Effect of Humidity on IDE Based WO₃/Nafion Polymer Sensing Structure Resistivity

1,2Amirul Abd Rashid, 1Nor Hayati Saad, 1Daniel Bien Chia Sheng, 1Lee Wai Yee

1NEMS and Photonics Lab, MIMOS Berhad, Technology Park Malaysia, Kuala Lumpur, 57000, Malaysia
2Micro-Nano Electromechanical System Laboratory (MiNEMS), Faculty of Mechanical Engineering, Universiti Teknologi MARA, 40450 Shah Alam Selangor Malaysia

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A B S T R A C T
Background: Nanostructured metal oxide material has widely been used as sensing element for gas sensor application. Their high surface to volume ratio allows better adsorption of target analyte at relatively lower concentration. However, they are also known to be very sensitive to the surrounding atmosphere, especially temperature and humidity. Objective: In this study, the effect of humidity on IDE based gas sensor inside a typical gas test chamber was investigated. The sensing element of the sensor consists of nanostructured WO₃ mixed with nafion polymer to create conductive networks across IDE. Results: It was found that when the nitrogen gas feed into the chamber, the resistance of the sensor increased significantly from ohm to kohm level while the RH level reduced from ~ 70% to less than 10%. This situation can be controlled by connecting a simple air bubble method where the resistance and RH was able to be maintained at certain value. Conclusion: Without any treatment, the usage of nitrogen gas as analyte carrier will have a big impact to the humidity inside the test chamber. This situation at the same time dictates the resistance value of the gas sensor prior exposing to any analyte. Therefore, it is worth to emphasize the importance to consider the actual level of humidity during characterization study to avoid misinterpretation of the metal oxide gas sensor performance result.

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INTRODUCTION
Since the commercialization of gas sensors in 1970s, the research in this field is continuously in growing mode (Yamazoe, 2005). The gas sensor was deployed not only to monitor the environment quality but has been used in industry, automotive as well as agriculture and food industry (Wang et al, 2006). There are many types of gas sensors but among them, metal oxide base gas sensors have many benefits to offer. They are relatively cheaper, more robust, lightweight and long lasting (Fine et al, 2010). With the advancement in nanotechnology, nanostructurated metal oxide gas sensor such as SnO, ZnO and WOₓ was extensively developed because of their high specific surface area makes them a very sensitive sensing element (Li et al, 1998).

Nevertheless, metal oxide is known to be sensitive to surrounding parameters such as temperature and humidity (Wang et al, 2010). Therefore, it is very important to understand how is the effect of these variables to the performance of the sensor to ensure the result obtains from the characterization study is accurate and representing the actual capability of the sensor. In this study, the focus is to understand how the WOₓ/nafion polymer sensor will respond to different level of humidity inside typical gas chamber. Nafion which is a type of conductive polymer (Gursuch et al, 2008) was used as a binder to create the conduction path in between the IDE electrodes.

Methodology:
WO₃ nanostructure was syntheesised via facile hydrothermal process reported elsewhere. WO₃ nanostructure was synthesized following the experimental procedure reported by Rashid et al (2013, 2014). All chemicals were of analytical reagent grade (Sigma-Aldrich) and used without further purification. The aqueous solutions were prepared by dissolving sodium tungstate dehydrate powder (Na₂WO₄·2H₂O) in DI water. The pH of this solution was controlled by mixing oxalic acid (H₂C₂O₄) to the solution while continuously stir at 350 r.p.m for 30 minutes. Right before the solution transferred into teflon-lined autoclave reactor, small amount of sodium chloride (NaCl) was added to the solution acting as directing agent of the nanostructure. The solution was transferred into teflon-lined autoclave and heated inside oven for 8 hours at 180°C. Once the synthesis...
process completed, the autoclave was cooled to room temperature naturally and the product obtained was washed in DI water and later dried in air for morphology characterization. Scanning electron microscopy (JEOL, JSM-7500 F) with 5 KV acceleration voltages was used to examine the morphology of as synthesized WO3 nanostructure.

To fabricate the sensing structure, WO3 nanostructure was mixed with 1% nafion in ethanol. The solution was then drop cast on silicon based IDE platform and spin coated at 2000 rpm for 30 seconds. To study the effect of humidity, the sensor was connected to LCR meter (Agilent model 4263B) and placed inside a 40 L test chamber. Commercial RH and temperature probe (Vernier, USA) was also placed inside the chamber to correlates the level of humidity and temperature to the sensor resistance. Initial resistance of the sensor was measured and compared to resistance when the humidity inside the chamber dropped when 10 sl/m nitrogen supplied to the chamber via mass flow controller (MFC). This nitrogen gas was used as the carrier to bring in analytes such as CO2, NO3 or C2H4 into the chamber during the actual characterization of the gas sensor. As an alternatives to control the humidity of the carrier gas (in this case nitrogen), the gas was flown through a simpler bubbler system as suggested by Forney and David (1992). A simplified schematic system for the apparatus used for this study shown in Figure 1.

![Fig. 1: Schematic drawing of gas sensor test chamber set-up apparatus in this study.](image)

**RESULTS AND DISCUSSIONS**

Scanning Electron microscopy (SEM) of the WO3/Nafion polymer sensing structure as shown in Figure 2. The bulk of the morphology consists of non-oriented 1D WO3 nanorods dispersed uniformly on the surface of IDE with average diameter of 65 nm and length of 1.2 microns. Theoretically, when analyte gas exposed the nanostructure, the surface reaction occur which will either increase or reduced the total resistance depends on the analyte type.

![Fig. 2: WO3/Nafion polymer sensing structure network on IDE.](image)

The initial resistance of such fabricated sensor was ~ 143 Ω with RH of ~ 75 % as shown in Figure 3 (a). When nitrogen gas feed into the chamber, the RH level dropped to ~ 10 %. Simultaneously, the resistance of the sensor increased significantly up to 4 kΩ. This suggests that the nitrogen gas somehow removed large amount of
water molecules from the chamber. Therefore, less oxygen molecule available at the surface of the nanostructure this reduced the mobility of the electron, hence the resistance of the sensor to increased.

Fig. 3: Initial RH vs resistance of the sensor (a) and significance increase of resistance when RH reduced upon nitrogen gas feeding (b). RH and resistance was controlled at certain level when nitrogen flown via bubler (c).

By connecting a self-made simple bubbler system to the nitrogen feeding line, it was found that the RH level was slightly increased from ~ 70% but it stabilized at ~ 88 % in 15 minutes as shown in Figure 3 (c). At the same time, the resistance value decreased from ~ 150 Ω to 85 Ω and remain unchanged for the next 20 minutes. This shows that the bubbler system was able to treat the nitrogen gas to absorb enough moisture to prevent RH loss inside the chamber.

Conclusion:
From this study, we can conclude that the nitrogen gas which was used as to carry the analyte to the metal oxide sensor inside the chamber will significantly reduced the RH level. If we not able to discriminate this phenomenon, the performance of the gas sensor towards the actual analyte is possibly misinterpreted by the effect of the sensor towards RH itself. A simple bubbler system was proven to mitigate this issue to ensure the RH level of the system can be controlled at certain level. As such, the actual performance of the sensor will be
isolated from the effect of humidity during actual characterization study of the fabricated metal oxide gas sensor.

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