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Dissolved hydrogen gas analysis in transformer oil using Pd catalyst decorated on ZnO nanorod array

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ABSTRACT

A resistivity-type hydrogen (H_2) sensor based on palladium (Pd)-decorated zinc oxide (ZnO) nanorod (NR) array has been developed to detect the dissolved H_2 gas in the transformer oil. The Pd catalyst decorated on the ZnO NRs not only enhanced the H_2 sensing properties at room temperature, but also played an important role to protect the ZnO NRs in liquid environment, which resulted long-term stability of the fabricated device. The H_2 sensor had a temperature coefficient of resistance (TCR) of $68.45 \times 10^{-4}/^{\circ}$C, and showed the potential to detect H_2 gas dissolved in transformer oil with a detection range of 5–1000 ppm. The sensor showed high response towards H_2 within 5–100 ppm gas concentrations, which is critical to develop a H_2 sensor for transformer oil. The kinetic H_2 absorption/desorption in sensors was also investigated at different oil temperatures. Moreover, the fabricated sensor achieved high response, good repeatability, and long-term stability in oil, making it a promising candidate for transformer oil applications.

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1. Introduction

Hydrogen (H_2) sensor is becoming more significant for safety purposes in the future hydrogen-based economy and widely used in various application fields [1]. In high voltage power system applications, power transformers are considered as the most critical and expensive component. Generally, oil is used inside the transformers for their operation and can release different gases such as hydrocarbons (C_mH_m), carbon oxides (CO_x), nitrogen oxides (NO_x), and hydrogen (H_2) without any warning [2]. This can cause the failure of transformers in such manners that can cause serious oil spills, fires, extensive damage to adjacent equipment and major disruptions in service [3–10]. As a consequence, it affects the reliability of the whole power system and causes the great damage to the national economy as well. Continuous monitoring of these gases (dissolved in transformer oil) using highly efficient sensors is a possible way to ensure the smooth functionalization of the power systems. Among of all these gases, the detection of hydrogen in the transformer oil is most important as the concentration of dissolved H_2 varies at different levels of faults in the power transformer [11]. In power transformers, H_2 is generated from the thermal decomposition of oil at high temperatures, which is a serious problem for transformer oil. It is mandatory to maintain H_2 gas concentrations within 3 to 3000 ppm in the oil for proper and safe function of transformer, which can be realized by reliable detection of H_2. To meet the demand an efficient and reliable H_2 sensor is required, which should be selective towards H_2 gas along with wide detection range (3–3000 ppm), high sensitivity, the ability to work well in high humid environment, and long-term stability in oil at various oil temperatures [3,5].

There are two methods to detect dissolved gases in transformer oil, namely, online monitoring and offline monitoring. In offline monitoring, a sample for analysis is taken from a transformer and transported to a chromatographic laboratory, where the measurement is carried out [3]. Using this method, the continuous monitoring of the transformer is practically impossible. Moreover, the period of time between the collection of the sample and the measurement can cause measurement errors [3]. On the contrary, in-situ gas detection in transformer oil can be possible through online monitoring. Thus, this measurement method is currently preferred to detect real-time dissolved gas in transformer oil. Different types of H_2 sensors have already been reported in the literature for the online detection of dissolved H_2 gas in transformer oil, such as resistivity-type [3,4], optical-type [3,6–9], and electrochemical-type [10]. Among of various sensor types, resistance-based sensors have several advantages including simple structure, high sensitivity, fast response, low cost, and low power consumption [3,5]. Popular hydrogen sensing nanomaterials including, polymer as polyaniline nanofibers, metal oxide such as zinc oxide (ZnO), tungsten oxide (WO_3), tin dioxide (SnO_2), and indium oxide (In_2O_3), carbon-based materials (carbon nanotube

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and graphene), and noble metals such as platinum (Pt) and palladium (Pd) [12–15] have already been used extensively for decades.

For the resistivity-type H_2 sensors, Pd-based thin films [3] and Pd nanowire (NW) arrays [5] were used and considered as the best catalyst materials for the detection of H_2 in transformer oil due to their high sensitivity towards H_2 and good stability in liquid environment. In comparison to Pd NWs, Pd thin films have lower sensitivity, because of its lower active surface area with compact films and they cannot satisfy the requirements for the characteristics of H_2 sensors used in transformer oil, especially when used to detect low H_2 concentrations down to 3 ppm. On the other hand, Pd NW based H_2 sensors satisfy the requirements for H_2 sensor characteristics in transformer oil; however, they require complex and expensive synthesis process to fabricate the end products [5].

Metal oxide-based gas sensors have huge advantages including simple synthesis mechanisms, low-cost raw materials, and nano-sized morphologies, which are controllable and compatible with micro/nano electromechanical system processes [13,14]. Among of various nanostructured metal oxides, one dimensional (1D) nanostructures including nanorods (NRs), NWs, and nanotubes (NTs) possess high surface-to-volume ratio, which provides more active sites to absorb H_2 molecules than other nanostructures and hence, facilitate the sensor performances [13,16]. In addition, 1D nanostructures exhibit additional quantum effects compared to other dimensional nanostructures and show profound influence on gas sensing performances, in which the grain size, defects, and oxygen-adsorption quantities are crucial [17].

Hence, in this work, we proposed a H_2 sensor for transformer oil monitoring using ZnO NRs array decorated with Pd catalyst. The discontinuous Pd nanoparticles (NPs) and ZnO NRs formed a core-shell like structure (ZnO NRs as the core and the Pd catalyst as the shell), which will ultimately reduce the cost of the final H_2 sensors (due to the use of lesser amount of expensive Pd material) than the pure Pd NWs array based sensors. In addition, a huge number of nanojunctions between the Pd NPs and ZnO NRs will enhance the sensitivity of the H_2 sensor. Our main target is to develop simple and low-cost H_2 sensors that will be capable to detect H_2 gas satisfactorily in transformer oil.

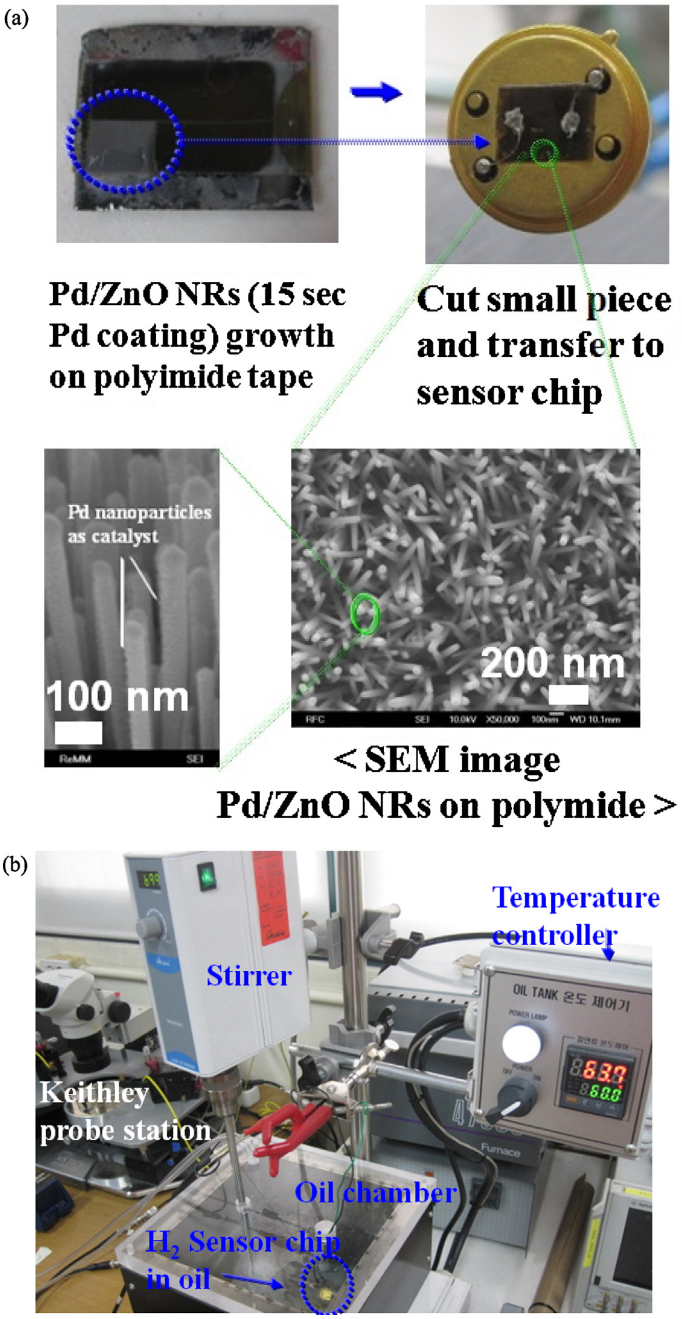

Fig. 1. (a) Fabrication process of the H_2 sensor based on Pd NPs/ZnO NRs/PI and (b) the setup of the measurement system for transformer oil.

2. Experiment

Based on our previous experiments, Pd NPs/ZnO NRs based H_2 sensor was fabricated by depositing Pd NPs (with an optimal Pd catalyst size of 8 nm) on ZnO NRs by radio frequency (RF) magnetron sputtering [14]. Commercial polyimide (PI) tape was used to grow the ZnO NR array to enhance the surface of the sensing layer [13,14]. The detailed experimental process of Pd NPs/ZnO NRs on PI tape based H_2 sensor was described in our previous work [14]. Two Ohmic contacts of gold (Au) were deposited on the surface of the Pd NPs/ZnO NRs/PI via a metal mask and RF magnetron sputtering (150 W, 7 mTorr working pressure). The diameter of the contacts was 1 mm, and the distance between the two contacts was 0.9 cm. After fabrication, the sensor was carefully attached on to a sensor chip (TOE-45). The H_2 sensor fabrication process was completed by a wire bonding process between the Au electrodes and the sensor chip using aluminum (Al) wire. The sensor fabrication process is described in Fig. 1(a).

An optical photograph of the measurement system set-up for dissolved H_2 gas in transformer oil is shown in Fig. 1(b). The H_2 sensor was mounted inside an oil chamber, which was filled by the real transformer oil (supplied by Hyundai Heavy Industry Co.). A Keithley probe station (SCS-4200) with a bias voltage fixed at 1 V was used for data acquisition and calculation. A computerized mass flow controller system (ATOVAC, GMC 1200) was used to vary the concentration of H_2 in nitrogen (N_2, 99.99%) (Deokyang Co. Ltd.). Gas mixtures with different H_2 concentrations were delivered to the oil chamber via two filter membranes at a constant flow rate of 50 standard cubic centimeters per minute (sccm). The filter membrane was used to create H_2 gas bubbles in the oil. The H_2 gas was dissolved into the oil by vigorous stirring. Oil temperature was supplied by an integrated heater inside the oil chamber and was controlled with an outside controller. The H_2 gas in the oil chamber was purged with N_2 between each H_2 pulse to allow the surfaces of the sensors to return to atmospheric conditions. The independence test on our H_2 sensor was performed in real transformer oil by professional equipment from an H_2 scan (also supplied by Hyundai Heavy Industry Co.).

The morphology of the Pd NPs/ZnO NRs/PI was characterized by field emission scanning electron microscope (FE-SEM; JSM-6500F), transmission electron microscope (TEM; JEOL JEM-2100F) and high resolution TEM (HRTEM). The crystalline characteristics and the

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