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Fabrication of ZnO nanorod-based hydrogen gas nanosensor

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Abstract

We report a first work on nanofabrication of hydrogen nanosensor from single ZnO branched nanorods (tripod) using in-situ lift-out technique and performed in the chamber of focused ion beam (FIB) system. Self-assembled ZnO branched nanorod has been grown by a cost-effective and fast synthesis route using an aqueous solution method and rapid thermal processing. Their properties were analyzed by X-ray diffraction, scanning electron microscopy, energy dispersion X-ray spectroscopy, transmission electron microscopy, and micro-Raman spectroscopy. These analyses indicate high quality ZnO nanorods. Furthermore, our synthesis technique permits branched nanorods to be easily transferred to other substrates. This flexibility of substrate choice opens the possibility of using FIB system for handling.

The main advantage of the proposed in-situ approach is a controllable lift-out procedure which permitted us to obtain a 90% success rate for building nanodevices. The fabricated nanosensor uses only single self-assembled ZnO branched nanorod (tripod) to gauge the 150 ppm H_2 in the air at room temperature. The hydrogen sensitivity is in the range of 0.6–2% depending on which two branches to use. The nanosensor has selectivity against other gases such as O_2, CH_4, CO and LPG, which shows sensitivity of $<0.02\%$. The single ZnO branched nanorod sensor can operate at low power of $<5\,\mu W$.

Keywords: Nanoscale materials and structures; Nanofabrication; Gas sensor; Hydrogen; ZnO branched nanorod

1. Introduction

Hydrogen (H_2) is expected to be the principal energy source or ''the fuel of the future'' [1] and can be used in the future power devices [2], solid oxide fuel cells [3,4], H_2 engine cars [1], etc. However, H_2 is a hazardous, odorless and highly inflammable gas and it is necessary to detect its leakage. A reliable and inexpensive sensor that can take advantage of nanoscale to detect hydrogen leaks is the focus of many research groups [3–8].

Common sensors proposed to use an indirect approach (e.g. Raman spectroscopy, etc.) or requiring complicated components to detect the presence of H_2. Many ideas have been proposed such as use of different metal wires [7], semiconductor oxides nanoarchitectures [8], etc. Gas sensors based on ZnO nanorods, SnO_2 nanowires, In_2O_3 nanowires, etc. showed excellent response and recover characteristics [9] and can potentially overcome obstacles of other type of sensors, such as sensitivity, selectivity, etc. Among different nanomaterials nano-ZnO is one of the most promising multifunctional materials for gas sensors, especially for H_2 sensing [3–5]. ZnO nanorods also have the advantages of large surface area, radiation hardness [10], thermal and mechanical stability [11]. The physical properties of the nano-ZnO materials depend on the microstructure including morphology, crystal size, orientation, aspect ration and crystalline density [12]. Sensing properties of nano-ZnO are directly related to its preparation history, particle size, surface to volume ratio, morphology and

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operating temperature. The signal consists of conductivity changes due to gas adsorption on the surface and permits to detect real-time events.

Recently, the branching growth phenomena such as nanojunction arrays have attracted great interest for achieving high degree of superior functionality via direct self-assembly. Thus, multiple ZnO nanorods [5] and single ZnO two-dimensional branched nanorods have attracted considerable attention due to their unique properties that strongly depend on their size, morphologies [13] and configurations [14], and their possible use as low-dimensional building blocks-functional units in H_2 nanosensors and nanodevices [8,15].

Different H_2 sensors have been demonstrated. Wang et al. used multiple ZnO nanorods with Pd and achieved $>4.2\%$ relative response ($\Delta R/R$) at 500 ppm H_2 in N_2 after 10 min exposure [3]. According to experimental results presented by Wang et al. [6], the H_2 sensitivity of nanowires have the highest sensitivity ($S\sim8$) at 250 °C [6]. Tien et al. [16] used single Pt-coated ZnO nanowires and achieved a relative response of ~20% to 500 ppm H_2 in N_2 after 10 min exposure. At the same time realized sensitivity ($S_{max} = R_{air}/R_{H_2}$ is between 1 and 2) to 100 ppm H_2 with ZnO nanorods is several times higher than the sensitivity of ZnO films at 300–400 °C obtained by Min et al. [17]. Tien et al. [4] demonstrated a current response of a factor ~3 larger for Pt-coated multiple ZnO nanorods versus ZnO thin films upon exposure to 500 ppm H_2 in N_2 at room temperature. They also found that the ZnO multiple nanorods sensors showed a faster response and a slower recovery in air after H_2 exposure than ZnO films. It has been presented single nanowires of different metal oxides [18] and metal catalyst coatings (Pt, Pd, Au, Ag, Ti and Ni) on multiple ZnO nanorods [19] which are easy to fabricate and possess enhanced sensing properties.

Although ZnO nanorods sensors had a high response, high selectivity and short response time to low concentrations of gas, at the current stage, it is still difficult to obtain single nanowire and to fabricate this kind of device in large quantities [20]. In this field are considered increased complexity, lengthy sample preparation and device fabrication, time consuming analysis and selectivity. Thus, for wide applications of ZnO nano/microrods in sensors, an inexpensive and environmentally benign self-assembly synthesis process is required in order to synthesize transferable nanorods which can be easily handled with modern equipment. Thus, the latest research efforts are directed towards obtaining alternative, lightweight and flexible nanodevices [5,14,21,22].

To overcome some of these limitations, we have focused on single ZnO branched nanorod-tripod as the material template for designing a H_2 sensor. We report a synthesis route for self-assembled ZnO branched nanorod easily transferable to other substrates and in-situ lift-out technique using focused ion beam (FIB) system to fabricate individual nanosensor with three electrodes to detect H_2 at room temperature and to be quite selective.

2. Experimental details

2.1. Synthesis and characterizations

ZnO branched nanorods have been synthesized using an aqueous solution deposition on glass or Si substrates cleaned using a procedure described in Refs. [14,23].

Staring materials are zinc sulfate and ammonia solution, all analytical grade (Fisher Scientific Corp.) without further purification. $Zn(SO_4)\cdot 7H_2O$ (0.01–0.04 M) and NH_4OH (29.6%) were mixed with 30 ml de-ionized (DI) water (~18.2 MΩ cm). The substrates were kept in solution of 0.2 g $SnCl_2$ in 200 ml DI water with 10 μl HCl for 3 min then were rinsed with a jet of DI water. After this procedure the substrates were placed in aqueous complex solution reactor and was mounted on pre-heated laboratory oven and kept at 90 °C for 10 min of heterogeneous reaction. Then the substrates were dipped in DI water to remove unreacted products from nanorods surface and then was dried in air at 150 °C for 5 min and rapid thermal processed at 650 °C for 60 s in a forming gas ambient.

The crystal structures of ZnO branched nanorods were investigated by X-ray diffraction (XRD; Rigaku 'D/B max') and transmission electron microscopy (FEI Tecnai F30 TEM) at an accelerating voltage of 300 kV. The elemental analyses were examined using energy dispersion X-ray spectroscopy (EDX) and the morphologies of the nanorods by scanning electron microscope (SEM) Hitachi S800. These investigations confirmed that these nanorods are highly crystallinity and regular distributed throughout the substrate surface.

For hydrogen detection using the ZnO branched nanorod, a measuring apparatus consisting of a closed quartz chamber connected to a gas flow system was assembled. The concentration of test gases was measured using pre-calibrated mass flow controller. Hydrogen and air were introduced to a gas mixer using separate mass flow controllers. The mixed gas was injected to a chamber in which the nanosensor was placed. A computer with suitable Lab–View interface handled all controls and acquisition of data.

Fig. 1 shows the XRD pattern of ZnO branched nanorods. The diffraction peaks in the pattern are indexed to hexagonal wurtzite structured ZnO (space group: $P6_3mc(186)$; $a = 0.3249$ nm, $c = 0.5206$ nm) and the data are in agreement with JCPDS card for ZnO [24].

The quality of the grown ZnO rods was also confirmed by the 1.0:1.0 stoichiometric composition deduced from the EDX analysis in all samples. TEM, XPS and micro-Raman characterization results have been discussed in details in our previous work [14,23].

Typical SEM images of ZnO branched nanorods are shown in Fig. 2. A close observation (Fig. 2a) reveals that the individual ZnO nanorod has a radius of about 200 nm and length about 12–17 μm for 10 min synthesis. Lowering the concentration of ammonia hydroxide and control

reaction process permit the growth of smaller ZnO nanorods with radius less than 100 nm, but smaller nanorods were difficult to be transferred, separated and picked-up in the FIB system (Fig. 2c).

According to our experimental results, the branched nanorods obtained by our process can be easily transferred to other Si/SiO_2 substrates and distributed on the surface (Fig. 2b) of another substrate for further processing by the in-situ lift-out needle.

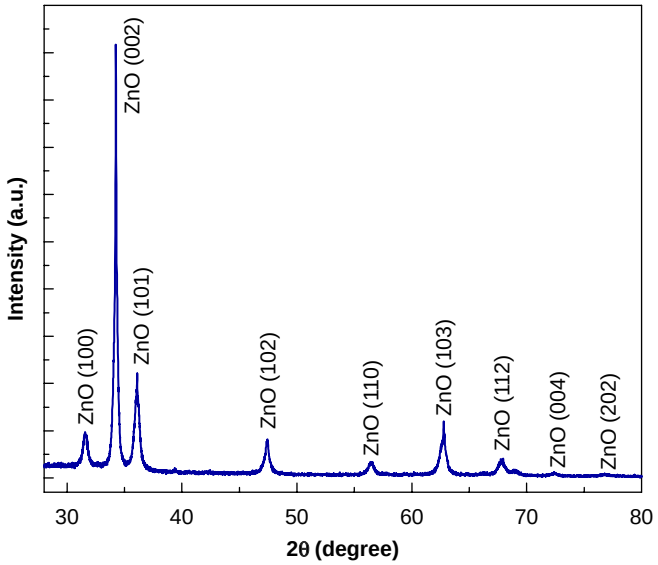

Fig. 1. Typical XRD pattern of the ZnO branched nanorods synthesized by aqueous solution method.

2.2. Nanofabrication of sensors by in-situ lift-out technique

In the following section, the in-situ lift-out technique is described. The Si/SiO_2 wafers were used as intermediate substrate for ZnO tripods transferring and distribution in order to avoid charging problems in the FIB system. For the nanosensor preparation, the glass substrate was used and Al electrodes were deposited as template with external electrodes/connections. Usually a microscope and a micromanipulator for the ex-situ lift-out technique have been used to separate individual ZnO nanorods in order to be easily attached to the in-situ FIB needle. A magnification of $\times 100$ was used to separate ZnO nanorods transferred to intermediate Si/SiO_2 substrate and distribute on the surface for an easy pick-up. A magnification of $\times 6500$ was used to position a needle on the ion optic axis and to lift the single ZnO branched nanorod-tripod away from the Si/SiO_2 substrate.

A micromanipulator mounted beside the stage used in our work permits movements in the nanometer regions along the x, y or z directions. Sample on the stage can be independently rotated perpendicular to ion beam, which enable easy arrangement of single ZnO branched nanorod

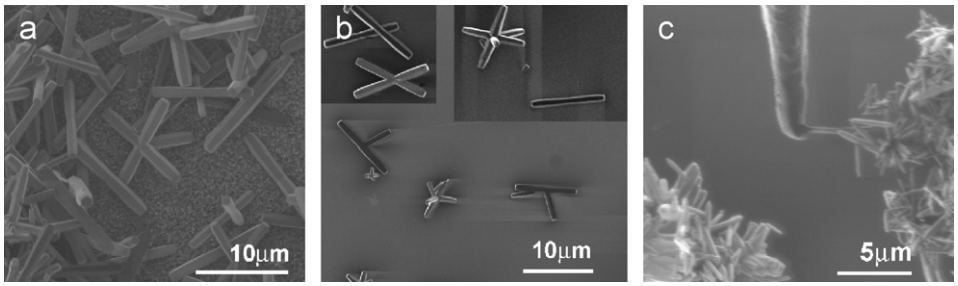

Fig. 2. The SEM images of the aqueous solution synthesized ZnO: (a) branched nanorods on initial glass substrate; (b) single and branched nanorods transferred to Si/SiO_2 substrate, insets are different regions of substrate showing the rods distributed on the surface in order to be picked up by the in-situ lift-out needle in the focused ion beam FIB/SEM system; and (c) attempt to pick up an individual nanorod from an agglomerations of ZnO nanorods.

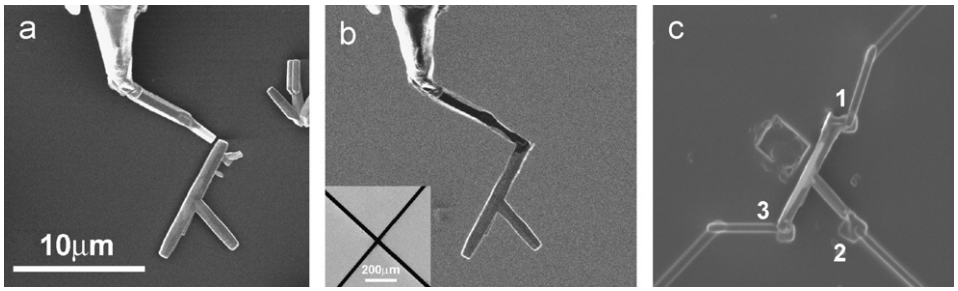

Fig. 3. Scanning electron micrographs showing the steps of the in-situ lift-out fabrication route in the FIB/SEM system for (a) branched ZnO nanorod transferred to Si/SiO_2 substrate and positioned FIB needle with intermediate connection from single ZnO nanorod, (b) branched ZnO nanorod picked up by in-situ lift-out needle to be used as tripod selected for nanofabrication, inset shows nanosensor substrate template (glass substrate with Al contacts as external electrodes) and (c) branched nanorod cutted from needle and brushed to the sensor template support and fixed to three electrode/external connection as final nanosensor.

on the nanosensor template with Al contacts as external electrodes.

In the in-situ process [25], we developed that attaching a single intermediate nanorod on the FIB needle (Fig. 3a) allows an easy pick-up of the branched nanorod for further handling. This intermediary nanorod can greatly enhance the nanofabrication capability and reduce the number of steps in the procedure and the total time for nanodevice fabrication, respectively.

The next step in our procedure is to scan the surface of the intermediate Si/SiO_2 substrate for convenient placed ZnO nanorod-tripod. Then the needle was lowered and bringing into the FIB focus and its tip positioned at the close end of the branched nanorod as shown in Fig. 3a. Before attaching of selected branched nanorod, it is recommended to push it in order to make sure that it is not attached firmly to the substrate and the branches are strong enough to be transferable. Then the needle was lowered until it touched the tripod and was attached to the end of the FIB needle as shown in Fig. 3b using Pt deposition of 0.5 μm thickness. Following this step (Fig. 3b), the needle and specimen was moved away from the substrate.

Fig. 3c shows the nanorod on the substrate using micromanipulator on the nanodevice substrate/template (see inset in Fig. 3b) with pre-deposited Al external electrodes. In this step, the needle was lowered and repeatedly swept across the substrate until nanorod brushes against the support and becomes attracted to it. If the nanorod does not initially lie flat on the substrate then it will be extremely difficult to realize good contacts with external electrodes. This situation will often happen in the case without using of an intermediate nanorod or if selected nanorod is not flat to the intermediate Si/SiO_2 substrate (Fig. 3a). The last step consists of positioning of tripod nanorod, then fixed to the one of the predeposited external electrodes. Then the nanorod has been cutted and needle raised away from the substrate. Fig. 3c shows the single ZnO branched nanorod-based sensor fabricated by our nanotechnology. The typical time taken to perform this in-situ lift-out FIB nanofabrication is 30–45 min. Taking in the account that nanorod synthesis was done in 10 min, we overcome the conception that single nanorod/nanowire are not convenient [20] for sensor production. In details, described technique makes easier to learn and apply nanofabrication steps using easy transferable nanorod (avoiding nanorods agglomerations) fabricated by our method, especially for new users/operators and will permit the highest success rate. Our success rate using this route is >90%. This minimizes the total time to machine time using FIB/SEM for the nanodevice fabrication and can be applied in other specific devices.

3. Gas sensing properties and discussions

For gas sensing characterizations, the fabricated single ZnO branched nanorod sensor were placed in a 1000 cm^3 gas chamber and investigated sensitivity to H_2 and also O_2, CH_4, CO, CO_2 and LPG gases at room temperature in the concentrations up to 2000 ppm. The readings were taken after the gases have been introduced in the test chamber. It was found that resistance change $|\Delta R| = |R_{gas} - R_{air}|$ increased linearly with H_2 gas concentration and is constant for other gases at room temperature.

The room temperature sensitivity of the self-assembled ZnO branched nanorod at 150 ppm H_2 is shown in Fig. 4 for three different possible connections. The relative resistivity changes after 50–80 s H_2 exposure becomes stable, on the other hand, were not restored to the 90% of the original level within 5 min suggesting a relatively longer recovery time, in comparison with the response time of ~1 min.

We repeat the sensor experiment for gas sensitivity to several common gases O_2, CH_4, CO, CO_2 and LPG under the same conditions and found that $|\Delta R/R|$ is less than 0.02% for these gases. Therefore, we establish that the ZnO nanosensor has certain degree of selectivity.

The multiple pure ZnO nanorod-based sensor presented in Ref. [3] has a sensitivity of ~0.25% at 500 ppm H_2 in N_2 after 10 min exposure. However, the Pd-coated ZnO nanowires gas sensors showed a higher H_2 sensitivity (4.2%) and fast response and recovery time at concentration up to 500 ppm at the room temperature [3]. Furthermore sensor based on ZnO multiple nanorods and exposed under 10% H_2 in N_2 at 112 °C, showed high sensitivity ~18% of current change which are quite good [5].

By comparison, our single ZnO branched nanorod sensor exposed to 150 ppm H_2 shows a relative response ~2% in 50–80 s, while Rout et al. [18] showed that single ZnO nanowires has a sensitivity of ~3 for 100 ppm H_2 at room temperature. The sensitivity $\Delta R/R$ of our sensor is attractive for further investigation for practical H_2 sensor applications.

The adsorption–desorption sensing mechanism is proposed on the base of reversible chemisorption of the hydrogen on the ZnO nanorod. It produces a reversible variation in the resistivity with the exchange of charges between H_2 and the ZnO surface leading to changes in the depletion length [26]. Thus, one way to improve sensitivity is to increase the change in the surface/volume ratio [27]. It is well known that oxygen is adsorbed on a ZnO nanorod surface as O^- or O^{2-} by capturing electrons [28–30]. Hydrogen atoms react with these oxygen ions and produce H_2O molecules $(O^-)_{ZnO} + 2H \rightarrow H_2O(g) + e^-$ and the released electrons contribute to current increase through the nanorod. The reaction is exothermic in nature (1.8 kcal mol^{-1}) [30] and the molecular water desorbs quickly from the surface [31].

4. Conclusion

In summary, an in-situ lift-out technique has been presented to fabricate single ZnO branched nanorod H_2

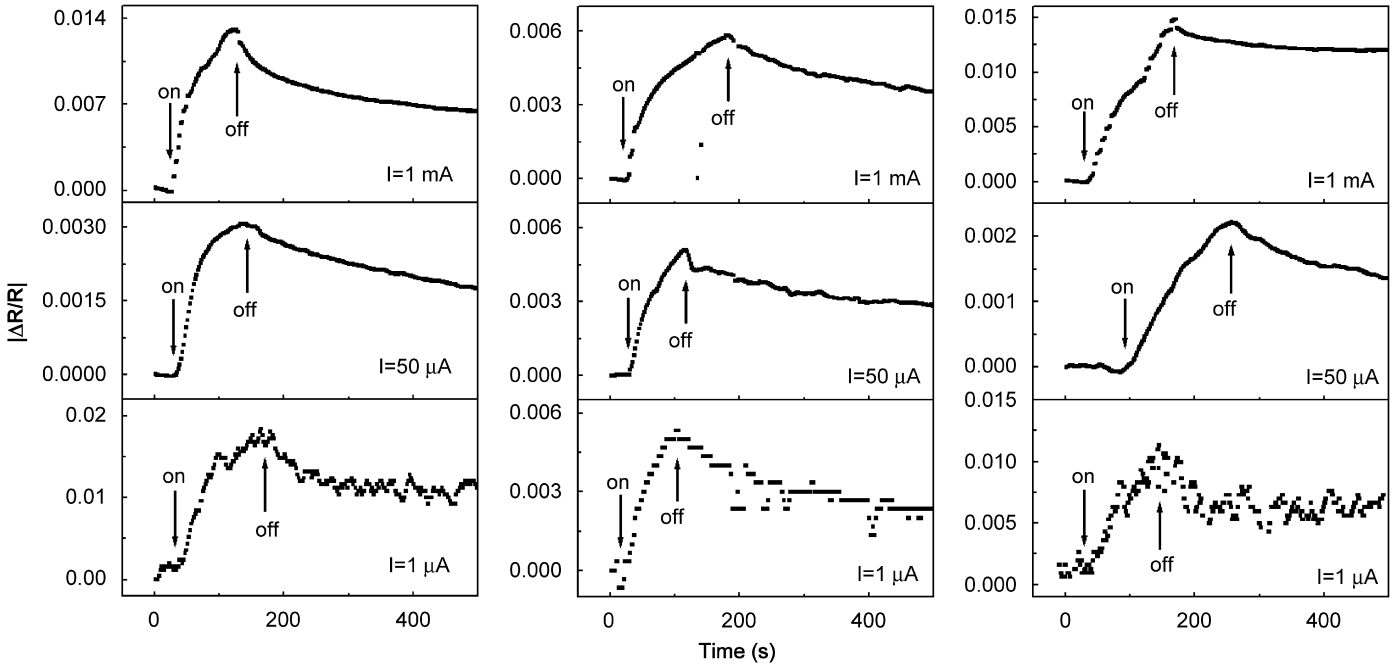

Fig. 4. The room temperature relative response $|\Delta R/R|$ of the single ZnO branched nanorod H_2 nanosensor at different current values and directions: (a) between branches 1 and 2; (b) between branches 1 and 3; and (c) between branches 2 and 3.

sensor. Self-assembled ZnO branched nanorod was synthesized through a low-temperature aqueous solution route. The main advantage of the proposed synthesis is its simplicity and fast growth rates (10 min versus several hours). Also showed an easy transfer of ZnO architectures to any substrate and pick-up by using in-situ lift-out FIB, opening the possibility of reproducibly fabrication and studying novel nanosensor and nanodevices.

The typical time taken to perform this in-situ lift-out FIB nanofabrication is 30–45 min. Also taken in the account that nanorod synthesis takes about 10 min, we contribute to overcome some obstacles for nanorods/nanowires sensor production. In details described technique makes easier to learn and apply nanofabrication steps using easy transferable nanorod (avoiding nanorods agglomerations) fabricated by our method, especially for new users/operators and will permit the highest success rate. Our success rate using this route with detailed described steps is >90%. This minimizes the total time to use FIB/SEM for the experimental nanodevice fabrication and can be extended for other specific devices. The relative resistivity changes after 50–80 s of H_2 exposure becomes stable, but did not restore to the 90% of the original level within 5 min suggesting a relatively longer recovery time, in comparison with the response time of ~1 min. The fabricated single ZnO branched nanorod sensor has a relatively higher H_2 sensitivity (~2%) comparable to the multiple ZnO nanorods-based sensors. Also we found gas selectivity to several common gases like O_2, CH_4, CO, and LPG considering that relative response $|\Delta R/R|$ is less than 0.02% in the same conditions. This selectivity was found to be useful for further development of H_2 nanosensor at room temperature. The single ZnO branched nanorod sensor can operate at low power conditions.

Acknowledgments

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