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Hydrogen sulfide detection properties of Pt-gated AlGaN/GaN HEMT-sensor

Robert Sokolovskij[a,b,c], Jian Zhang[b,d], Elina Iervolino[b], Changhui Zhao[b], Fabio Santagata[a], Fei Wang[b,e], Hongyu Yu[b,e], Pasqualina M. Sarro[a], Guo Qi Zhang[a,*]

[a] Department of Microelectronics, Delft University of Technology, 2628 CD Delft, The Netherlands
[b] Department of Electrical and Electronic Engineering, Southern University of Science and Technology, 518055 Shenzhen, China
[c] State Key Laboratory of Solid State Lighting, 213161 Changzhou, China
[d] State Key Laboratory of ASIC and System, School of Microelectronics, Fudan University, 200433 Shanghai, China
[e] Shenzhen Key Laboratory of the Third Generation Semi-conductor, 518055 Shenzhen, China

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ABSTRACT

AlGaN/GaN high electron mobility transistor (HEMT)-based sensors with catalytic platinum gate were micro-fabricated on commercially available epitaxial wafers and extensively characterized for ppm level hydrogen sulfide (H_2S) detection for industrial safety applications. High operating temperature above 150 °C enabled large signal variation (ΔI_{DS}) of 2.17 and sensing response of 112% for 90 ppm H_2S in dry air as well as high stability across a wide range of biasing conditions. Transient response measurements demonstrated stable operation, superb response and recovery, with good repeatability. The measured sensing signal rise (fall) times reduced from 476 (1316) s to 219 (507) s when the temperature was increased from 200 °C to 250 °C. The response to 90 ppm H_2S was 4.5x larger than to H_2 and the device showed stable operation over an extended time period.

1. Introduction

With the continuous and rapid industrial growth, environmental pollution monitoring and assurance of worker safety raise increasing concerns. H_2S is a toxic, flammable, colorless gas with characteristic pungent odor [1]. It is naturally produced from sewage, liquid manure, sulfur hot springs and biogas [2]. H_2S is also a by-product of coal mining, petroleum and natural gas refinement industries [3]. Extended exposure to 10–500 ppm H_2S concentrations can cause symptoms varying from rhinitis to loss of consciousness and even respiratory failure. Furthermore, continuous inhalation of concentration above 100 ppm will cause inhibition of sensing the characteristic odor, due to olfactory fatigue [2,4], which can result in false assumption of the gas being dissipated. Hence, portable, wearable and reliable detectors, capable of sensing low ppm levels of H_2S are of crucial importance to ensure worker safety for the fossil fuel and other energy production industries.

The first solid state gas sensor based on silicon metal oxide semiconductor (MOS) field effect transistor (FET), using palladium (Pd) as hydrogen (H_2) sensitive layer, was reported in 1975 [5]. Numerous modifications of the original transducer were further developed to enhance performance in terms of sensor sensitivity and expand the range of detectable gases. These devices include the suspended gate FET (SG-FET) [6], hybrid SG-FET [7], capacitively controlled FET (CCFET) [8], floating gate FET (FG-FET) [9] and most recently the horizontal FG-FET [10]. However, because of the narrow energy bandgap of Si (1.12 eV), these GasFETs are not able of operating at temperatures above 200 °C. In fact, initial reports on silicon Pd-MOS H_2S sensors have demonstrated operation at temperature up to 150 °C [11,12]. To overcome the limitations related to the use of Si, wider bandgap 2nd generation compound III-V semiconductors, including InP [13], GaAs [14,15], InGaP [16], InAlAs [17], have been previously investigated for gas sensing applications. While these devices achieved improved sensing performance, the requirement of costly and fragile GaAs or InP substrates for layer epitaxy limits their large-scale adoption. 3rd generation wide bandgap (> 3 eV) silicon carbide (SiC) and gallium nitride (GaN) materials are favorable for development of high performance gas sensors. The price of SiC substrates is currently very high, while GaN can be grown on cost effective sapphire or Si wafers. Hence gallium nitride, with a bandgap of 3.4 eV, is particularly advantageous for harsh environment, high temperature electronics and sensor applications [18]. AlGaN/GaN heterostructure Schottky diode and high electron mobility transistor (HEMT) based H_2 sensors have been demonstrated operating at 800 °C under N_2 ambient [19,20]. While most studies refer to H_2

* Corresponding author.
E-mail addresses: R.Sokolovskij@tudelft.nl (R. Sokolovskij), G.Q.Zhang@tudelft.nl (G.Q. Zhang).

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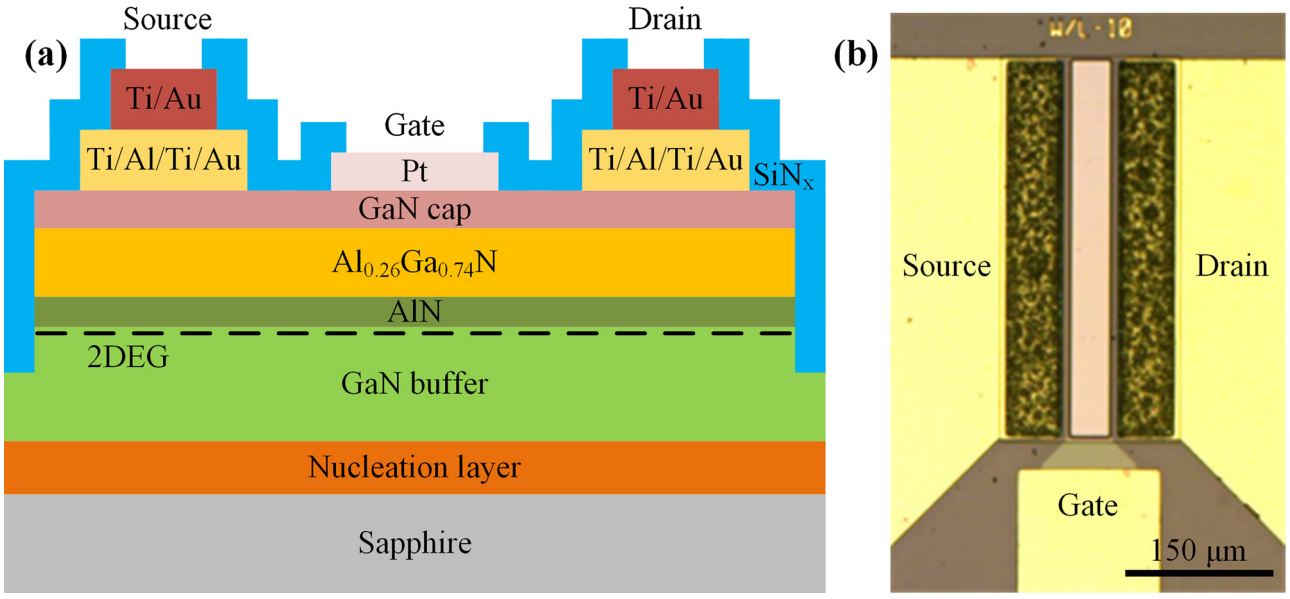

Fig. 1. (a) Schematic cross-section of the studied Pt-AlGaN/GaN HEMT H_2S sensor. (b) Top view optical micrograph of the fabricated sensor.

sensors [21–23], AlGaN/GaN sensors were also demonstrated for CO, CO_2, NO, NO_2, NH_3, Cl, CH_4, C_2H_2 [24–30]. Very few results of H_2S detection with wide bandgap semiconductor devices have been reported so far. A GaN Schottky diode with Pt gas sensing layer has been demonstrated for 0.1 ~ 10 ppm H_2S detection under N_2 ambient [31]. A Silicon carbide FET with Pt and Ir gates was also shown to sense H_2S, however signal saturation was observed at low concentration of 6 ~ 8 ppm under 5% oxygen atmosphere [32]. Recently we presented successful sensing of H_2S with Pt-AlGaN/GaN HEMT based sensor [33]. In this work, we further expand on our preliminary results by comprehensively studying DC and transient characteristics at different temperatures and H_2S concentrations in dry air atmosphere. Moreover, the measured signal variations and sensing responses at different bias points are evaluated and sensor stability, selectivity and repeatability are demonstrated.

2. Experimental

2.1. Fabrication of sensors

The epitaxial structure used for device fabrication was purchased from a commercial vendor, Suzhou Nanowin Co. The material was grown by MOCVD on 2 inch C-plane sapphire wafers. Starting from the substrate, the stack consisted of a proprietary nucleation layer, for lattice mismatch compensation, a 1.8 µm GaN buffer, 1 nm AlN interlayer, followed by an undoped 21 nm $Al_{0.26}Ga_{0.74}N$ barrier and 1 nm GaN capping layer. The basis for HEMT operation is the formation of a high electron density channel, two-dimensional electron gas (2DEG), at the AlGaN/GaN heterojunction interface, due to polarization effects [34].

The sensor fabrication started with wet chemical cleaning of the substrate using acetone, isopropanol and DI water rinsing. Afterwards 100 nm deep mesa etching was performed by ICP BCl_3/Cl_2 plasma to isolate individual devices. Then ohmic contacts consisting of a Ti/Al/Ti/Au stack with thickness of 20/110/40/50 nm, were e-beam evaporated and patterned by lift-off. A 60 s dip in HCl:H_2O solution was done right before loading the wafers into the deposition chamber to remove any surface oxide [35]. After pattering, the contacts were annealed for 47 s at 870 °C in N_2 ambient. The gas sensing gate electrode was then formed by e-beam evaporation and lift-off of a 10 nm Pt layer. Then a bi-layer of 30/300 nm Ti/Au interconnect metal was evaporated and patterned by lift-off to guarantee reliable wire bonding. Finally, the devices were passivated by a 500 nm PECVD SiN_x layer followed by combined RIE and wet BOE etching to open the sensing area and the bonding pads. The schematic cross-section and top view optical micrograph of the fabricated device are shown in Fig. 1. The gate dimensions exposed to gas were 40 µm × 400 µm and the gate-source and gate-drain spacing was 6 µm, based on our earlier report [36]. After fabrication, the wafers were diced and individual devices were wire bonded to ceramic substrates for high temperature measurements.

2.2. Testing of sensors

Gas testing was performed using a commercial gas mixing system from Beijing Elite Tech Co., which consists of mass flow controllers (MFC) to dilute the calibration gas, a 1.8 L volume chamber with temperature controlled hotplate, temperature and humidity sensors and electrical feedthroughs. The sensors were tested at different temperatures using H_2S reference gas diluted with dry synthetic air (O_2/N_2 = 21%/79%) to ensure 0% relative humidity. The combined total gas flow was kept at 310 sccm. Electrical sensor measurements were conducted using a pair of Keithley 2450 source meters. Prior to gas sensing experiments the sensors underwent a burn-in procedure in dry air ambient for 24 h at 150 °C with gate and drain bias voltages of 0 V and 5 V respectively in order to minimize baseline drift. Afterwards sensor activation was carried out with H_2 pulses of increasing concentration from 100 to 900 ppm with air purges in-between at 250 °C. We observed that such treatment allowed to extend the upper limit of H_2S detection before signal saturation occurred. Exposure to H_2 can reform the surface morphology of Pt [37] and increase the number of surface sites for gas adsorption. Further investigations into the mechanism of H_2 based activation are currently ongoing.

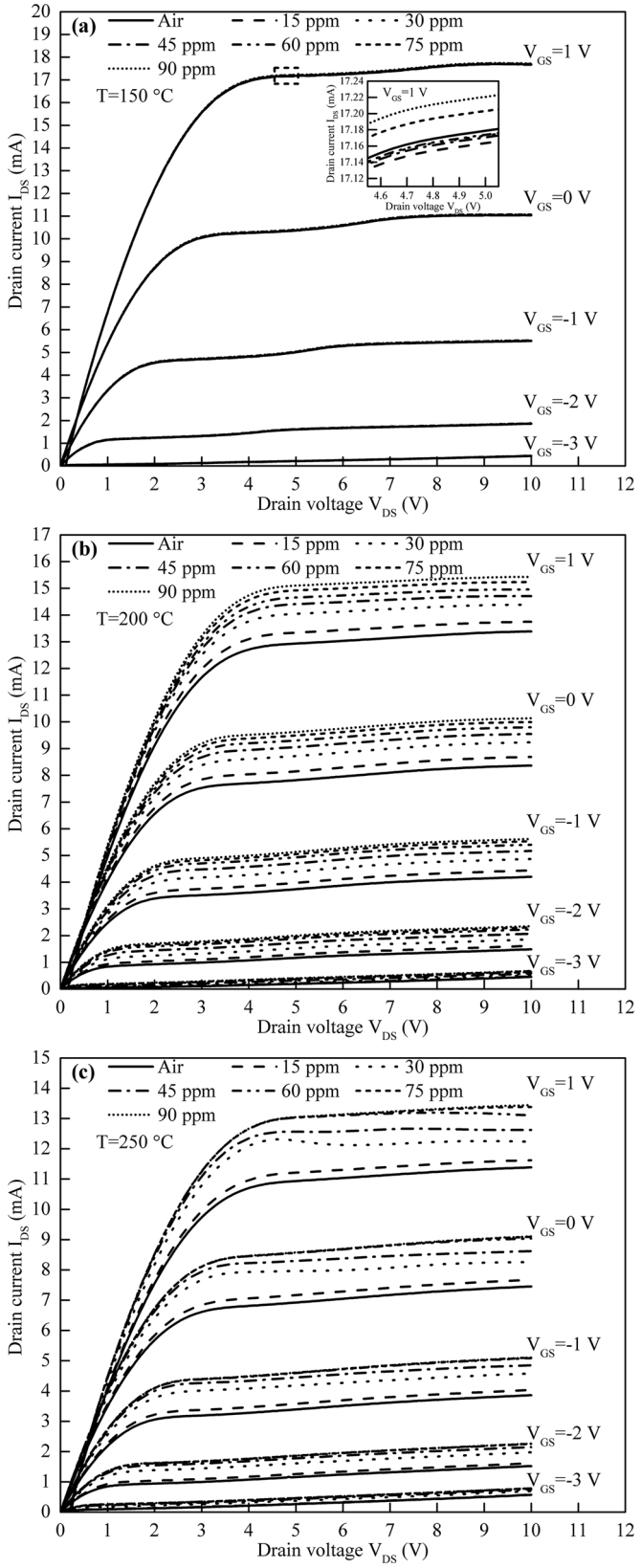

Fig. 2. Output (I_{DS}-V_{DS}) characteristics of Pt-HEMT sensors exposed to different H_2S concentrations at (a) 150 °C, with the inset showing a magnified view of the box area, (b) 200 °C and (c) 250 °C.

3. Results and discussion

The steady state sensing characteristics of the Pt-HEMT sensor were studied by measuring the drain current versus drain-source voltage (I_{DS}-V_{DS}) and the drain current versus gate-source voltage (I_{DS}-V_{GS}). Output (I_{DS}-V_{DS}) characteristics upon exposure to H_2S/Air concentrations of 15–90 ppm at temperatures of 150 °C, 200 °C and 250 °C are shown in Fig. 2(a–c). The gate-source voltage (V_{GS}) was stepped from −3 V to 1 V with 1 V increments. Proper transistor operation is clearly observed with distinct linear and saturation regions and the ability to module output current via the gate terminal is maintained at all tested temperatures. A profound rise in drain current was observed at 200 °C and 250 °C with increasing H_2S concentrations. At 250 °C the sensing signal started to saturate at concentrations above 60 ppm/air. The magnitude of current variation at 150 °C was much lower, with noticeable instability below 75 ppm/air. The corresponding transfer (I_{DS}-V_{GS}) and transconductance (g_m) characteristics at V_{DS} = 7 V are shown in Fig. 3(a–c). Upon exposure to H_2S the curves shift towards more negative voltages for the same I_{DS} levels. The maximum transconductance ($g_{m,max}$) values increased from 6.82, 5.35, 4.54 mS in air ambient to 6.83, 5.63, 4.89 mS when exposed to 90 ppm/air at temperatures of 150 °C, 200 °C and 250 °C respectively. Higher transconductance in H_2S containing ambient is attributed to increased number of electrons in the 2DEG channel due to gas interaction with the Pt gate which is also evident from Fig. 2. Threshold voltage (V_{TH}) values at tested concentrations were extracted from Fig. 3(a–c) using the linear extrapolation method by fitting a tangent line at the point of $g_{m,max}$ to the V_{GS} axis intercept [38]. The threshold voltage shift, defined as $\Delta V_{TH} = V_{TH,air}$-$V_{TH,H2S}$, for the tested H_2S concentration range is shown in Fig. 3(d). Clearly the magnitude of ΔV_{TH} increases with increasing test gas concentration at 200 °C and 250 °C. At 150 °C ΔV_{TH} mostly unchanged, due to low catalytic dissociation efficiency of H_2S on Pt at this temperature. Output and transfer characteristics before and after exposure to 90 ppm H_2S/Air are shown in Fig. 4(a) and (b) respectively. After the initial baseline measurements in dry air at 250 °C, H_2S was injected into the test chamber for 20 min followed by an air purge for 60 min. From Fig. 4(a) an increase of 0.26 mA was observed for the baseline current (at V_{GS} = 0 V, V_{DS} = 5 V) and a corresponding -0.06 V shift (Fig. 4(b)) of the transfer curve (at I_{DS} = 5 mA). It was observed that after the sensor was exposed to ambient conditions for several hours the baseline values were restored. To understand the current and threshold voltage variations and analyze the gas sensing mechanism we first look at the saturation drain current of an AlGaN/GaN HEMT expressed as:

$$I_{DS,sat} = \frac{\mu C_b W_g}{2L_g}(V_{GS} - V_{TH})^2 \quad (1)$$

where μ is the 2DEG mobility, W_g/L_g the gate width/length, C_b is gate to channel capacitance, which is the sum of capacitance contributions from each layer between the gate metal and 2DEG ($1/C_b = 1/C_{cap} + 1/C_{AlGaN} + 1/C_{AlN} + 1/C_{2DEG}$). The μ and C_b are determined by the quality and structure of the epitaxy, while W_g/L_g are defined by sensor design. The shift in threshold voltage towards more negative value would result in the observed I_{DS} increase in H_2S containing atmosphere. The expression of V_{TH} for an AlGaN/GaN HEMT is:

$$V_{TH} = \Phi_b - \frac{\Delta E_C}{q} - \frac{qn_s}{C_b} \quad (2)$$

where Φ_b is the Schottky barrier height, ΔE_C is the conduction band discontinuity, q is the elementary charge and n_s is the sheet charge carrier density. The Schottky barrier height is in turn dependent on the

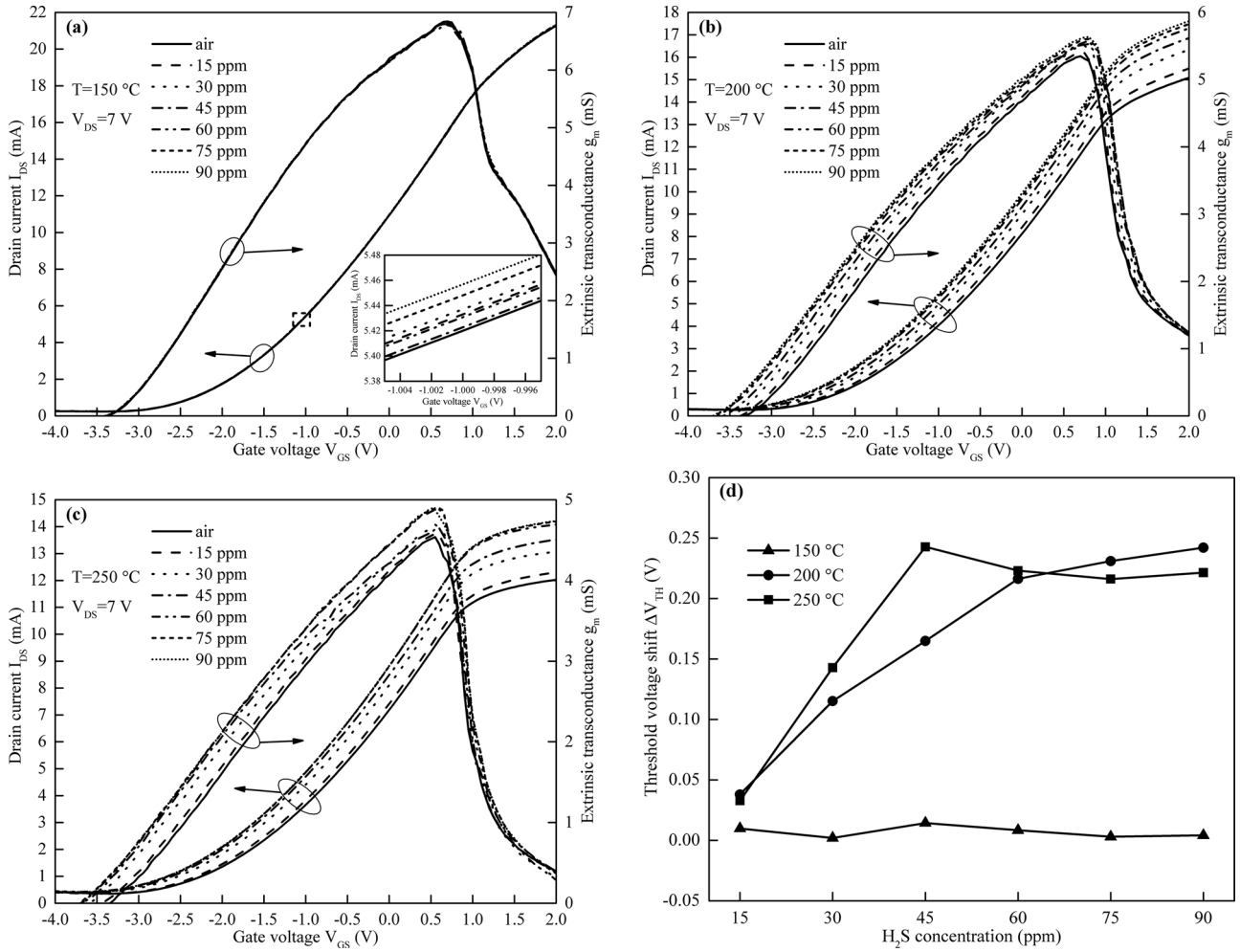

Fig. 3. Transfer (I_{DS}-V_{GS}) and transconductance characteristics of Pt-HEMT sensors exposed to different H_2S concentrations at (a) 150 °C, with the inset showing a magnified view of the box area, (b) 200 °C and (c) 250 °C. (d) Threshold voltage shift versus H_2S concentration.

work function (Φ_m) of the gate metal and the semiconductor electron affinity (χ_s), $\Phi_b = \Phi_m$-χ_s. Previous research has found that hydrogen containing gas molecules upon adsorption on the surface of catalytic metals (e.g. Pt, Pd or Ir) dissociate and release hydrogen atoms [21,27,29]. In the case of H_2S adsorption in air ambient the probable reaction mechanism is as follows [39]:

$$H_2S_{(ad)} \rightarrow SH_{(ad)} + H_{(ad)} \quad (3)$$

$$SH_{(ad)} \rightarrow S_{(ad)} + H_{(ad)} \quad (4)$$

$$S_{(ad)} + O_{2(ad)} \rightarrow SO_{2(gas)} \quad (5)$$

The S—H bonds are broken sequentially as described by (3) and (4) and the remaining sulfur reacts with O_2 present at the Pt surface and forms SO_2 which can then desorb form the surface. The hydrogen ions rapidly diffuse through the Pt to the M–S interface. It is assumed that there is an interfacial oxide layer present on the GaN surface, since it was exposed to ambient conditions during sensor fabrication for several hours and no chemical or plasma treatments were performed prior to Pt deposition. The oxide layer supplies bonding sites for the diffused H resulting in a dipole layer at the interface [40]. This causes the reduction of metal work function (Φ_m) and the lowering of the Φ_b, which results in the observed ΔV_{TH} and I_{DS} increase in H_2S containing atmosphere. To evaluate the hydrogen sulfide detection performance of our Pt-HEMT sensor, we calculated the sensing response defined as:

$$S(\%) = \frac{\Delta I_{DS}}{I_{DS,air}} \times 100\% \quad (6)$$

where $\Delta I_{DS} = I_{DS,H2S}$-$I_{DS,air}$ is the drain current variation between H_2S and air ambient. Fig. 5(a) shows the ΔI_{DS} as a function of drain-source voltage (V_{DS}) for V_{GS} = 0 V at temperature of 200 °C. Measurements indicated that the ΔI_{DS} increases linearly with V_{DS} in the transistor linear region until it reaches the maximum value at the transition point to the saturation region at approximately 4 V, and then reduces by only 4% at V_{DS} = 10 V. This demonstrates that our sensor exhibits high stability and allows for a wide selection of biasing conditions without diminishing sensing performance. The drain current variation at different H_2S concentrations and gate bias voltages is shown in Fig. 5(b). The magnitude of sensing signal variation is greatly impacted by the gate bias. For 90 ppm H_2S concentration the measured ΔI_{DS} increased tenfold, from 0.21 mA at V_{GS} = −3 V to 2.17 mA at V_{GS} = 1 V. The ΔI_{DS} increase is due to larger baseline current ($I_{DS,air}$) with increasing gate bias. Fig. 6(a) shows the hydrogen sulfide sensing response at different gate bias voltages, while Fig. 6(b) shows response at different temperatures. Looking at Eq. (6) the reduction of S is due to larger increase of $I_{DS,air}$

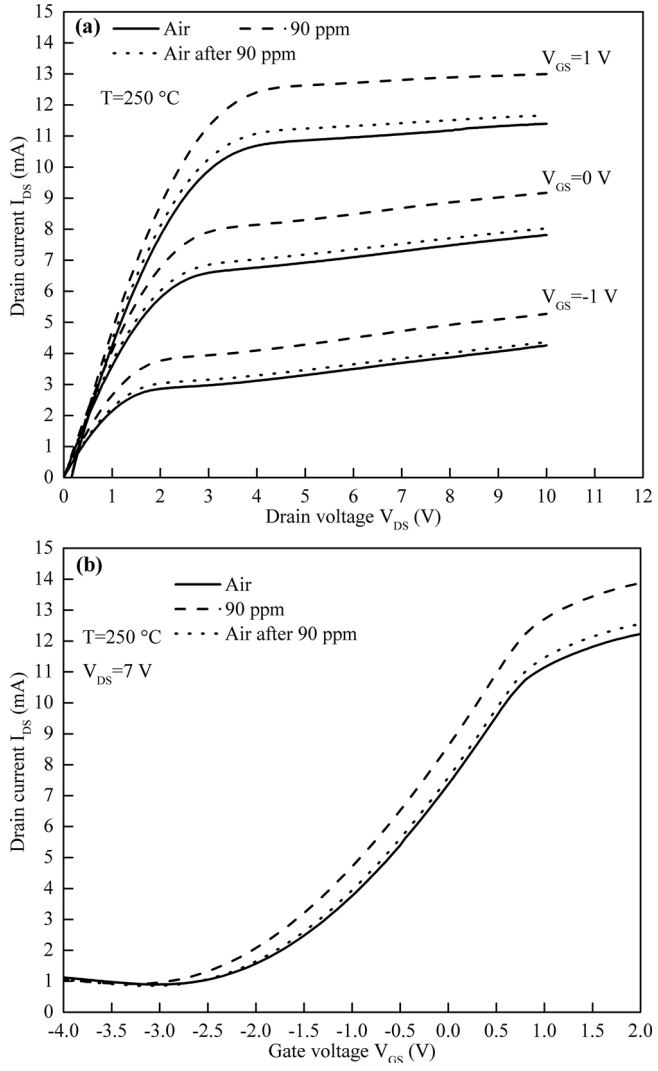

Fig. 4. (a) Output (I_{DS}-V_{DS}) and (b) transfer (I_{DS}-V_{GS}) characteristics of the Pt-HEMT sensor before and after exposure to 90 ppm of H_2S at 250 °C.

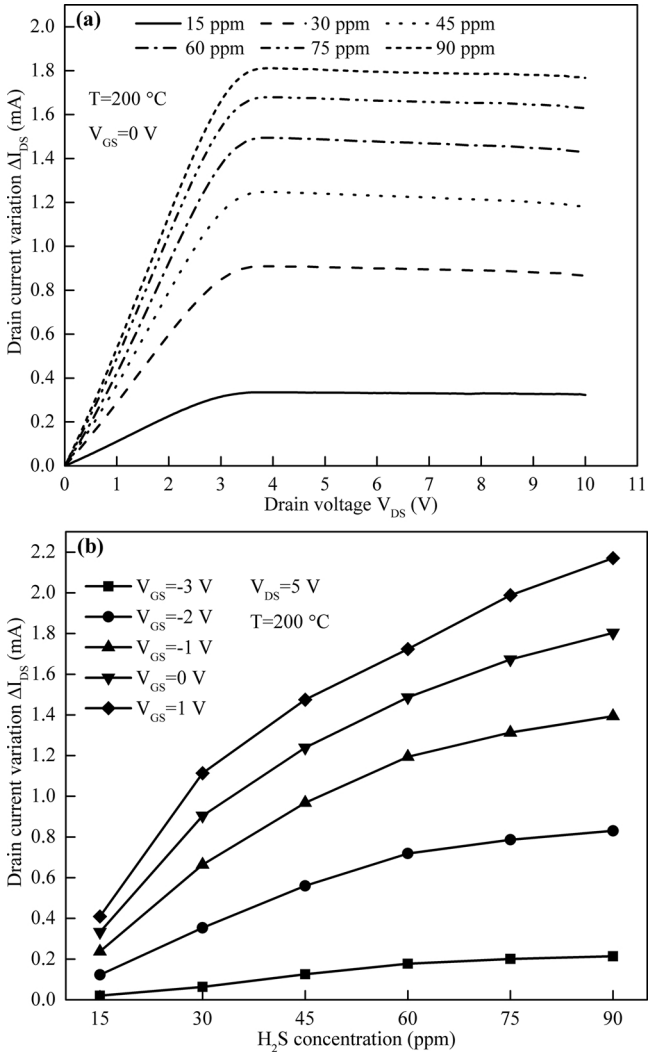

Fig. 5. (a) Drain current variation (ΔI_{DS}) as a function of drain-source (V_{DS}) voltage for different H_2S concentrations at 200 °C, V_{GS} = 0 V. (b) Drain current variation (ΔI_{DS}) versus H_2S concentration at 200 °C, V_{DS} = 5 V.

with higher V_{GS} than the increase in ΔI_{DS}. Based on Fig. 5(b) we conclude that HEMT type sensor can be operated at high signal amplitude (ΔI_{DS}) conditions ($V_{GS} > 0$ V) to achieve the lowest gas detection limits. Alternatively, as shown in Fig. 6(a), operating in high response mode is possible when gate bias is approaching pinch-off state thereby minimizing sensor power consumption. GaN Schottky diode type H_2 sensor had been previously demonstrated with very high response of ~3500% at 150 °C, however the signal amplitude was on the order of nA [41]. From Fig. 6(b) it is evident that response towards H_2S increased with higher temperature, however saturation started to occur earlier, namely above 60 ppm concentration.

Fig. 7 shows transient characteristics of the Pt-HEMT sensors at the tested temperatures and increasing analyte gas concentrations. The bias conditions used were V_{DS} = 5 V, V_{GS} = 0 V to obtain high ΔI_{DS} and operate the sensor as 2-terminal device with gate and source terminals shorted. The drain current increased immediately upon injecting H_2S, with the operating temperature having a significant impact on the observed response. At 150 °C there is substantial baseline value drift at 15 ppm and 30 ppm, followed by minimal signal variation with increasing concentration. It is believed to be caused by sulfur poisoning of the Pt surface due to incomplete oxidation to SO_2 and desorption at this temperature [12]. Raising the temperature to 200 °C resulted in an increased signal and improved recovery of the sensor, while at 250 °C the sensor rapidly reached steady state after gas introduction and returned to the baseline level during air purge steps. Moreover signal saturation was less profound compared with DC measurements. The increased magnitude of response current at 200 °C and 250 °C is due to enhanced reaction rate of H–S bond cleavage and H atom diffusion through the Pt gate. The baseline current value in air ambient reduces with rising temperature due to reduction of electron mobility in 2DEG channel.

Response and recovery rates were estimated using rise (t_R) and fall (t_F) times, defined as the time required for the signal to rise/fall from 10% to 90% of the steady state values. This method was used to reduce the influence of the delay necessary to equilibrate gas concentration inside the testing chamber. Fig. 8(a) and (b) shows the t_R and t_F as function of H_2S concentration at tested temperatures. The transient

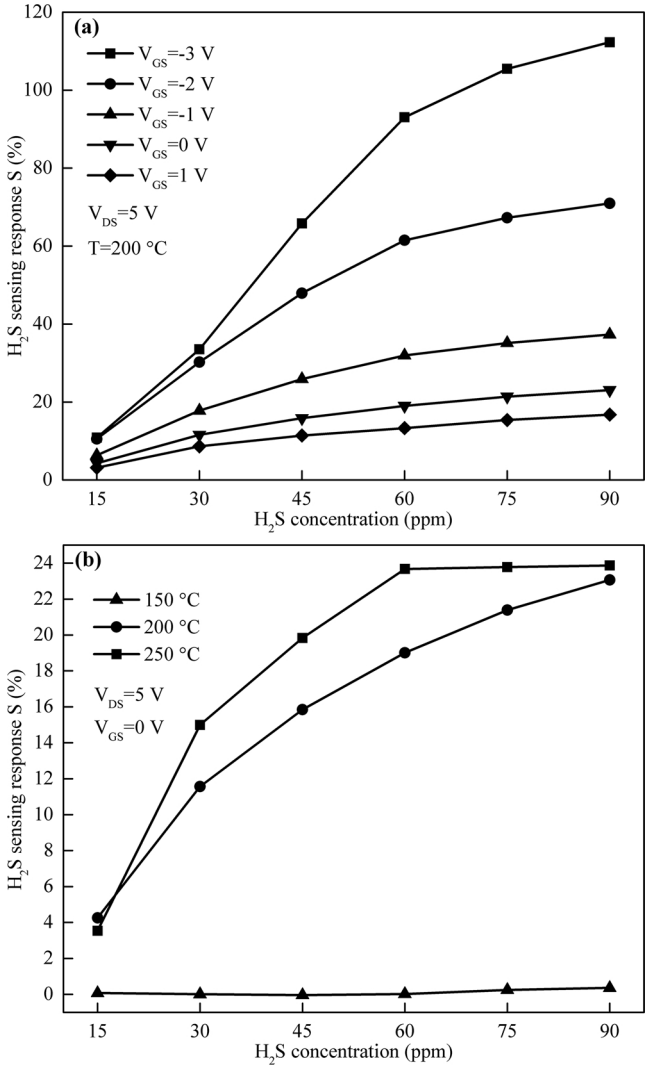

Fig. 6. Hydrogen sulfide sensor sensitivity at (a) various gate bias voltages and (b) at different temperatures.

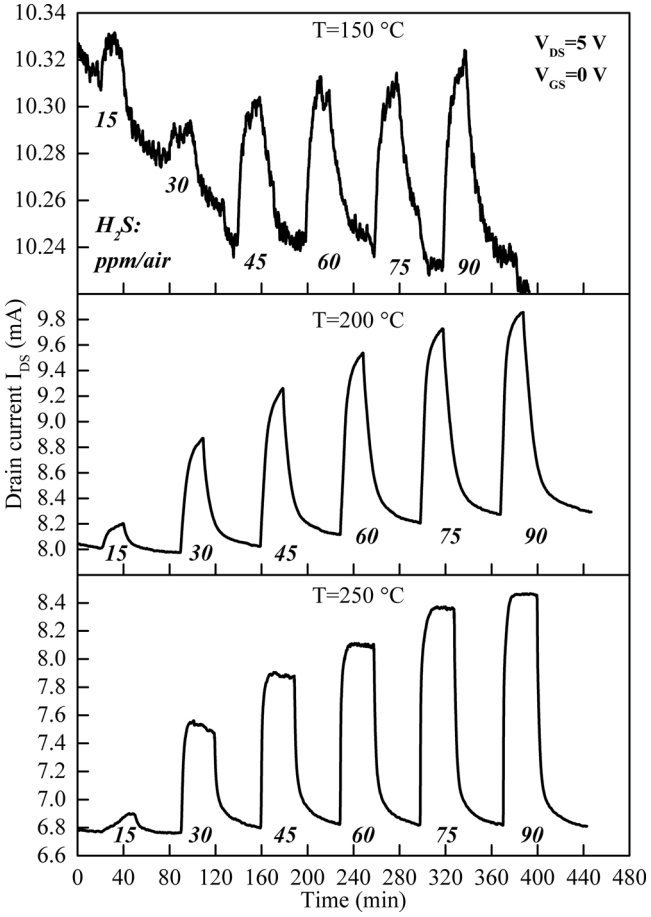

Fig. 7. Transient response characteristics upon injection and purge of H_2S in dry air ambient at 150 °C, 200 °C and 250 °C (from top to bottom). During all measurements V_{DS} = 5 V, V_{GS} = 0 V.

times decreased with increasing gas concentration for 200 °C and 250 °C. The results at 150 °C were irregular due to low signal variation and baseline current value drift at 15 ppm and 30 ppm gas concentrations which resulted in quicker t_R and t_F. Starting with 45 ppm the baseline stabilized and the response (recovery) times became more consistent. At 90 ppm H_2S concentration t_R (t_F) reduced from 476 (1316) s at 200 °C to 219 (507) s at 250 °C. The shorter rise/fall times are attributed to faster gas adsorption and desorption kinetics at the Pt surface and M–S interface with increasing temperature. Further decrease of t_R and t_F is expected with downsizing the volume of the testing chamber. Fig. 9 shows 5 consecutive exposure and purge cycles of 90 ppm H_2S. Obviously the sensor demonstrates repeatable and reversable current variation characteristics under continuous operation at 250 °C.

Pd and Pt gate field effect devices are known to detect H_2 gas, therefore a comparison of response between H_2S and H_2 was carried out. Fig. 9(a) shows the transient drain current curves of the tested sensor exposed to 60 ppm and 90 ppm of H_2S, H_2 and NO_2 at 250 °C. The sensing response to 90 ppm for each gas is summarized in Fig. 9(b). It is evident that the response to H_2S was 4.5x higher than to H_2, while the response to NO_2 was negligible and I_{DS} decreased upon gas exposure (Fig. 10).

Long-term operation stability of the Pt-HEMT sensor was tested by performing the H_2S tests over a period of 15 days. Fig. 11 shows the daily drain current variation (ΔI_{DS}) for 60 ppm and 90 ppm H_2S at 250 °C. There was no significant deterioration of the sensing signal during the testing period. We did observe however that the Au-plated pads of our ceramic testing substrates and bond-pads of the HEMT chip were corroded after extended exposure to H_2S, therefore packaging reliability needs to be further investigated to ensure stable sensor operation.

4. Conclusions

Sensors based on AlGaN/GaN HEMTs with Pt catalytic gate were fabricated and characterized for detecting ppm levels of hydrogen sulfide at high temperature. The developed devices exhibited a significant drain current increase and threshold voltage shift upon exposure to the test gas. At 200 °C maximum ΔI_{DS} of 2.17 mA (V_{GS} = 1 V) as well as high sensing response of 112% (V_{GS} = −3 V) for 90 ppm H_2S was obtained. High stability was observed with only 4% reduction of ΔI_{DS} across the tested drain saturation voltage range, which enables a wide selection of biasing conditions. At 150 °C very low response and baseline drift were likely caused by sulfur poising effect of the Pt gate. Transient measurements confirmed stable operation with excellent response, recovery and repeatability properties. The rise (fall) times reduced from 476 (1316) s to 219 (507) s when the temperature was elevated from 200 °C to 250 °C. Sensing response of H_2S was 4.5x greater than H_2 for 90 ppm concentration. The operating stability was validated over 15 days with no significant reduction of sensing signal. Our findings firmly suggest that AlGaN/GaN HEMT sensors are a promising technology for industrial wearable worker safety detectors.

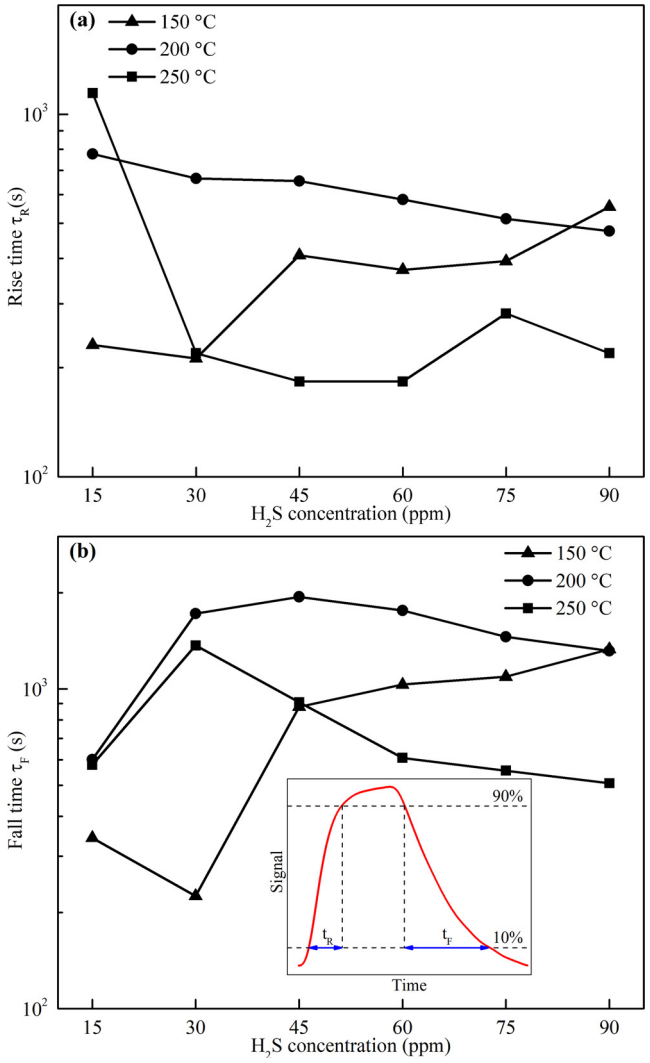

Fig. 8. (a) Rise time (t_R) and (b) fall time (t_F) versus H_2S concentration at 150 °C, 200 °C and 250 °C. The inset schematically shows the definition of t_R and t_F.

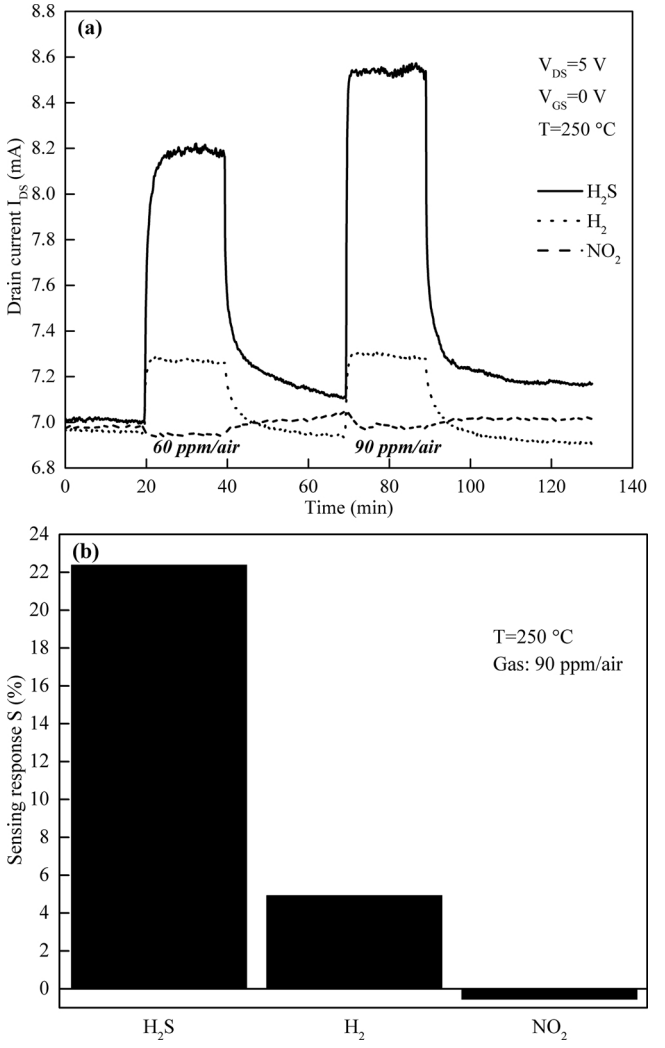

Fig. 10. (a) Transient response characteristics upon exposure to 60 ppm and 90 ppm of H_2S, H_2 and NO_2 at 250 °C in dry air ambient. (b) Sensing response towards 90 ppm of H_2S, H_2 and NO_2.

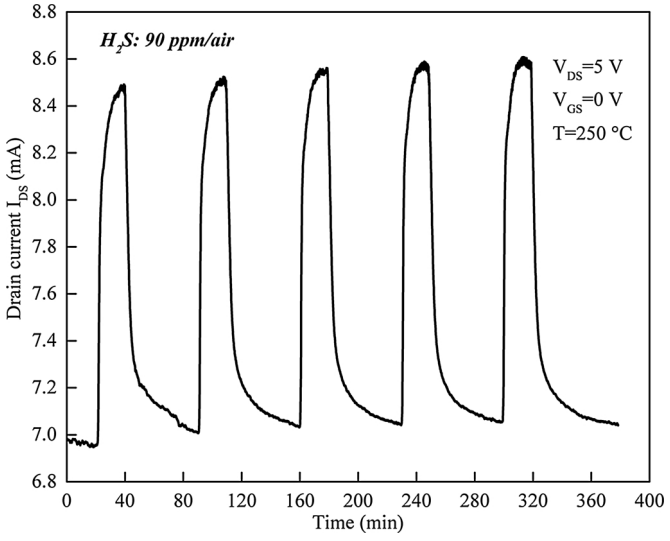

Fig. 9. Five repetitive cycles of sensor exposure to 90 ppm of H_2S at 250 °C.

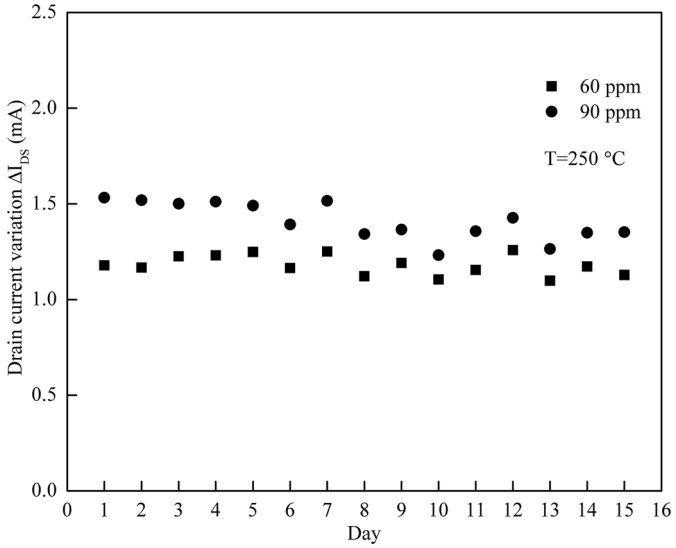

Fig. 11. Sensor drain current variation (ΔI_{DS}) characteristics towards 60 and 90 ppm of H_2S over 15 day testing period at 250 °C.

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Robert Sokolovskij received the B.S. degree in electronics engineering from Vilnius University, Vilnius, Lithuania in 2010 and the M.S. degree in electrical engineering from Delft University of Technology, Delft, the Netherlands in 2013, where he is currently working towards his Ph.D. degree. From 2014 he is also with the State Key Laboratory of Solid State Lighting, Changzhou, China. From 2018 he is part-time research assistant at Southern University of Science and Technology, Shenzhen, China. His current research interests include design, fabrication and characterization of wide bandgap gallium nitride (GaN)-based power electronic devices and chemical sensors.

Jian Zhang received the B.S. degree from Fudan University, Shanghai, China, in 2015. He is currently pursuing the Ph.D. degree in Fudan University. His current research interests

include electrical properties of metal/semiconductor interfaces, and fabrication and characterization of wide bandgap gallium nitride (GaN)-based power electronic devices and chemical sensors.

Elina Iervolino holds a Ph.D. degree in MEMS systems from Delft University of Technology since 2012. Her research interests include novel sensor development for environmental and wellness applications.

Changhui Zhao received his B.S. and Ph.D. degrees from the School of Physical Science and Technology, Lanzhou University, China, in 2011 and 2016, respectively. Since 2016, he joined in the Department of Electrical and Electronic Engineering, Southern University of Science and Technology, China, as a post-doctor. He is interested in the field of low-dimensional nanomaterials for low power micro-gas sensors.

Fabio Santagata received his Ph.D. degree from Delft University of Technology in 2012. He has extensive experience in the field of MEMS design and fabrication. He currently holds a position as technology marketing senior manager at 3NOD.

Fei Wang received the B.S. degree in mechanical engineering from the University of Science and Technology of China, Hefei, China, in 2003, and the Ph.D. degree in microelectronics from the Shanghai Institute of Microsystem and Information Technology, Chinese Academy of Science, Shanghai, China, in 2008. He was a Post-Doctoral Researcher with the Department of Microtechnology and Nanotechnology, Technical University of Denmark, where he has been an Assistant Professor since 2010. Since 2013, he has been an Associate Professor with the Department of Electronic and Electrical Engineering, Southern University of Science and Technology, China. His current research interests include micro energy harvesting, MEMS and NEMS sensors, MEMS probes for IC and semiconductor testing. Dr. Wang served as a TPC Member for the 19th International Conference on Solid-State Sensors, Actuators and Microsystems (Transducers 2017), the International Conference on Manipulation, Manufacturing and Measurement on the Nanoscale (IEEE 3M-NANO) from 2014 to 2017, and the International Multidisciplinary Conference on Optofluidics (IMCO) from 2016 to 2017.

Hongyu Yu received his BSc, MSc and Ph.D. from Tsinghua University, University of Toronto and National University of Singapore, respectively. He was a Senior Researcher at IMEC, Belgium from 2004 to 2008. From 2008 to 2011 he served as an Assistant Professor in the Department of Electrical and Electronics Engineering, Nanyang Technological University, Singapore. Since 2011 he is a Professor and Deputy Chair of the Department of Electrical and Electronic Engineering at Southern University of Science and Technology, Shenzhen, China. Prof. Yu has achieved a series of innovative results in integrated circuit technology and devices, including CMOS, new ultra-high density memory, Gallium Nitride (GaN) HEMT and system integration. He has authored and co-authored more than 350 papers (&160 journal papers + &160 conference papers), written 4 book chapters, edited 2 books and holds 20 US/European and 2 Chinese patents. He has been awarded with the "1000 Talents Program for Young Scholars", Peacock Project Shenzhen. Prof. Yu is a Pengcheng scholar, Fellow of IET and Deputy Editor of Science Bulletin Journal, Elsevier.

Pasqualina M. Sarro received the Laurea degree (cum laude) in solid-states physics from the University of Naples, Italy, in 1980, and the Ph.D. degree in electrical engineering from the Delft University of Technology, The Netherlands, in 1987. From 1981 to 1983, she was a Post-Doctoral Fellow in the Division of Engineering, Photovoltaic Research Group, Brown University, Providence, RI, USA. She joined the EE Faculty, Delft University of Technology, to establish and lead research on silicon micromachining, integrated sensor, MEMS, and material processing. In 2001, she was an appointed Antoni van Leeuwenhoek Full Professor for research merits. She has (co)-authored over 500 publications. Her main research interests include novel materials and structures for MEMS and NEMS to be applied in health, automotive, environmental applications, and scientific instrumentation. She is a member of the International Steering Committee of Eurosensors, Transducers, and IEEE MEMS. She serves on various international advisory boards and panels for research. She is an elected member of the Royal Netherlands Academy of Sciences, a Knight in the Order of the Dutch Lion, and a Knight in the Order of the Italian Star. She was the General Chair and the Technical Program Committee Chair for all major international conferences in the field of sensors, MEMS, and micro systems several times. Prof. Sarro was elected as IEEE fellow in 2007. From 2006 to 2009, she was an Associate Editor for the IEEE SENSORS JOURNAL. She is an Associate Editor for the IEEE JOURNAL OF MICROELECTROMECHANICAL SYSTEMS.

Guo Qi Zhang received his Ph.D. degree in aerospace engineering at Delft University of Technology, Delft, the Netherlands, in 1993. He is currently a Chair Professor in the Department of Microelectronics at Delft University of Technology since 2013. His research focuses on heterogeneous micro/nano-electronics packaging, system integration and reliability. Prof. Zhang had been with Philips for 20 years: Principal Scientist (1994–1996), Technology Domain Manager (1996–2005), Senior Director of Technology Strategy (2005–2009), and the Philips Fellow (2009–2013). From 2002, he also had part-time appointments as Professor at Technical University of Eindhoven (2002–2005), and Chair Professor at Delft University of Technology (2005–2013). Prof. Zhang is one of pioneers in developing More than Moore (MtM) strategy when he served as Chair of MtM technology team of Europeans Nanoelectronics Platform (ENIAC) in 2005. Prof. Zhang has published more than 350 papers including more than 140 journal papers, 3 books, and 17 book chapters, and more than 100 patents. He was awarded Outstanding Contributions to Reliability Research by European Center for Micro/Nanoreliability, Berlin in 2007. He received Excellent Leadership Award at EuroSimE and Special Achievement Award at ICEPT. He was elected as IEEE Fellow in 2014, and received IEEE CPMT Outstanding Sustained Technical Contribution Award in 2015.