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Novel hydrogen gas sensing by palladium electrode on dielectric capacitor coupled with an amorphous InGaZnO thin-film transistor

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ABSTRACT

Palladium (Pd) is well known for its capability to selectively detect hydrogen (H_2) gas, where the detection process involves absorbing hydrogen gas molecules to form compound palladium hydrides. Such Pd–H interaction leads to the increase of electrical resistance and volume of Pd, simultaneously lowering its work function. These Pd-based hydrogen sensors would be more beneficial when connected to conventional semiconductor integrated circuits. Here, we utilize the Pd film as H-sensing electrode for metal/SiO_2/p^+-Si (MIM) capacitor, since we found the H-induced chain reactions in Pd/SiO_2/p^+-Si capacitor:Pd volume expansion, Pd-SiO_2 contact are change, and the capacitance change. This capacitance change is connected to the gate of an electrically stable amorphous InGaZnO (a-IGZO) thin-film transistor (TFT). As a result, H-induced output as the drain current of a-IGZO TFT was statically and dynamically measured through the capacitance signal change from Pd-MIM sensor. This output current signal was converted to voltage when a load resistor was connected to the a-IGZO TFT in series. These sensor circuit configurations are regarded promising and novel because of their simplicity and practicality.

1. Introduction

In recent years, hydrogen gas (H_2) has become an important energy resource as one of the environmentally green fuels along with solar energy. However, it is flammable, explosive, and even dangerous when the maintenance and monitoring are so ill cared that its concentration exceeds over 4% in air. In this regards, there have been so many efforts to avoid any H-induced risk by developing H_2 gas sensor for safety [1–7]. For hydrogen gas sensing applications, palladium metal (Pd) has long been considered and extensively studied as a promising hydrogen sensing material because of their simple fabrication, low cost, size reduction, and compatibility with the conventional semiconductor integration process [8–11]. In particular, Pd is also well known for selective detection of H_2 gas, changing its electrical resistance, volume, and work function by interaction with H_2 molecules [12–16]. Among the H-induced property changes, the lattice expansion mechanism of Pd has recently been proposed, since such changes were accompanied by electric resistance change and were reversible with quite a fast response time and a high sensitivity [17–22]. In the present work, we utilize the volume expansion mechanism of Pd film, which is now H-sensing electrode for metal/SiO_2/p^+-Si capacitor. So, the present work uses Pd/SiO_2 contact area (or capacitance) variation induced by Pd-to-PdH_x phase transformation (which leads to volume expansion). This would be quite a difference from the previous report, which mainly used a Pd nanogap in standalone thin film as an electrical switching media or resistor [22]. We name the H-sensing capacitor part as Pd-induced metal–insulator–metal (Pd-MIM) sensor, because here we do not use the electrical resistance change of Pd but rather use capacitance change of Pd-MIM capacitor. This sensor part is connected to the gate of an electrically stable amorphous InGaZnO (a-IGZO) thin-film transistor (TFT). As a result, H-induced output drain current (I_D) of a-IGZO TFT was statically and dynamically measured through the capacitance signal change from Pd-MIM sensor. This output current signal was converted to voltage when a load resistor was connected to the a-IGZO TFT in series. These hydrogen sensors utilizing a-IGZO TFT

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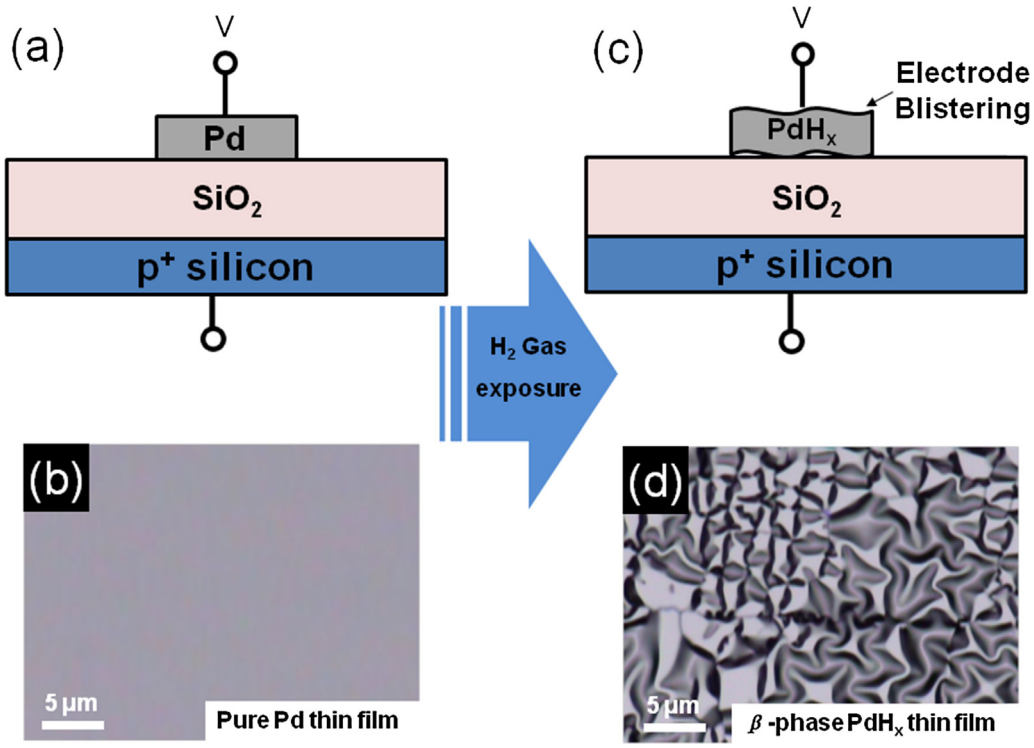

Fig. 1. (a) Schematic of $Pd/SiO_2/P^+$-Si (Pd-MIM) device and (b) optical microscopy image on the surface of pure thin film. After H_2 exposure, (c) schematic illustration of $PdH_x/SiO_2/P^+$-Si device and (d) optical microscopy images on the surface of β-phase PdH_x thin film.

can be highly beneficial owing to their good compatibility with conventional semiconductor integrated circuits at room temperature under atmospheric pressure.

2. Experimental

In order to fabricate the Pd-MIM type sensor, 50 nm-thin Pd film was deposited on the cleaned 200 nm-thick SiO_2/p^+-Si substrate by using a direct current (DC) magnetron sputtering system under 2 mtorr of working pressure. The Pd electrode is patterned through shadow mask as shown in Fig. 1a (area dimension: 1.1 mm [L] × 1.0 mm [W]). After fabrication of sensor, optical microscopy images of Pd thin film before and after H_2 exposure in Fig. 1b and d, respectively. Capacitance–time (C–t) characteristics of Pd-MIM were measured along with capacitance–voltage (C–V) characteristics using a semiconductor parameter analyzer (HP 4284A, Agilent Technologies) at room temperature (RT: 300 K) with 10 kHz. The capacitance–time (C–t) measurement was performed under controlled gas conditions: alternate cycles of H_2 gas (96% N_2 + 4% H_2 mixture) and air (or N_2) ambient at 1 atm with relative humidity of 15%, while The C–V measurement was performed under controlled gas conditions: alternate cycles of H_2 gas (96% N_2 + 4% H_2 mixture) and air ambient at 1 atm.

In the next place, a 40 nm-thick a-IGZO thin film was deposited on cleaned substrate using RF magnetron sputtering system. And then, the IGZO active channel was carried out by photo-lithography that involved wet chemical etching process. To define the channel, BOE 200:1 etchant was used and the etching rate was ~4 nm/s at RT. After that, Au/Ti (50 nm/50 nm) source (S) and drain (D) electrodes was deposited by DC magnetron sputtering system and patterned by a combination of photo-lithography and lift-off processes. A 50 nm Al_2O_3 gate insulator layer was deposited on the patterned Au/Ti (S/D) electrodes and IGZO active channel by atomic layer deposition (ALD) system. And 50 nm-thick Au top-gate electrode was formed in the same way as S/D electrode. Then, the Pd-MIM sensor was connected to an electrically stable a-IGZO TFT device (to the gate electrode) in a probe station in the dark. All drain current–gate voltage transfer curve and dynamic drain current were measured using a semiconductor parameter analyzer (HP 4155C, Agilent Technologies). Besides, load resistor (100 MΩ) was connected to the drain electrode of a-IGZO TFT for consisting of inverter system according to the hydrogen response at RT within 1 atm. And all output voltage transfer characteristics and dynamic output voltage also were measured using same analyzer and under controlled gas conditions of a cycle of exposure to H_2 gas as same as in C–V measurement.

3. Results and discussion

3.1. H_2 sensing mechanism and time-dependent capacitance behavior of $Pd/SiO_2/p^+$-Si (Pd-MIM) during H_2 infusion

We fabricated the simple $Pd/SiO_2/p^+$-Si device: metal–insulator–metal (Pd-MIM) capacitor as hydrogen sensor to investigate the physical and electrical changes before and after high concentration of H_2 gas exposure. Fig. 1a and b shows the schematic cross section of Pd-MIM capacitor and the top view optical microscope image of 50 nm-thick pure Pd thin film surface, respectively. After 4% H_2 exposure, large blisters are observed throughout the entire surface of the Pd film as Fig. 1d and c illustrates the phenomena which present the phase transition from α-phase to β-phase [23]. According to the reports, Pd can easily absorb H_2 gas to preserve H atoms in its lattice, but as a result, its volume expands eventually leading to phase transition which occurs from pure Pd to palladium hydride α-phase in the first stage but then to β-phase if the Pd is under the high H_2 concentration above 1.5%. In the second stage, the α-phase becomes to β-phase PdH_x compound and excess H atoms bring about the dislocation defects and serious lattice expansion. The lattice parameter can increase to ~4.03 Å in maximum, which means that volume of Pd thin film can expand to

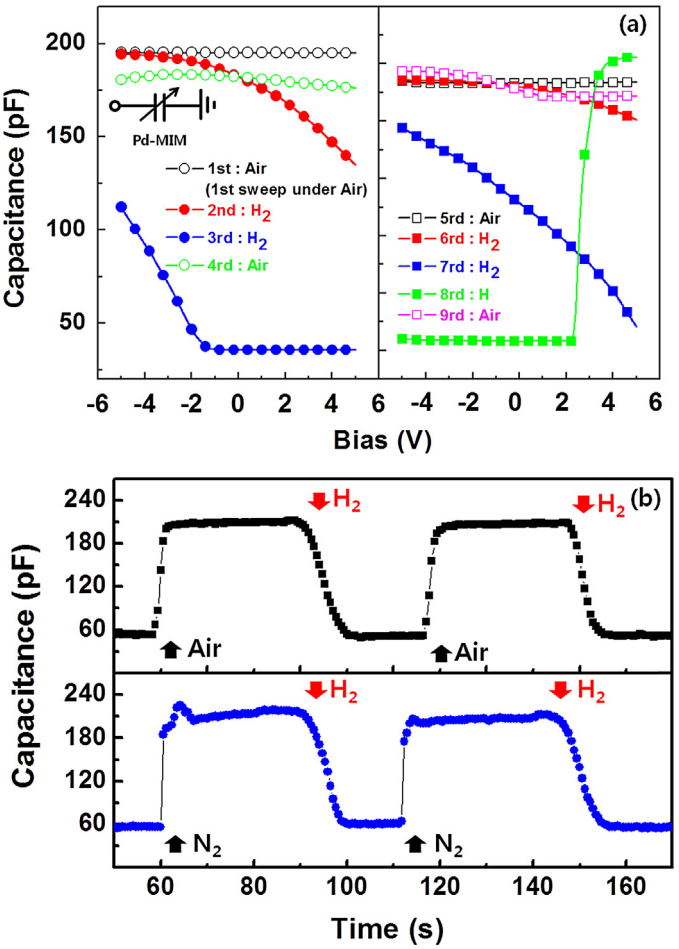

Fig. 2. (a) Capacitance–voltage (*C*–*V*) characteristics of Pd-MIM, obtained at 10 kHz alternately under 4% H_2 exposure and under ambient air at room temperature. Inset is the corresponding equivalent circuit of variable capacitor which depends on H_2 exposure. (b) Capacitance vs. time (*C*–*t*) plots of Pd-MIM obtained by repeated 4% H_2 exposure. We here used two different purge gases for H_2-free ambiences: air with relative humidity of 15% and pure N_2 gas at room temperature (300 K).

about 110% of its original volume in Fig. 1c and d [24–28]. Such volume expansion results in a partial break-off between Pd thin film and the substrate, which is called blistering or peeling off.

The Pd-MIM type devices as hydrogen sensor were studied once by researchers but they only utilized the H-induced work function change in Pd, according to which their device form was Pd-SiO_2/p-Si (MOS capacitor) and the work function change induced flat band voltage in capacitance–voltage (*C*–*V*) plots [29–34]. However, our Pd-MIM uses capacitance changes originating from the changes of Pd volume and Pd/SiO_2 contact area that occurs when exposed to high concentration H_2 gas ambient. *C*–*V* plots of Pd-MIM capacitor are measured at 10 kHz under the repeated conditions of H_2 infusion (*C*–*V* sweep stage 2, 3, 6, 7 and 8) and H_2 removal (stage 4, 5, and 9) as shown in Fig. 2a, where the curve was initially flat as ~195 pF but decreased to ~35 pF with the 4% H_2 gas exposure while it recovers to more than 90% of the initial capacitance value with H_2 removal. The $C_{Pd\text{-}MIM}$ can be expressed as:

$$C_{Pd\text{-}MIM} = \varepsilon \frac{A}{d} \quad (1)$$

where A is the contact area (1.1 m^2) between Pd film and SiO_2 layer, d is the thickness (200 nm) of insulator layer, and ε is the dielectric constant in SiO_2. The $C_{Pd\text{-}MIM}$ is thus estimated to be ~190 pF in calculation, and was measured to be 195 pF in the *C*–*V* measurement, which is quite similar to the calculation. According to the *C*–*V* measurements, the capacitance decreases to ~35 pF soon after H_2 exposure, indicating that the remaining contact area between PdH_x and SiO_2 would be only 15% of initial area. However, the capacitance gets back to ~180 pF after removing H_2 exposure (going back to air ambient), which is almost 93% of initial contact area. Interesting to note here was that the ~35 pF and 180 pF values were reproducible in our repeated H_2 sensing processes. Such reproducibility of *C*–*V* plots was well supported by *C*–*t* plots, when implemented with other sets of Pd-MIM samples under the repeated conditions of H_2 infusion/removal in air and N_2 ambient (Fig. 2b). In both cases of air and N_2 ambient, the capacitance was as high as ~210 pF but decreased to ~60 pF with the 4% H_2 gas exposure in 8 s while it rapidly recovers the initial capacitance value in 1–2 s with H_2 removal (by air or N_2). As expected in the optical microscopy image in Fig. 1d, the change of contact area respectively by H_2 infusion and removal is attributed to the blister developed after H_2 exposure. Since we regard that the reproducible *C*–*t* and *C*–*V* characteristics are important findings, we here implemented a H_2 sensing circuit application by connecting our Pd-MIM capacitor to the gate of an electrically stable a-IGZO TFT.

3.2. H_2 sensing circuits comprised of Pd-MIM and a-IGZO TFT

Based on the capacitance change of Pd-MIM sensor by H_2 infusion, we constructed a H_2 sensing circuit comprised of the Pd-MIM capacitor and a-IGZO TFT, of which the top gate is connected to the Pd-MIM capacitor (see the TFT cross section and the equivalent circuit of sensor in Fig. 3a and b). Hydrogen-induced transfer curves (drain current–gate voltage; I_D–V_G) are obtained under H_2 exposure and air condition as shown in Fig. 3c. As shown in the transfer curves of Fig. 3c and S1, our a-IGZO TFT was initially very stable with less than ~10 pA of leakage current, turning on at ~1.3 V. (ON/OFF current ratio was more than ~10^7 and the linear field-effect mobility was ~1.5 cm^2/V s). When the gate of our a-IGZO TFT is connected to Pd-MIM, the total gate capacitance (C_{total}) of the equivalent circuit should become smaller than the dielectric oxide capacitance C_{ox} because of the incorporation of a Pd-MIM with its capacitance ($C_{Pd\text{-}MIM}$) as shown below:

$$C_{total} = \frac{1}{(1/C_{Pd\text{-}MIM} + 1/C_{ox})} \quad (2)$$

The smaller C_{total} leads to a slightly reduced drain current than that by C_{ox} alone at the same V_G, as shown in Fig. 3c. The ON state drain current of our oxide TFT without Pd-MIM (at V_G = 20 V) was ~10^{-4} A but apparent decreases to ~2×10^{-5} A with the gate-connected Pd-MIM. Further decease of I_D (to ~2×10^{-6} A) is shown at 20 V of V_G after H_2 exposure/infusion (H_2 ON), which results from H_2-induced capacitance reduction of Pd-MIM. Interestingly, drain current precisely recovers to previous value under H_2 removal (H_2 OFF). Now we selected a voltage condition of V_D = 1 V and V_G = 7 V to obtain a time-domain response of H_2 ON/OFF behavior (as indicated by a vertical dashed line in Fig. 3c). Fig. 3d shows the dynamic response of the Pd-MIM-connected TFT, which appears quite consistent with the I_D results of Fig. 3a; $I_D = 5 \times 10^{-8}$ A for H_2 ON, and 3×10^{-6} A for H_2 OFF, so H_2 ON/OFF ratio was more than ~100 in I_D). Faster response (falling time ~5 s) was observed for H_2 ON condition than for H_2 OFF (rising time ~25 s), which is somewhat in accordance with the previous report. According to the report, the Pd-to-PdH_x phase transition includes α-to-β phase transition of PdH_x depending on H content, *x*, and the α-to-β transition is known to faster than its reverse transition (β-to-α) [35].

As a final experimentation, we constructed an inverter-type device to extract out a voltage (V_{OUT}) as a H-sensing signal by combining Pd-MIM/a-IGZO TFT couple and a load resistor of R = 100 MΩ in series (see the inset equivalent circuit in Fig. 4a). So, by measuring V_{OUT} signal with respect to input voltage (V_{IN}) under H_2

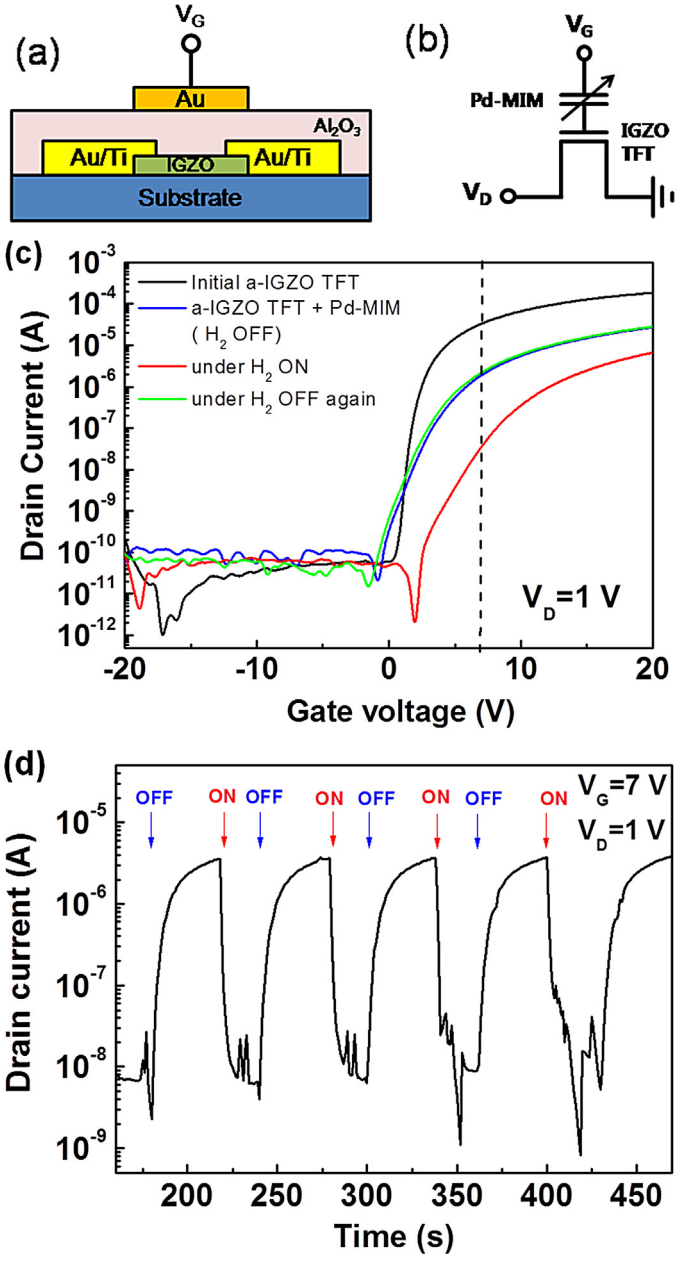

Fig. 3. (a) Schematic view of a-IGZO TFT (Au/Al_2O_3/a-IGZO) and (b) the corresponding equivalent circuit of a-IGZO TFT linked to Pd-MIM variable capacitor. (c) Drain current-gate voltage (I_D–V_G) transfer curves obtained from the initial a-IGZO TFT (black line) and from the a-IGZO TFT linked to Pd-MIM variable capacitor under 4% H_2 exposure (H_2 ON) and under air condition (H_2 OFF) under V_D = 1 V. The vertical dash line indicates V_G = 7 V we used for measurement of dynamic current. (d) Drain current vs. time plot achieved by H_2 exposure or not (ON/OFF) in a-IGZO TFT linked to Pd-MIM.

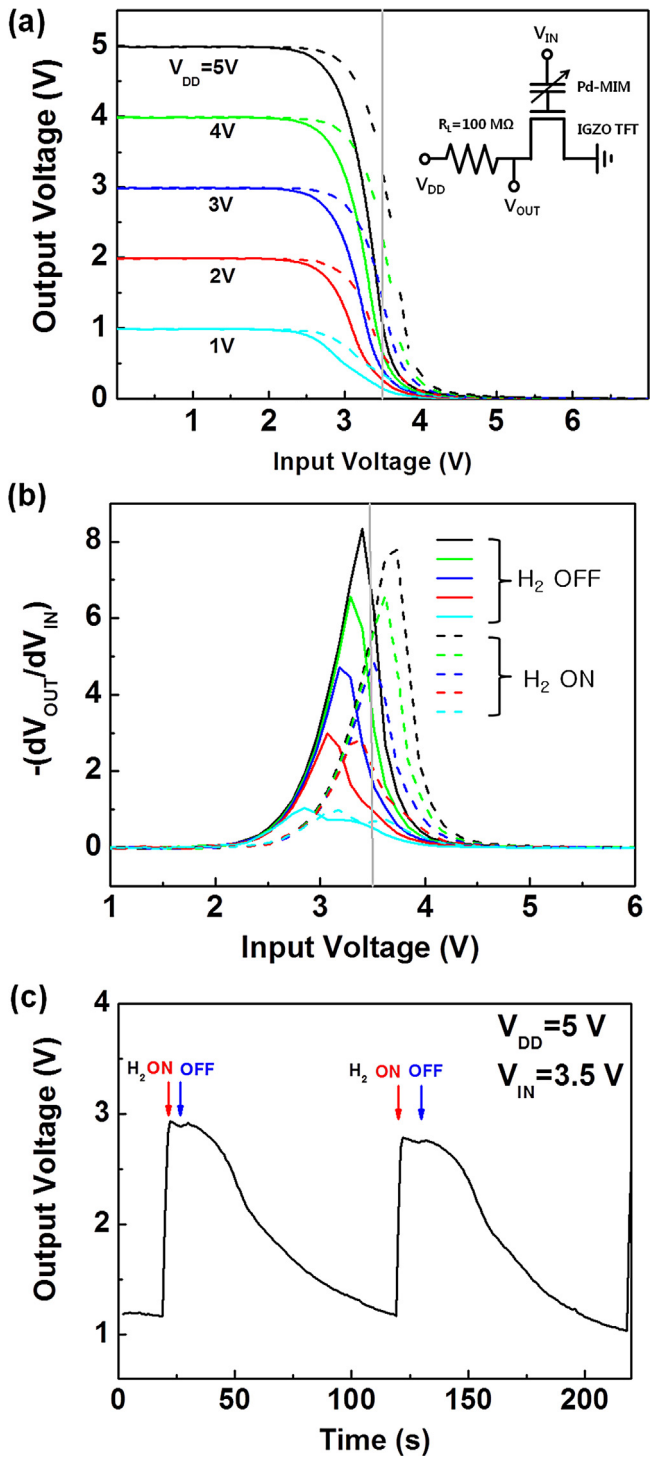

Fig. 4. (a) Voltage transfer curve and (b) voltage gain ($-dV_{out}/dV_{in}$) plots of our H_2 sensing inverter system when H_2 is exposed on the Pd-MIM of drive TFT from 1 V to 5 V of drain voltage V_{DD}. The vertical gray line indicates V_{IN} = 3.5 V we used for measurement of dynamic voltage and inset is the corresponding equivalent circuit. (c) Dynamic H_2 sensing plot in time domain in our inverter under H_2 On/Off condition.

exposure (dashed line) and removal (solid line), voltage transfer curves (VTCs) of our inverter are nicely acquired under a low supply voltage (V_{DD}) from 1 V to 5 V, as shown in Fig. 4a. Maximum voltage gain was obtained to be ~8 at a V_{DD} of 5 V (Fig. 4b). As expected, the VTC was more shifted toward positive-side with H_2 infusion, reflecting the hydrogen-induced threshold voltage shift of transfer curves in Fig. 3c. We here selected V_{DD} = 5 V and V_{IN} = 3.5 V (vertical line) as a voltage condition to obtain a time-domain ON/OFF response behavior of our H-sensing inverter. Fig. 4c shows the dynamic hydrogen sensing behavior, which appears consistent with the results of Fig. 4b in respect of V_{OUT} level. Again, the turn on speed (H_2 ON) was quite fast (rising time ~1 s) although H_2 OFF speed was slow (falling time ~40 s) and comparable to the result (25 s) from I_D in the same H_2 OFF condition (Fig. 3d).

4. Conclusions

We fabricated a H_2-sensing device utilizing a Pd film as H_2-detecting electrode for metal/SiO_2/p^+-Si (MIM) capacitor, since we

found the H-induced chain reactions in $Pd/SiO_2/p^+$-Si capacitor: Pd volume expansion, Pd-SiO_2 contact are change, and the capacitance change. This capacitance change is connected to the gate of an electrically stable amorphous InGaZnO (a-IGZO) thin-film transistor (TFT). As a result, H-induced output as the drain current of a-IGZO TFT was statically and dynamically measured through the capacitance signal change from Pd-MIM sensor, demonstrating H_2 ON/OFF I_D ratio of ~100. This output current signal was then converted to voltage when a load resistor was connected to the a-IGZO TFT in series. We conclude that our capacitor-added H_2-sensor circuits are promising and novel due to their simplicity and practicality.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.snb.2014.12.005.

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