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Highly selective and ultra-low power consumption metal oxide based hydrogen gas sensor employing graphene oxide as molecular sieve

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A R T I C L E   I N F O

Keywords: Graphene oxide, Molecular sieving, Hydrogen gas sensors, 2D nanomaterials, Zinc oxide

A B S T R A C T

The excellent gas sensing performance of metal oxide based nano- and microstructures, including a fast response time and good sensitivity, is typically limited by their low selectivity. Therefore, novel approaches and strategies are required to gain a precise control of the selectivity. Here, we introduce a nanoporous few-layer graphene oxide (GO) membrane with permeability only to specific gas molecules to improve the selectivity of individual zinc oxide microwires (ZnO MWs) toward hydrogen (H2) gas. The fabricated GO-covered ZnO MWs showed ultra-low power consumption (60–200 nW) and an excellent room temperature H2 gas sensing properties with fast response (114 s) and recovery (30 s) times, and a low detection limit of ∼4 ppm, while no gas response was measured to all other tested gases. As proposed, the gas sensing mechanism is based on selective sieving of H2 gas molecules through the GO membrane and further diffusion to the Schottky contacts, resulting in a decreased barrier height. Being based on a bottom-up fabrication approach, the presented results could have great potential for further technological applications such as high-performance and highly selective ultra-low power metal oxide-based gas sensors, opening new opportunities for the design of nanosensors and their integration in wireless and portable devices.

1. Introduction

Metal oxide based nano- and microstructures are one of the most prominent studied systems for the detection of gases owing to their high surface area, fast response as well as good sensitivity, with the possibility to integrate them into “electronic nose” systems [1–5]. Typically, commercial metal oxide based gas sensors require a power supply of tens to hundreds of mW in order to generate the relatively high operating temperatures of 200–400 °C using micro-machined heating elements [6], which are too high for portable applications and remote systems such as Internet of Things (IoT), lab on a chip and electronic nose devices [6,7]. Therefore, it is preferable to develop such devices based on individual metal oxide microstructures with minimized size and the ability to operate at room temperature, as reported by many authors [1,3,8,9]. However, the fabrication of high performance metal oxide nano- and microstructures able to detect different gaseous species at room temperature is strongly limited by their low selectivity [10–14]. Many methods have already been reported for the increase of the selectivity of metal oxide based gas sensors, including (i) doping of the semiconducting oxides with metal ions [15–17], (ii) surface functionalization/decoration with metal (oxide) nanostructures and polymers [16,18–20], (iii) formation of heterostructures, core-shell structures or nanocomposites by combination of n- and p-type metal oxides [4], (iv) the use of permeable membranes [14,21–23], (v) the use sensor array, such as “electronic nose”, and pattern recognition algorithms such as principal component analysis or artificial neural networks [24]; (vi) combination of the already mentioned methods [4,17]. Membranes are key elements in industrial-scale chemical and gas purification [14], as well as in selective gas sensing [21]. For instance, Zhang et al. successfully prepared a new zeolitic imidazolate framework-8 membrane architecture supported on vertically aligned zinc oxide (ZnO) nanorods demonstrating a considerable increase of the ideal selectivity to H2/CO2 [22]. Günnter et al. demonstrated that zeolite membranes are suitable filters for gas sensors, removing
undesired species from mixtures like exhaled breath, exhibiting an exceptional selectivity (> 100) for formaldehyde (down to 30 ppb) at 90% relative humidity [23]. Recently, 2D nanomaterials such as graphene and graphene oxide have also emerged as promising candidates for the fabrication of selective gas membranes due to their high mechanical strength, relative inertness and impermeability to all standard gases [14]. Koenig et al. investigated the transport of a range of gases (H₂, CO₂, Ar, N₂, CH₄ and SF₆) through suspended porous graphene demonstrating selective molecular sieving based on effusion through Ångstrom-sized pores [14]. Jiang et al. observed that subnanometer pores in graphene sheets are superior to standard polymer and silica membranes, where bulk solubility and diffusivity dominate the transport of gas molecules through the material [25]. Formation of pores in graphene has been performed by different methods like ultraviolet-induced oxidative etching, oxygen plasma etching [14,26], and focused ion beam (FIB) [10]. Next to the use of graphene, graphene oxide (GO) has been shown to be another excellent candidate for the fabrication of selective membranes [27]. Compared to graphene sheets, GO sheets stack non-uniformly and can be permeable to small gases without the need for additional pore formation [11]. These results demonstrate that membranes based on graphene and related 2D nanomaterials in combination with the high sensitivity of metal oxide nano- and microstructures are promising candidates to increase the selectivity of the latter.

In this work, we demonstrate that wet-chemical coating of individual ZnO microwires (MWs) with a thin (< 20 nm), nanoporous GO membrane leads to a strong reduction in permeability toward specific gas molecules, resulting in a highly selective and ultra-low power H₂ gas sensor (up to 200 nW). The gas response and its dependence on the gas concentration was studied in detail for different gases and volatile compounds, including methane, ammonia, ethanol, acetone, methanol and hydrogen. While the GO-covered ZnO MWs showed a high gas response toward H₂ with a detection limit of ~4 ppm, no gas response was measured for all other gases. The proposed gas sensing mechanism is based on the effusion of gas molecules through nanopores (< 50 nm diameter) and the modification of the Schottky barrier at the electrical contacts, enabling a highly selective gas response. In addition to the study of GO/ZnO MWs, other devices based on electrochemically exfoliated graphene covered ZnO MWs (EG/ZnO), reduced graphene oxide covered ZnO MWs (rGO/ZnO), and as individual EG, GO and rGO structures were fabricated and investigated in order to gain a deeper understanding of the gas sensing mechanism.

2. Experimental: materials and methods

2.1. Fabrication of carbon 2D nanomaterial dispersions

Graphene oxide (GO) was prepared according to previously reported procedures [28,29]. Afterwards, the as produced GO powder was dispersed in deionized water by tip sonication to achieve a homogenous and stable dispersion. Aqueous dispersions of electrochemically exfoliated graphene-covered ZnO MWs (EG/ZnO), reduced graphene oxide covered ZnO MWs (rGO/ZnO), as well as individual EG, GO and rGO structures were fabricated and investigated in order to gain a deeper understanding of the gas sensing mechanism.

2.2. Fabrication of ZnO microstructures

First, highly porous macroscopic networks of interconnected, tetrapodal zinc oxide (t-ZnO) microparticles were prepared using the flame transport synthesis which is described in detail elsewhere [30–32]. In brief, a mixture of polyvinyl butyral and zinc powder with grain sizes of 1 – 5 µm (mass ratio 2:1) was prepared in a crucible and heated in a muffle furnace for 30 min at 900 °C (heating rate 60 °C min⁻¹). The obtained loose t-ZnO powder was pressed into cylindrical tablets (diameter 6 mm, height 3 mm) and sintered at 1150 °C for 5 h, resulting in a network of interconnected ZnO tetrapods.

2.3. Fabrication of macroscopic ZnO-EG/rGO networks

To coat the t-ZnO network with carbon 2D nanomaterials, aqueous dispersions of GO or EG sheets (each with a concentration of 2 mg mL⁻¹) were dropped on the highly porous template (94% porosity) to fill the free volume until no more dispersion was taken up. Afterwards, the infiltrated t-ZnO template was dried at 50 °C for 4 h to promote evaporation of the solvent (water) and simultaneous deposition of the nanomaterials on the surface of the template. SEM images of t-ZnO networks coated with thin GO and EG layers are presented in Figs. S1 and S2, respectively. For preparation of a reduced GO (rGO)-covered t-ZnO network, the template was first coated with GO and subsequently reduced by immersion in diluted L-ascorbic acid (0.1 mg mL⁻¹) for 24 h (at 50 °C). Then, the template was washed thoroughly in deionized water.

For fabrication of hollow microtubes or nanosheets comprised of GO, rGO and EG sheets, the wet-chemically coated t-ZnO networks were etched in diluted HCl (1 M) for 24 h, washed thoroughly in absolute ethanol, and dried by critical point drying using an EMS 3000. The resulting networks consisted of interconnected hollow microtubes comprised of GO, rGO or EG sheets. Further details regarding the fabrication as well as mechanical and electrical characterization of the t-ZnO hybrid networks and the aero-networks can be found in another work [33,34].

2.4. Fabrication of nano devices

Nano devices based on individual structures, i.e., ZnO MWs, exfoliated graphene-covered ZnO MWs (EG/ZnO, from samples with ZnO microstructures with a density of 300 mg cm⁻² and EG with a density of 1.1 mg cm⁻²), graphene oxide-covered ZnO MWs (GO/ZnO, from samples with ZnO microstructures with a density of 300 mg cm⁻² and GO with a density of 2 mg cm⁻²), reduced GO-covered ZnO MWs (rGO/ZnO, from samples with ZnO microstructures with a density of 300 mg cm⁻² and rGO with a density of 2 mg cm⁻²), EG, GO and rGO microtubes/microsheets were fabricated in a FIB/SEM system using the procedure reported by Lupan et al. [3,8,9,15,35,36].

2.5. Gas sensing measurements

For gas sensing measurements, nearly practical gaseous environments were intentionally used, i.e., the measurements were performed under atmospheric pressure [3,8,9], and dry air from the background (relative humidity of 30–40%) was used as a carrier gas to dilute the necessary concentrations of gases. All measurements with devices based on individual structures were performed at room temperature (20 – 24 °C) under a dc bias voltage of 1 V. The gas response was defined as the ratio of the sensor resistance upon exposure to air (R空气) and upon exposure to the sample gas (R样) for n-type response and as R样/R空气 for p-type response [37]. More details can be found in previous works [37–39].

3. Results and discussion

The fabrication of the ZnO MWs covered with thin membranes of GO, rGO and EG is based on a simple wet-chemical coating technique which has recently been published and discussed in detail [33]. In brief, a highly porous (~94%) 3D network of interconnected ZnO MWs was infiltrated with an aqueous dispersion of the carbon 2D nanomaterials, resulting in filling of the entire free volume of the network. During evaporation of the solvent, the 2D flakes assembled on the surface of the ZnO MWs to form a uniform nanoscopic layer. Figs. S1 and S2 show scanning electron microscopy (SEM) images of the ZnO MW networks coated with a thin GO and EG membrane, respectively. By variation of the number of infiltrations or the concentration of the nanomaterial dispersion, the coverage could be easily controlled from less than a
monolayer (no interconnected layer of nanomaterial flakes) up to several layers of GO, rGO and EG. SEM images of gas sensing devices based on individual pristine and GO-covered ZnO MWs are presented in Fig. 1a,b and d,e, respectively. Additionally, SEM images of GO/ZnO MWs with different magnifications are shown in Fig. S1. Strikingly, the SEM images of GO-covered ZnO MWs (Figs. 1c and S3a–e) reveal a high density of wrinkles within the assembled GO layer. Wrinkling is known to be a ubiquitous phenomenon in large 2D membranes [40] and has already been reported for other graphene-based structures. For instance, Grosse et al. observed a small temperature rise at wrinkles in CVD grown hexagonal graphene grains using scanning Joule expansion microscopy [41], which is very important for gas sensing applications. Zhang et al. also observed a reversible wrinkling and unwrinkling of graphene sheets on the Pt (111) surface upon cycled heating and cooling treatments [42]. The high concentration of wrinkles can also be observed on the surface of rGO-covered ZnO MWs (Fig. S4).

While the diameter of both the pristine and GO-covered ZnO MWs (Fig. 1a and b) was 0.57 μm at one end and 1.2 μm at the other end, the diameter of the rGO-covered ZnO MW was 1.15 μm. Thus, all individual structures had practically the same diameter allowing the comparison of their gas sensing properties without considering the geometrical parameters. This is very important due to the high dependence of the gas response on the diameter in the case of individual structures, which was demonstrated experimentally and theoretically [39,43,44]. The structure of the fabricated devices based on pristine and GO-covered ZnO MWs is illustrated in Fig. 1c and f, respectively.

The current–voltage (I–V) characteristics of devices based on individual ZnO, EG/ZnO, GO/ZnO and rGO/ZnO MWs (range of −5 to +5 V) are presented in Fig. 2a.

The graph directly demonstrates that the coating of carbon nanomaterials on the ZnO MWs has a strong influence on the I–V characteristics of the individual devices. The pristine ZnO MW had the highest resistance of all the fabricated devices, resulting in a rather low current at the same applied bias. By covering the ZnO MW with graphene-based nanomaterials (EG, GO, rGO), the resistance decreased, probably by adding their lower resistance to the higher resistance of the ZnO MW in parallel. The EG/ZnO MW showed the lowest resistance of all fabricated devices, which can be associated with the high conductivity of the graphene sheets [45,46]. The rGO-covered ZnO MW exhibited a slightly higher resistance compared to EG/ZnO, while the addition of GO to the ZnO MW decreased the resistivity only marginally. The latter can be attributed to the fact, that GO is insulating by nature [47], thereby only slightly contributing to the conductivity of the device. The room temperature gas sensing properties of all devices to different types of reducing gases and vapors with a concentration of 1000 ppm are presented in Fig. 2b. The error bars represent the deviation in the gas response $R_{\text{air}}/R_{\text{gas}}$ ($R_{\text{air}}$: sensor resistance in air, $R_{\text{gas}}$: sensor resistance in sample gas) after several measurements at the same concentration of the sample gas. In the case of ZnO MWs and GO/ZnO MWs, an n-type gas response was observed, i.e., the decrease in electrical resistance upon exposure to reducing gases, which is typical for ZnO nano- or microwires [48,49]. This indicates that the gas response can be mainly attributed to the ZnO MW. The gas response of the pristine ZnO MW to 1000 ppm of H2, ethanol, CH4, NH3, methanol and acetone was 7.6, 4.4, 1.6, 2.9, 4 and 5.1, respectively, showing no selectivity toward any of the gases. In contrast to that, the addition of a GO membrane resulted in a drastic increase in selectivity toward H2 gas (gas response 3.4), while there was no gas response to all other investigated gases. Compared to the pristine ZnO MW, however, the gas response of GO/ZnO was by a factor of 2.2 lower. The measurements with the EG/ZnO and rGO/ZnO MWs revealed a p-type response, i.e., the increase in electrical resistance upon exposure to reducing gases, which is typical for carbon based nanomaterials [46,50,51]. Therefore, the gas response can be mainly ascribed to the sorption and gas sensing properties of the EG and rGO membrane. For the rGO/ZnO MW, the gas response to 1000 ppm of H2, ethanol, CH4, NH3, methanol and acetone was 1.77, 1.26, 1, 2.3, 1.06 and 1, respectively, showing no selectivity. For the EG/ZnO MW only a low response to NH3 (−1.1) was observed, which is in accordance with the literature, but not of importance in the scope of this study. It can be assumed that the lower resistance and poor gas sensing properties of the EG membrane (compared to ZnO) lead to a considerably lower gas response, i.e., the change in resistance of the ZnO MW is shunted by a lower parallel resistance of the EG layer [52]. Since the GO/ZnO system represents the highest interest due to its high selectivity, only the gas sensing properties of ZnO (for comparison) and GO/ZnO MWs are investigated in detail in the following paragraphs.

Fig. 2c shows the dependence of the room temperature gas response on the concentration of H2 gas for pristine and GO-covered ZnO MWs. A power law relationship of gas response ($S$) and H2 gas concentration ($p_{\text{H2}}$) can be observed ($S \propto p_{\text{H2}}^\beta$), where $\beta$ is the slope of log $S$ versus log $p_{\text{H2}}$ [53]. The obtained $\beta$ values for pristine and GO-covered ZnO MWs are 0.3 and 0.25, respectively. These values depend on the charge of the surface-adsorbed species and the type of chemical reaction on the surface [54,55]. Since it is nearly the same for both investigated systems, the same type of surface chemical reaction between chemisorbed oxygen species and the H2 gas molecules can be expected [54,55]. The detection limits were measured using the signal/noise ratio, as reported by Dua et al. [50], and yielded values of ∼2 ppm for the pristine
ZnO MW and ~4 ppm for the GO/ZnO MW. The calculated power consumption of pristine and GO-covered ZnO MWs at 1 V applied bias voltage versus the concentration of H2 gas is also presented in Fig. 2c. In a passive state, i.e., when no gas is applied, the power consumption of pristine and GO-covered ZnO MWs was ~4 nW and ~60 nW, respectively. By increasing the H2 gas concentration up to 1000 ppm, the power consumption increased up to ~28 nW and ~200 nW, respectively. This demonstrates that the fabricated devices can operate at extremely low power levels in the nW region, even at application of relatively high gas concentrations of 1000 ppm (0.1%). The dynamic gas response of individual pristine and GO-covered ZnO MWs to different concentrations of H2 gas at room temperature is presented in Fig. 2d. The gas response of the pristine ZnO MW to 10, 50, 250, 500, 1000 and 2000 ppm H2 was 1.5, 2.8, 4.7, 6.1, 7.6 and 10 s, respectively (Fig. 2c and e). In comparison, the gas response of the GO-covered ZnO MW to 10, 25, 50, 125 and 1000 ppm H2 was 1.15, 1.4, 1.71, 2.1 and 3.42 s, respectively (Fig. 2c and f). In the case of the pristine ZnO MW, the signal recovered completely after evacuating the H2 gas from the test chamber (Fig. 2e), while it did not fully recover for GO/ZnO (Fig. 2f) (at least not in the investigated time intervals). Furthermore, the response and recovery times for the pristine ZnO MW decreased from 200 s to 43 s and from 68 s to 31 s, respectively, by increasing the concentration of H2 gas from 60 to 2000 ppm (Fig. 2d). In contrast, it can be seen that the response and recovery times of the GO-covered ZnO MW to H2 were significantly reduced (Fig. 2d), i.e., from 211 s to 114 s (response) and from 106 s to 30 s (recovery) upon increasing the H2 concentration from 125 ppm to 1000 ppm. Thus, the GO membrane did not only strongly enhance the selectivity of the ZnO MW, it also improved the response and recovery times.

The dynamic gas response to 1000 ppm of different gases and vapors (hydrogen, ethanol, methane, ammonia, methanol and acetone) of pristine and GO-covered ZnO MWs is presented in Fig. 3a and b (dynamic resistance measurement in Fig. S6a and b), respectively. The graphs confirm the high selectivity of the GO-covered ZnO MW toward H2 gas compared to the pristine ZnO MW, showing no gas response to all tested gases except H2 with a significant increase in signal. In order to investigate the stability of the GO/ZnO sensors, the gas response of the device was also measured under cyclic H2 gas exposure.
with the same concentration (1000 ppm, Fig. 3c and Fig. S6c). The graph demonstrates the repeatability of the gas response with a residual standard deviation smaller than 5%. The dynamic response of the individual rGO/ZnO MW to 1000 ppm of H2, ethanol, NH3, and methanol is presented in Fig. S7.

In order to exclude the potential influence of the GO membrane on the gas response of GO-covered ZnO MWs, gas sensing measurements were also performed on individual GO membranes. These were prepared by wet-chemical etching of the GO/ZnO MWs, resulting in nanoscopic thin GO microsheets (inset in Fig. 3d). The gas response of the GO membrane was measured toward 1000 ppm of different gases at room temperature. As revealed from the results in Fig. 3d, the membrane did not show any response to all of the tested gases. This confirms that the GO membrane itself does not contribute to the gas response of the GO/ZnO system. The gas response and current-voltage characteristics of devices based on individual EG and rGO membranes are presented in Fig. S8, showing minor gas responses to NH3 typical for carbon-based nanomaterials with enhanced conductivity [56].

The gas sensing mechanism of individual ZnO structures has already been proposed and described in detail [15]. In this case, the surface-dependent electrical properties of the ZnO MWs are dominated by the formed Schottky contacts at the Pt/ZnO interface [49,52,57]. As confirmed by the current-voltage characteristics of the sensing device based on the ZnO MW (Fig. 2a), the higher work function of Pt ($\phi_{Pt} = 6.1$ eV) compared to the electron affinity of ZnO ($\chi = 4.5$ eV) [58] leads to the formation of double Schottky contacts. Owing to the strong catalytic effect of noble metals like Pt or Pd, the formation of Schottky contacts with ZnO are known to contribute significantly to the H2 gas response [43,52,57,59,60]. This can explain the higher gas response to H2 gas of device based on individual ZnO MW compared to other tested gases, observed in our case (see Fig. 2b) and reported previously [3,8,9]. Similarly, this effect has even been reported for sensing devices based on carbon nanotubes and silicon, which are usually not sensitive to H2 gas at room temperature [43,57,61,62]. This can explain the observed higher gas response of the ZnO MWs toward H2 gas compared to the other sample gases, which has already been reported in previous experimental studies [3,8,9]. The high selectivity of the GO-covered ZnO MWs, however, has not been investigated yet and may be explained as follows. A perfect monolayer graphene sheet is impermeable to gases as small as He [63]. Therefore, to use graphene or GO as a membrane material, it is necessary to form nanosized pores in the GO layer in order to achieve a certain gas permeability [25]. In this work, the formation of these pores might be related to the fabrication of the GO membrane on the ZnO MW. Since the GO membrane is produced by wet-chemical assembly of individual GO sheets on the surface of a ZnO MW [33], the assembled layer constitutes nanosized pores between the interconnections of the GO sheets which originate from partial overlapping and non-uniform assembly. Additionally, during the deposition of Pt for the formation of electrical contacts between the GO/ZnO MW and Au pads in the FIB/SEM system, nanopores could be formed within the GO membrane near the Pt contacts [10]. For example, Celebi et al. used Ga-based FIB to perforate apertures between 14 nm and 1 mm in diameter and He-based FIB for < 10 nm-pore drilling [10]. In addition to the pore formation within the GO membrane, the irregular stacking in few-layer GO causes an interplanar spacing larger than that of graphitic layers (0.335 nm) [11]. As a result, both the nanopores and interlayer region of the GO sheets play a central role for size-selective molecular sieving of the tested gas molecules. In particular, smaller molecules with weaker interaction with GO (i.e., H2) diffuse faster than larger molecules (i.e., all other tested gas molecules, see molecular diameters in Table S1), giving rise to the observed response of the GO-covered ZnO MW only to H2 gas among all tested gases [13,63]. As schematically shown in Fig. 4a, the diffusion of the H2 molecules can occur through the nanopores and the interlayer region of the GO sheets [13].

The interlayer diffusion could be the rate-limiting process compared to the cross diffusion through nanopores in GO layers, because the lateral size of the 2D diffusion channel is the order of micrometers, while the interlayer distance is the order of sub-nanometers [13,64]. In the case of nanopores (< 50 nm), the transport of molecules occurs via effusion, because the mean free path ($\lambda$) becomes larger than the nanopore diameter (d) [10]. The effusion mechanism is known to be

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**Fig. 3.** Dynamic gas response at room temperature (20–24 °C) to different gases with a concentration of 1000 ppm for (a) a ZnO MW and (b) a GO-covered ZnO MW. (c) Dynamic response under cyclic exposure to H2 gas with a concentration of 1000 ppm. (d) Gas response of an individual GO membrane to 1000 ppm of different gases at room temperature. The inset micrograph shows the fabricated device used for the measurement.
4. Conclusions

In summary, we have demonstrated a simple concept for the fabrication of a highly selective and ultra-low power (60–200 nW) H₂ gas sensor, which combines the excellent sensitivity of metal oxide microstructures with the selectivity of a few-layer GO membrane based on molecular sieving. The devices were prepared by a scalable wet-chemical synthesis strategy in which a self-assembled layer of 2D nanomaterial sheets, such as GO, formed a homogeneous coating on the metal oxide surface. We propose that the induced nanopores in the GO membrane act as a size-selective sieve, which only allows permeation of hydrogen molecules among the tested gases, while other gases (e.g., ethanol, methane, ammonia, acetone and methanol) cannot pass the membrane. The obtained experimental results support this hypothesis and are consistent with theoretical models developed in the literature based on diffusion through nanometer-sized (< 50 nm) pores. The gas sensing mechanism is explained based on the modulation of the Schottky barrier due to dissociation of H₂ gas molecules at the Pt/ZnO interface and subsequent diffusion, leading to the formation of a dipole layer. The obtained results demonstrate the great potential to use GO with engineered nanopores for the development of highly selective hydrogen gas sensors. Combined with the small size, light weight and possibility to integrate such MWs with a high density, such devices can become the essential building blocks for mobile breath analysis systems, air quality monitoring systems, and sensor networks technology for nanosensing applications.

Author contributions

OL, RA and FR came up with the concept and identified the idea. FR, VP, FS, YKM, MRL, ASN, RA, XF and OL designed the study. FR and OL fabricated the samples. OL fabricated the sensors on individual microwires. FR, VP, FS, RA, MRL, ASN and OL analyzed the data. FR and OL carried out the SEM measurements. VP and OL carried out the I–V and gas sensing measurements and analyzed the data. FR, VP, FS, RA and OL finalized the study and wrote the paper. All the authors have contributed to the discussion of the results and reviewed the manuscript.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to
influence the work reported in this paper.

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