



Vanadium Pentoxide Nanostructures for Sensitive Detection of Hydrogen Gas at Room Temperature

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ABSTRACT

In this paper, novel vanadium pentoxide (V_2O_5) nanostructures of wire and star morphologies were synthesized by a daylong simple hydrothermal route using vanadyl sulphate hydrate and potassium bromate as precursors. The detection of trace quantities of Hydrogen (lower explosion limit is 4% in air) is performed at room temperature using thin films of the as synthesized novel nanostructures. Sensing behaviour of V_2O_5 nanostructures has been studied in details confirming sensitivity enhancement depending upon different morphologies. A 40% increase in sensitivity was recorded just by changing the shape of nanostructures from nanowires to nanostars. Detailed characterization was carried out for these morphologies using various techniques like FESEM, XRD, and FTIR etc. Sensor performance was dynamically evaluated under trace gas concentrations in room temperature condition and was recorded to possess a high sensitivity of 80%. The work shows a promising nature of the V_2O_5 nanostructures as sensing element for the detection of trace hydrogen gas at room temperature.

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1. Introduction

In recent years hydrogen gas has been globally accepted as a clean energy source of future and finds innumerable applications in industrial and automobile sectors (Akansu et al., 2004). Hydrogen possess an almost negligible carbon footprint and releases water as a by-product during combustion which is not detrimental for the environment (Ma and Zhou, 2010). However, low explosion limit (4% in air) in case of hydrogen raises critical safety issues even at room temperature like conditions (Sanger et al., 2016). Hence it becomes important to detect trace hydrogen leakages particularly in storage, transportation, and other utilities. Various gas sensors have been used and some of them even commercialized over years for sensitive detection by using optical, electrochemical, thermal, and mechanical methods (Liu et al., 2002; Al-mashat et al., 2010). Apart from these sensors, resistive gas sensors based on Transition metal oxides (TMOs) have been extensively studied and reported and because of their low cost, simple fabrication and operating methodology, and overall compact size (Huang and Wan, 2009; Wang et al., 2010a). TMOs possess multiple oxidation states to facilitate the charge transport in presence of reducing as well as oxidizing gases. TMOs are semiconducting in behaviour and their electrons can easily transit from valence to conduction band through extremely small amounts of energy. An appropriate selection of the sensing element to obtain better response at trace concentrations of the target gases particularly in room temperature conditions is highly desirable for the realization of gas sensor platforms. Metal oxides such as ZnO, SnO₂, and TiO₂ etc. have been widely reported earlier for hydrogen

gas sensing (Ruhland et al., 1998; Imawan et al., 2001; Karthik et al., 2016). To achieve zero tolerance levels of explosions the operating temperature of the sensor for Hydrogen gas should be low, which is a major challenge for researchers. This is mainly owing to the high temperature dependence of the adsorption/desorption kinetics and dissociation of the gas molecules on the sensor surface. Low operating temperature also eliminates the heating element from the sensor system which reduces complexity of fabrication and power consumption issues. Wang et al. (2015) showed V_2O_5 , a transition metal oxide having relatively narrow band gap of 2.2 eV in comparison with other metal oxides such as ZnO (3.4 eV) and SnO₂ (3.7 eV), to be high performing sensing element for the room temperature detection of hydrogen. The performance of the sensing element was mainly attributed to the high surface area obtained from the nano-structures of core shell type morphology.

Vanadium is a transition metal oxide which exists in multiple oxidation states (ranging between 2 and 5) (Haber et al., 1997). One of its oxides Vanadium Pentoxide (V_2O_5) possesses semiconducting behaviour and may applied widely to resistive gas sensing, photocatalytic degradation of organic dyes due to an abundance of electron hole pairs as discussed above (Yamazoe and Miura, 1994; Manno et al., 1997; Choi and Jang, 2010; Wang et al., 2010). V_2O_5 nanostructures have been used widely for sensing different gases such as ethyl alcohol (Liu et al., 2005; Dhayal Raj et al., 2010; Jin et al., 2010), NH₃ (Dhayal Raj et al., 2010), H₂ (Wang et al., 2015; Sanger et al., 2016), NO_x (Ishihara et al., 1989) etc. V_2O_5 possess a crystal structure comprising of orthorhombic unit cells with edge connected square pyramids of V_2O_5 (Ramana et al., 1998). It shows n-

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type semiconducting behaviour and has electronic conductivity of (~ 0.5 S/cm) at room temperature (Muster et al., 2000). Electrical transport mechanism through V_2O_5 nanostructures was studied by Mott's research group in detail and was explained by the Polaron hopping model (Mott, 1968; Muster et al., 2000). Various defect states of vanadium such as V^{5+} and V^{4+} are present in the lattice and are responsible for the charge transport through hopping of electrons between these impurity states even at room temperature (Livage, 1991). As any particularly gaseous species is absorbed over the V_2O_5 nanostructure it results in shortening of the overall hopping distance of the polaron leading to an overall change in resistance of the element.

Various research groups have developed different methods of fabrication such as surfactant assisted solution method, thermal evaporation method, hydrothermal method, solvothermal synthesis method etc. for vanadium pentoxide nanostructures (Pavasupree et al., 2005; Lavayen et al., 2007; Wu and Lee, 2009; Zhai et al., 2010). Among different morphologies, the one dimensional nanostructures are found to provide larger surface area and excellent catalytic performance. Various techniques such as refluxing and hydrothermal synthesis has been used earlier by the researchers to grow V_2O_5 nanotubes (Lavayen et al., 2007). A four days long reaction using V_2O_5 powder and hydrogen peroxide via hydrothermal route to synthesize centimetre long crystalline nanowires of V_2O_5 was also studied (Zhai et al., 2010). Another intriguing method to develop nanowire structure was given, where pyrolytic deposition of vanadium polyoxometalate anions was carried over a conducting glass substrate and the growth mechanism was shown to be dependent upon the reduction of V_2O_5 in the presence of H_2O and NH_3 vapours which were supplied abundantly by the precursor $NH_4OH \cdot HCl$ (Wu and Lee, 2009). The methods used so far by various research groups in developing one dimensional nanostructures of V_2O_5 have been either time consuming or too complex to follow. From the studies conducted in past the hydrothermal route is found to be a widely used method to grow one dimensional nano-structure. In this work we have utilized a day long simple hydrothermal method suggested in earlier study (Zhou et al., 2010), by using $VOSO_4 \cdot xH_2O$ as a vanadium source and $KBrO_3$ as an oxidizing agent. We have developed nanowires of V_2O_5 by using the same technique and nano-star morphology by changing the process parameters so that the growth is not confined to one direction. These two morphologies developed have been compared for hydrogen gas sensing and interestingly nano-star shape showed a better performance in terms of enhanced room temperature sensitivity. Various characterization techniques such as field emission scanning electron microscopy (FESEM) (Zeiss Supra 40V, Germany), X-ray diffraction technique (XRD) (PANalytical XRD) and FTIR have been used to analyze the synthesized products. Dynamic gas sensing experiments have been conducted in a setup consisting of a controlled flow of gas mixture.

2. Materials and Methods

2.1 Fabrication of V_2O_5 Nanowires and Nanostars

To synthesize the V_2O_5 nanowires, we have followed a simple hydrothermal method which was earlier used (Zhou et al., 2010). In the synthesis process vanadyl sulphate hydrate (10 mmol) (purchased from Alfa Aesar) and $KBrO_3$ (5 mmol) (purchased from Merck specialities private limited) was dissolved into 40 ml of distilled water. The obtained solution was magnetically stirred for 30 minutes followed by addition of HNO_3 to change the pH to 1. Obtained vanadium gel was transferred to a Teflon lined stainless steel autoclave for hydrothermal synthesis at $160^\circ C$ for 24 hours. Finally the solution was cooled to room temperature with the following filtration and centrifugation steps on the precipitates multiple times and washing the precipitate with DI water and ethanol subsequently. The product was then vacuum dried at $80^\circ C$ for 12 hours and named V_2O_5 -NW.

V_2O_5 nano-stars were synthesized by changing the process parameters used for nanowires. The method was described in details in one of our communicated paper (Chouhan et al., 2016), briefly to obtain nanostar shape we used same precursors in same amount but hydrothermal treatment was done at atmospheric pressure and $98^\circ C$ instead of high pressure inside an autoclave and high temperature as in case of nanowires. After 24 hours the precipitate was filtered and washed thoroughly in the above mentioned manner and the final product was named V_2O_5 -NS.

2.2 Sensor fabrication and Experimental set-up

Hydrogen gas sensing measurements have been performed by depositing the V_2O_5 sensing element on interdigitated copper electrodes, fabricated with the help of standardized lithography and chemical etching techniques over a printed circuit board (Size = 2.5cm X 2.5cm) (Arshak et al., 2005).

In a typical process, pre-cleaned PCB was spin coated by a positive photoresist (Microposit™ S1813™) and heated to $80^\circ C$ for 2~3 minutes followed by exposure to UV light for 45 seconds. Before exposing to UV light the PCB was masked in a certain manner to block the light for patterning. After this step the PCB was developed in a suitable developer Microposit™ MF-319. The obtained photoresist patterned over the surface was chemically etched using ferric chloride ($FeCl_3$) for 1~2 minutes. The copper electrode made by this method was further used as a sensing platform where the sensing elements (V_2O_5 -NW/ V_2O_5 -NS) were drop casted on the electrodes. To deposit thin films of V_2O_5 nanostructures over interdigitated copper electrodes, powdered material (10 mg) mixed with ethanol solution (5 ml) and ultrasonicated for 30 minutes was used to get a homogenous suspension, 20 μl of which was drop casted onto the electrode surface following which a curing step was initiated at $40\sim 45^\circ C$ for drying of the film and ensuring good contact between the sensing element and copper electrodes. The process is repeated multiple times to fill the space over the interdigitated electrodes completely. After this the prepared sensor was kept at room temperature for 12 hours.

The gas sensing performance of the sensor was investigated inside a test chamber where the flow of gas was controlled by using two mass flow controllers (MFC's) (M/S D07 SERIES Sevenstar), one for injecting the test gas and the other one for carrier gas (Nitrogen). The sensor response was measured by observing the resistance change across the electrodes through a LCR meter (M/S MASTECH MS5300). The measurement set up also consisted of a data acquisition system (DAQ) to record the sensor response for further analysis. Initially the test chamber was evacuated completely and then the carrier gas was supplied to record the resistance for base lining purpose of the sensing element in the absence of the target gas. After that hydrogen gas (target) was passed in the test chamber the sensor response was observed to change (come down). The concentration of hydrogen gas in the gas mixture was varied by varying the flow of hydrogen gas through MFC in order to obtain different PPM levels of the target gas. The performance of the sensor was measured in terms of sensitivity denoted by S (% change in resistance) as in equation (1).

$$S (\%) = \left(\frac{R_o - R_g}{R_o} \right) \times 100 \quad (1)$$

Here, R_g is the instantaneous resistance of element in presence of target gas and R_o is the initial resistance of the element measured in the presence of only the carrier gas.

3. Results and Discussion

3.1 Scanning Electron Microscopy Analysis

The scanning electron microscopy (SEM) analysis was performed for the synthesized nanostructures of vanadium pentoxide. SEM images, shown in figure 1 describe the morphology of the nanostructures in form of nanowires and nanostars. As synthesized nanowires and nanostars are shown in figure 1 (a) and (b), while figure 1(c) shows the EDAX data revealing both Vanadium and Oxygen. The length of wires is observed to be several micrometers and their diameter falls in the range of 26 ~ 30 nm. The growth mechanism of the nanowire shape is well explained through the Ostwald ripening process. VO^{2+} cations present in the solution get oxidized and form V_2O_5 nanoparticles in presence of $KBrO_3$ which is a strong oxidizing agent (Zhou et al., 2010). As mentioned above we obtained nanowires of better aspect ratio due to lower diameter and larger length which offers larger surface area for enhanced interactions with the target gas molecules.

The well grown V_2O_5 nanostar morphology can be observed in figure 1 (b). The SEM image show that modified synthesis process results into the formation of nanostar morphology. Due to their shape the nanostars are entangled with each other and come together to assembly as a porous film while nanowires are well stacked together showing lesser porosity. We have already mentioned that to fabricate nanostar shapes we have changed the process parameters from high pressure and temperature ($160^\circ C$) to atmospheric pressure and low temperature ($98^\circ C$). From our synthesis and previous work that has been reported by researchers we hypothesize that hydrothermal synthesis of V_2O_5 under high pressure and high temperature conditions favours the formation of one dimensional structures, whereas when the temperature is lowered from $160^\circ C$ to $98^\circ C$ and the synthesis carried out at atmospheric pressure conditions it leads to multidimensional growth which is recorded in the nanostar morphology. From this observation we can say that the operating parameters such as pressure and temperature play a critical role in determining the morphology.

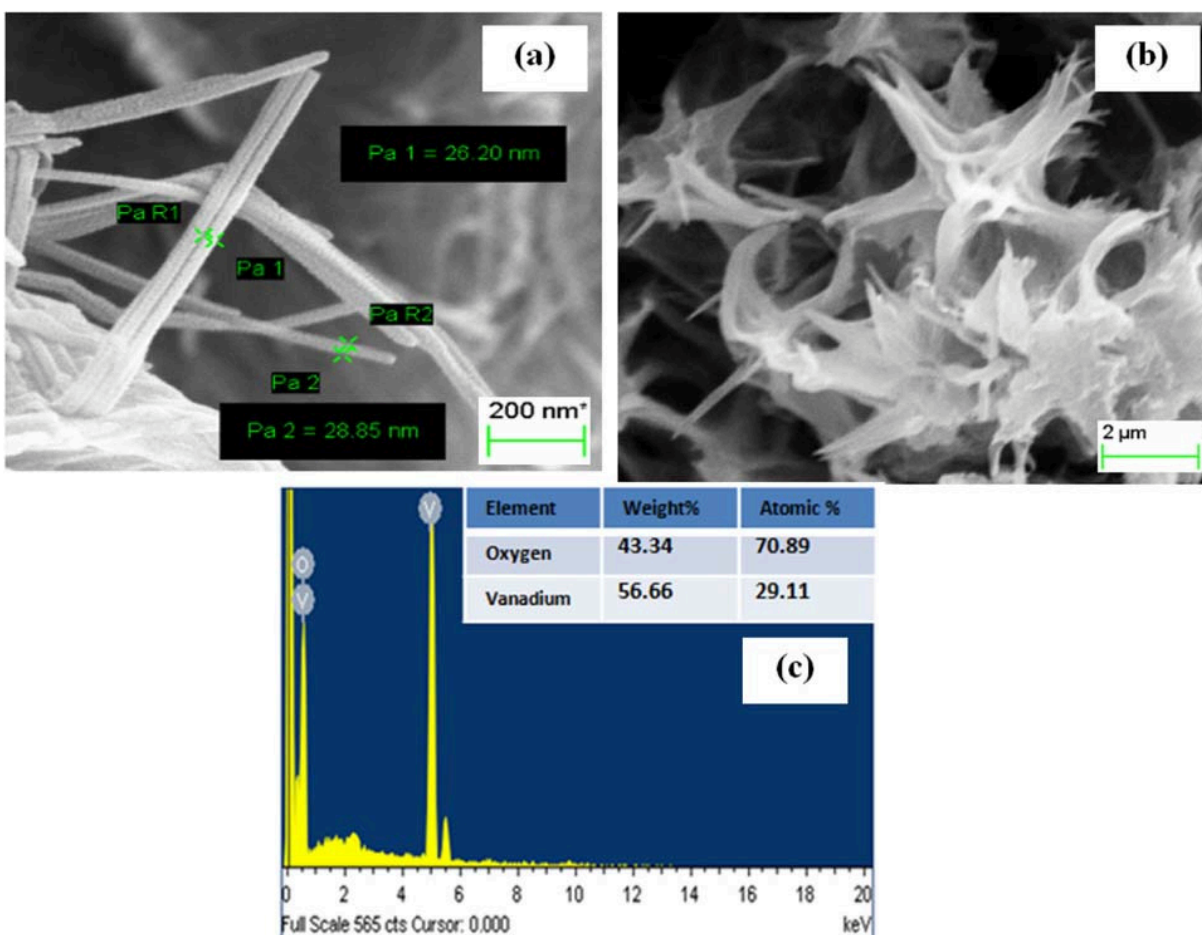


Figure 1: SEM images of as synthesized (a) V_2O_5 Nanowires and (b) V_2O_5 Nanostars at different magnifications. (c) EDAX confirms the presence of Vanadium and oxygen elements.

3.2 X-Ray Diffraction analysis

X-ray diffraction (XRD) analysis of fabricated nanostructures of V_2O_5 is shown in supplementary figure S1. The XRD pattern shows various characteristic peaks of vanadium pentoxide crystal structure. The high intensity peak represented by $\{001\}$ plane confirms the hydrated layered structure of V_2O_5 (JCPDS No. 40-1296) (Xiong et al., 2008; Du et al., 2011). The position of appearance of this peak shows the presence of intercalated water molecules into the interlayer of nanostructures which is attributed to the hydrothermal synthesis process (Kittaka et al., 1991; Oliveira et al., 2000, 2014). The lamellar and hydrated form of the structure of V_2O_5 nanostructures is represented by the peak at $2\theta = 260$ which is designated to the $\{003\}$ plane (Zampronio et al., 2003). The XRD analysis of both morphologies (V_2O_5 -NW and V_2O_5 -NS) shows the same pattern of peaks confirming the same crystal structure (Orthorhombic) of the synthesized products.

3.3 Infrared Spectroscopy Analysis

The Fourier Transform Infrared Spectroscopy (FTIR) analysis was performed to analyze the presence of different bonds in the synthesized product. Supplementary Figure S2 shows different peaks observed at their respective absorption bands for the material. The peak represented by the absorption bands from 3200~3600 cm^{-1} and at 1619 cm^{-1} represents presence of -OH bonds and bending vibration attributed to the presence of free water molecules respectively (Sediri and Gharbi, 2007). An asymmetrical stretching band of -CH₂ bond can be observed through a small peak at 2927 cm^{-1} . This indicates that the organic elements are present at trace level even after thorough washing of the test sample (Xiong et al., 2008). Various vibrations of vanadium-oxygen bond are represented by absorption peaks from 500 to 1000 cm^{-1} . The FTIR spectrum also shows stretching vibration of V=O type of bond by observing the peaks in the range of 995~1050 cm^{-1} band which indicates

the presence of V⁵⁺ and V⁴⁺ intermediate states of vanadium in the crystal structure of V_2O_5 (Dhayal Raj et al., 2010). Another peak observed at the position of 760 cm^{-1} can be attributed to V-OH₂ bond representing the intercalation of water molecule into the interlayers of V_2O_5 nanostructures (Sediri and Gharbi, 2007; Xiong et al., 2008). These type of vibrations cause oxygen atoms to move in-plane with angular bond of V-O-V (Fomichev et al., 1997). While the V=O type bond vibration is also observed at 1012 cm^{-1} usually observed at 1015 cm^{-1} in crystalline V_2O_5 (Choo et al., 2000; Guerra et al., 2009).

3.4 Gas sensing performance

The sensor response in form of resistance variation was recorded by flowing in the test gas (hydrogen) and carrier gas (Nitrogen) in the test chamber. The gas concentration was varied by changing the mass flow rate of test gas with respect to the carrier gas. The electrodes of the sensor were connected to the resistance-meter followed by chamber evacuation and subsequent supply of the carrier gas. The resistance value was allowed to stabilize before supply of the test gas for measurement of R_0 . Following this the test gas was loaded with carrier gas at a required concentration level and the sharp change in resistance of the sensing element was observed. After few seconds the resistance value stabilized to a new value and at this point of time the flow of test gas was stopped. Resistance re-changed back to the baseline value and such real time variations in resistance was recorded cyclically by the injection and clearances of test gas in the chamber dynamically. Figure 2 (a) and (b) show such responses at 50 ppm concentration level of hydrogen gas for V_2O_5 nanowire and nanostar sensing elements respectively. To record the effect of concentration variation and also to obtain the limit of detection of the sensing element tests were carried out by varying concentration of hydrogen in the chamber from 10-300 ppm. Figure 2 (c) and (d) shows the sensor response under varying concentration of hydrogen for V_2O_5 nanowire and nanostar sensing elements respectively. These results show

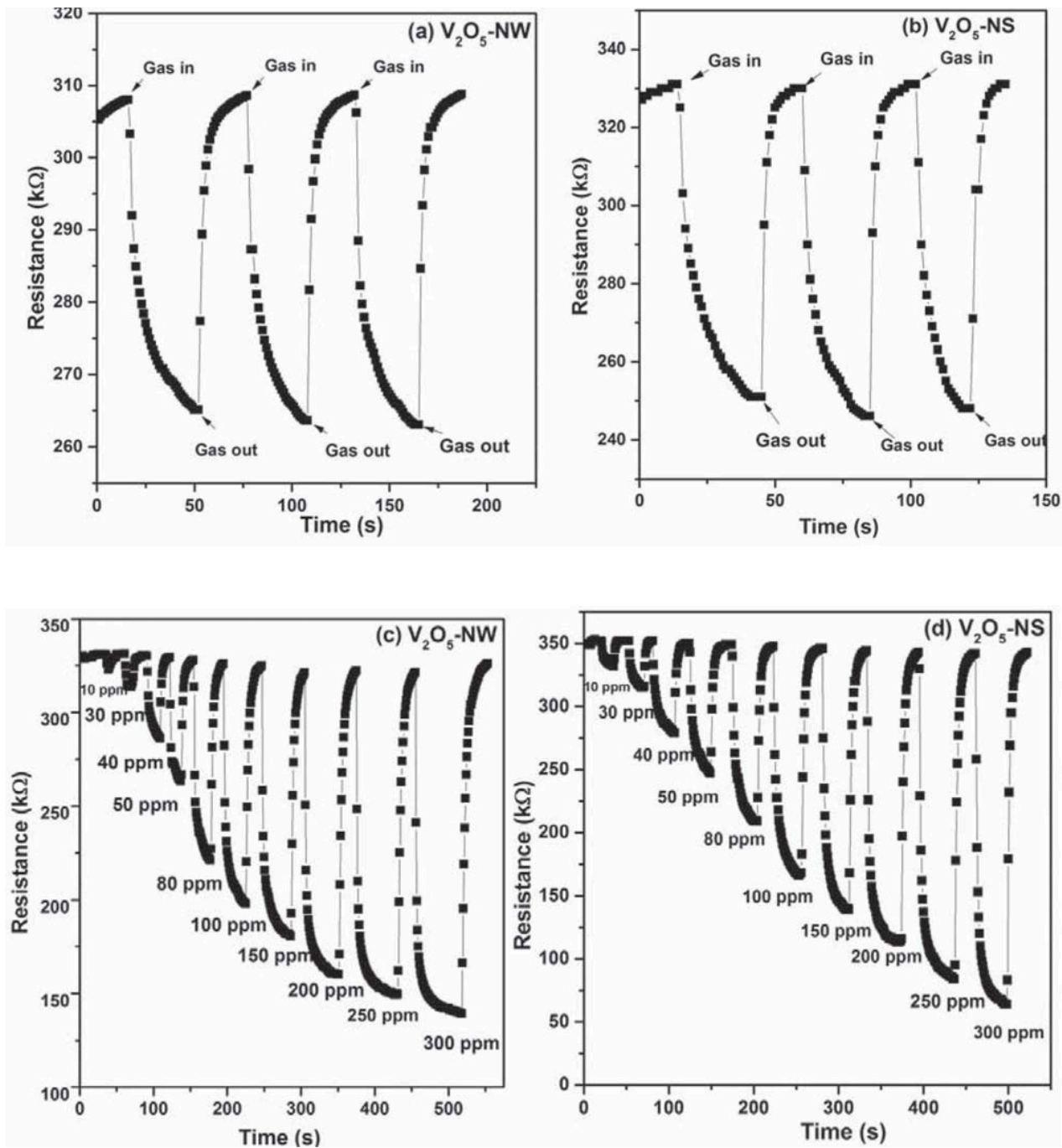


Figure 2: Sensor response with respect to hydrogen gas by using (a) V_2O_5 -NW and (b) V_2O_5 -NS under 50 ppm, (c) V_2O_5 -NW and (d) V_2O_5 -NS under 10-300 ppm concentration level.

that the fabricated V_2O_5 nanostructures are having sharp response and recovery characteristics towards hydrogen gas for low as well as high concentration levels under room temperature operating condition.

To compare the performance of both the morphologies of vanadium pentoxide as described above, the percentage sensitivity was calculated by using a general formula (discussed earlier in section 2.2). The sensitivity results for both morphologies (figure 3) show enhanced sensitivity for the V_2O_5 nanostar structures. We hypothesize that the reason behind such a behaviour to be the differences in morphology as a similarity is recorded in crystal structure and chemical composition of both morphologies. The nanowires are having uniform cross section, well developed structure and good stacking levels resulting in reduced porosity. On the other hand the nanostars possess high surface roughness, sharp edges and also an overall entangled structure with high porosity. These parameters provide

a large overall surface area and high diffusivity to the target gas molecules thus enhancing their overall interaction level. Thus the nanostar morphology demonstrates an overall enhanced level of sensitivity to Hydrogen gas. Large range of gas concentration was examined and fabricated sensing elements were found to be responsive at room temperature conditions which indeed qualify the V_2O_5 material for MEMS grade sensing. In comparison of the two structures of V_2O_5 , the nanostar shape was found to be more sensitive (81% at 300 ppm) in comparison with nanowire shape (58% at 300 ppm) towards hydrogen gas. The V_2O_5 nanostructures were thoroughly investigated for sensing of hydrogen gas and the results show sharp response and recovery response for different gas concentrations. The room temperature gas sensing results of V_2O_5 nanostructures make it a potential material for sensing of other combustible gases as well.

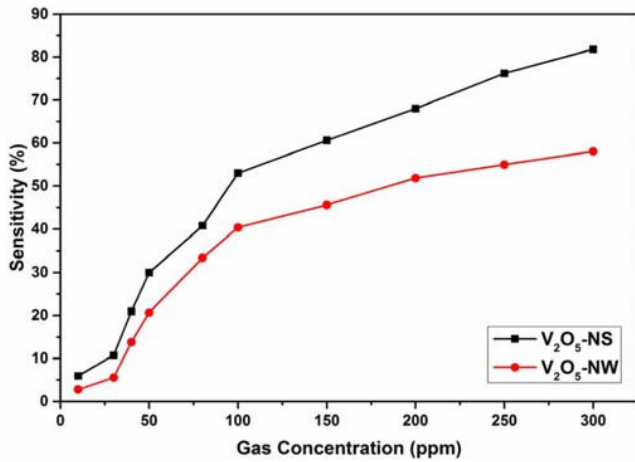
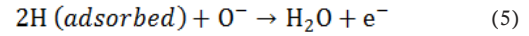
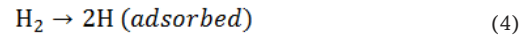
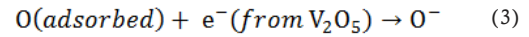
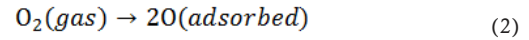


Figure 3: Variation in percentage sensitivity of V₂O₅ nanowires (V₂O₅-NW) and Nanostars (V₂O₅-NS) under different gas concentration.

3.5 Sensing Mechanism

Hydrogen gas is having relatively low atomic mass and small size which allows it to flow through the solid nanostructures at high rate. The hydrogen gas molecules get adsorb physically into the interlayer of the V₂O₅ nanostructures resulting into a high performance sensing characteristics. The high surface area of the V₂O₅ nanostructures offers the maximum adsorption of the hydrogen gas molecules. The reactions which are responsible for the resistive change of the nanostructures on adsorption and desorption of hydrogen gas are explained in following steps: As discussed earlier V₂O₅ possesses a hydrated layered structure which allows oxygen molecules to chemisorb into the interlayers thus

capturing free electrons and creating O⁻¹/O⁻² states due to their high electron affinity. This reduces the electron concentration level in the n-type V₂O₅ nanostructure creating a depletion region. During exposure to hydrogen gas, chemisorbed oxygen reacts with hydrogen gas molecules resulting into release of electrons and formation of water molecules as expressed in following reactions [Eqs. (2)-(5)] (Boudiba et al., 2013).



The release of electrons causes an overall decrease in the resistance of the sensor which reaches a saturation level after sometime of exposure and during the recovery process in absence of hydrogen gas the electrons are again re-captured by chemisorbed oxygen resulting in an increase in the resistance.

3.6 Response and recovery time constants with respect to concentration

To understand the sensor performance with respect to gas concentration in terms of response and recovery characteristics a theoretical analysis was performed. The response and recovery times are calculated by recording the time required for the sensor to attain 90 % of original values. The general formulae which are used to find out the response and recovery time constants are given in equations (6) and (7) respectively.

$$S(t) = S_{max}(1 - e^{-t/\tau_{res}}) \tag{6}$$

$$S(t) = S_{max}(e^{-t/\tau_{rec}}) \tag{7}$$

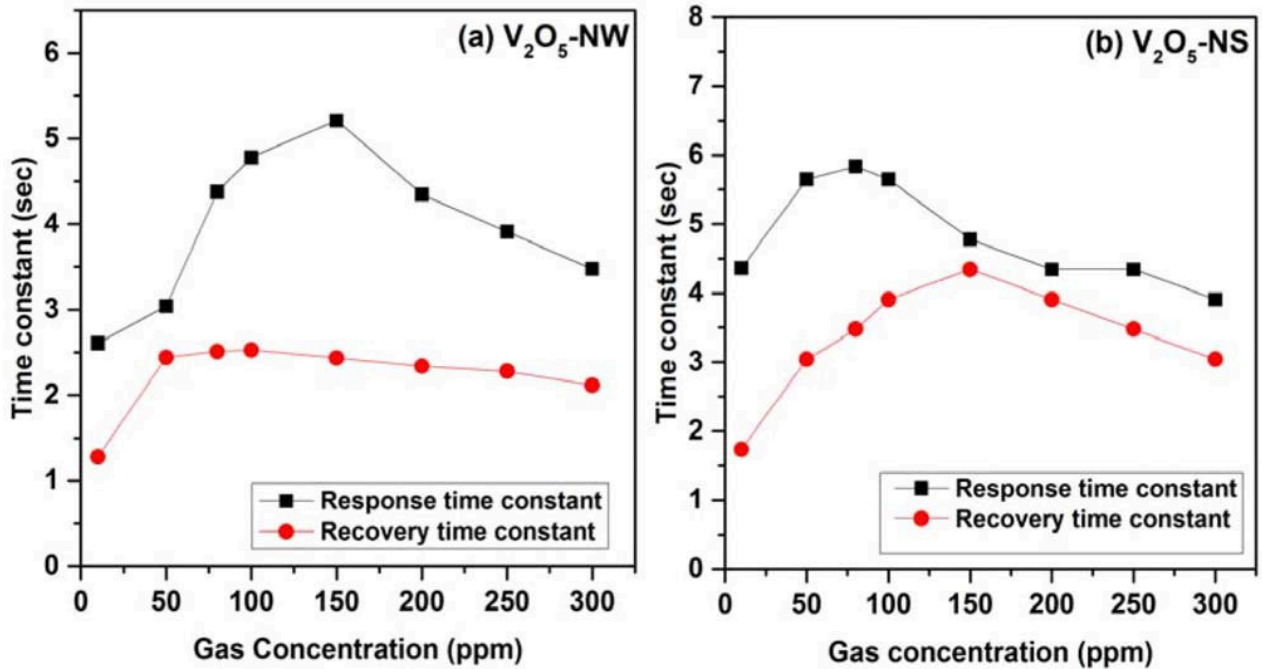


Figure 4: Response and recovery time constants of (a) V₂O₅-NW and (b) V₂O₅-NS with respect to hydrogen gas concentration, respectively.

Where $S(t)$ is the sensitivity with respect to time, S_{max} is the maximum sensitivity of the sensor at a particular gas concentration, and t is the response or recovery time for the sensing cycle (Gupta et al., 2014). The terms τ_{res} and τ_{rec} are the response and recovery time constants of the sensing element respectively. Interestingly, the results obtained by this analysis show that hydrogen gas molecules react with V₂O₅ nanostructures in a very different manner which depends upon the gas concentration. Overall values of response and recovery time constants are quite low (1

to 6 seconds) for the various gas concentrations, which is an essential characteristic for a MEMS grade gas sensor. Both the structures (V₂O₅-NW and V₂O₅-NS) demonstrate an overall low value of time constants for trace gas concentrations (less than 50 ppm) of Hydrogen. The time constant increases with increase in gas concentration and after certain threshold concentration level (100 to 150 ppm) the time constant values for both the structures decrease. The possible explanation of such behaviour for the gas sensor can be given by the interaction between

hydrogen gas molecules and the oxygen defect sites present on the surface of thin film metal oxide semiconductor. Initially when the gas concentration is lower there is a less number of gas molecules present to interact with large number of oxygen defect sites hence the response time is low. As the gas concentration is increased the reaction times are also increased upto a certain threshold value because there are more number of gas molecules present to react with competing oxygen defect sites resulting into higher response and recovery time. When the gas concentration is very high the response and recovery time constants are reduced gradually which is attributed to a quick saturation of the films due to the presence of abundantly available gas molecules. All the adsorption and desorption related reactions occur at the thin film surface at lower concentration level and at higher concentration, porous structure of the bulk provides larger surface area (more defect sites) to interact with large number of gas molecules resulting into quicker response and recovery time constants (Gupta et al., 2014).

4. Conclusion

The novel nanostructures of vanadium pentoxide were synthesized and successfully validated for hydrogen gas sensing at room temperature for trace concentrations. Thin films of as synthesized nanostructures were found to be highly sensitive with fast response (~11 seconds) and recovery time (~7 seconds). Novel nanostar shape morphology was developed by altering the synthesis process for nanowires and resulted in a high sensitivity yield (40% enhancement) over the nanowire morphology. Detailed studies of gas sensing performance towards hydrogen gas showed that the vanadium pentoxide nanostructures formulate a class of potential sensing element with enhanced sensitivity for different gas concentrations of Hydrogen.

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