Development of Chemical Sensors for H$_2$ Detection

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Abstract

In the last decade there has been an increased interest in using palladium (Pd) nanostructures for hydrogen (H$_2$) sensing devices. Nanostructured based sensors promise shorter response times and higher sensitivity due to their high surface to volume ratio. With this in mind, an anodic alumina membrane (AAM) Pd nanostructured based sensor has been developed and tested. The Solid State Reduction method used formed a combination of a thin film and nanowires in the AAM. The fabrication method and preliminary test results are discussed.

Background

- Hydrogen occupation of the interstitial sites in the Pd lattice of continuous films results in an increase in the resistance [1]: Scattering of conduction electrons.
- The $\alpha$ to $\beta$ phase transition provokes structural changes in the Pd lattice causing the resistance of the film to decrease as the lattice expands: Percolation effect [2].
- Anodic aluminum oxides (AAO) act as exceptional substrates for H$_2$ sensing devices because on top of providing a rough surface with large surface area for the formation of nanostructures, it is also weakly conductive providing means for better electronic measurements [3].

H$_2$ Leak Sensor Technology (existing technology)

- Accurate leak detection for safety applications.
- Microfabricated using MEMS-based technology for minimal size, weight and power consumption.
- Highly sensitive in inert or oxygen-bearing environments, wide concentration range detection.
- Two sensor system for full range detection: from ppm level to 100%.
- “Lick & Stick” approach.

Synthesis and Characterization

- Solid Pd(NO$_2$)$_2$xH$_2$O is used as a precursor.
- The precursor was deposited over the AAM and left to absorb the humidity from the air.
- After the metal salt has absorbed enough water, it was spread evenly over the surface of the membrane.
- To reduce the metal, solid NaBH$_4$ was put in contact with the membrane on the opposite side to where the salt was spread.

Sensor Response to H$_2$

- Current increases initially exposed to H$_2$.
- During N$_2$ flow, current increases and then decreases as time passes.
- Exposure to H$_2$ in the second cycle causes the current to increase.
- If during N$_2$ flow, at the point where the current reaches a maximum the sample is exposed to H$_2$, the current decreases.
- Scattering effect governs the response.
- Addition of silver epoxy to the contact points improves recovery of the sensor.
- Current is not stable under H$_2$ flow: drifting.
- The behavior of the sensor is improved by imposing the scattering effect.

Electrode Deposition

- Gold (Au) electrodes deposited by electron beam evaporation could provide better contacts and thus improve reliability. The Au electrodes are 1µm thick.
- The metal precursor is then spin coated at 1500 rpm on the side of the sample opposite to the Au electrodes.
- A solution of 0.2 M of NaBH$_4$ is spin coated at 1500 rpm on the electrode side and washed with distilled water.

Conclusions

- Two sensing mechanism govern the response of the samples.
- Eliminating the phase transition could improve reliability.
- Using metal (Ag) epoxy on the contact points improves the recovery of the sensor.
- Gold electrodes greatly improve the response of the sensor.

References


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