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Atomic layer deposition of tin dioxide sensing film in microhotplate gas sensors

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ABSTRACT

We report the use of atomic layer deposition (ALD) to produce the gas-sensitive tin dioxide film in a microhotplate gas sensor. The performance of the device was demonstrated using ethanol, acetone and acrylonitrile as model analytes. Fast response times and low drift rates of the output signal were measured, indicating a structurally stable tin dioxide film and reflecting the capabilities of ALD in gas sensor applications. Fabrication of the microhotplate using tungsten metallization and plasma deposited silicon dioxide dielectrics is also detailed.

1. Introduction

Microhotplate gas sensors employing interdigitated electrodes covered by a gas-sensitive film have long been used as sensors for various gases [1]. The most common sensing material is tin dioxide, which is an *n*-type granular material whose electrical conductivity is dependent on the density of preadsorbed oxygen ions on its surface [2–5]. The microhotplates are used for keeping the sensing material at an elevated temperature, usually 200–500 °C [5], ionizing oxygen atoms adsorbed on the surface of the film. Reducing gases are generally sensed based on their reaction with these oxygen ions, leading to an increase in film conductivity as the ions are consumed [2,5]. The behavior is opposite with oxidizing gases. The response can be tailored for various analytes by doping the film surface with a suitable catalyst material [2–4,6].

The tin dioxide material itself can be deposited on the sensor device in several ways. Screen printing and drop coating of thick films are well established methods in the industry, but thin films by chemical vapor deposition (CVD) and sputtering methods have also been studied [1]. Thin and thick film sensing materials differ greatly in composition and morphology and therefore also in their gas response, as well as their heating power requirements.

In this paper, we report on atomic layer deposition (ALD) for depositing the tin dioxide film in a microhotplate gas sensor process. ALD differs from other vapor phase deposition methods, as the substrate is exposed to each precursor material individually, with an inert gas purge between the precursor pulses [7]. Due to this single monolayer approach to deposition, excellent control over deposition rate and film composition is possible. Multi-layer films can also be deposited simply by alternating different precursor materials. On α-Al$_2$O$_3$ (0 1 2) substrates, ALD has been shown to produce higher-quality epitaxial films than CVD [8]. While ALD tin dioxide has been used in gas sensing applications, *e.g.* [9–11], this is the first time ALD SnO$_2$ has been used in a microhotplate design.

We also investigate the use of tungsten metallization, and inter-metal dielectrics deposited by plasma-enhanced chemical vapor deposition (PECVD) as a convenient microhotplate technology for prototyping and testing purposes.

The response of the device to ethanol, acetone, and acrylonitrile vapors is tested. These are all widely used chemicals in many fields of industry, and are representative of the kind of flammable or toxic materials that are easily vaporized into the workplace air.

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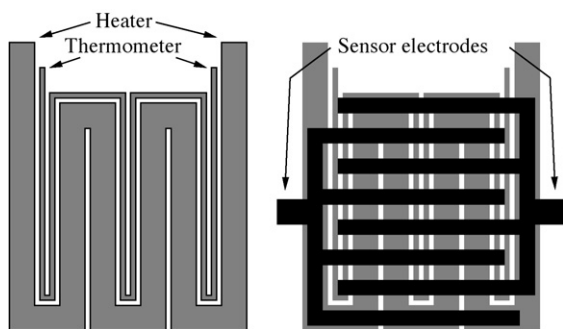
Fig. 1. The heater and thermometer resistors (*left*) and sensor electrodes (*right*) of the device.

2. Experimental methods

2.1. Sensor design and fabrication

Two sizes of sensor devices were processed on the same wafer, with active device areas of 500 μm × 500 μm and 1000 μm × 1000 μm. Both components were 3 mm × 3 mm in outer dimensions, with identical bonding pads at the chip edges.

Silicon wafers of (1 0 0) orientation were first thermally oxidized and then coated with silicon nitride in an LPCVD furnace. The tungsten heater metallization was deposited by sputtering, beginning and ending with a reactive sputtering step in nitrogen to produce a thin film of tungsten nitride above and below the metal film. Heater and thermometer resistors (Fig. 1, left) were patterned by standard optical lithography and reactive ion etching in an SF_6 plasma. A silicon dioxide intermetal dielectric was deposited by PECVD, followed by a thermal annealing step in a nitrogen atmosphere to stabilize the film. The temperature was ramped at approximately 1.7 °C/min, and the annealing temperature of 500 °C (same as the ALD deposition temperature later in the process) was held for 24 h. Interdigitated sensing electrodes (Fig. 1, right) were sputtered gold, with an adhesion-promoting film of titanium underneath. They were patterned by optical lithography and wet etching in diluted aqua regia, and the thin titanium layer was removed from the exposed areas with dilute H_2O_2 after removal of photoresist. The linewidth and spacing of the electrodes was 30 μm and 80 μm in 500 μm × 500 μm devices, and 60 μm and 160 μm in 1000 μm × 1000 μm devices, respectively.

The wafer backside was patterned by lithography and reactive ion etching of the nitride and wet etching of the oxide, to facilitate bulk etching of the microhotplate later in the process. The nitride and oxide films were patterned either simultaneously or separately to produce different suspended structures (Fig. 2).
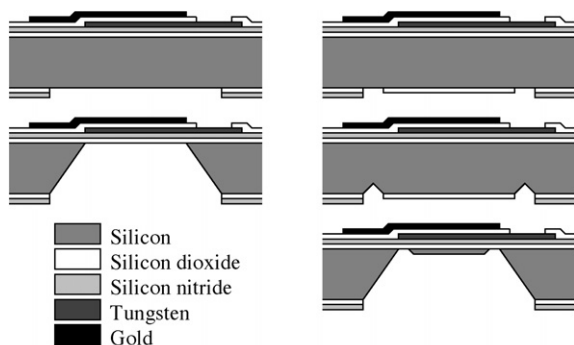

Fig. 2. A cross-sectional diagram of the microhotplate gas sensor with single-step KOH etching (*left*) and two-step KOH etching (*right*).
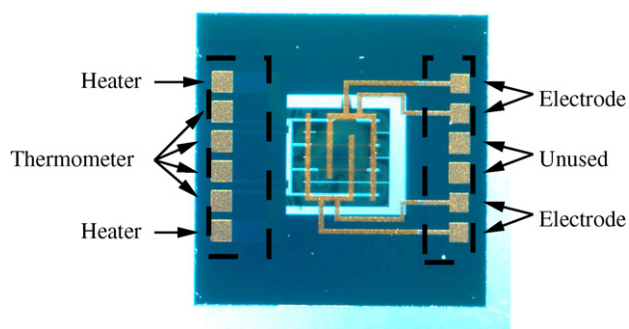

Fig. 3. The completed microhotplate chip. Double contact pads are provided for the thermometer resistor and electrodes, to facilitate four-wire resistance measurements. The dashed lines indicate areas from which the ALD-deposited SnO_2 film was removed by etching.

The gas sensing layer was deposited and patterned prior to bulk etching of the microhotplates. Tin dioxide was deposited by atomic layer deposition using tin(IV) chloride (Sigma–Aldrich, 99.995% pure) and deionized water as precursors. The precursors were temperature stabilized at 20 °C, and their own vapor pressures were sufficient to transport them in vapor phase to the reaction chamber, using 500 sccm of oxygen as the carrier gas. The reaction temperature was 500 °C, and after deposition, the coated substrates were allowed to cool to room temperature in an atmosphere of 250 sccm of oxygen gas flow. This has been shown to eliminate the formation of an oxygen-deficient surface layer [15], giving a high resistance sensor. The film deposition rate was ∼0.35 Å /cycle and the total film thickness was 17.5 nm. Film thickness was measured using a M-2000 FI spectroscopic ellipsometer from J.A. Woollam Co. Surface topography was measured with a Veeco CP-II atomic force microscope (AFM) in non-contact mode. Grain size was further confirmed by transmission electron microscopy (TEM) using a JEOL 2010 F microscope operating at an accelerating voltage of 200 kV. For TEM analysis, ALD tin dioxide was deposited directly on an electron-transparent Si/SiO_2 membrane TEM window. The chemical composition of the film was verified by Rutherford backscattering analysis. The tin dioxide film was patterned to expose contact pad areas by optical lithography and wet etching, using a Cr^{2+}-containing HCl-based etchant activated by zinc, as detailed in [12]. The photoresist mask was able to withstand the etchant for 30 s, long enough to fully etch through the SnO_2 film.

Finally the wafer front surface was spin coated with ProTEK B1-18 etch-protective material (Brewer Science), and the sample was etched in aqueous 20% KOH solution at 80 °C, in a special wafer holder exposing only the wafer backside to the etchant. A single etching step was used to produce membrane structures (Fig. 2, left), or two bulk etching steps with intermediate wet etching of oxide, to produce suspended island structures (Fig. 2, right), in a similar manner to the process proposed in [13].

The ProTEK layer is not strictly necessary during bulk etching, as only the wafer backside was exposed in the wafer holder. It did, however, serve as additional protection if minor leaks occurred during etching, and also provided added mechanical strength to the released membrane during dicing of the wafer into individual chips. After dicing, the ProTEK was removed at the chip level using a sequence of acetone, isopropanol and deionized water. Fig. 3 shows the finished gas sensor chip.

The chips were mounted on an alumina printed circuit board (PCB), shown in Fig. 4. The PCB is equipped with an aluminum shielding ring and an electrical connector for instrumentation used in sensor operation and characterization. The shielding ring together with an o-ring allows gas-tight connection to the gas
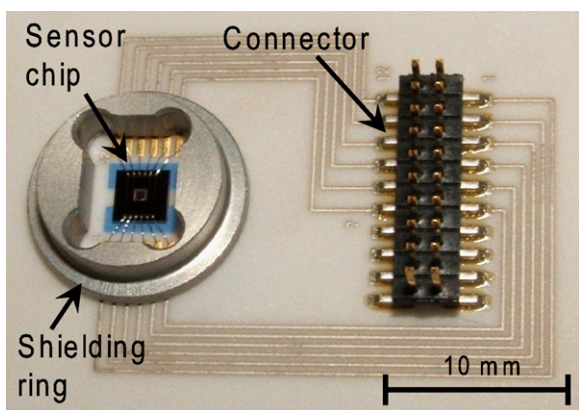
Fig. 4. The sensor chip mounted inside the aluminum shielding ring on the alumina printed circuit board.

testing equipment. The devices were mounted on the board using emission-free epoxy glue.

2.2. Sensor characterization

The characteristics of the sensors were measured both in laboratory air and in a controlled atmosphere generated by gas testing equipment. The response of the device to ethanol, acetone, and acrylonitrile vapors was tested.

The controlled atmosphere was generated from clean dry air provided by the cleanroom facilities of Micronova [14]. The clean dry air was humidified to the value of 32% relative humidity (RH) with a gas washer bottle filled with deionized water. Ethanol, acetone, and acrylonitrile vapors were produced by a vapor generation system, where the liquid chemical is injected into clean air flow. The concentrations of vapors in the gas phase were calculated based on the known injected quantity per time unit and volume flow (2.8 l/min) of the clean air. The temperature of the air mixture was 20–23 °C. The humidity of the resulting gas mixture was measured with a Vaisala HUMITTER 50 Y humidity probe.

The PCB on which the sensor was mounted (Fig. 4) was connected to an aluminum block with a small chamber equipped with gas line connectors. The connection between the shielding ring and the aluminum block was sealed with an o-ring. This setup was connected to the gas testing apparatus and the air flow was kept at 0.2 l/min with the help of a rotameter.

The temperature sensing resistor of the microhotplate was calibrated in an oven by measuring the temperature with a regular thermometer and the resistance with a Fluke 111 multimeter. This calibration was performed in the range of 20–100 °C, but since the resistor was fabricated from tungsten and its temperature–resistance curve was linear in the measured range, its linearity was assumed to extend to the operating temperature of 300 °C.

The conductance of the sensing film and the resistance of the thermometer resistor were measured with Keithley 236 and Keithley 237 source/measure units operating as voltage sources. The measurement voltages were 0.1 and 0.5 V across the sensing film and thermometer resistor, respectively. Before the gas response experiments, the sensors were kept at the operating temperature of 300 °C for at least 1 h in order to stabilize the sensing film. The heating power for the microhotplates was provided by a Keithley 228 A operating as a voltage source. The heating power and the resistance of the heater resistor were calculated from the measured current. The heater voltage was controlled so that the thermometer resistor indicated a temperature of 300 °C.
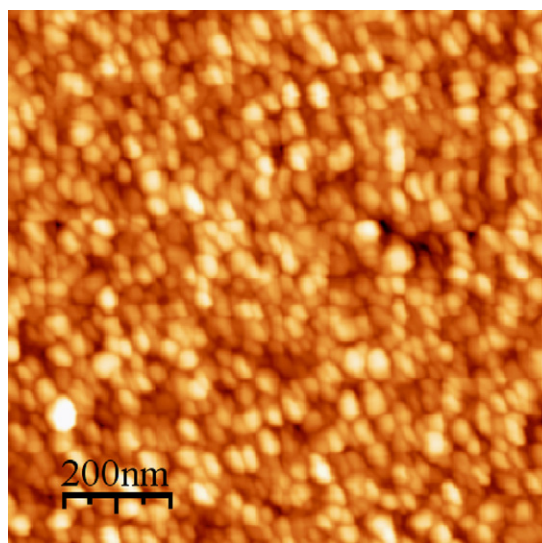

Fig. 5. AFM height image of ALD-deposited tin dioxide, measured in non-contact mode. rms surface roughness is approximately 3.6 nm.

3. Results

The chemical composition of ALD-deposited tin dioxide layers was verified by Rutherford backscattering analysis, and there was no evidence of any chlorine content from the $SnCl_4$ precursor. An AFM height image of a 17.5 nm thick tin dioxide film is shown in Fig. 5. The films consisted of nanosized grains in the size range of 35–50 nm. The rms surface roughness was around 3.6 nm. The grain structure was also confirmed by TEM (Fig. 6). The presence of grains with a relatively large lateral dimension of 35–50 nm in a 17.5 nm thick tin dioxide film shows that they have a platelet-like structure, as would be expected from a Stranski–Krastanov type film growth mechanism. Further details of the deposition process and the resulting film structure can be found in [15].

The characteristics of the microhotplates were comparable to the typical characteristics reported in the literature [1]. At room temperature, the resistance of the thermometer resistors was 2.4–2.5 kΩ for the small (500 μm × 500 μm) devices, and 4.4–4.6 kΩ for the large (1000 μm × 1000 μm) devices. The temperature coefficient of the resistance was 5.7–5.8×10^{-4} K^{-1}. Based on these calibrated thermometer values the heating voltages for the samples were selected. They are listed in Table 1. The stabilization time in Table 1 is the time required to reach stable operating temperature. Even though our microhotplates are
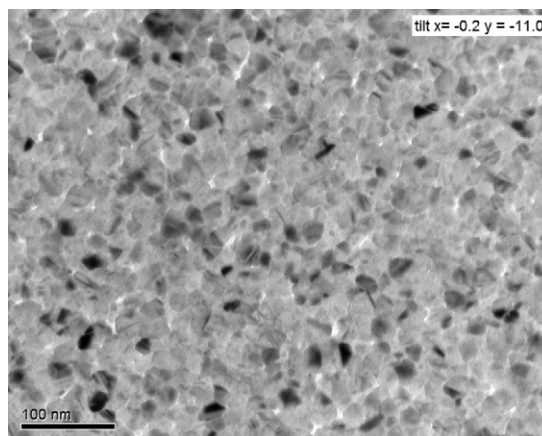

Fig. 6. TEM image of ALD-deposited tin dioxide showing the grain structure.

Table 1
Heater characteristics of the sensors operating at 300 °C.

Sensor area (μm)	500 × 500	1000 × 1000
Heater resistance (Ω)	200	250
Heating voltage (V)	4.5	6.0
Heating power (mW)	80	180
Stabilization time (s)	< 2	< 4

Table 2
Sensitivity of small (500 μm × 500 μm) sensor operating at 300 °C.

Gas	20 ppm	100 ppm	500 ppm
Ethanol	–	1.51	1.64
Acetone	–	1.23	1.38
Acrylonitrile	1.008	1.035	–

larger than the common ones [1], the required heating power is within the range of typical values for microhotplates (30–150 mW [1]). As Table 1 suggests, even smaller heating power requirement can be achieved by reducing the size of the microhotplate, since the required heating power decreases with decreasing sensor area.

Fig. 7 shows the response of the device to pulses of ethanol, acetone, and acrylonitrile at different concentrations. The measurements were made at the operating temperature of 300 °C in clean air (32% RH). Being reducing gases, ethanol, acetone, and acrylonitrile rapidly increase the conductivity of the sensing film. This is in agreement with other *n*-type metal-oxide gas sensors [4]. Compared to the responses to acetone and 100 ppm acrylonitrile, the responses to ethanol and 20 ppm acrylonitrile differ in shape. When ethanol or 20 ppm acrylonitrile is introduced, the conductance increases rapidly. After peaking briefly, the conductance descends to a steady-state value. These kinds of responses are often characterized by a second-order differential equation,
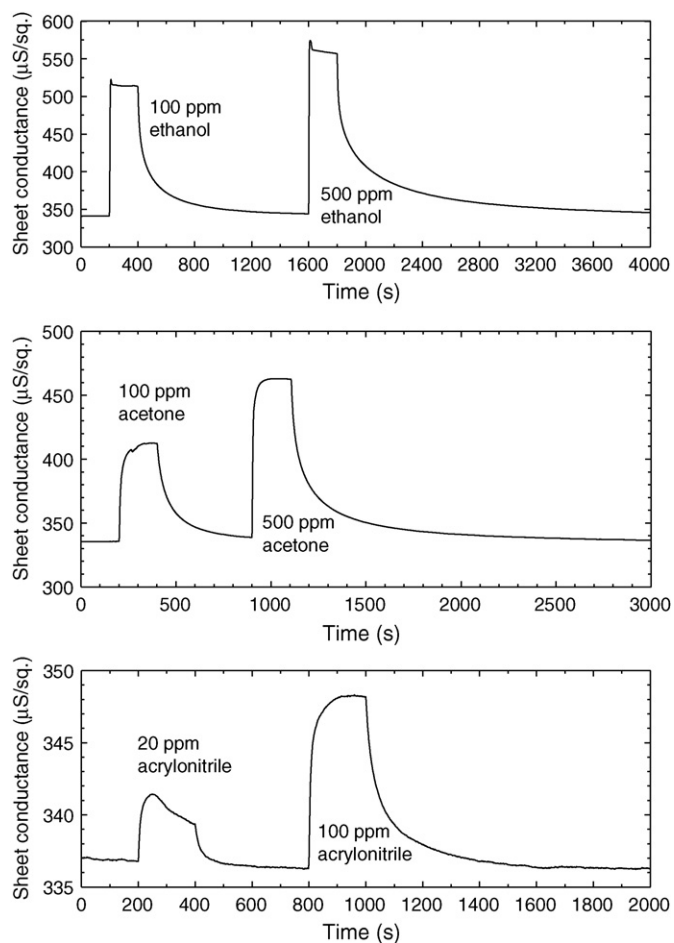

Fig. 7. The response of a 500 μ × 500 μm device at 300 °C operating temperature to 200 s pulses of two different concentrations of ethanol (*top*), acetone (*center*), and acrylonitrile (*bottom*). The sheet conductance of the sensing film is shown. Measured in clean air (32% RH).

i.e. a second-order response. In turn, the responses to acetone and 100 ppm acrylonitrile show only a rapid increase of conductance, which suggest that the response is of first order. The difference in the order of the response could be caused by the complex reaction pattern of ethanol [2,16]. In general, however, the response of a resistive metal-oxide gas sensor consists of diffusion steps [17] and multiple adsorption, ionization, and chemical reactions [2,16,18,19], which is a highly nonlinear high-order system *per se*. The recovery, on the other hand, is similar after all pulses, and the recovery time is proportional to the concentration of the gas.

The sensitivity of the sensor to a specific gas can be calculated with the formula $S = G_{sh}^{gas}/G_{sh}^{air}$, where G_{sh}^{gas} is the sheet conductance of the sensing film in the presence of the gas and G_{sh}^{air} in clean air. Table 2 presents the sensitivities calculated for the pulses shown in Fig. 7.

The sensitivity appears relatively low compared to values reported in literature, *e.g.* [20]. However, the current devices have not been doped with any catalyst material, as is commonly done in this type of sensors to improve sensitivity. Also the dense thin film produced by the ALD process may be expected to have lower response than a thick porous film produced from a powder or paste, with its inherent large surface area. However, responses to the gases are extremely fast, as seen from the $t_{50\%}$ and $t_{90\%}$ response times (the time taken for 50% and 90% of the overall response to occur, respectively) listed in Table 3. Furthermore, the recovery and base resistance stability during measurement cycles of several hours were excellent, exhibiting a drift of only −0.4%/h, (*i.e.* decreasing sheet conductance). After 11 days of continuous operation at 300 °C in clean air (30–33% RH), the drift rate reduces to −0.02%/h. Such behavior can be an indication of a structurally stable, very dense and thin SnO_2 layer, which reflects the extraordinary capabilities of the ALD process in gas sensor applications.

After prolonged operation of 12–16 days, however, the operation of the devices began to deteriorate. Further investigation revealed a long-term decay in the tungsten heater resistors, which led to an increase in their resistance and therefore higher voltage requirements to maintain the operating temperature of the device. Fig. 8 shows this increase in heater resistance in two different devices operating at 300 °C. A similar increase in resistance was observed in the thermometer resistors. Fig. 9 shows a microscope image of a heater resistor exhibiting oxidation from the line edge inwards, probably initiating at a stress-induced defect in the surrounding dielectric material. The decay of the heater resistors was, however, slow enough to enable investigation of the devices' operation and the properties of the sensing film. Data in Fig. 8 allows the lifetimes of the sensors to be estimated as 12 and 16 days for

Table 3
Response times of small (500 μm × 500 μm) sensor operating at 300 °C.

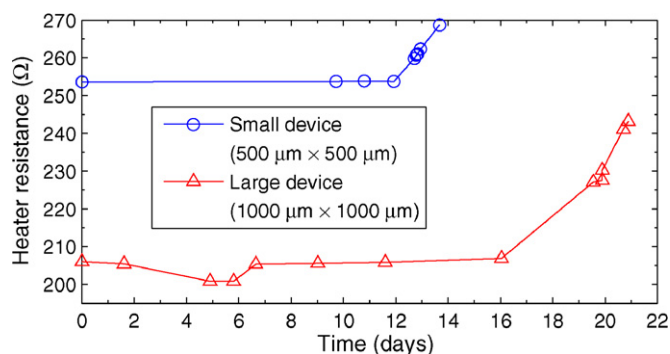| Gas	$t_{50\%}$ (s)	$t_{90\%}$ (s)
Ethanol 100 ppm	3.4	5.9
Ethanol 500 ppm	2.3	3.0
Acetone 100 ppm	8.1	46.2
Acetone 500 ppm	4.2	20.2
Acrylonitrile 20 ppm	3.0	5.6
Acrylonitrile 100 ppm	5.2	43.5

Fig. 8. Resistances of the heaters of small and large devices during a long operation test in laboratory air. The devices are operating at 300 °C until the heater resistance begins to increase. The increase of the heater resistance is caused by the oxidation of the tungsten heater.
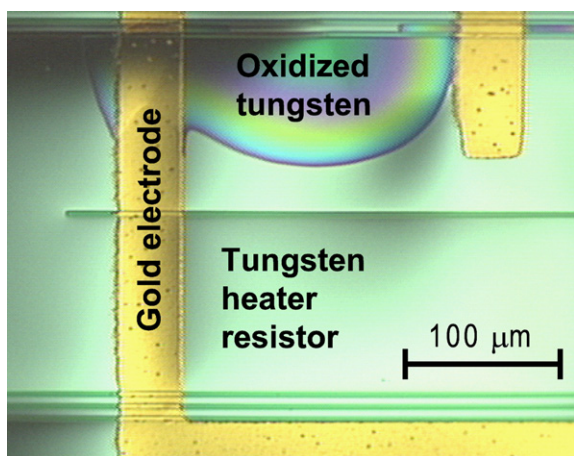

Fig. 9. Optical microscope image of a partially oxidized tungsten heater resistor in a large sensor device.

the small and large sensors, respectively. No changes in the sensing films, the thermometers, nor the heater resistors of the sensors were observed before these times. While tungsten was, apparently, not an appropriate choice of heater material in the long term, a demonstration run of the fabrication process was also conducted using platinum as the heater metallization. No obvious incompatibilities in the fabrication process were observed with this change of metal.

4. Conclusions

Tin dioxide deposited by ALD has been integrated for the first time on a microhotplate gas sensor. Due to special demands of the ALD process, new solutions were required for the fabrication sequence, including film patterning methods and reordering of process steps. We have demonstrated that devices with ALD SnO_2 sensing films exhibit good response and recovery to gases, as well as good operational stability in the short term. This would alone facilitate many applications where short operational lifetime is sufficient, *e.g.* low-cost and disposable sensor applications.

Long-term stability of the current devices was limited by the degradation of the heater resistors, not by the ALD SnO_2 sensing film *per se*. Degradation of the heater metallization initiates from the line edges where the tungsten is not protected by tungsten nitride, and proceeds slowly inwards. However, for experimental and testing purposes, tungsten was an adequate material. Likewise, silicon dioxide intermetal dielectrics deposited by PECVD were found to work when annealed after deposition, provided that the

annealing temperature is at least as high as the SnO_2 deposition temperature and the intended operating temperature of the device.

To allay stability concerns in production devices, compatibility of the fabrication process with platinum heater metallization was verified. This is expected to improve the long-term stability of the devices overall. The sensitivity of the devices presented here is considerably lower than what has been reported for conventional SnO_2 gas sensor devices, but optimization of the deposition process and application of catalytic additive materials, possibly deposited *in situ* during the ALD process itself, is expected to improve device performance. In general, the technology concept was successfully demonstrated, and combined with the intrinsic high quality of ALD-deposited SnO_2, steps towards commercialization of the device are feasible.

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Biographies

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Aapo Varpula received his Master's degree with distinction in Engineering Physics and Mathematics in 2006 from the Helsinki University of Technology (HUT) in Espoo, Finland. Since the beginning of 2006 he has been working on his doctoral thesis on metal-oxide based gas sensors in the Electron Physics Group of Department of Micro and Nanosciences at HUT, where he obtained his L.Sc. in 2009. His main research interests are the physical and chemical modelling of metal-oxide based gas sensors, development of measurement techniques of sensors, impedance spectroscopy, and characterization of sensor materials.

Mikko Utriainen, Doctor of Science and Technology (chemical engineering, Helsinki University of Technology 1999). Current position: Senior Technology Adviser at Tekes, Finnish Funding Agency for Technology and Innovation (since 2008) and in Employment and Economic Development Centre for North Savo, Kuopio that provides Tekes's services in North Savo region. His specialty is research, technology and innovations in the field of security and safety. Before current position Mikko was R&D manager in Environics Oy 2000–2008 specializing in sensor technology development for CBRNE security area. Before Environics he was carrying out solid state chemical sensor research in the Helsinki University of Technology and wrote his D.Sc. thesis of the same topic.

Gomathi Natarajan received her Master's degree in Materials Science in the year 2002 from PSG College of Technology, Coimbatore, India. In 2007, she received her Ph.D. from Dublin City University, Ireland on "Novel UV emitting layers lattice matched with Si". Her research interests include deposition and analysis of functional thin films. She is presently working as a Humboldt guest scientist at RWTH Aachen University, Germany.

David Cameron obtained his bachelor's degree in Electronics and Electrical Engineering in 1972 from the University of Glasgow. After working in industry, he returned to the University of Glasgow and received his Ph.D. in 1980. He was a Senior Research Fellow at the Royal Signals and Radar Establishment (UK) before joining the School of Electronic Engineering of Dublin City University in 1982 where he became Associate Professor. He joined Lappeenranta University of Technology as Professor of Materials Technology in 2004. His research has been in thin film deposition: magnetron sputtering, atomic layer deposition and plasma CVD.

Sergey Novikov received his Master degree in Physical chemistry from Leningrad Institute of Technology (USSR) in 1982. He obtained his Ph.D. from the A. F. Ioffe Institute (St. Petersburg, Russia) in 1994 on the growth and characterization of the alkali-earth fluorides on silicon and gallium arsenide. Since 1995 he is working in the Helsinki University of Technology, where he is currently the senior research scientist. His main field of interest is growth of II–IV compounds and epitaxial oxides.

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Juha Sinkkonen (M.Sc. 1967, D.Sc. 1976) has been Professor in the Electron Physics Group of Department of Micro and Nanosciences, Helsinki University of Technology (Finland) since 1984. He is specialized in semiconductor and device physics. Currently he is interested in physico-chemical characterization of semiconductor surfaces and interfaces.

Sami Franssila studied physics and chemistry at the University of Helsinki and obtained B.Sc. and M.Sc. degrees in 1985 and 1986. His Ph.D. degree is from Helsinki University of Technology, School of Electrical Engineering (1995) with a thesis on plasma etching. He worked as a research scientist at VTT Microelectronics from 1986 till 1998, with 1993–1994 at IMEC, Belgium, and since 1998 at Helsinki University of Technology. He has worked on CMOS, MEMS and microfluidic devices and has published 80 peer reviewed journal articles and a textbook "Introduction to Microfabrication". He is currently a Professor of Materials Science, with research interests in materials and fabrication technologies for fluidic, bio, chemical and thermal devices, and flexible electronics.