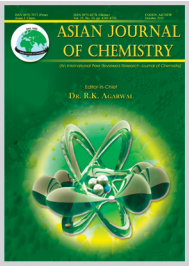
Asian Journal of Chemistry; Vol. 24, No. 5 (2012), 2087-2089

ASIAN JOURNAL OF CHEMISTRY

www.asianjournalofchemistry.co.in



Enhancement of H_2 Sensing *via* Silica Medium Embedded with Nano Platinum as Gas Sensor

H. GHORBANI[1], E. BINAIAN[1,*], H. TAYEBI[1], A. SHOKOOHIRAD[1] and M. GILAKI[2]

[1]Department of Engineering, Qaemshahr Branch, Islamic Azad University, Qaemshahr, Iran
[2]Chemical Engineering Department, Faculty of Engineering, Science and Research Branch, Islamic Azad University, Tehran, Iran

*Corresponding author: Tel/fax: +98 111 2287828; E-mail: ehsanbinaian@gmail.com

(*Received*: 6 May 2011; *Accepted*: 12 December 2011) AJC-10834

A new permeable silicon sensor with nano structures of platinum as hydrogen sensing layer is accessible in this study. This sensor works at room temperature. p-Type silica substrate is prone to permeable silica etching. The substrate is coated with a thin layer of platinum and annealed at 800 ºC. This results in a quantity of platinum getting oxidized on permeable silica and slender platinum oxide layer appearances on the exterior of the substrate. The sensor was tested in the range of 0-1.5 % H_2. The sensor responded in real time. Dissimilar conformist thin film-based resistive hydrogen sensors this sensor showed an opposite association along with increased hydrogen concentration *versus* resistance.

Key Words: Nano, Platinum, Permeable silica, Sensor, H_2.

INTRODUCTION

Many research centres such as NASA which employ great magnitude of hydrogen and expansion of the technology, have draw round a comprehensive presentation decisive factor for an satisfactory hydrogen sensor[1]. Plentiful move toward are being examined to expand these hydrogen sensors, counting sol-gel-based sensors, thin-film-based sensors, oxide-based sensors and audio wave sensors. Lately sensors with the based of nanowire for sensing hydrogen have reported[2]. Nevertheless they lack the sensitivity required and do not respond to small concentration of hydrogen. Furthermore, the performances used to manufacture these nanowire sensors necessitated complex trial, such as the transport of nano-structures and their prearranged congregation[2,3]. These manufacturing methodologies have high price and don't look like appropriate for profitable production. Permeable or porous silicon substrates have been engaged in the earlier period to construct functional hydrogen sensors[4,5]. By employing permeable silicon substrates to construct sensors is useful for the reason that bulk amount of the substrate can be shaped through effortlessness. In the current sensor, platinum was selected as the dynamic sensing element as it can adsorb up to 1000 times than volume of hydrogen gas. The adsorption of hydrogen reason a bulge of the platinum altering its electrical property. Current paper presents a simple to make, resistance-based permeable silica sensor with platinum nano-structures as the sensing layer that do something in reply to small concentrations of hydrogen. The permeable silica etching process is a restricted electrochemical etching process that consequences in isles of silica, nanometers separately. The thin layer of platinum on the surface of these silica isles, perform something as sensing layer. The permeable silica etching exchanges the silica surface to a layer with very high electrical impedance. As platinum on pinnacle of this high impedance layer absorbs hydrogen, its volume expands fetching it in get in touch with adjoining platinum, noticeably plummeting this impedance. The amend in impedance is connected to hydrogen concentration. The great surface to volume relative amount in platinum nanoparticles in this sensor exploits the hydrogen adsorption region, whilst minimizing the diffusion of hydrogen owing to reduced width of the particles. This consequences in more sensitivity, quicker response and shorter desorption times.

EXPERIMENTAL

The permeable silicon substrate was made-up from a p-type (100) silicon wafer of low resistively (0.001-0.004 Ω cm). The wafer was anodized in an electrochemical etching solution consisting of 20 % HF/methanol/H_2O. The wafer and platinum cathode were located in a Teflon leap. The surfaces of silica substrate and platinum cathode were kept analogous to each other and the current flow in the etchant was usual to the wafer surface. The wafer was etched at a existing density of 25 mA/cm^2 for 1 h. The wafer was then detached from the etching bath, washed with water and cleaned. This resulted in formation of an array of nanopores normal to the wafer, all being analogous to each other. Fig. 1 illustrates the (a) surface and (b) cross-sectional SEM of the wafer behind permeable silica etching. Fig. 1a exemplify the allocation of the nanopores.

It was obviously that the average pore width is about 10 nm. Fig. 1b explains that the pores are leaning normal to the wafer and branching. The branching outcome in an augment is baseline resistively. The permeable-silica bulk silica-interface is obviously noticeable. After that, the wafer is placed in an electron-beam evaporator and vacuum is stabilized at 10 Pa. Four nanometers of platinum is deposited over the permeable silica side of the substrate. The platinum thin film is then stabilized on the permeable-silica surface from side to side an annealing. A tailored annealing cycle is used to make sure no platinum reduction takes place on the substrate. The sample is placed in a tube heater and annealed at 1000 ºC for a 50 min.

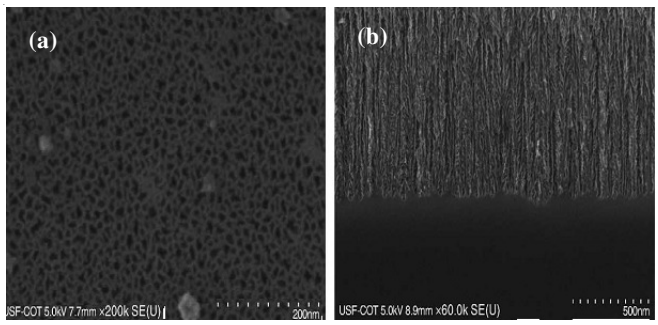


Fig. 1. (a) Surface and (b) cross-section of the substrate right away after permeable (electrochemical) etching. The etch process can be controlled to adapt the pore diameter and porosity of the substrate. The deepness of the pores is able to be tainted to change baseline resistively

RESULTS AND DISCUSSION

Energy dispersive spectroscopy (EDS) spectrum of obtained sensor was in use to conclude the analytical composition of the sensing interface. Table-1 showed the energy dispersive spectroscopy information for the film. As illustrated, the sensing surface is prosperous in oxygen excluding diverse from the standard silica. The process of compound configuration is the next.

TABLE-1
ENERGY DISPERSIVE SPECTROSCOPY
SPECTRUM OF THE SENSORE SURFACE

Element	Wt %	At %	K- ratio
O	31.5	46.3	0.1558
Si	59.3	50.5	0.5887
Pt	9.2	3.2	0.0672
Total	100.0	100.0	-

The bare silica on the surface of the pores oxidizes upon annealing. Moreover, the platinum covering the silica oxidizes to platinum oxide. The additional platinum deposited on the surface remains in the native form. While the platinum directly absorbs hydrogen, platinum oxide gets reduced by hydrogen and possibly will return to platinum and donate to the sensing of the hydrogen. In this film processes the reduction of platinum oxide to platinum takes place at 120 ºC. The current sensor is operating at room temperature and the likelihood of room temperature change is small. The sensor was tested in the described setup and resistivity revolutionize was experiential at different percentages of hydrogen in a nitrogen environment in the midst of 0 and 1.5 % hydrogen. The sensors baseline resistivity was deliberate to be 2.20 M Ω cm. This big baseline resistivity conflicted to that as of the starting wafer is steady with the formation of oxide and the permeable nature of the substrate. The changing in resistivity as a task of hydrogen concentration in nitrogen is shown in Figs. 2 and 3. The left side of Figs. 2 and 3 showed the percentage revolutionize in resistivity calculated as $\frac{\rho - \rho_{min}}{\rho_{max} - \rho m_{min}} \times 100$.

where ρ is resistivity, ρ_{min} the resistivity at saturation and ρ_{max} the baseline resistivity (per cent hydrogen = 0) and the right hand side showed the per cent of hydrogen in nitrogen. The figures demonstrate that the sensor responds to concentration revolutionize of hydrogen in genuine time together with rising concentrations and declining concentrations. The sensor rejoinder is considerably enhanced than those reported earlier[4]. The permeable silica pattern is the input to this stability and sensor presentation.

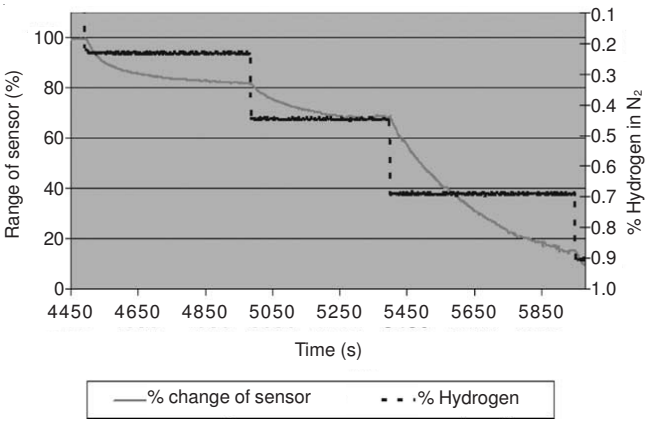


Fig. 2. Retort of platinum-based sensor at what time percent hydrogen is greater than before from 0 to 1% in 4 steps. Per cent hydrogen is exhibit in opposite order (right hand scale square steps) to emphasize curve dependence at 300 K

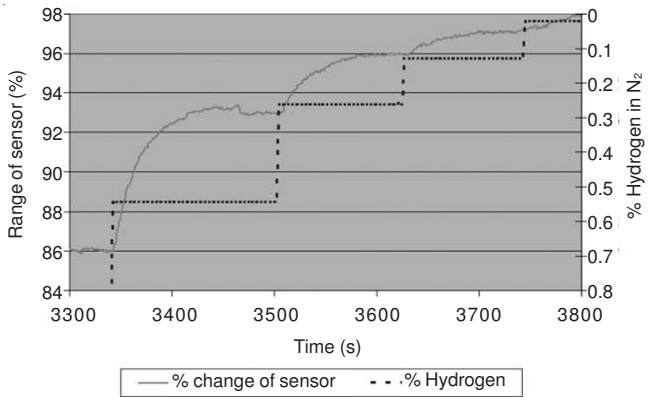


Fig. 3. Response of platinum-based sensor at what time percent hydrogen is begin at 0.8 % then reduce in four steps. Per cent hydrogen is displayed in opposite order (scale on the right square steps) to emphasize curve dependence at 300 K

The permeable silica pattern supplies two targets; it considerably augments the surface region for adsorption of the gas and concurrently augments the baseline resistivity of the permeable-silica film by eliminating the silica and generates voids. It look like that once properly sized and spaced, the particles contact each other at what time bareed to hydrogen

considerably reducing its resistance. This current work trial propose a close relationship along with the volume of the pores/particles and the reply of the sensor. To revision the sensor answer and adsorption and desorption rates, the hydrogen was pulsed back and onward surrounded by zero and a haphazard value. The revolutionizing in resistivity and the concentration of the sensor of the feed gas are shown in Fig. 4. It can be seen that the adsorption and desorption times of the sensor are less than 2 s additionally it can be illustrated that the sensor uphold a stable baseline following recurring cycling. This stability and lack of float is an significant characteristic that is critical for industrial requests. The sluggish rate of amend at the bottom and top of the resistivity curves given in Fig. 4 recommend a shift of hydrogen from effortlessly admission surface particles to less reachable pore particles. This surveillance advised that the deepness of the pores possibly will also influence the reply time of the sensor. This mechanism is part of a continued investigation.

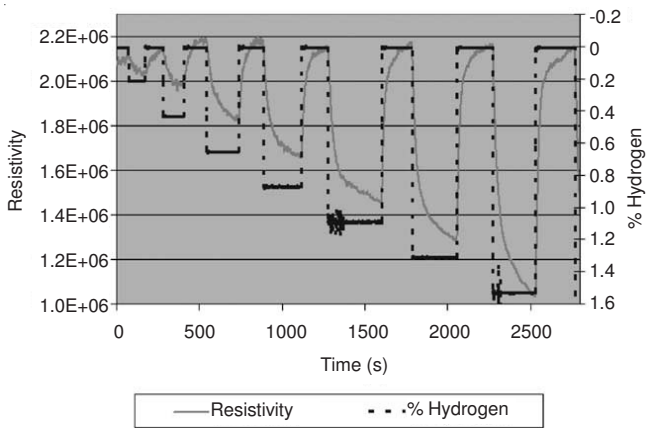


Fig. 4. Response of platinum-based sensor, when per cent hydrogen is started at zero and gradually more pulsed to diverse concentrations among 0 and 1.5 % and then back to zero. Per cent hydrogen is exhibited in inverse order to emphasize curve dependence at 300 K

Conclusion

Platinum/permeable silica sensor for detection H_2 has been constructed and experienced in the 0-1.5 % variety. It has been observed that sensors constructed with permeable silicon and platinum nanoparticles showed a reduction in resistively with regard to at what time exposed to hydrogen. The platinum nanoparticles reduce the adsorption and desorption times, which enhances the sensitivity, sensing and renewal times of the sensor. The elevated selectivity, cost usefulness and effortlessness of manufacturing, platinum/permeable silicon has the potential of flattering a truthfully worldwide hydrogen sensing system.

REFERENCES

1. F. Petra, W. Marion, C.B. Mihaela, P. Marlis, K. Deistung and B. Erika, *Sens. Actuators B*, **100**, 151 (2004).
2. F. Favier, E. Walter, M. Zach, T. Benter and R. Penner, *Science*, **293**, 2227 (2001).
3. M. Zach, K. Ng and R. Penner, *Science*, **290**, 2120 (2000).
4. V. Polishchuk, E. Souteyrand, J. Martin, V. Strikha and V. Skryshevsky, *Anal. Chim. Acta*, **375**, 205 (1998).
5. K. Luongo, S. Aravamudhan, S. Bhansali, A Novel Approach to Integrating nanowires to Transductors, without Surface Contamination, Fifth Topical Technology Workshop on MEMS, Related Microsystems and Nanopackaging, Boston, November, pp. 20-22 (2003).