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Palladium nanogap-based H_2 sensors on a patterned elastomeric substrate using nanoimprint lithography

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ABSTRACT

Here we report a new nanogap formation manner at edges of a palladium (Pd) thin film on highly patterned arrays of an elastomeric PDMS substrate fabricated nanoimprint lithography (NIL) by absorption/desorption cycles of H_2 for use in the detection of hydrogen (H_2) gas. A 10-nm-thick Pd layer was deposited on the patterned PDMS substrate using an ultra-high vacuum DC magnetron sputtering system. The Pd nanogaps (~100 nm (W)) formed on the edges of the grating structure by expansion and contraction of the film through a few cycles of H_2 absorption and desorption. Such nanogaps are crucial to the performance of the sensors. The H_2 sensors were found to exhibit a fast response time (~1 s), low detection limit (0.1%), wide linear range (0.1–2%), and an ON–OFF switching operation in air. These properties are attributed to the synergistic combination of nanogap break junction control in Pd and the patterned elastomeric substrate.

1. Introduction

Hydrogen (H_2) gas is one of the promising resources for renewable clean energy that offers an alternative to the use of fossil fuels [1,2]. However, amounts in excess of 4% (40,000 ppm) in ambient atmosphere are highly explosive or flammable and therefore, accurate detection of H_2 gas is extremely important [3]. To date, many H_2 sensors have been developed using materials such as metal oxides [4,5], conducting polymers [6] and CNTs [7,8]. Palladium (Pd) is especially widely used as an H_2 sensing material since it absorbs up to 900 times its volume thereby forming Pd hydride at room temperatures. These Pd-based sensors exhibit enhanced sensitivities, high selectivities for H_2 gas, reversible hysteresis behavior for H_2 absorption/desorption at room temperature and fast response times. These attributes have led to the use of Pd-based H_2 sensors in various nanostructures including thin films [9], mesowires [10,11], nanowires [12,13], nanoparticles [14], nanotubes [15,16], clusters [17] and several others [18].

A recent work introduced a nanogap-based sensing method that utilizes crack formation in a Pd (and PdNi) thin film [19]. The cracks were generated by stretching the thin film onto an elastomeric substrate. This setup was referred to as highly mobile thin film on elastomer (MOTIFE) [19]. The MOTIFE sensors functioned perfectly in the ON–OFF mode. However, insufficient control of the nanogap width resulted in high detection limits (12,000 ppm H_2 in air and 4000 ppm H_2 in N_2) compared to other sensors and therefore, limited use in commercial applications. As a result, the nanogap-based H_2 sensors were alloyed with Pd and Ni in order to overcome this drawback [19,20]. Alloying with Ni hinders the volume expansion of Pd by reducing the H_2 solubility of the film. Sub-10 nm wide nanogaps in the Pd/PDMS substrate were obtained by controlling the initial H_2 concentration [21]. In that case, the width of the nanogaps decreased with decreasing concentration of the initial feed of H_2 gas. Liquid nitrogen freezing of the Pd/PDMS elastomer substrate and hybrid thin films have also been investigated in relation to the control of nanogaps [22]. In hybrid films, a Pd thin film is sandwiched between layers of poly(methyl methacrylate), PMMA, which upon exposure to H_2, prevent the stretching-recovery of Pd to an equilibrium dimension [23]. A novel cracked Pd film on an elastomeric substrate (CPE) was developed as an alternative method to generate nanogap formation by absorption/desorption of H_2 [24]. Mechanical treatments are not needed for the CPE. Strain

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localization resulting from adherence (by means of H_2 absorption) of the PDMS to the silica-like surface leads to random crack propagation in the Pd film [24]. Although mechanical treatments are not needed, the CPE fails to achieve both nanogap break junction control and lower detection limits.

In this study, we demonstrate nanogap formation at edges of the Pd thin film on the patterned elastomeric PDMS substrate fabricated nanoimprint lithography (NIL) via repeated absorption/desorption cycles of H_2. Such a NIL technique is a very precise and cost-effective, which provides high-throughput patterning of polymer nanostructures [25]. Grating structures on patterned elastomeric substrates facilitate the control of Pd break junctions and promote the formation of nanogaps at the edge of the gratings without mechanical stretching. Through the synergistic combination of the nanogap break junction control in Pd and the patterned elastomeric substrate, these sensors exhibit a significantly improved performance, including faster response time, lower detection limit, higher sensor response, and ON–OFF switching mode.

2. Experimental details

2.1. Preparation of a Pd thin film on a patterned elastomeric substrate

Fig. 1 schematically illustrates the fabrication process of Pd nanogap sensors using an elastomeric substrate patterned by nanoimprint lithography (NIL). The patterned elastomeric substrate was fabricated using PDMS (Dow Corning Corp., Sylgard 184) through a conventional NIL technique. Prior to the fabrication of the PDMS layer, the Si template with line patterns was manufactured by electron beam lithography. The grating structures were spaced 2 μm apart in the patterned Si template. The patterned array on

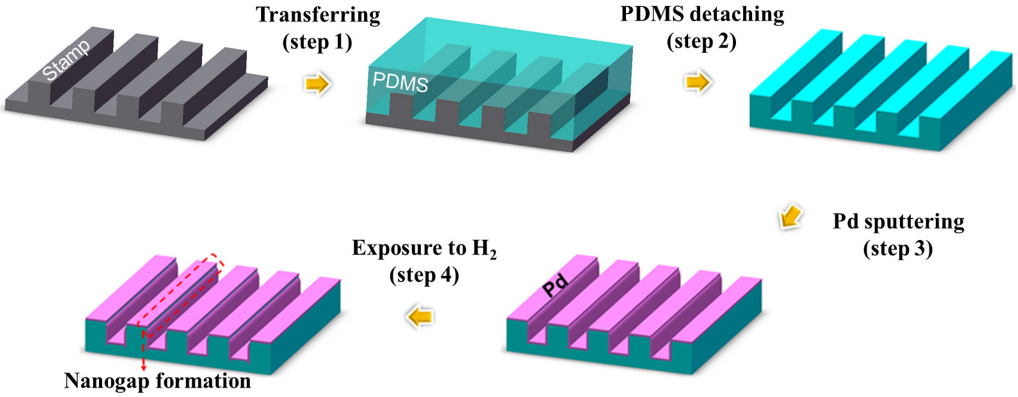

Fig. 1. The schematic fabrication process of Pd nanogap sensors using an elastomeric substrate patterned by nanoimprint lithography (NIL). The elastomeric substrate is imprinted (step 1) and peeled off from a Si stamp (step 2). A 10 nm thick Pd layer is then sputtered onto the detached PDMS substrate (step 3). H_2 absorption–desorption cycles are performed (step 4). Finally, nanogaps are formed at the edges of gratings on the Pd/PDMS sample.

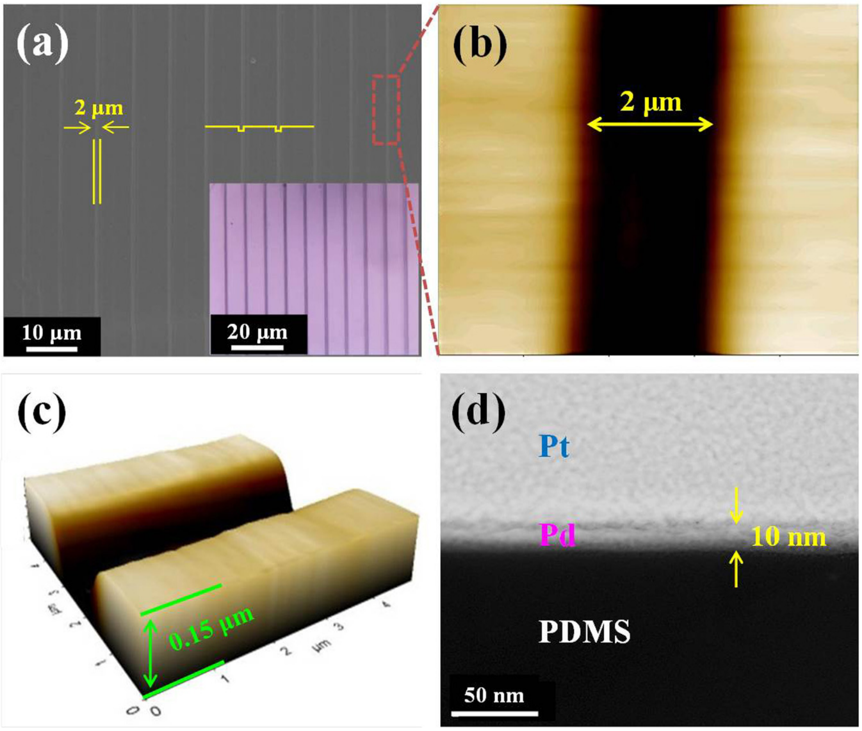

Fig. 2. (a) SEM image showing the top view of the 10 nm thick Pd film deposited on the patterned PDMS substrate and the inset of an optical image, (b) an AFM image and (c) corresponding depth profile of Pd/PDMS grates, (d) a cross-sectional STEM image of a Pd/PDMS grate.

the PDMS substrate consisted of 2-μm-spaced gratings, each with dimensions of 6 μm (*W*) × 0.15 μm (*H*). The fabrication process consists of several steps. PDMS precursor and curing agent (10:1 ratio, w/w) were mixed and poured into the prepared Si template (step 1). The PDMS layer (0.75 mm) was subsequently peeled off from the template after curing on a hot-plate at 75 °C for 4 h (step 2). The patterned PDMS was diced into a square size (10 mm × 10 mm) and a 10 nm-thick Pd film was deposited on the patterned PDMS substrate using a power of 20 W by an ultra-high vacuum (UHV) DC magnetron sputtering system (SNTEK Co., Ltd) under 34 sccm Ar flow (step 3). The chamber base pressure was retained at 4.0×10^{-7} Torr and the working pressure was 2.3 mTorr. The deposition rate of the Pd film at RT was ~3.7 Å/s at 20 W. The deposited film was repeatedly exposed to a few H_2 absorption–desorption cycles in air (step 4), giving rise to the formation of nanogaps at the edges of gratings on the Pd/PDMS sample. In more detail, the formation mechanism of Pd nanogaps is explained as the following 3 stages. On 4% H_2 exposure, the Pd film expands firstly through the H_2 absorption. It was attributed to phase transition from α to β by the absorbed hydrogen atoms, which corresponds to 3.5% lattice expansion. In the second stage, the strain in the Pd film is transferred down to grating of PDMS and the random crack is initiated on its edge. Finally, the Pd nanogaps are produced through the crack propagation during desorption of H_2 and clearly generated after several cycles of H_2 absorption/desorption.

2.2. Analysis of the Pd/PDMS sample

The morphology of the deposited Pd layer was investigated by field-emission scanning electron microscopy (FE-SEM; JSM-6701F, JEOL Ltd.), transmission electron microscopy (TEM; JEM-2100F, JEOL Ltd.), and tapping-mode atomic force microscopy (TM-AFM; XE-BIO, Park Systems). A TEM sample was prepared by a dual beam focused ion beam system (FIB, Nova 600, FEI Co.). A FIB-deposited Pt layer was used to protect the surface of the Pd/PDMS TEM sample from damage during sample preparation. The presence of the Pd layer was confirmed by energy dispersive X-ray spectra (EDX; JSM-6701F, JEOL Ltd.).

2.3. Measurements of H_2 sensing properties

The 10 nm Pd film on patterned PDMS was loaded onto a printed circuit board (PCB). Two electrical contacts were formed at ends of the Pd film and connected to PCB pad using silver paste (P-100, Cans Inc.). The H_2 sensing performance of the Pd/PDMS sample was determined from electrical measurements performed using a workstation composed of a Keithley 236 source-measurement unit (Keithley Instruments, Inc., Ohio, USA) and a compatible PC with Labview. All electrical measurements were carried out at room temperature (22 °C). The concentration of H_2 was controlled by a mass flow controller (MFC) using air as a carrier gas. A gas chamber (250 ml) was maintained at atmospheric pressure during the measurements.

3. Results and discussion

Fig. 2(a) shows a SEM image (top view) of the 10 nm thick Pd film deposited on the patterned PDMS substrate and the inset of an optical image. The PDMS substrate was patterned on an area with a spacing of 2 μm between the gratings. AFM analysis (Fig. 2(b) and (c)) confirms that each grating has dimensions of 6 μm × 0.15 μm (width × height). The cross-sectional STEM image of the Pd/PDMS grate (Fig. 2(d)) reveals that the 10 nm Pd thin film uniformly covered the patterned PDMS substrate. For ON–OFF switching operation of the H_2 sensors, the optimal thickness of Pd film was reported to be 9–11 nm [21]. If the Pd thickness exceeds 12 nm, then the sensors would always operate in the on-state. Pd films thinner than 8 nm lose functionality and would always be in the off state. These results were applied in the current study in H_2 sensing operations under air.

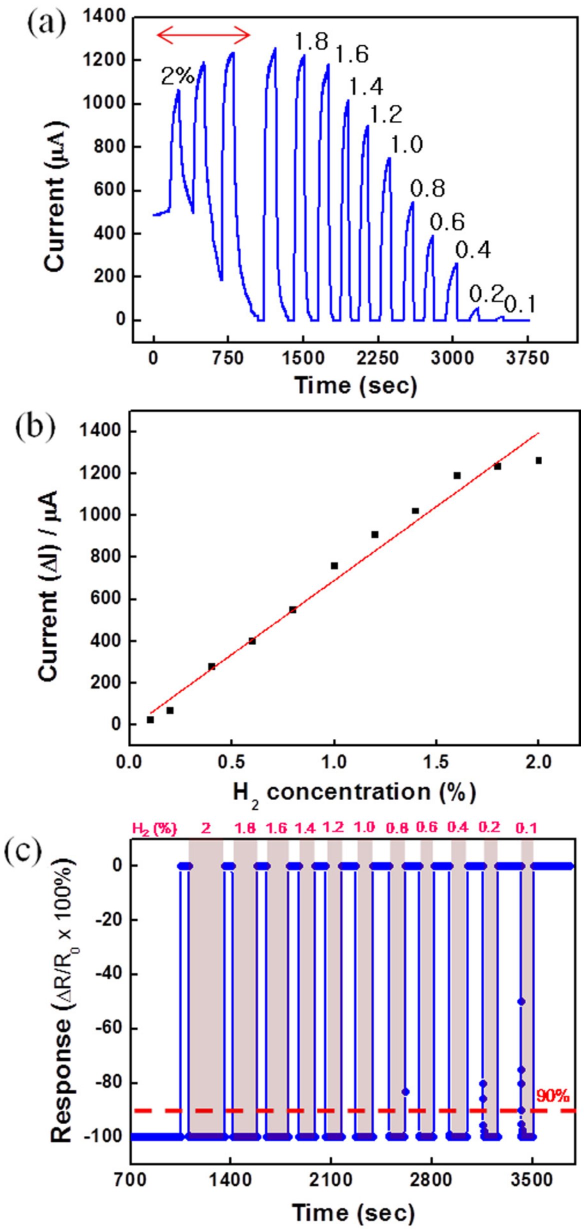

Fig. 3. (a) The real-time electrical responses of a Pd/PDMS sensor at room temperature in air, and (b) plot of the response curve of changes in current vs. H_2 concentration. (c) The sensor response vs. time curve for the Pd/PDMS sensor for H_2 concentrations of 2–0.1% (the time interval is 1 s).

To measure the electrical current of the Pd/PDMS samples for detection of H_2 gas, a real-time response was monitored at room temperature with various H_2 concentrations (Fig. 3). This sample was cyclically exposed to 2% H_2 in air in order to promote the formation and maintain the stability of Pd nanogaps. The nanogap formation mechanism will be discussed in more detail in Fig. 5. Fig. 3(a) plots the response of this Pd/PDMS sample as measured in 1 s intervals. This sample exhibits perfect ON–OFF sensing behavior. After three cycles of H_2, the baseline current level was set to zero and perfect ON–OFF sensing was measured for decreasing (from 2 to 0.1%) H_2 concentrations. The changes in current were plotted as a function of the H_2 concentration (Fig. 3(b)) using the data from

Table 1
Comparison of sensing performance to the Pd based H_2 gas sensors reported in the literature.

Device type	Operating mode	Low detection limit (%)	Detection range (%)	Response time (s)	Sensitivity (ppm^{-1})	Ref.
Pd gratings on PC	ON	0.005 (air base)	0.005–0.35	18 (0.35% H_2)	0.00046	[25]
Pd-NPs/SWNT on PET	ON	0.01 (air base)	0.01–1	3 (1% H_2)	0.065	[26]
Pd-NCs on SCG	ON	0.0025 (N_2 base)	0.0025–5	0.70 (2% H_2)	0.0108	[27]
Dendritic Pd on Si	ON–OFF	0.11 (N_2 base)	0.11–9.6	23 (9.6% H_2)	0.0000625	[28]
Cracked Pd on PDMS	ON–OFF	1.2 (air base)	1.2–2	<1 (2% H_2)	–	[19]
Pd on patterned PDMS	ON–OFF	0.1 (air base)	0.1–2	<1 (2% H_2)	0.8	This work

Fig. 3(a). Fig. 3(b) reveals a linear dependence of changes in current on H_2 concentrations in the range of 0.1–1.6% (correlation constant, $r = 0.97877$ with low detection limit of 0.1%). The sensor response (Fig. 3(c)) to various H_2 concentrations is another measure of the performance of the sensor and is generally expressed as Eq. (1),

$$\text{Response }(\%) = \frac{R_{H_2} - R_a}{R_a} \times 100 \qquad (1)$$

where R_{H_2} and R_a are the electrical resistances in H_2 gas and dry air, respectively. The response of the sensor was also determined. This value is defined as the time needed to reach 90% of the total change in electrical resistance for a given concentration [19,20]. Response times of less than 1 s were obtained for H_2 concentrations in the range of 0.4–2%. These values increased for concentrations lower than 0.4% but the recovery time never exceeded 1 s. Compared to other Pd based sensors reported in the literature, our sensor shows enhanced sensing performances in air, such as lower detection limit, wider detection rage, faster response, higher sensitivity and ON–OFF operating mode (Table 1). Although Ho's group developed a flexible Pd-based H_2 sensor with the similar structure to those considered in this work, it showed, however, lower sensitivity, slower response time and always ON operating mode. It might be attributed to no formation of Pd nanogaps. In our previous study, the cracked Pd-based H_2 sensors were fabricated by stretching a flexible substrate [19]. The Pd nanogaps on PDMS were generated, however, this sensor exhibited higher detection limit and narrow detection range under air in comparison to our sensor. It was originated from wide nanogaps that were obtained owing to the grooves (ca. 300 nm wide and 50 nm deep) formed during stretching.

The exact position of Pd nanogap formation on a Pd/PDMS sample was investigated using TEM (STEM) after FIB milling process (Fig. 4). STEM images compare the deposited Pd layer before and after the H_2 cycling treatment (Fig. 4(a) and (b)). H_2 cycling (Fig. 4(b)) results in the formation of a gap (~100 nm wide) in the initially, uniformly deposited Pd layer (Fig. 4(a)). The resulting lower detection limit can be attributed to the small width of the nanogap. STEM elemental mapping images (Fig. 4(c)) were used to confirm the exact disconnection of the Pd films from the edge of PDMS grating. Fig. 4(c) confirms that the nanogap formed in a particular region (especially at the edge of the grating structure) by

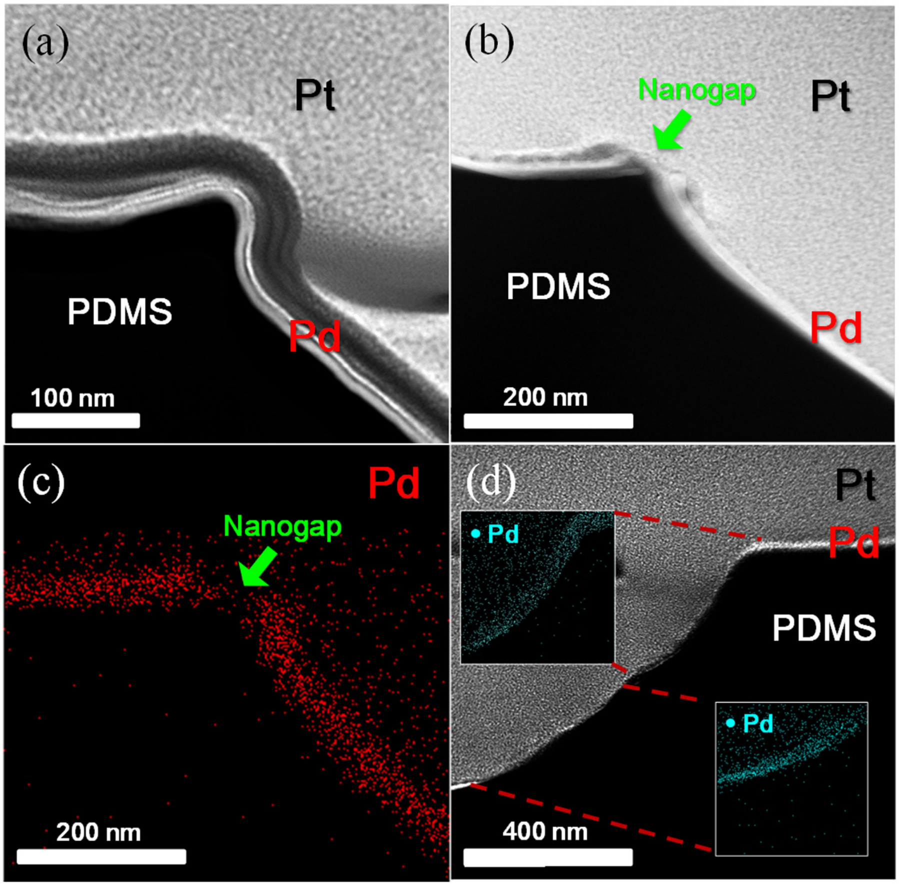

Fig. 4. Cross-sectional STEM images of the edge of the Pd/PDMS sensor (a) before and (b) after H_2 cycling treatment, (c) elemental mapping images of nanogap formation (disconnected Pd layer, red dots), and (d) no nanogap formation at the edges of the Pd layer on PDMS after repeatable H_2 treatment cycles. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

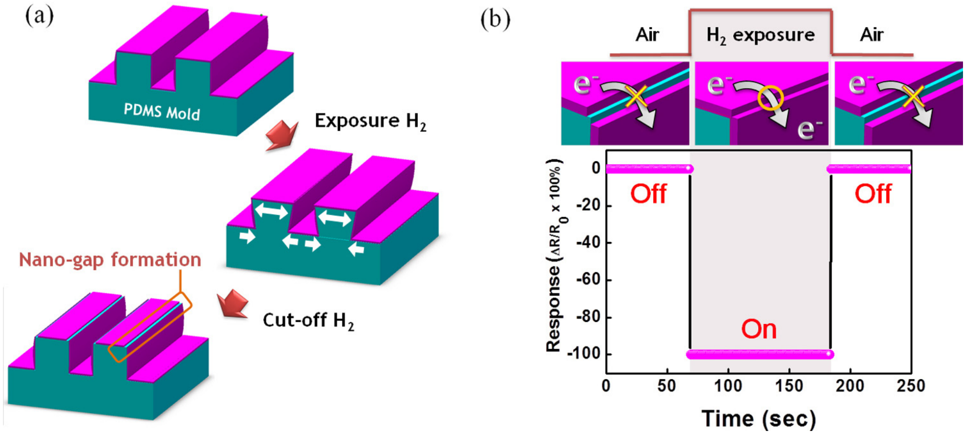

Fig. 5. (a) Schematic images of the sensing mechanism for Pd nanogap formation using volumetric expansion through H_2 absorption and desorption, and (b) the changes in current response with ON–OFF operation depending on electron transfer at the edge of the Pd/PDMS sensor.

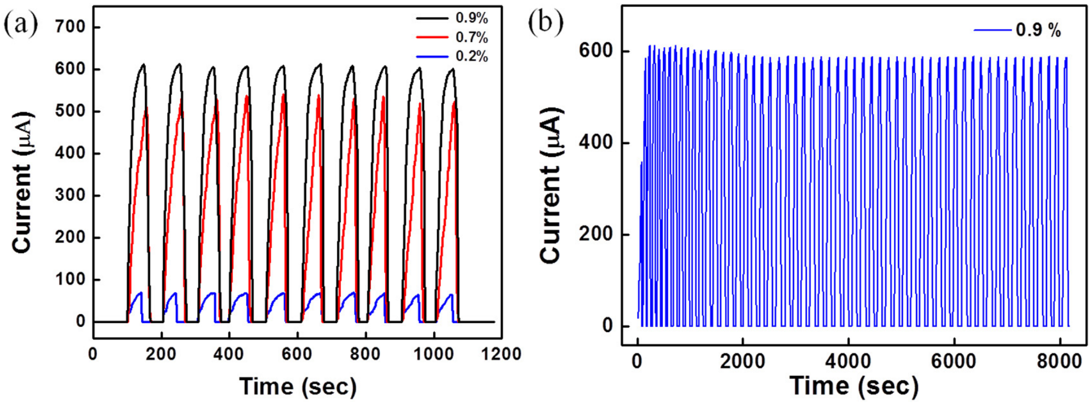

Fig. 6. Repeatability tests of the real-time electrical responses vs. time of the Pd/PDMS sensor (a) in the H_2 concentration ranges of 0.2–0.9% at 10 on/off cycles and (b) under 0.9% concentration H_2 at 55 on/off cycles.

expansion and contraction of the Pd film. In addition, STEM image with elemental mapping images of lateral surface of the Pd grating showed continuous connection of the Pd layer and no nanogap formation at the edge of the Pd layer on PDMS, as shown in Fig. 4(d). Based on the STEM observation, the cross-sectional view of the Pd/PDMS grating revealed that nanogaps randomly formed at the edges of the structure after repeatable H_2 treatment cycles. To summarize, the ON–OFF sensing property of the H_2 sensors results from Pd nanogaps which formed at the edges of the grating structure.

Pd-based H_2 sensors function by means of the carrier scattering effect. This effect originates from the formation of PdH_x which, in turn, generates differences in the resistance in the system. The current system, however, is based on the strong connection which results from volume expansion of the Pd layer when H_2 is absorbed. Fig. 5 presents the mechanism of the nanogap formation on the Pd/PDMS sensor with the pattern (a) and its ON–OFF operation principle for detection of H_2 (b). Pd nanogap formation results from the absorption and desorption of H_2 (Fig. 5(a)). When exposed to H_2, Pd converts to Pd hydride (PdH_x) through absorption of H_2 molecules and the thin film expands. PdH reconverts to Pd upon the desorption of H_2 and the film contracts. The consequent formation of nanogaps is facilitated by the flexibility of the PDMS [19]. These nanogaps form especially easily at the edges of the grating structures owing to strain localization. This strain localization occurs as a result of the difference in expansion between the thin Pd layer and the PDMS substrate. Strain in the Pd layer extends to the PDMS substrate during lattice expansion or contraction. Based on the Pd nanogap, these sensors operate as a perfect ON–OFF mode according to absorption and desorption of H_2 (Fig. 5(b)). This ON–OFF switching operation is facilitated by the flexibility of the PDMS substrate [19]. The entire operation depends on the presence of H_2. For example, when H_2 is absent from ambient air, the electron transfer pathway is cut owing to the nanogaps (OFF-switching mode) which form on the edge of the grating. On the contrary, lattice expansion of the film during H_2 exposure results in closure of the nanogaps and hence, electron transfer (ON-switching mode) at the edge of the Pd/PDMS sensor (Fig. 5(b)). This ON–OFF switching occurs repeatedly and the extent of nanogap closure is proportional to H_2 concentrations lower than 1.6%.

Fig. 6 shows the repeatability tests of the Pd/PDMS sensor with various concentration of H_2. At 0.2, 0.7, and 0.9% H_2 concentrations, the current response showed obviously constant with no significant change during 10 repetitions with a perfect ON–OFF sensing behavior, as shown in Fig. 6(a). The response and recovery times maintained almost constant at high H_2 concentrations (0.7 and 0.9%, respectively). A more detailed repeatability test was carried out on the best performing H_2 concentration (0.9%) during 55 ON–OFF cycles as shown in Fig. 6(b). This result showed a consistent repeatability in each cycle, indicating that the Pd/PDMS sensor was stable and fast for an extended period of ~8000 s.

4. Conclusion

Novel Pd-based H_2 nanogap sensors on a patterned elastomeric substrate were fabricated by nanoimprint lithography (NIL). The grating structures in these sensors facilitate the control of Pd break junctions. Narrow (width less than 100 nm) Pd nanogaps were generated on the edge of the grating by expansion and contraction of the film. Furthermore, the flexible substrate enables the formation of the nanogaps owing to the expansion difference between Pd and PDMS. These nanogap sensors exhibit a perfect ON–OFF switching operation and significantly improved performance, including fast response time, low detection limit, and high sensor response.

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Biographies

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