A Novel Molybdenum Disulfide Based High-Precision Microwave Sensor for Methanol Gas Detection at Room Temperature

Mohammad Mahmudul Hasan, Onur Alev, Eda Goldenberg, Michael Cheffena

***Abstract*—This letter reports, for the first time, a molybdenum-disulfide (MoS_2)-coated microwave gas sensor for methanol gas detection. A wideband monopole antenna, resonating at 2.45 GHz for wireless applications is fitted with a 15-mm long copper-based grating sensing element for high precision gas sensing. When the sensor surface is coated with a highly sensitive layer of hydrothermally grown MoS_2 and exposed to methanol gas, the electrical properties of the sensing material change. The developed sensor confirms the detection of methanol gas in parts-per-thousand (ppt) range from 5.3 to 42.6 ppt with a sensitivity of ∼1.0 MHz/5 ppt. Furthermore, the gas sensing functionality of the sensor does not affect the communication performances of the antenna, suggesting seamless integration into wireless sensor networks. The simulation results, experimental data, and sensing mechanism are provided to support the claims.**

***Index Terms*—Microwave Antenna sensor, methanol gas sensor, monopole antenna, Molybdenum disulfide, MoS_2.**

I. Introduction

TODAY, air pollution is one of the world's deadliest threats and can be considered a pandemic, killing around nine million people every year (one in six deaths worldwide) [1]. Among volatile organic compounds (VOCs), methanol is extremely toxic and, despite its use in many applications, can cause serious adverse health effects in indoor environments [2]. According to the Occupational Safety and Health Administration (OSHA), its permissible exposure limit (PEL) is 200 parts-per-million (ppm) (260 mg/m^3). Overexposure can cause bronchoconstriction [3], neurological disorders [4], visual impairments [5], and blood disorders [6]. Research also shows that 90% of these poisonings occur due to lack of awareness [4]. There is therefore an urgent need for the development of methanol gas sensors. In recent decades, several novel sensors have been developed based on chemo-resistive, electrochemical, and optical devices [7], [8]. However, these technologies have some disadvantages such as high operating temperature, high power consumption, and complex electronic systems [7]. Therefore, there is an urgent need for a new generation of sensors for methanol detection. Recently, microwave-based sensors have attracted much attention in gas sensing, medical applications, environmental monitoring, etc. due to their inherent advantages such as simplicity, low-cost, low-power, real-time sensing at room temperature (RT), passive operation, self-recovery, etc. [9], [10]. Furthermore, antenna-based microwave sensors can be functionalized for dual operation: communication and sensing and can thus be integrated into wireless sensor networks (WSNs) [11], [12].

Another important part of a gas sensor is the sensitive layer. So far, various materials such as metal-oxides, polymers, alloys, and transition metal dichalcogenides (TMDs) have been used in sensing applications [7]. Among them, molybdenum disulfide (MoS_2), a semiconductor TMD, is one of the most promising sensing materials due to its large specific surface area, high carrier mobility, and surface activity [13]. In addition, MoS_2 has shown excellent results against various gases at RT. Zhang *et al.* reported that the MoS_2-based sensor exhibited sensor response to ethanol gas at RT [14]. Halvaee *et al.* showed that MoS_2-based gas sensor can be a good candidate for RT detection of methanol gas. Kim *et al.* developed a MoS_2-based gas sensor for the detection of VOCs at RT [15]. These properties of MoS_2 make it a powerful tool for new generation gas sensors. When microwave sensors are exposed to the target gas, the electrical properties such as conductivity, permittivity, etc. of the sensing material change. This change proportionally perturbs the antenna resonance frequency, phase, magnitude of the reflection coefficient ($|S_{11}|$), etc. [16] and is used to detect the presence of the gas along with its concentration. For example, nanomaterial-loaded antenna sensors have been used for gas detection using either the changes in the ($|S_{11}|$), or the resonance frequency [17]. However, the ($|S_{11}|$)-based detection technique may not be reliable for sensor network integration, as ($|S_{11}|$) can also be sensitive to environmental changes [18], [19]. Therefore, researchers are putting most emphasis on sensors with frequency-based detection to facilitate seamless integration with WSNs [20], [21].

This letter presents a novel, low-cost gas sensor that couples hydrothermally grown MoS_2 to a microwave transducer. The developed microwave sensor detects methanol gas with a sensitivity of ∼1.0 MHz per ∼5 parts-per-thousand (ppt) at RT. This high-precision sensing capability was achieved by using a 15-mm long grated-copper (GC) patch and a highly sensitive layer of MoS_2. During the gas detection (or recovery) process, the observed frequency shifts do not affect the transmission bandwidth, suggesting it as a viable option for WSNs. To the best of our knowledge, there have been no reports of VOC detection studies using MoS_2-based microwave platform.

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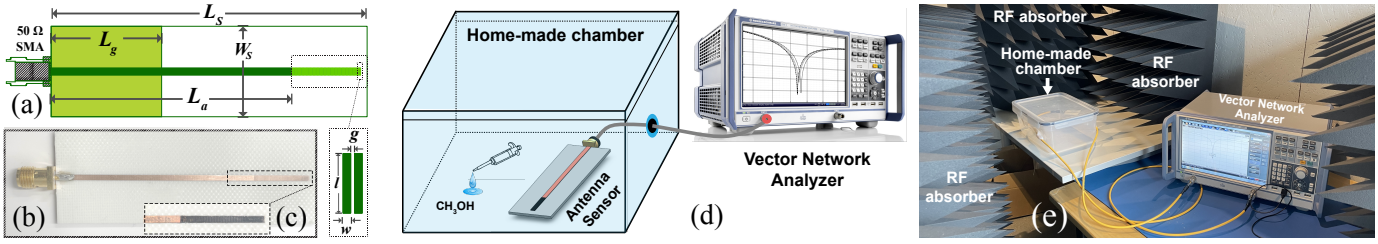

Fig. 1. (a) Geometry of the proposed antenna-sensor with grated-copper patch (sensing area). Fabricated antenna: (b) before and (c) after MoS_2 coating. Experimental setup: d) a conceptual diagram, and e) the real scenario of sensor measurements.

II. Experimental Details

A. Sensor-Integrated Antenna Design

A broadband monopole microstrip patch antenna was designed for microwave sensing and wireless applications at 2.45 GHz, as shown in Fig. 1(a). Fig. 1(b) shows the fabricated antenna on Rogers 4003C substrates with relative dielectric constant ε_r = 3.55, dielectric loss tangent tan δ = 0.002, substrate thickness h = 1.52 mm, and copper thickness t = 0.035 mm. The substrate has a length L_s = 71 mm, width W_s = 27 mm and a ground plane of L_g = 20.75 mm. The antenna is fed by a 50 Ω SMA connector. At the end of the antenna, a 15-mm long sensing area, consisting of a sequence of 50 discrete GC patches, is incorporated for precision detection. The size parameters of each unit of the GC patch are as follows: length l = 1.56 mm, width w = 0.2 mm, and gap g = 0.1 mm. The geometric parameters (l, w, g) of the GC patches were optimized to increase sensitivity. First, the sensor area was coated with a thin film of MoS_2 covering the GC pattern, as shown in Fig. 1(c). The conductivity (G) and susceptivity (B) of the sensing area were then measured with R&S HM8118 LCR Bridge/Meter and used for optimization by HFSS full-wave simulation. To achieve perfect resonance at 2.45 GHz without any chemical layer loading, the antenna dimensions were readjusted after adding (and optimizing) the sensor area.

B. Synthesis of MoS_2

MoS_2 was synthesized by a simple hydrothermal process [22]. To prepare MoS_2 structures, 390 mM thiourea and 80 mM sodium molybdate dihydrate (Na_2MoO_4•$2H_2O$) were dissolved in 52.5 mL ethanol and 52.5 mL deionized (DI) water. The resulting solution was then transferred to a stainless-steel autoclave. The autoclave was then placed in a temperature-controlled oven at 175°C for 24 hours. After the hydrothermal process, the solution was filtered and rinsed with ethanol and DI water. The resulting black precipitates were then dried in a temperature-controlled oven at 100°C. For gas sensor measurements, the prepared MoS_2 powders were suspended in DI water to achieve a constant concentration of 5 mg/mL. In addition, 4% wt. poly (vinyl alcohol) was added to the solution to improve the adhesion of the MoS_2 film to the substrates [23]. The final solution was then dropped onto the copper patch of the antenna (see Fig. 1(c)).

C. Experimental Setup

To better understand the electrical behavior and methanol sensing characteristics of the MoS_2 film, electrical measurements were performed. A potentiostat (PalmSens4) attached to a commercial MoS_2 coated electrode (ItalSens Gold SPE, PalmSense) was placed inside the chamber and antenna measurements were carried out at the same concentrations. The time dependent current curves were monitored using chronoamperometry measurements under a constant applied voltage of 1 V. Gas sensor measurements were conducted at RT in a homemade polypropylene chamber with a volume of 5.2 L (width: 32 cm, length: 21 cm, and height: 12 cm), as shown in Fig. 1(d)-(e). All measurements related to $|S_{11}|$ and frequency were carried out using an R&S ZNB8 Vector Network Analyzer (VNA), with a sweep frequency ranging from 1 to 4 GHz with a frequency spacing of 100 kHz, and a power level set to -10 dBm. After connecting the MoS_2-coated antenna to the VNA and placing it in the chamber, the base frequency was first recorded. Methanol was then added to the chamber in volumes of 50, 100, 150, 200, 250, 300, 350, and 400 μL using a micropipette. After evaporation of the methanol, the resulting concentrations in the closed box were calculated as 5.3, 10.7, 16.0, 21.3, 26.7, 32.0, 37.3 and 42.6 ppt. The $|S_{11}|$ was recorded every 5 seconds using MATLAB for all subsequent concentrations.

III. Results and Discussions

A. Sensor Performances

After MoS_2 coating, G and B of the sensor area (measured between the 1st and 50th copper contacts) increase by 0.0050 μS and 0.0025 μS, respectively. This increased admittance also increases the conductivity between the GC patches, thereby increasing the current flow. This increases the effective length of the antenna and shifts the resonance frequency to 2.441 GHz (see Fig. 2(a)). This frequency is used as the baseline for the sensor. Prior to the antenna gas sensing measurements, chemiresistive measurements were performed to better understand the sensing behavior of the MoS_2 film and to confirm its sensitivity to methanol gas. The time-dependent current curve under different methanol concentrations is shown in Fig. 2 (b). It is observed that the resistivity of MoS_2 increases when the sensor is exposed to methanol gas. Furthermore, MoS_2 showed a sensor response to methanol in the concentration range of 1.6 – 27 ppt. Although the sensor-antenna was designed to resonate at 2.45 GHz, the measured return-loss of the uncoated sensor was -51.99 dB at 2.462 GHz, as shown in Fig. 2(a). This 12.0 MHz difference in resonant frequency is due to the dimensional tolerance of the fabrication process. After the MoS_2 coating, the measured 10-dB return-loss bandwidth is 417 MHz (from 2.300 GHz to 2.717 GHz). This wideband operation enables

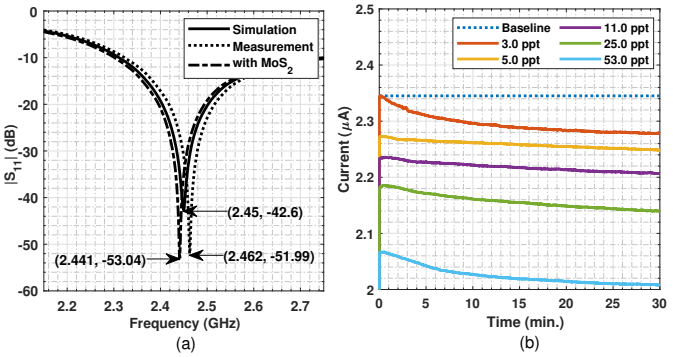

Fig. 2. (a) Simulated and measured antenna return-loss before and after MoS_2 coating. (b) Time-dependent current measurement with varying methanol exposure.

uninterrupted wireless connectivity without the need for tuning circuitry, despite the frequency shifts during gas exposure. This ensures that the communication performance of the antenna is unaffected by the gas detection, and vice versa. The sensor response to different concentrations of methanol gas at room temperature was then investigated. First, the sensor was exposed to 5.3 ppt methanol for 15 minutes, resulting in a frequency shift of ∼1.0 MHz. This procedure was repeated for all subsequent concentrations (10.7, 16.0, 21.3, 26.7, 32.0, 37.3 and 42.6 ppt). The return-loss, instantaneous frequency, frequency-shifts, and response-time corresponding to each concentration were recorded, as shown in Fig. 3. The sensor demonstrated consistent performance when tested with the same concentrations of methanol on different days over the span of a week (see Fig. 3(c). The total response times ranged from 7 to 14 minutes, as shown in Fig. 3(d). The relatively high sensor response times may be attributed to the time methanol takes to evaporate. Additionally, the observed increase in response time with rising concentration further supports this conclusion. Table I presents a performance comparison of MoS_2-based methanol sensors from literature and the proposed MoS_2-based microwave sensor in this work.

B. Sensing Mechanism

It is observed that antenna frequencies increased with methanol exposure due to the increased resistivity of the MoS_2 (see Fig. 3(a)-(c)). This indicates that MoS_2 exhibits p-type behavior, as evidenced by electrical measurements (see Fig. 2(b)). The p-type behavior of MoS_2 synthesized via the hydrothermal method has been demonstrated in various studies [29]. The sensing mechanism of the semiconductor materials can be explained by the reaction between the reducing gas and the surface adsorbed oxygen species (see Fig. 4) [30]–[33]. In air, absorbed oxygen species trapped electrons from surface states. Thus, negative charged carriers decrease, and

TABLE I
COMPARISON OF MoS_2-BASED SENSORS FOR METHANOL SENSING

Material	Transducer	Temperature	Concentration	Reference
MoS_2	Chemi-resistive	130°C	100 ppm	[24]
MoO_3/MoS_2/rGO	Chemi-resistive	200°C	100 ppm	[25]
ZnO/MoS_2	Chemi-resistive	240°C	100 ppm	[26]
MoS_2/Mxene	Chemi-resistive	140°C	100 ppm	[27]
MoS_2/TiO_2	Chemi-resistive	240°C	500 ppm	[28]
MoS_2	Microwave	RT	5000 ppm	This work

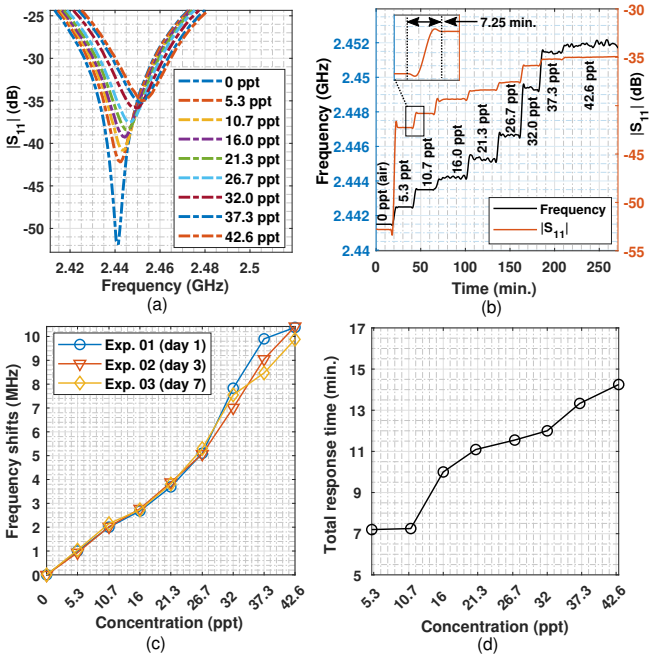

Fig. 3. Measured a) return-loss b) instantaneous resonance frequency, c) frequency shifts, and d) response time for 5 to 43 ppt of methanol

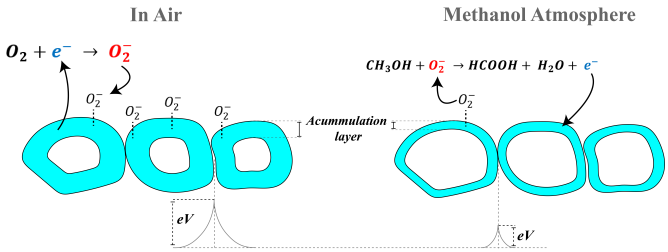

Fig. 4. Schematic for MoS_2 sensing mechanism.

the hole concentration increases. As a result of this process, a hole accumulation layer forms on the MoS_2 surface and the conductivity of MoS_2 increases. When methanol molecules are introduced, they react with surface oxygen species and trapped electrons are released back to the conduction band. Thus, the hole concentration decreases and accumulation layer narrows. Thereby, conductivity of the MoS_2 decreases because of a decrease in band bending. Decreasing conductivity leads to a reduction in the length of the patch antenna due to GC strips design (see Fig. 1(a)). Consequently, reduction of the antenna patch length increases the resonance frequency, accordingly.

IV. CONCLUSIONS

This letter presents our findings on the design, fabrication, and investigation of a novel, simple, low-cost, microwave sensor with frequency-based detection technique, integrated into a 2.45 GHz wideband monopole antenna. A 15-mm-long sensing element combines a grated pattern of copper-patch coated with a hydrothermally grown MoS_2 chemical layer. The developed sensor shows great potential in quickly detecting 5.3-42.6 ppt of methanol gas at RT with a sensitivity of ∼1 MHz/5 ppt. Further, gas sensing does not impact the 10-dB transmission bandwidth of the antenna, implying seamless integration into wireless sensor nodes for uninterrupted communication.

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