# Smart Chemical Sensor application of ZnO Nanowires grown on CMOS compatible SOI Microheater platform

Jonghyurk Park*, Junghwan Huh**, S.Z. Ali***, Florin Udrea***, J.W. Gardner****, G.-T. Kim**, Sunglyul Maeng* and W.I. Milne***

*ETRI, 161 Gajeong-Dong, Yusong-Gu, Daejeon, Korea 305-700
**Department of Electrical Engineering, Korea University, Korea 136-701
***CAPE, Electrical Engineering Division, Department of Engineering, Cambridge University, 9 JJ Thomson Ave., Cambridge, CB3 0FA, UK
****School of Engineering, University of Warwick, Coventry, CV4 7AL, UK

## ABSTRACT

Smart chemical sensor based on CMOS(complementary metal-oxide-semiconductor) compatible SOI(silicon on insulator) microheater platform was realized by facilitating ZnO nanowires growth on the small membrane at the relatively low temperature. Our SOI microheater platform can be operated at the very low power consumption with novel metal oxide sensing materials, like ZnO or SnO2 nanostructured materials which demand relatively high sensing temperature. In addition, our sol-gel growth method of ZnO nanowires on the SOI membrane was found to be very effective compared with ink-jetting or CVD growth techniques. These combined techniques give us the possibility of smart chemical sensor technology easily merged into the conventional semiconductor IC application. The physical properties of ZnO nanowire network grown by the solution-based method and its chemical sensing property also were reported in this paper.

***Keywords***: ZnO nanowire, CMOS compatible SOI microheater, smart chemical sensor and sol-gel growth.

## 1 INTRODUCTION

CMOS compatible microhotplate gas sensors enables us to develop the low cost, low power and fast switching devices which can be used in battery operated and wireless applications.[1-3] Common semiconducting-type gas sensors which should be operated at relatively high temperatures, includes metal oxide sensing materials on the membrane heated by a microhotplate structure. CMOS compatible microhotplates typically utilize either polysilicon or MOSFET heaters, but polysilicon has poor long-term stability, while MOSFETs cannot operate at higher temperature required by current metal oxide sensors. Although platinum, however, is commonly used for microheaters due to its high operating temperature and good long-term thermal stability, platinum is not CMOS compatible. Hence, there is manifestly strong need for a high temperature CMOS compatible microheater platform with good long-term stability. In fact, tungsten is an interconnect metal used in high temperature SOI-CMOS process and therefore ideal for CMOS compatible high temperature gas sensors. In addition, being CMOS compatible and manufactured by a commercial foundry, it can be easily integrated with the drive and control circuitry in a single chip.

As the smart sensing materials, metal oxide sensing materials were investigated for a long time. Futhermore, the nanostructured materials can be more effective sensing materials in terms of sensitivity and response time since they have large surface to volume ratio compared with the conventional film structure. Especially, ZnO is most promising material because of its various interesting physical and chemical properties. Various kinds of chemical sensors were reported using ZnO sensing materials.[5,6] For the practical reason, nanowire networks can be better candidate compared with single nanowire device.

In order to realize the smart chemical sensor based on CMOS compatible SOI microheater platform, we have to deposit densely noble metal oxide nanowire sensing materials on the small SOI membrane at the relative low temperature. Hence, we tried to utilize solution-based ZnO nanowire networks selectively grown on the SOI membrane and characterize this fully CMOS compatible smart chemical sensor..

## 2 EXPERIEMNTALS

### 2.1 CMOS compatible SOI Microheater platform fabrication

Circular microhotplates have been designed with approximately 5μm thick SOI membrane having diameters of about 600 and 300 μm and heaters of 150 and 20 μm, respectively. The hotplates have been fabricated using an XAFB (Germany) SOI-CMOS fabrication facility using tungsten metallization process and back etched to the buried oxide at Silex (Sweden), by DRIE (deep reactive ion etching). In collaboration with the foundry, we have developed a very accurate back to front alignment and minimized the undercut effect of DRIE. The heaters have excellent reproducibility and very low DC power consumption (34mW for the large heater and 12mW for the small heater at 600℃). Figure 1 shows the schematic diagram of our microheater with front passivation layer on top of the membrane, the power consumption and time-response to reach 600℃ of the larger membrane. We believe this is the lowest power consumption of microhotplate reported. The heaters have very fast response times (for small heater 10-90% rise time of 2.0 ms and fall time of 6.2ms, for larger heaters 10ms and 20ms, respectively). These outstanding characteristics allow more reliable operation of our microheater sensor platform with very fast switching electronics even at higher temperature in excess of 400℃.

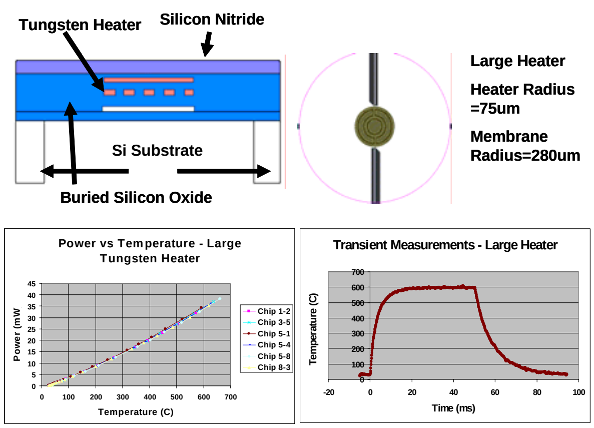


Figure 1: Schematic diagram of microheater and its typical performance

The tungsten sensing electrodes were designed having a interdigitated pattern as shown in the Figure 2. To prevent the short circuit formation between electrodes and protect other interconnecting metal lines, SiNx passivation layer was deposited over the metal electrodes and only part of the metal electrodes was open using dry etching process (RIE). Under these sensor structures, we chose the proper technique to deposit the smart nanostructure sensing materials on the non-planar sensing electrodes. More details can be found in the previous work.[4]

## 2.2 Solution-based ZnO Nanowire Growth

To deposit the sensing materials on the microhot plate, we used the solution-based nanowire growth method. For example, Zinc nitrate hexahydrate (HMTA, Zn(NO3)2) 6H2O) and Methenamine (HMTA, C6H12N4) are dissolved in the distilled water (MilliQ, 18.2MΩ cm) to a concentration of 0.01M. The microhotplate samples were placed in the mixed solution consisting of an equimolar ingredients and heated at a constant temperature of 95℃ for several hours. Then, ZnO nanowires were found to grow on the ourter area over the sensing electrodes patterned on the microhotplate and others could be removed using lift-off process.[4]

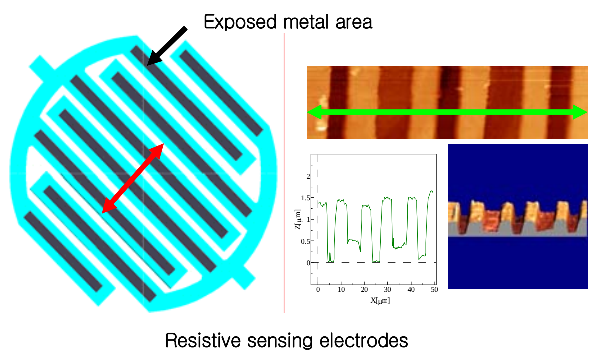


Figure 2 Design of tungsten IDT sensing electrodes and AFM images showing 3D structure with SiNx passivation layer

The selective area patterning was performed using high-resolution E-beam Lithography as follows. Double layer electron beam resist, for example, copolymer (MMA(8.5)MAA) and PMMA (polymethyl methacrylate) 950K were very carefullly spin-coated as thick as 120nm and 300nm, respectively. Then, electron beam was irradiated on the microheater and the exposed region was developed to bring out the microheater in a circular shape.

## 2.3 Physical and Electrical Characterization

Optical images of ZnO nanowire networks grown on the microheater are shown in the Figure 3. The ZnO nanowires were grown only over the membrane with heaters, which is importance to reduce the power consumption of the sensors. We observe the dark region covered with much denser nanowires which can be clearly seen in the SEM image. The shape of individual ZnO nanowires was found to be very straight, but sometimes packed densely to form a cluster. The typical size of ZnO nanowires varies up to a few hundred nanometers in diameter and several microns in length. XRD data showed the nanowire growth was not preferential in the direction compared with the substrate.

These nanowires were found to be mechanically stable even under solution process like stirring.

Electrical measurement showed the typical resistance of a few Mohms at the room temperature which is found to be common in the undoped ZnO nanowires. As previously known, ZnO nanowires have a n-type semiconductor characteristic and are expected to have a much less resistance at higher temperature like 500 ℃. In the atmospheric condition, the humidity can affect the electrical property of ZnO nanowires since ZnO is known to be sensitive to the water concentration of environment. In fact, we found the resistance of ZnO nanowires on the microheaters showed the decreasing tendency according to the temperature of the membrane but the change was not so large as expected. This means that we still have to improve the other parasitic resistances, for example, the interfacial component between electrodes and ZnO nanowires.

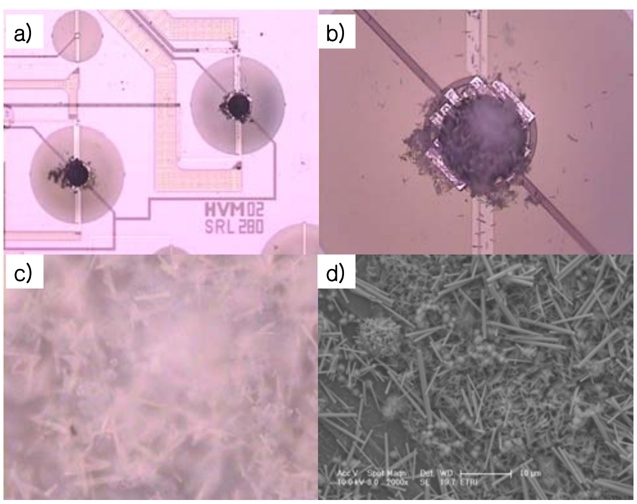


Figure 3 Optical and SEM images of solution-based ZnO nanowire networks growth on SOI membrane.

## 2.4 Sensor Performance

To test our ZnO nanowires sensors, we investigated hydrogen gas sensing property. The hydrogen gas was balanced with dry air to simulate the realistic gas sensing environement. The hydrogen gas concentration was set to 1% of flowing gas volume which was, for example, fixed to 300 sccm(standard cubic centimeters). The temperature of the sensing element was elevated up to 500℃. Before introducing hydrogen/air gas, the gas chamber was purged by flowing dry air and maintained to be stabilized having a constant resistance of 150kOhm. The measured gas sensing property was shown in the Figure 4.

After introducing 1% diluted hydrogen gas, the resistance change was estimated about 20kOhm and it reached the minimum in about 100 seconds. When hydrogen gas was closed and air flowed again, the resistance was raised and restored the almost initial value but it took longer time compared with the falling case. In the first run, it reached the maximum in several minutes. In the repeated runs, the response curve was found to be a little bit deformed and took more times to reach the minimum which means the reliability of our sensing materials needs to be improved. Despite this small deviation, our sensor was found to work very successfully even at the higher concentration of hydrogen gas balanced with air.

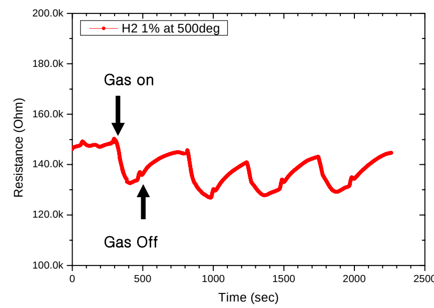


Figure 4 Hydrogen gas sensing characteristics of ZnO nanowire network sensor: H2 gas balanced with dry Air

## 3 DISCUSSIONS

Solution-based ZnO nanowires were found to be very effective sensing material on our high-temperature CMOS compatible SOI tungsten microheaters. Though the higher temperature growth techniques like CVD tend to show the same nanostructured materials of better quality in terms of size and shape controllability, these fine nanometerials are very difficult to apply to the conventional semiconductor technology platform, not to speak of the state-of-the-art CMOS IC application.. This is very important issue to integrate the smart sensor to the pre-existing IC circuitry for the future smart technology.

To enhance overall performance of our smart sensors, we have to optimize several aspects, for example, determine the proper sensing electrodes, apply the post-growth annealing process and introduce some catalytic materials (noble metals such as Pt, Pd). Some groups are reporting Ti/W electrodes covered with platinum. In this case, these materials cannot be fully CMOS compatible. Hence, we're trying to get more reliable sensor performance by applying these additional processes.


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## 4 CONCLUSION

We have fabricated the ZnO nanowire network chemical sensor based on the high temperature CMOS compatible SOI tungsten micro-heater platform. Our solution-based growth method was found to be very effective even in the sensor structure of non-planar electrodes like, in our case, deep grooved pattern. And we observed successfully the hydrogen gas sensing property of ZnO nanowire network which proves that our smart sensor technology can be very promising and powerful in the future smart sensor technology.


## ACKNOWLEDGMENT

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