This is the published version:


Available from Deakin Research Online:

http://hdl.handle.net/10536/DRO/DU:30055923

Reproduced with the kind permission of the copyright owner.

Copyright: 2009, IEEE.
A Pt/oriented-C hydrogen gas sensor  

A. Moafi, J. G. Partridge and D. G. McCulloch  
Applied Physics, School of Applied Sciences, RMIT  
University, Melbourne, Australia.  
Ali.Moafi@rmit.edu.au  

A. Z. Sadek, K. Kalantar-zadeh and W. Wlodarski  
School of Electrical and Computer Engineering, RMIT  
University, Melbourne, Australia.  

Abstract—A Pt/C heterojunction has been fabricated and tested as a conductometric hydrogen gas sensor. The carbon layer, deposited between planar Au substrate contacts using a filtered cathodic vacuum arc (with an applied substrate bias of -500 V), consisted largely of vertically oriented graphitic sheets. These sheets have good through-film electrical conductivity and contributed to a low device resistance of ~ 60 Ω. The Pt layer (of 3 nm thickness) was deposited using a Gatan Model 682 Precision Electron Coating System (PECS). A change in the device resistance of > 2% was exhibited upon exposure to 1 % hydrogen gas (in synthetic, zero humidity air) at room temperature.  The time for the sensor resistance to decrease by 2 % under these conditions was 60 s and the baseline (zero hydrogen exposure) resistance remained constant to within 0.03% during and after the exposure tests.

I. INTRODUCTION

Developments in hydrogen fuel cell technology [1] have led to increased interest in devices which can effectively monitor the concentration of hydrogen gas in air. This is largely because concentrations of hydrogen gas in air above 4% are highly explosive [2] and therefore rapid leak detection is a mandatory safety feature.

Hydrogen gas sensing has been demonstrated by functionalized carbon nanotubes (CNTs) [3,4] and graphene [5]. These materials offer good sensitivity and low power consumption and importantly, they can offer these attributes at room temperature, thereby simplifying the construction of sensor chips and associated circuits. Recently, the ultimate in sensitivity has been achieved using graphene. Single molecules adsorbing to and desorbing from graphene flakes have been sensed [5].

Whilst functionalized CNT and graphene sensors have demonstrated high performance, their preparation methods typically include the manipulation of individual sheets/tubes, precluding compatibility with large-scale silicon device processing. The aim of this work was to produce a high performance hydrogen sensor from a readily deposited carbon film, patterned simply in a CMOS compatible process. Carbon thin films can be prepared with a wide variety of mechanical and electrical properties depending on the energy of deposition [6]. A filtered cathodic vacuum arc (FCVA) was used to deposit the carbon film for the sensor as this system allows fine control over the deposition energy via an applied substrate bias. An eventual aim of the investigation is to determine the gas sensing response as a function of the deposition energy and in particular, the microstructure of the carbon film.

II. SENSOR DESIGN

The layout selected to produce the sensors consisted of a P-type Si (20 × 20 mm²) substrate with a 1 μm thick layer of SiO₂ separating dual Ti/Au planar contacts from the Si substrate.  The planar contacts were deposited in a standard (DynaVac) thermal evaporator using a photo-plot mask and optical lithography. A second layer of photoresist was spun and exposed/developed to define the area of the Pt/C layers.

The carbon film was energetically deposited from a 70 mm high purity graphite cathode within the FCVA system. The deposition conditions for the C film were: Arc current = 70 A, arc voltage = 28 V, substrate bias = -500 V and substrate temperature = 30 °C. The substrate bias was selected to produce a carbon film consisting of a high density of graphitic sheets aligned perpendicular to the substrate [7]. After the carbon film was deposited onto the contacted substrate, a Pt layer of thickness 3 nm was deposited using a Gatan Model 682 Precision Electron Coating System instrument. Finally, a lift-off process was used to reduce the Pt/C film to the dimensions indicated in Fig. 1.

![Figure 1. Schematic cross-section and plan view diagrams of the sensor.](image-url)
III. GAS SENSING MEASUREMENTS

The sensing measurements were performed in a multi-channel gas test chamber (see Fig. 2). A gas calibration system, with computer interfaced mass flow controllers, was used to expose the sensor to different concentrations of hydrogen gas. The sensor resistance was measured continuously using a multimeter (Keithley 2001) interfaced with a personal computer. The total flow rate was kept constant at 200 sccm and synthetic air (certified zero humidity) was used as the reference gas. The various concentrations of gas were inlet as pulses of duration 1800 s with zero exposure periods of 3600 s separating them.

IV. GAS SENSING RESULTS

Figure 3 shows the resistance of the Pt/C heterojunction during exposure to various concentrations of hydrogen gas in synthetic air. These measurements were performed at room-temperature. The resistance decreased by ~ 2.5 % when the hydrogen concentration in air was 1% (well below the ~4% combustion threshold) and the response to the varying gas concentrations was reproducible. The response and recovery times are longer than those obtained from some CNT sensors [4] however upon exposure to 1 % hydrogen, the resistance of the sensor changed by > 2% within 60 s.

We attribute the decrease in resistance of the back-to-back heterojunctions to a lowering of the potential barrier between the oriented carbon and the platinum [3]. (The work function of Pt is known to reduce upon exposure to hydrogen [8,9]). At higher operating temperatures, the sensor resistance decreased more upon hydrogen exposure (see Fig. 4) however, instabilities in the baseline (zero hydrogen exposure) resistance occurred. This behavior suggested that hydrogen storage was occurring in the oriented carbon film, an effect commonly observed in graphitic carbon and CNTs [10]. As shown in Fig. 3, the baseline resistance at room temperature was stable to within 0.3 %.

![Figure 3. Room temperature resistance of the back-to-back Pt/oriented-C heterojunctions in the presence of various indicated hydrogen concentrations in synthetic air.](image1)

![Figure 4. Percentage change in resistance of the Pt/oriented-C heterojunction to 0.13% hydrogen gas at various operating temperatures.](image2)
V. CONCLUSION

We have demonstrated a simple fabrication method to produce gas sensing heterojunctions consisting of carbon and Pt films supported on a contacted substrate. The carbon films were deposited using a filtered cathodic vacuum arc and consisted largely of perpendicularly oriented graphitic sheets.

A 2% change in the device resistance in the presence of 1% hydrogen in air compares favorably with published results for functionalized CNTs. The results suggest that the sensor would be suitable as an alarm device to indicate the presence of combustible concentrations of hydrogen. Further optimization of the geometry of these devices is underway and is expected to yield improved performance.

ACKNOWLEDGMENT

The authors wish to thank Peter Rummel for assisting in maintenance of the FCVA and PECS deposition systems.

REFERENCES


