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Ultra low power temperature compensation method for palladium nanowire grid

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Abstract

An interface was designed to independently measure two palladium nanowires on one substrate to decrease the cross sensitivity for temperature. The circuit was evaluated in a test chamber where temperature, humidity and hydrogen concentration could be controlled. The palladium sensors showed no response to humidity's as high as 80%, but did show an expected response to temperature.

One of the nanowires was covered with a 2-Hydroxyethyl methacrylate based compound to prevent hydrogen from reaching the wire. The compound was dried by a UV source and tested in chamber for comparison with previous measurements. The results shows that temperature effects can be reduced by a digital signal processing algorithm without measuring temperature near or at the substrate. With this method no additional temperature probes are necessary making this solution a candidate for ultra low power wireless applications.

Keywords: DSP; digital signal processing; nanowire; palladium; DEA; amplifier; ultra low power; temperature compensation

1. Introduction

In the near future an increased demand is expected for ultra low power hydrogen sensors when the utilization of hydrogen powered devices will rise. Hydrogen sensors that will be used for safety reasons are likely to work in a wireless sensor grid. For this reason power management is a serious issue. With this method a 1‰ detection limit is achieved even at low temperature fluctuations with conventional electronics.

Several research groups are reporting the use of palladium nanowires as a sensing material[2-4] and some reported a response to temperature variations [3]. Offermans *et al.* report a change in the temperature coefficient of resistance of 3.9 ±0.1 x 10^{-4} in 2.4% H_2 which changed to 5.6±0.2 x 10^{-4} in N_2. This change in temperature coefficient suggests a linear digital correction algorithm at high concentrations of hydrogen. Classical temperature compensation can be done by measuring temperature nearby or on the substrate but involves integration and calibration. This method uses

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a nanowire on the same substrate for compensating the temperature effects in the readout. The data suggest that the system does not need calibration for trash hold detection depending on the process of chip[1] .

2. Experimental

The palladium nanowires are fabricated on a silicon wafer with SiO_2 as an insulation layer by using deposition and etching under angles (DEA) [1]. In this work each chip consists of a double array of 14 Pd nanowires with different lengths (Fig 1). Two identical wires were selected with a resistance of approximately 8300Ω. The resistance of the wires differ within a 100Ω range and explain together with resistor variations the difference in the steady-state voltage of a single wire.

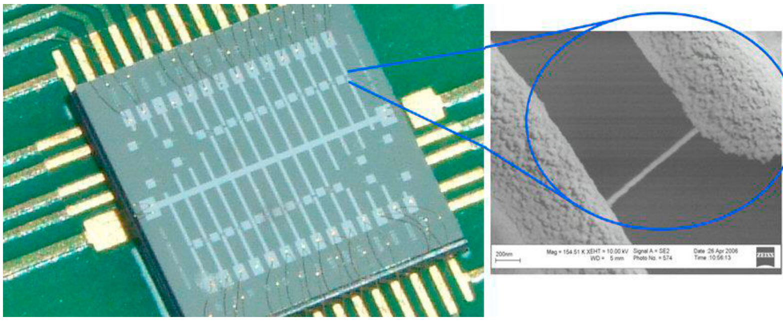

Fig. 1. Wedge bonded palladium nanowires on a silicon wafer in flip chip directly bonded on the PCB with a close up of a Pd wire

One half of the nanowires was covered with a 2-Hydroxyethyl methacrylate based compound to temporarily prevent hydrogen from reaching the wire. In time Hydrogen will reach the wire and the covered wire will respond.
The wires were measured with a differential amplifier connected to a 12Bit AD differential analog to digital converter . The reference voltage of this converter was used to power the bridge to minimize fluctuations of the power line. The bias current was set to 50uA at an excitation voltage of 0.4V resulting in a response time of approximately 15 seconds [1]. Conversion and power control was performed by an embedded processor and the values recorded by computer intervention. Measurements were done in a custom built measurement chamber. Temperature is computer controlled using a 100W Peltier element and the humidity is set using a bubbler and computer controlled valve. The exit valve is controlled by a pressure sensor keeping the ambient pressure and chamber pressures equal. Furthermore the amount of H_2 is administered using a syringe. The gasses are kept in constant motion using a small fan to ensure a homogeneous mixture in the chamber.

3. Results

The experiments were done in a nitrogen environment with periodic admission of 2‰ H_2. The 7.5 liter test chamber was flushed when the Pd wire reached a steady state. The chamber temperature was changed between 19.3°C and 24.1°C at an interval during 150 minutes as shown in Fig 2 and 2‰ H_2 was added two times during the largest temperature change. One admission was done at the falling slope of the temperature curve and one at the rising slope. Samples were taken on a 6 seconds based interval of the open Pd and the covered wire as shown in Fig 3.

The algorithm that was developed for this application corrects the amplitude of the compensation wire by subtracting its DC component. The simplified formula shows no redefining of the baseline but only the primary definition:

$$Pdcorrected_i = S2_i - \left[\left[\frac{1}{i} \sum_i S1_i \right] \frac{\min(S2) - \max(S2)}{\min(S1) - \max(S1)} \right]$$

Were S1-2 is the palladium nanowire sensor data and min-max are embedded functions. The min-max functions need to be build up in the processor during the sampling process. The best candidate for reference data is the covered wire due to its temporal immunity to hydrogen. Fig 4 shows the corrected response together with the response of both wires.

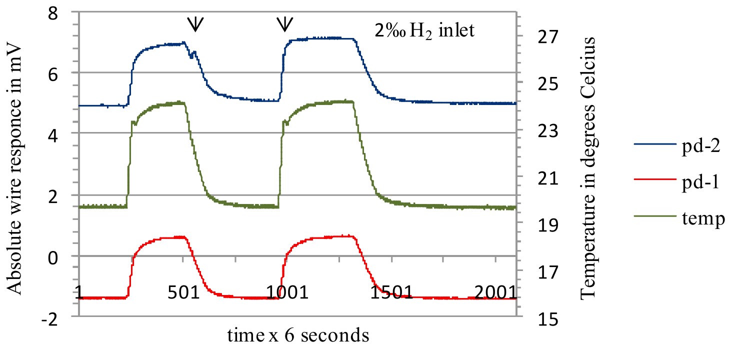

Fig. 3 Temperature variation and absolute response from the Palladium wires.

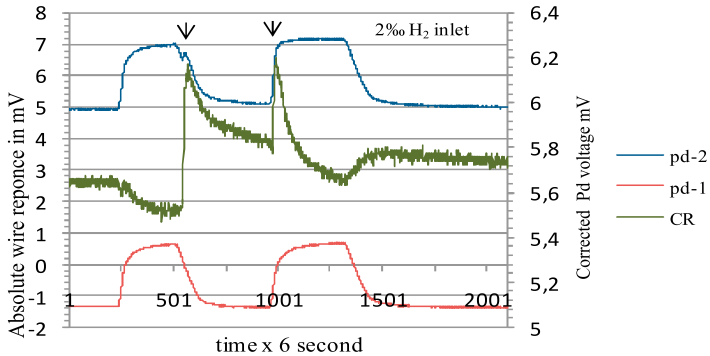

Fig. 4 Corrected response from two palladium wires

Experiments show a drastic improvement of the detection limit with small temperature fluctuations (<5°C) combined with small (≤2‰) H_2 variations making the response data suitable for threshold detection with a microcontroller. Data show the possibility to isolate a 500µV signal from a 2mV unwanted signal caused by temperature fluctuations.

4. Discussion

As shown in Fig 4, temperature variations can be detected by a relatively simple setup. This method was followed so that the DSP algorithm could be performed with as little overhead as possible making it a suitable candidate for implementation on low-end 8 bit microcontrollers. The application in which this principle will be used aims on long periods (>2 year) of use. This means that even calculations done by the microcontroller have to be kept as short as possible from an energy perception. Apart from a heartbeat of the wireless system to ensure a correct link every predefined period this setup will only start transmitting a H_2 value when a certain threshold is exceeded. Further work includes the writing microcontroller code and work on power saving by switching the bridge excitation voltage. The use of a detection limit without having to calibrate the system needs to be investigated.

5. Conclusion

The experiments have shown that temperature compensation of palladium nanowires using a grid of identical wires is possible. The detection limit of 2‰ H_2 can be met using commercial available electronics while maintaining the ultra low power issue. The DSP algorithm can be executed with a microcontroller with limited resources. This means that a serious step is made towards an ultra-low power wireless hydrogen detection system.

Acknowledgements

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