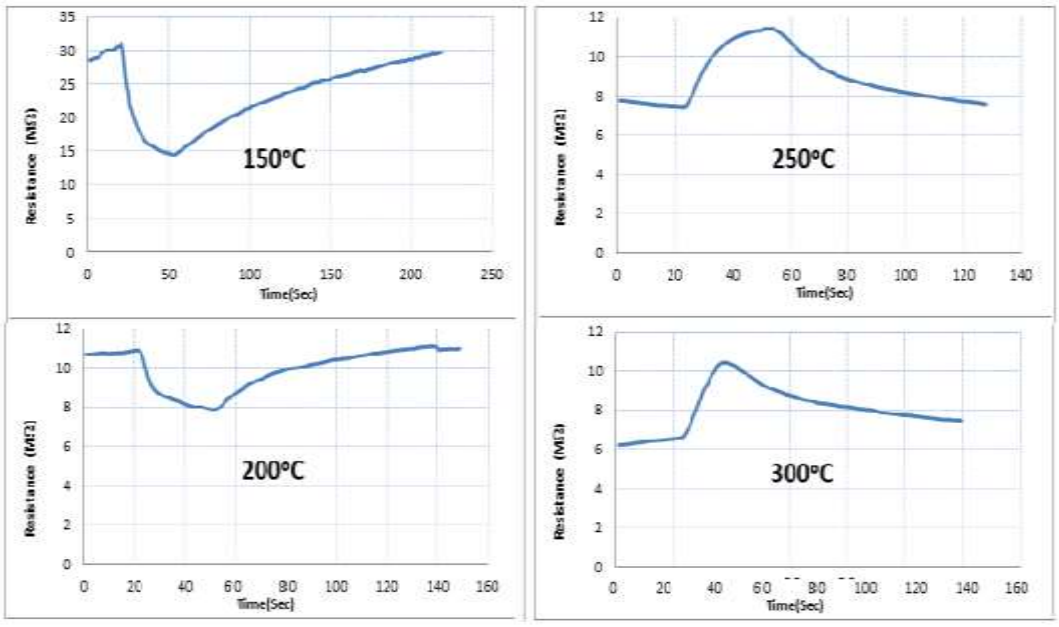

Figure 9: The variation of resistance of V_2O_5: Ag thin film with time for different temperatures using NO_2 as the tested gas.

The resistance of the thin film decreased with increasing temperature according with the semiconducting nature. The predictable reduction in resistance of V_2O_5 is attributed to the desorption of adsorbed oxygen from V_2O_5 film surface under vacuum thus emitting the trapped electrons [27]. Since V_2O_5 is an n –type semiconductor, its electrical manner upon exposure to NO_2 oxidizing gas can be described by a reduction of conduction carrier density. The quantity of oxygen ions obtainable on the V_2O_5 surface rises at the operating temperature. The adsorbing NO_2 molecules react immediately with the adsorption locations at the oxide surface [5]. The increasing of film resistance is typical for n-type semiconductor oxide gas sensors [28].

The change of the thin film resistance under the influence of NH_3 gas at temperatures of 150 ^oC, 200 ^oC, 250 ^oC and 300 ^oC is presented in Figure 10. The resistance decreased with increasing the operating temperatures and its values were (35, 11, 3.1 and 0.375 MΩ) at (150, 200, 250 and 300 ^oC), respectively. The resistance slightly increased at operating temperature 150 ^oC and 200 ^oC then decreased till 55 second then return to increased, while at 250 ^oC and 300 ^oC it increased then decreased with further time. When the reducing gas is exposed to the detecting element, it decreases its resistance, which emphasizes the standard property of an n-type semiconductor [5].

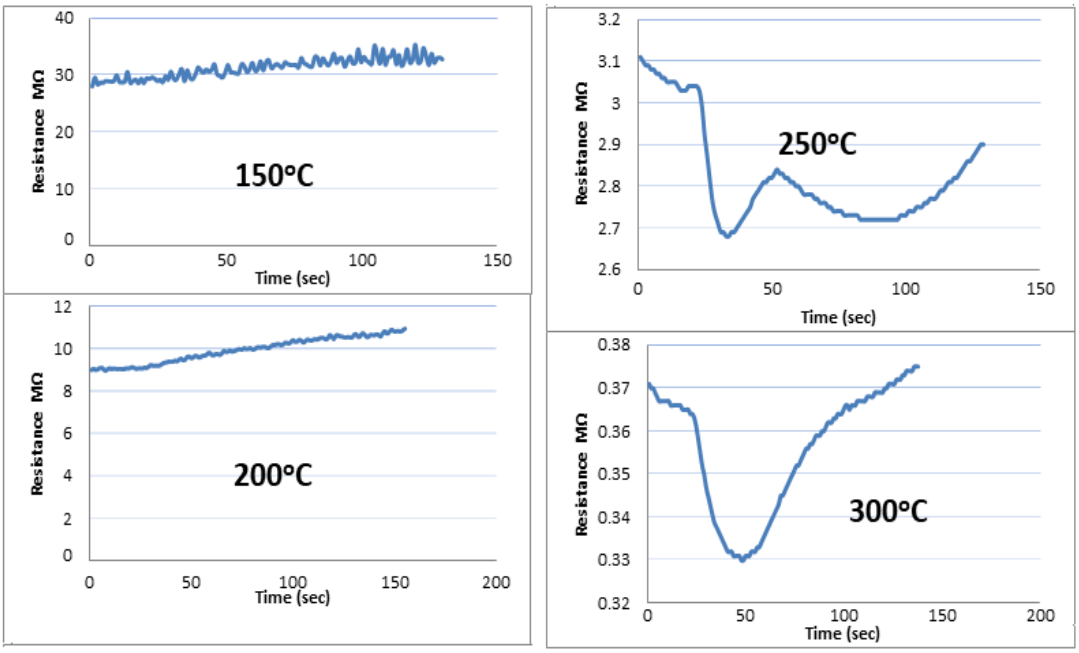

Figure 10: The variation of resistance of V_2O_5: Ag thin film sensor with time for different temperatures using NH_3 as the tested gas

4. Conclusion

There are much larger particles formed in the annealed films compared with as-deposited films. The sensors were exposed to NO_2 and NH_3 gases separately at various temperatures and its sensitivities were recorded. The changes of the response and recovery time of the V_2O_5: Ag film with operating temperature using NH_3 gas is less than their counterparts when using NO_2 gas. The increase of the thin film sensor resistance when exposed to the oxidizing gas NO_2 and the decrease when exposed to the reducing gas NH_3 emphasize the standard property of n-type semiconductor. It was found that the performance of the sensor with the thin film as the sensing element was remarkably better than that of the sensor with the bulk pellet as the sensing element state.

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