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# Fast-responding ethanol sensor with extremely low detection limit: Influence of Pt film thickness on gas sensing properties

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## ABSTRACT

To facilitate alcohol detection in exhaled breath, high-performance gas sensors capable of rapidly responding to low-concentration target gases in highly humid environments are required. In this study, we developed  $In_2O_3$  nanorods (NRs) decorated with Pt nanoparticles (NPs) to improve selective  $C_2H_5OH$  detection under dry and 80 % relative humidity (RH) conditions. Using glancing angle deposition (GLAD), we fabricated  $In_2O_3$  NRs with Pt film of varying film thicknesses (0.5, 1, and 2 nm), which transformed to NPs after the subsequent annealing process, to systematically identify the optimal Pt NPs size and distribution. Optimized  $In_2O_3$  NRs with an initial Pt thickness of 1 nm exhibited the highest ethanol response at 450 °C, with a theoretical detection limit (DL) calculated to be 0.42 parts per billion (ppb) in dry air and 6.1 ppb under 80 % RH. This enhanced performance is attributed to the expansion of the electron depletion layer (EDL) due to Schottky barrier formation at the Pt  $In_2O_3$  interface, alongside a spillover effect that enhances gas adsorption and reaction on the  $In_2O_3$  surface, where Pt NPs contribute both electronic and chemical sensitization effects. These findings indicate that  $In_2O_3$  NRs with Pt NPs are promising candidates for next-generation ignition interlock devices (IIDs), offering high selectivity, rapid response times, and stability in high-humidity environments.

# 1. Introduction

The National Highway Traffic Safety Administration (NHTSA) reports that approximately 10,000 people die each year in the U.S. due to drunk driving-related crashes, accounting for 29 % of all traffic fatalities. There has been a notable increase in these numbers since 2014, indicating a decline in the effectiveness of traditional prevention methods [1]. Law enforcement agencies have intensified efforts to combat drunk driving through stricter enforcement of driving under the influence (DUI) laws, including sobriety checkpoints and harsher

penalties. However, studies suggest that while these measures have led to temporary reductions in drunk driving-related fatalities, their longterm effectiveness remains limited [2]. The implementation of IIDs, which require drivers to pass a breathalyzer test before starting their vehicle, has shown promising results in significantly reducing repeat offenses and fatalities [3]. Despite this success, widespread implementation has been challenging due to high costs and logistical issues, such as installation expenses and the relatively large size of the devices [4]. Therefore, various types of gas sensors, such as electrochemical, optical, catalytic combustion-type, and chemoresistive-type have been

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being investigated for the integration into vehicles with high performance and low cost [5–7].

Metal-oxide-semiconductor (MOS)-based gas sensors offer costeffective, easy integration, and potential for miniaturization, making them promising candidates for integration into the IIDs systems [8]. Among various MOS (SnO<sub>2</sub>, ZnO, CuO, and NiO), indium oxide (In<sub>2</sub>O<sub>3</sub>) has attracted significant attention due to its wide bandgap (3.5-3.7 eV), exceptional thermal stability, and superior electrical conductivity, which operate by detecting changes in the electrical properties when exposed to target gases [9,10]. However, due to this simple operation mechanism, MOS-based sensors tend to respond to a wide range of volatile organic compounds (VOC) gases, resulting in low selectivity toward specific targets gas.

To address these limitations, researchers have developed techniques such as noble metal functionalization, doping, defect control, the formation of heterojunctions, and the use of novel nanomaterials to improve selectivity and sensitivity by tuning the potential barrier and employing catalytic activity [11–15]. Among these approaches, noble metals such as platinum (Pt), palladium (Pd), ruthenium (Ru), and silver (Ag) enhance the gas-sensing performance of MOS-based sensors through key mechanisms of electronic and chemical sensitization. A Schottky barrier forms at the interface between the metal and MOS, which amplifies changes in electrical conductivity by facilitating electron transfer (electronic sensitization). Additionally, the spillover effect of noble metals improves gas selectivity and response time (chemical sensitization) by facilitating the decomposition of specific gases on the metal surface. Notably, Pt is well known as a catalyst with superior chemical and mechanical stability compared to other metals and noble metals. Furthermore, the low binding energy of Pt catalysts makes them suitable candidates for realizing a highly selective C<sub>2</sub>H<sub>5</sub>OH sensor [16]. For example, Liu et al. synthesized SnO<sub>2</sub> nanoflowers decorated with Pt NPs using a hydrothermal technique, resulting in a 3.4-fold improvement in C2H5OH response at 320 °C compared to pure SnO2 nanoflowers [17]. Similarly, Zhao et al. showed that doping SnO<sub>2</sub> hollow nanospheres with 0.3 wt% Pt NPs significantly enhanced C2H5OH sensing, with the gas response for 1 ppm H<sub>2</sub>S increasing dramatically from 19 (pristine SnO<sub>2</sub>) to 250 at 120 °C, highlighting the critical role of Pt in enhancing sensing performance [18]. Moreover, the dimensions and spatial arrangement of noble metal catalysts have a significant impact on gas sensing performance, yet few studies have systematically evaluated the effects of Pt NPs size and distribution.

Herein, we fabricated 1-dimensional In<sub>2</sub>O<sub>3</sub> NRs with Pt NPs using a simple GLAD to improve gas response and selectivity. To determine the optimal initial Pt film thickness and operating temperature, we evaluated gas-sensing characteristics at various temperatures up to 450 °C. In<sub>2</sub>O<sub>3</sub> NRs with 1 nm thick Pt NPs exhibited the highest gas response at 450 °C and showed enhanced selectivity towards C<sub>2</sub>H<sub>5</sub>OH compared to In<sub>2</sub>O<sub>3</sub> NRs. Additionally, the DL in both dry air and 80 % RH were calculated to be 0.42 ppb and 6.1 ppb, respectively, demonstrating the potential strategy for integration into IIDs applications.

# 2. Experimental section

## 2.1. Sample fabrication

Au interdigitated electrodes (IDEs), serving as substrates for gas sensors, were fabricated using a photolithography process. A Si wafer with a 3000 Å dry oxide layer was cleaned sequentially in a sonicator using CH<sub>3</sub>COCH<sub>3</sub>, isopropanol, and deionized (D.I.) water, 5 min per step. A photoresist layer (LOR 5A, MicroChem Corporation) was then spin-coated onto the Si wafer at 3000 rpm for 30 s and baked at 180 °C for 5 min. An additional photoresist layer (AZ GXR 601, AZ Electronic Materials) was subsequently spin-coated under the same conditions. After a 1-minute bake at 150 °C, the IDEs were patterned using a mask aligner system (MDA-400S, MIDAS System). The Si wafer was then developed (AZ 300MIF, MicroChemicals), rinsed with D.I. water, and

placed in an e-beam evaporator (EBX-1000, ULVAC) for deposition of an Au/Pt/Cr layer (180 nm/70 nm/30 nm). The Au-IDE-patterned substrate was cleaned again in a sonicator with CH<sub>3</sub>COCH<sub>3</sub>, C<sub>2</sub>H<sub>5</sub>OH, and D. I. water, for 5 min per step. Afterward, the samples were transferred to an electron beam evaporator (Korea Vacuum Tech) for deposition of a 600 nm-thick In<sub>2</sub>O<sub>3</sub> NRs film (99.99 %, Taewon Scientific Co.). The deposition was carried out at a glancing angle (80°) with a rotation speed of 6 rpm, a base pressure of  $3 \times 10^{-6}$  Torr, and a growth rate of 1 Å/s. To crystallize the  $In_2O_3$  NRs, the sample was annealed in a box furnace with a temperature ramp of 5 °C/min up to 500 °C, holding at this temperature for 1 h in ambient air. For Pt NP decoration on the top surface of the  $In_2O_3$  NRs, a Pt film (0.5, 1, or 2 nm thick; 99.99 %, Taewon Scientific Co.) was deposited at the base position (0°) with a deposition rate of 0.1 Å/s. The Pt film-deposited In<sub>2</sub>O<sub>3</sub> NRs were then annealed again (5 °C/min, 500 °C, 1 h) to transform the Pt film into Pt NPs, resulting in In<sub>2</sub>O<sub>3</sub> NRs with Pt NPs.

# 2.2. Characterization

The top-view and cross-sectional morphologies of the  $In_2O_3$  NRs with Pt NPs were analyzed using field-emission scanning electron microscopy (FE-SEM; JSM-7610F-Plus, JEOL) at an acceleration voltage of 15 kV and a working distance of 8 mm and high-resolution transmission electron microscopy (HR-TEM; JEM-ARM 200F NEOARM, JEM-F200, JEOL). The surface FE-SEM images were analyzed using MATLAB R2023a (MathWorks, Natick, MA, USA), and custom scripts were written to calculate the porosity. The crystal structures of the  $In_2O_3$  NRs and Pt NPs were obtained using X-ray diffraction (XRD; Empyrean, PAN-alytical) with Cu-K\alpha radiation ( $\lambda = 1.5418$  Å), a tube voltage of 40 kV, and a current of 30 mA.

## 2.3. Gas-Sensing measurement

The gas sensing measurement setup consisted of a mini probe chamber equipped with an 8-channel probe system (Fig. S1, MPS4000, PHOCOS), a gas controller (PHOCOS) for accurate adjustment of gas concentration between 1 and 50 ppm, and a temperature controller (PHOCOS). The sensing chamber had dimensions of 140 mm in width, 80 mm in length, and 40 mm in height. Gases were sequentially injected in alternating cycles of dry air and target gases (C2H5OH, HCHO, C3H8, C<sub>5</sub>H<sub>8</sub>, C<sub>7</sub>H<sub>8</sub>, H<sub>2</sub>, NH<sub>3</sub>, CH<sub>4</sub>, and H<sub>2</sub>S; balanced with dry air, RIGAS) through an automated gas control system. A silicon carbide heater maintained the operating temperature at 150 °C, 200 °C, 250 °C, 300 °C, 350 °C, 400 °C, and 450 °C to assess the gas-sensing properties of the sensors across various temperatures. The gas response was evaluated using a source meter (Keithley 237, KEITHLEY) under a constant DC bias voltage of 1 V, while the sensor resistance was measured with high accuracy using the 7001 switch system (KEITHLEY). Resistance changes were monitored at intervals of 2 s to evaluate the response to the target gases.

#### 3. Results and discussion

## 3.1. Characterization of In<sub>2</sub>O<sub>3</sub> NRs with Pt NPs

The transducer function in gas sensors refers to the process by which chemical interactions between gas molecules and the sensing material are converted into measurable electrical signals. Previous studies have reported that nanostructures enhance this transducer function by increasing the surface area and providing more active sites for gas interactions, thereby improving sensor performance [19]. Additionally, nanostructures often form multiple neck regions where grains or particles connect which are highly sensitive to changes in electrical signals and further amplify the sensor's response to gas adsorption. To maximize these effects, we fabricated one-dimensional NRs structures using electron beam evaporation through the GLAD technique at an 80° angle



Fig. 1. A schematic representation of the fabrication procedure for a gas sensor based on In<sub>2</sub> O<sub>3</sub> NRs with Pt NPs.

(Fig. 1). The GLAD technique operates by positioning the substrate at a high angle relative to the incoming vapor flux, resulting in a self-shadowing effect. This effect occurs when the elevated regions of the growing structures block vapor from reaching certain areas, promoting anisotropy and guiding the growth of columnar structures like NRs [20,21]. To decorate the  $In_2O_3$  NRs with Pt NPs, the substrate was positioned at a base angle of 0°, and Pt film were deposited at thicknesses of 0.5, 1, and 2 nm. A subsequent annealing process was applied to promote the crystallization of the  $In_2O_3$  and induce the aggregation of the Pt film into Pt NPs simultaneously, finalizing the formation of  $In_2O_3$  NRs with Pt NPs.

Fig. 2 presents the surface and cross-sectional FESEM images, the transmission electron microscopy (TEM) image, along with X-ray diffraction (XRD) analysis results of In2O3 nanorods (INRs) and In2O3 NRs with different Pt NPs (PINRs). The Pt film was first deposited at different initial thicknesses of 0.5, 1, and 2 nm, referred to as 0.5-PINRs, 1-PINRs, and 2-PINRs, respectively. The deposition mechanism through GLAD provides highly porous nanostructures, as confirmed by the top surface FE-SEM images (Fig. 2a-d). Each porosity was calculated by binary digitization of the SEM images and found to be 61.8 %, 61.4 %, 61.2 %, and 61.0 % for INRs, 0.5-PINRs, 1-PINRs, and 2-PINRs, respectively. This indicates that the overall porosity decreased as the initial Pt film thickness increased. The deposited INRs were grown with an approximate height of 600 nm, perpendicular to the substrate (Fig. 2e-h). The INRs exhibited a sharp, elongated shape, leading to the formation of numerous necks between the structures, with no noticeable changes in total thickness observed with varying initial Pt film thicknesses.

To analyze the detailed structure of the 1-PINRs, a cross-sectional scanning transmission electron microscopy (STEM) image was obtained. The STEM image shows both the grain sizes of  $In_2O_3$  and Pt NPs, indicating that 1-PINRs are porous structure (Fig. 2i). The HRTEM images are displayed in Fig. 2j, with magnified views provided in Fig. 2k–l ( $In_2O_3$  and Pt, respectively). The  $In_2O_3$  lattice fringe is associated with the (400) plane, showing a fringe spacing of 0.25 nm, while the Pt lattice fringe is related to the (111) plane with a spacing of 0.23 nm, confirming the successful formation of Pt NPs [22,23].

Fig. 2m demonstrates conventional crystallinity of the  $In_2O_3$  by showing the diffraction peaks at 21.4°, 30.6°, 35.4°, 37.8°, 41.9°, 45.7°, 51.1°, and 56.0° (PDF No. 00–044-1087). Additionally, the peak of deposited Pt observed at the 44-45° region corresponds to the (200) plane (PDF 00-033-0875). Collectively, it is clear that the INRs were

effectively crystallized during the annealing process, with the Pt film aggregating into Pt NPs and no additional phase formation observed throughout the process.

# 3.2. Gas sensing properties of INRs and PINRs

The MOS-based gas sensors interact with target gases through the adsorbed oxygen species ( $O_2^-$ ,  $O^-$ , or  $O^{2-}$ ) on the surface of MOS, which generates an electrical depletion layer (EDL) at n-type MOS the surface [24]. This reaction is significantly influenced by the operating temperature, as the type of adsorbed oxygen species varies with temperature. At temperatures below 100°C,  $O_2^-$  is predominantly formed. Between 100°C and 300°C, the more reactive  $O^{2-}$  species are primarily present. When reducing gases, such as CH<sub>3</sub>COCH<sub>3</sub>, HCHO, and C<sub>5</sub>H<sub>8</sub>, react with the adsorbed oxygen ions on the n-type MOS surface, electrons are injected into the material, thereby reducing the resistance of sensors. In these reactions, the gas response (*S*) of gas sensors was defined using the ratio of saturated resistance in air ( $R_a$ ) to resistance in target gas ( $R_g$ ), as represented by equation:

$$S = (R_a - R_g)/R_g \tag{1}$$

The decorated Pt NPs on the top surface of INRs form a Schottky barrier between the Pt-In<sub>2</sub>O<sub>3</sub> interface, leading to the expansion of the EDL. The size and distribution of Pt NPs also significantly affect the gas sensing properties of all PINRs due to arrangement of EDL. To find optimal operating temperature and Pt film thickness, INRs and all PINRs were exposed to 50 ppm C<sub>2</sub>H<sub>5</sub>OH at different temperatures (150, 200, 250, 300, 350, and 450 °C), as shown in Fig. 3a-g. Upon exposure to the C<sub>2</sub>H<sub>5</sub>OH as a reducing gas, all samples show a decrease in resistance, followed by a recovery to  $R_a$ , indicating n-type semiconductor behavior. As the temperature increased, the  $R_a$  of INRs and all PINRs gradually increased, reaching maximum values at 400°C for INRs and 450°C for all PINRs. Compared to INRs, all PINRs exhibited higher  $R_a$  values at all tested temperatures due to the expanded EDL induced by the Schottky junctions, with 1-PINRs showing the maximum  $R_a$ . To clearly confirm the optimal conditions and sample, Fig. 3h shows the responses as a function of Pt film thickness. Similar to  $R_a$ , maximum values of response indicate at 400°C and 450°C for INRs and all PINRs, respectively. At 450°C, the responses were 3.04, 21.39, 36.98, and 21.37 for INRs, 0.5-PINRs, 1-PINRs, and 2-PINRs, respectively, with 1-PINRs exhibiting the



**Fig. 2.** Top-view SEM micrographs of (a) INRs and (b-d) 0.5-PINRs, 1-PINRs, 2-PINRs, respectively. The inset shows the converted top-view SEM images by MATLAB. (e-h) Cross-sectional SEM images of INRs and PINRs. (i) STEM image showing the top region of 1-PINRs; (j) HRTEM image of 1-PINRs; (k-l) magnified views of the selected areas, with corresponding to  $In_2 O_3$  and Pt respectively. (m) XRD patterns for INRs and PINRs.

highest response. Interestingly, the  $R_a$  and response of INRs decreased at 450 °C, which will be discussed in detail in future sections.

Since IIDs required selectively detect  $C_2H_5OH$  in human breath, which includes various gases, such as NH<sub>3</sub>, CH<sub>4</sub>, and H<sub>2</sub>S, we investigate selectivity of INRs and 1-PINRs by exposing them to 50 ppm  $C_2H_5OH$ , HCHO,  $C_3H_8$ ,  $C_5H_8$ ,  $C_7H_8$ ,  $H_2$ , NH<sub>3</sub>, CH<sub>4</sub>, and H<sub>2</sub>S at 450 °C (Fig. S2). The 1-PINRs show response values of 37.0, 21.7, 2.1, 14.1, 10.9, 3.2, 4.7, 2.4, and 20.1 for  $C_2H_5OH$ , HCHO,  $C_3H_8$ ,  $C_5H_8$ ,  $C_7H_8$ , H<sub>2</sub>, NH<sub>3</sub>, CH<sub>4</sub>, and H<sub>2</sub>S, respectively, whereas the INRs exhibited responses of 3.0, 4.0, 1.1, 3.8, 2.1, 2.3, 1.8, 1.0, and 6.6 for the same gases, indicating that 1-PINRs have enhanced response across all target gases. To clearly evaluate the gas selectivity and the effects of Pt NPs, the radar plots of response for INRs ( $S_{INRs}$ ) and 1-PINRs ( $S_{PINRs}$ ) to all exposed gases were obtained, as shown in Fig. 4a–b. The INRs exhibited the highest response to H<sub>2</sub>S, followed by HCHO, C<sub>5</sub>H<sub>8</sub>, C<sub>2</sub>H<sub>5</sub>OH, H<sub>2</sub>, C<sub>7</sub>H<sub>8</sub>, NH<sub>3</sub>, C<sub>3</sub>H<sub>8</sub>, and CH<sub>4</sub>. Meanwhile, the highest response of 1-PINRs change to C<sub>2</sub>H<sub>5</sub>OH from H<sub>2</sub>S at INRs, followed by HCHO, H<sub>2</sub>S, C<sub>5</sub>H<sub>8</sub>, C<sub>7</sub>H<sub>8</sub>, NH<sub>3</sub>, H<sub>2</sub>, C<sub>3</sub>H<sub>8</sub>, and CH<sub>4</sub>. H<sub>2</sub>S showed the third-highest response of 6.6 for 1-PINRs, which exhibited the highest response of 20.1 for INRs. The C<sub>2</sub>H<sub>5</sub>OH response of 37.0 for 1-PINRs was approximately 37 times higher than the lowest response of 1 observed for CH<sub>4</sub>. To investigate the effect of Pt NPs, we calculated *S*<sub>INRs</sub>/*S*<sub>PINRs</sub> value which indicate approximately 12 as highest value to C<sub>2</sub>H<sub>5</sub>OH followed by HCHO, C<sub>7</sub>H<sub>8</sub>, C<sub>5</sub>H<sub>8</sub>, NH<sub>3</sub>, H<sub>2</sub>S, NH<sub>3</sub>, CH<sub>4</sub>, C<sub>3</sub>H<sub>8</sub> (Fig. 4c). Compared to *S*<sub>INRs</sub>/*S*<sub>PINRs</sub> values for other gases, which range between 1 and 5, C<sub>2</sub>H<sub>5</sub>OH exhibits a remarkable improvement. As shown in Fig. 4d, we calculated response time ( $t_{res}$ , 90 % of the response)) for INRs and 1-PINRs, with 1-PINRs achieving lower  $t_{90\%}$  to all exposed gases compared to INRs. Especially,  $t_{90\%}$  decreased by



**Fig. 3.** Resistance curves of the INRs, 0.5-PINRs, 1-PINRs, and 2-PINRs to 50 ppm  $C_2 H_5$  OH at different temperatures; (a) 150 °C, (b) 200 °C, (c) 250 °C, (d) 300 °C, (e) 350 °C, (f) 400 °C, and (g) 450 °C. (h) Gas responses to  $C_2 H_5$  OH of INRs, 0.5-PINRs, 1-PINRs, and 2-PINRs-based gas sensors at different temperatures.



**Fig. 4.** Radar plot of responses of (a) INRs and (b) 1-PINRs to 50 ppm various gases at 450 °C (E:  $C_2 H_5 OH$ , F: HCHO, P:  $C_3 H_8$ , I:  $C_5 H_8$ , T:  $C_7 H_8$ , H:  $H_2$ , A: NH<sub>3</sub>, M: CH<sub>4</sub>, and HS:  $H_2$  S). (c) The response ratios (*S*<sub>*PINRs*</sub> / *S*<sub>*INRs*</sub>) between INRs and 1-PINRs. *S*<sub>*INRs*</sub> and *S*<sub>*PINRs*</sub> represent the gas responses of INRs and 1-PINRs, respectively. (d)  $t_{res}$  of INRs and 1-PINRs. (e) 3D PCA graphs based on the results of base resistance, target gas resistance, response, response time, and recovery time of two repeated measurements to nine gases for 1-PINRs.

approximately 62 s (from 70 s to 7.9 s) for  $C_2H_5OH$ , representing the most significant reduction among all exposed gases. To further enhance gas selectivity, we performed principal component analysis (PCA) based on  $R_a$ ,  $R_g$ , S,  $t_{res}$ , and recovery time ( $t_{rec}$ , 90 % of the original state) of each response curve (Fig. 4e). Data points of each gas are distinctly separated and form clusters for the same gas, allowing the distinction of each target gas. Enhanced  $C_2H_5OH$  sensing performance like high response and selectivity and fast  $t_{res}$  attributed to electronic and chemical sensitization of Pt NPs, which will be discussed in detail within the following section.

For practical applications, both reliability and accuracy of the gas sensor are one of the most important factors. To confirm repeatability with long-term stability, the 1-PINRs were alternately exposed to air and 50 ppm of C<sub>2</sub>H<sub>5</sub>OH over 20 cycles (Fig. 5a). During the 20 cycles, the 1-PINRs fully recovered to their initial resistance after the multiple exposures to target gases. To quantify the differences in response for 20 curves, the standard deviation ( $\sigma$ ) was 41.63. To consider relative magnitude of the  $\sigma$  in comparison to the mean ( $\mu$ ), coefficient of variation (C.V.) was calculated 2.73 % based on the following equations:

$$C.V. = -\frac{\sigma}{\mu}$$
(2)

Additionally, the response of the 1-PINRs to 50 ppm of C<sub>2</sub>H<sub>5</sub>OH was measured over a period of 10 days at 24-hour intervals to evaluate reliability, with the C.V. was calculated to be 5.26 % (Fig. 5b-l). Compared to commercial gas sensors, which typically exhibit C.V. values between 16.7 % and 41 %, our results demonstrate that the 1-PINRs possess significant potential for commercialization [25,26]. In addition, we investigated the humidity dependence of the 1-PINRs, finding that both  $R_a$  and gas response progressively decreased with increasing humidity. Notably, the response at 80 % RH was reduced to approximately 16 % of that under dry conditions (Fig. S3). Despite this degradation, the 1-PINRs maintained a high gas response of 6.15, highlighting their robustness under varying humidity conditions.

To calculate the DL of C<sub>2</sub>H<sub>5</sub>OH, the 1-PINRs were tested under both dry and 80 % RH conditions, with exposure to multiple pulses of C<sub>2</sub>H<sub>5</sub>OH at concentrations ranging from 3 ppm to 7 ppm (Fig. 6). In both dry and 80 % RH conditions, the response linearly increased with increasing gas concentration, indicating exceptional linearity and stability. Under dry conditions, the sensor responses were 9.9, 11.6, 13.8, 15.6, and 17.1 for 3, 4, 5, 6, and 7 ppm C<sub>2</sub>H<sub>5</sub>OH, respectively, while in 80 % RH, response values were 2.0, 2.3, 2.7, 3.0, and 3.5. After measuring the slope in a linear fit plot of response and concentration, the DL was then determined using a signal-to-noise ratio of 3, based on the following equations [27]:

$$R_{x^2} = \sum (y_i - y)^2$$
(3)

$$RMS_{noise} = \sqrt{\frac{R_{x^2}}{N}}$$
(4)

$$DL = 3 \frac{RMS_{Noise}}{Slope}$$
(5)

here,  $y_i$  represents discrete values from the response curve before C<sub>2</sub>H<sub>5</sub>OH exposure, and *y* is their average. The  $R_{x^2}$  value was obtained through a fifth-order polynomial fitting, using ten data points extracted from  $y_i$ . The root mean square ( $RMS_{noise}$ ) was calculated from these values, where *N* indicates the number of data points. These calculations yielded a slope of 1.84 ppm<sup>-1</sup> and a DL of 0.42 ppb under dry conditions (Fig. 6a), while under 80 % RH, the slope was 0.37 ppm<sup>-1</sup>, with a DL of 6.1 ppb (Fig. 6b).

Considering that IIDs standards typically require DL over 200 ppb, particularly for monitoring the  $C_2H_5OH$  concentrations in exhaled breath, despite the reduced sensor response in humid conditions, the sufficient DL of 6.1 ppb under 80 % RH can offer powerful solution for

being integrated into the IIDs as driver safety applications.

# 3.3. Surface chemical states of INRs and PINRs

Fig. 7 presents the XPS analysis INRs, 0.5-PINRs, 1-PINRs, and 2-PINRs to investigate the influence of chemical state, including oxygen species and the ionic states of Pt and In, on gas sensor performance. As shown in Fig. 7a, the In3d spectra displayed peaks at 444.4 eV and 452.0 eV, corresponding to In3d<sub>5/2</sub> and In3d<sub>3/2</sub>, respectively [28]. After Pt deposition, the In3d peaks exhibited a shift toward lower binding energies, which can be attributed to electron transfer from In<sub>2</sub>O<sub>3</sub> to Pt. The extent of the binding energy shift was found to increase with Pt film thickness, reaching a maximum value of 0.6 eV for the 2-PINRs. Fig. 7b illustrates the Pt4f spectra of PINRs with varying Pt decoration thicknesses. Five distinct peaks were observed at 71.1, 72.4, 74.4, 75.5, and 77.5 eV, corresponding to  $Pt^0$ ,  $Pt^{2+}$ , the overlap of  $Pt^0$  and  $Pt^{4+}$ ,  $Pt^{2+}$ , and Pt<sup>4+</sup>, respectively [29–32]. Notably, we observed an increase in the intensity of oxidized Pt states (Pt<sup>2+</sup> and Pt<sup>4+</sup>) for the 2-PINRs, suggesting that higher Pt thickness promotes oxidation, which could influence its sensing performance. Furthermore, the valence states of oxygen species in INRs and PINRs with varying Pt decoration thicknesses were analyzed using the O1s spectra, as shown in Fig. 7c. Three distinct peaks were observed, corresponding to lattice oxygen in the oxide (O<sub>a</sub>, 529.2 eV), adsorbed oxygen ions related to oxygen defects (Ob, 530.2 eV), and surface hydroxyl groups (O<sub>c</sub>, 531.4 eV) [33–35]. Among the various oxygen species, adsorbed oxygen ions play a particularly critical role in semiconductor-based gas sensors [36]. These adsorbed oxygen ions were actively involved in gas-sensing reactions, interacting with target gas molecules and significantly affecting sensor performance. Therefore, we calculated the ratio of  $O_b$  to the total oxygen species  $(O_a + O_b + O_c)$  and presented the results in Fig. 7d. Interestingly, the 1-PINR sample exhibited the highest ratio of adsorbed oxygen, which we interpret as an indication of the largest number of defect sites available for adsorption and desorption processes. These findings are consistent, where 1-PINRs demonstrated higher gas response compared to 0.5-PINRs and 2-PINRs.

# 3.4. Structural and catalytic effect of PINRs

Our results clearly demonstrate that the PINRs significantly improve gas sensing performance. We obtained two key effects of the PINRs, including structural effects and catalytic effects. Firstly, the large specific surface area of INRs enhanced both the transducer function and the utility factor, translating signals from chemical adsorption on the oxide surface into electrical signals and indicating the capacity of MOS grains to interact with target gases, respectively. In addition, the sharp and elongated shape of the INRs forms numerous necks (15-40 nm) that are integrated into current pathways, influencing more efficient resistance changes when exposed to target gases (Fig. S4). Secondly, the catalytic effects of Pt NPs can derive two mechanisms, namely electronic and chemical sensitization which improved the receptor function. Electronic sensitization is caused by the interaction between the Pt and In<sub>2</sub>O<sub>3</sub> surface. Due to the difference in their work functions, electron transfer occurs from In2O3 to Pt until the Fermi levels equilibrate, which extends the EDL on the In<sub>2</sub>O<sub>3</sub> surface and enhances gas response (Fig. 8a) [37]. At the narrow necks, this effect is further enhanced, resulting in a significantly higher response (Fig. 8b-c). The distribution and size of Pt NPs are crucial factors that significantly affect the gas sensing performance of PINRs. Our previous study showed that the initial film thickness resulted in different sizes and distributions of NPs, with optimal performance achieved at a specific film thickness [38]. For the thick Pt film (2-PINRs), the size and distribution of Pt NPs are relatively large and sparse, which cannot be utilized for the catalytic effects because the surface area of sensing material to react with target gases decreases and the widths of the interfacial EDL were not overlapped (Fig. 8d). On the other hand, when the thickness of Pt film (0.5-PINRs) is extremely thin, the Pt NPs are too dense and thus the surface area of In2O3 for the



Fig. 5. (a) Resistance curves of 1-PINRs to 50 ppm  $C_2$   $H_5$  OH at 450 °C measured over 20 cycles. (b-k) Response curves of 1-PINRs to 50 ppm  $C_2$   $H_5$  OH at 450 °C analyzed over 10 days. (l) The response of 1-PINRs to 50 ppm  $C_2$   $H_5$  OH at 450 °C over the measurement period.



Fig. 6. Sensing curves of 1-PINRs toward low concentration  $C_2 H_5 OH (3, 4, 5, 6, and 7 ppm)$  in different conditions with (a) dry and (b) 80 % RH at 450 °C. The inset shows responses of 1-PINRs as a function of  $C_2 H_5 OH$  concentration under dry and 80 % RH, including the calculation of the DL.



Fig. 7. XPS spectra of INRs, 0.5-PINRs, 1-PINRs, and 2-PINRs; (a) In3d spectra for both INRs and PINRs, (b) Pt4f spectra of PINRs and (c) O1s spectra for INRs and PINRs. (d)  $O_b / (O_a + O_b + O_c)$  ratio of INRs, 0.5-PINRs, 1-PINRs, and 2-PINRs.

adsorption of gas molecules is significantly reduced, resulting in a low response relatively. Therefore, our results suggest that 1-PINRs possess an optimal size and distribution of Pt NPs. The chemical sensitization effect of Pt NPs plays a critical role in dissociation of gas molecules, such as C<sub>2</sub>H<sub>5</sub>OH. For instance, 1-PINRs exhibited a response time of 7.9 s compared to 70 s for INRs to 50 ppm C<sub>2</sub>H<sub>5</sub>OH at 450 °C, representing the most significant decrease among the target gases. The decrease in response time experimentally indicates the chemical sensitization effect of Pt NPs toward C<sub>2</sub>H<sub>5</sub>OH. Moreover, to clearly observe this effect, we introduced the Arrhenius equation:

$$S = \exp\left(-\frac{E_a}{kT}\right) \tag{6}$$

In this equation,  $E_a$ , k, and T represent the activation energy, Boltzmann constant, and operating temperature, respectively [39]. The activation energy was calculated as 32.18, 24.75, 22.27, and 25.85 kJ/ mol for INRs, 0.5-PINRs, 1-PINRs, and 2-PINRs, respectively. As Pt NPs were decorated, the activation energy decreased, with 1-PINRs showing the lowest activation energy of 22.3 (Fig. 8e-f). R. Costa-Amaral et al. reported that DFT studies on Pt (111) surfaces revealed significant



**Fig. 8.** (a) The energy band structure of Pt/In<sub>2</sub> O<sub>3</sub> junctions, illustrating the formation of the EDL in the narrow necks between the NRs; (b) INRs and (c) PINRs. (d) Effect of Pt NPs size on the EDL formation. (e) Plots of ln(S) vs. 1000/T (K) measured for the INRs, 0.5-PINRs, 1-PINRs, and 2-PINRs. (f) Calculated activation energy of INRs, 0.5-PINRs, 1-PINRs, and 2-PINRs.

changes in the electronic density distribution upon  $C_2H_5OH$  adsorption [40]. These findings support the observed chemical sensitization of Pt NPs in this study. Furthermore, the decomposition of  $C_2H_5OH$  during the gas sensing process can be described by the following reactions [41]:

$$C_2H_5OH(gas) + 6O^-(ads) \rightarrow 2CO_2 + 3H_2O + 6e^-$$
 (7)

In summary, the large specific surface area of INRs enhanced both the transducer function and the utility factor, whereas the catalytic effects (electronic and chemical sensitization) of Pt NPs improved the receptor function [42]. The highest catalytic effect was observed at the optimal catalyst distribution (1-PINRs), with each effect being maximized for  $C_2H_5OH$ , thereby enabling highly selective gas detection.

# 4. Conclusions

We successfully developed high-performance  $C_2H_5OH$  gas sensors based on PINRs, utilizing the GLAD technique to optimize Pt NPs size and distribution. In<sub>2</sub>O<sub>3</sub> NRs with an initial Pt thickness of 1 nm exhibited the greatest enhancement in C<sub>2</sub>H<sub>5</sub>OH response, achieving DL of 0.42 ppb in dry air and 6.1 ppb at 80 % RH at 450 °C, far surpassing the 200 ppb detection requirement for IIDs. These improvements are attributed to (i) the expansion of the EDL resulting from Schottky barrier formation (electronic sensitization) and (ii) the spillover effect of Pt NPs (chemical sensitization). The strategic engineering of PINRs, including optimized NPs size and operating temperature of sensors, enabled stable and highly selective C<sub>2</sub>H<sub>5</sub>OH detection even under high humidity. This work provides valuable insights into enhancing MOS-based gas sensor performance through noble metal functionalization, paving the way for the development of advanced C<sub>2</sub>H<sub>5</sub>OH sensors for IIDs and other gas detection applications.

# CRediT authorship contribution statement

Jae Han Chung: Methodology, Investigation, Formal analysis, Data curation. Eunsol Lee: Methodology, Investigation. Junho Hwang: Investigation, Data curation. Junha Park: Methodology, Investigation. Seungjong Hwang: Methodology, Investigation. Dongwook Youm: Methodology, Investigation. Dohyeon Shin: Methodology, Investigation. Ki Chang Kwon: Methodology, Investigation. Soonmin Yim: Methodology, Investigation. Wooyoung Lee: Methodology. Donghwi Cho: Writing – review & editing, Writing – original draft, Conceptualization. Kwangjae Lee: Writing – review & editing, Writing – original draft, Conceptualization. Young-Seok Shim: Writing – review & editing, Writing – original draft, Conceptualization.

# 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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#### Appendix A. Supplementary material

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## Data availability

Data will be made available on request.

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