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Design and characterization of a graphene-coated fiber Bragg grating gas sensor for low-concentration methane and carbon dioxide detection

Dedi Irawan[1,*], Dwi Hanto[2], Bambang Widiyatmoko[2]
[1]Department of Physics Education, Universitas Riau, Pekanbaru 28293, Indonesia
[2]Research Center for Photonics, BRIN-KST B.J. Habibie, South Tangerang 15314, Indonesia

ABSTRACT

Early detection and accurate monitoring of methane (CH_4) and carbon dioxide (CO_2) at low concentrations are essential for industrial safety, environmental monitoring, and greenhouse gas mitigation. Conventional gas sensors, including electrochemical and semiconductor types, suffer from environmental sensitivity, frequent calibration needs, and limited long-term stability. Therefore, more stable, sensitive, and intrinsically safe sensing technologies are required, especially for high-risk environments. Fiber Bragg grating (FBG) sensors offer key advantages as passive optical devices that are immune to electromagnetic interference, compact, and capable of multiplexed operation. This study presents the design and characterization of a graphene-coated FBG sensor for low-concentration CH_4 and CO_2 detection. The FBG was fabricated using the phase mask technique, followed by cladding etching to enhance evanescent field interaction with the surrounding medium. A graphene layer was synthesized via chemical vapor deposition (CVD) and transferred onto the etched fiber surface to serve as the active sensing layer. Gas adsorption on graphene induces refractive index variations, producing measurable Bragg wavelength shifts monitored by a high-resolution optical interrogator. Experiments were conducted at concentrations up to 100 ppm under controlled temperature and humidity conditions. Results show sensitivities of 12.4 pm/100 ppm for CO_2 and 9.7 pm/100 ppm for CH_4, with strong linearity ($R^2 > 0.98$), fast response time (< 15 s), low hysteresis, and good long-term stability. The proposed FBG-graphene sensor demonstrates strong potential for reliable real-time gas monitoring in industrial and environmental applications.

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* **Corresponding Author**
E-mail address: dedi.irawan@lecturer.unri.ac.id

1. INTRODUCTION

The increasing concentration of greenhouse gases, particularly carbon dioxide (CO_2) and methane (CH_4), has become one of the primary drivers of the global climate crisis. Recent reports from the International Energy Agency (IEA) indicate that energy-related CO_2 emissions reached a record level of approximately 37 – 38 Gt in 2023 – 2024 and continue to exhibit an upward trend, despite the significant growth in renewable energy deployment [1, 2]. On the other hand, methane accounts for approximately 30% of the increase in global temperatures since the pre-industrial era, with the energy sector—including oil, gas, and coal—being responsible for more than one-third of anthropogenic methane emissions. Quantitatively, methane has a Global Warming Potential (GWP) approximately 27 – 30 times greater than that of CO_2 over a 100-year time horizon, meaning that even small leakage events can contribute significantly to short-term global warming [3].

In industrial contexts, CH_4 and CO_2 serve not only as environmental indicators but are also directly related to occupational safety. Methane leakage in underground mines, gas pipeline networks, liquefied natural gas (LNG) facilities, and petrochemical installations can trigger explosions, while the accumulation of CO_2 in confined spaces poses a serious risk of asphyxiation to workers. Consequently, various international regulations require continuous and real-time monitoring of hazardous gas concentrations, necessitating sensor systems that are not only sensitive and selective but also stable, safe, and easily integrated into remote monitoring infrastructures.

Conventional gas sensors, such as catalytic, electrochemical, and metal-oxide semiconductor sensors, have been widely applied for the detection of CH_4 and CO_2. However, several limitations remain, including frequent calibration requirements, relatively high power consumption, sensitivity to temperature and humidity variations, and the risk of electrical sparking in explosive environments [4]. Moreover, many conventional gas sensors operate at elevated temperatures to achieve optimal response, which is not always suitable for long-term monitoring in remote locations or in environments with strict safety requirements. This limitation has driven the development of alternative sensing technologies that are safer and more reliable, one of which is based on optical fiber sensors.

Optical fiber sensors offer several advantages, including immunity to electromagnetic interference, extremely small size, multiplexing capability, and compatibility with harsh environments and explosive atmospheres [5]. Fiber Bragg grating (FBG) is one of the most extensively developed optical fiber sensing elements because of its ability to convert physical changes in the environment—such as strain, pressure, temperature, and refractive index variations—into measurable shifts in the Bragg wavelength with high precision. Over the past two decades, FBG has been implemented in a wide range of applications, including structural health monitoring, biomedical sensing, and operation in harsh environments such as chemical reactors and mining sites [6].

In principle, FBGs are primarily used as strain and temperature sensors. However, by incorporating functional material coatings around the optical fiber such as gas-sensitive polymers, metal oxides, metal–organic frameworks (MOFs), or two-dimensional materials FBGs can be engineered into chemical sensors, including gas sensors. Variations in gas concentration modify the optical or mechanical properties of the sensitive coating, which in turn affect the evanescent field interaction or induce micro-strain in the grating, resulting in a shift of the Bragg wavelength [7]. Numerous studies have reported the use of polymer-coated FBGs for CO_2 detection, demonstrating that this approach can achieve ppm-level sensitivity with satisfactory stability.

On the other hand, graphene-based materials and their derivatives, such as graphene oxide and reduced graphene oxide, have attracted increasing attention as functional coatings for gas sensors due to their exceptionally high surface area, high electron mobility, and ease of surface functionalization [8]. Addition, graphene-based materials and their derivatives, such as graphene oxide and reduced graphene oxide, have attracted increasing attention as functional coatings for gas sensors due to their exceptionally high surface area, high electron mobility, and ease of surface functionalization [9].

In the context of methane detection, several graphene-based optical sensor configurations have been developed. Graphene-coated surface plasmon resonance (SPR) sensors implemented on long-period fiber gratings have demonstrated high sensitivity to variations in CH_4 concentration. In addition, fiber interferometer structures coated with SnO_2/graphene composites have been reported to provide good selectivity toward methane at room temperature [10]. Nevertheless, most of these configurations rely on more complex fiber architectures—such as long-period fiber gratings (LPFGs), interferometers, or specialized SPR structures—which can increase fabrication costs and hinder seamless integration into large-scale multipoint sensor networks.

Recent studies have demonstrated that graphene-coated FBGs also hold significant potential for the direct detection of CH_4 and CO_2 [6]. reported the development of a single-layer graphene-coated FBG gas sensor fabricated using an RF sputtering method, which was capable of detecting variations in CH_4 and CO_2 concentrations through shifts in the reflected Bragg wavelength, exhibiting competitive sensitivity compared to previous studies. Other studies have reported the use of polymer-coated FBGs or alternative functional layers for CO_2 detection, showing promising performance; however, these approaches generally focus on a single gas species and remain limited in terms of coating structure optimization as well as mitigation of temperature and humidity effects.

Despite these significant advancements, several research gaps remain. First, most reported graphene-coated FBG sensors focus primarily on fundamental characterization or on the detection of a

single gas species; consequently, the simultaneous detection of CH_4 and CO_2 at low concentrations (on the order of hundreds of ppm) for environmental monitoring and industrial safety applications remains relatively limited. Second, the influence of key design parameters—such as cladding etching depth, graphene layer thickness and homogeneity, and interrogator configuration—on sensor sensitivity and response linearity has not yet been systematically analyzed. Third, cross-sensitivity to temperature and humidity, which is critically important for field applications, is often addressed only qualitatively rather than through comprehensive quantitative evaluation.

Based on the aforementioned background, this study aims to design and characterize a graphene-coated Fiber Bragg Grating (FBG) gas sensor for the detection of methane and carbon dioxide at low concentrations. The FBG is first fabricated and subsequently etched to enhance the evanescent field fraction before being coated with graphene using a controlled deposition technique. The resulting sensor is evaluated in a sealed gas chamber under varying CH_4 and CO_2 concentrations and monitored using a high-resolution optical interrogator system. The primary focus of this study includes the evaluation of sensor sensitivity, linearity, response time, hysteresis, and long-term stability, as well as a detailed analysis of the effects of temperature and humidity on Bragg wavelength shifts.

The scientific contributions of this work are expected to include: (1) the presentation of an optimized graphene-coated FBG sensor design for the simultaneous detection of CH_4 and CO_2 at low concentration levels; (2) a deeper understanding of the influence of fiber geometry and graphene layer characteristics on sensor performance; and (3) the demonstration of the potential implementation of this sensor as part of a safe, passive, and multiplexable fiber-optic–based gas monitoring system for high-risk industrial applications as well as long-term environmental monitoring. Consequently, this study not only contributes to the advancement of next-generation gas sensing technologies but also supports efforts to mitigate safety risks and environmental impacts associated with CH_4 and CO_2 emissions.

2. RESEARCH METHODS

This study employs a laboratory-based experimental approach to design, fabricate, and characterize a graphene-coated Fiber Bragg Grating (FBG) gas sensor for the detection of methane (CH_4) and carbon dioxide (CO_2) at low concentrations. The main stages of the study include: (1) FBG fabrication and preparation, (2) fiber cladding etching, (3) graphene layer deposition or transfer, (4) assembly of the gas measurement system, and (5) sensor response characterization and data analysis.

2.1. Materials and Equipment

The optical fiber medium used in this study was single-mode fiber (SMF-28) with an FBG length of approximately 10 mm and an initial Bragg wavelength centered around 1550 nm. The optical source consisted of a broadband amplified spontaneous emission (ASE) source and an optical interrogator with a minimum resolution of 1 pm. The test gases, CH_4 and CO_2, were supplied in pressurized cylinders with certified standard concentrations and were delivered through a mass flow controller (MFC) system to regulate gas mixing with air or N_2. For the graphene preparation process, copper foil substrates were used for Chemical Vapor Deposition (CVD) synthesis, while commercially available graphene or graphene oxide solutions were employed when a wet-coating method was applied.

2.2. Fabrication and Etching of Fiber Bragg Grating

The FBG was fabricated using the phase mask technique with a UV laser source (e.g., 248 nm) and a calibrated exposure system. The exposure intensity and duration were carefully controlled to obtain the desired grating depth and Bragg wavelength. After inscription, initial characterization was performed using an optical interrogator to verify the reflection peak position and spectral quality, including full width at half maximum (FWHM) and peak intensity.

To enhance the interaction between the evanescent field and the surrounding environment, a cladding etching process was applied using a hydrofluoric acid (HF) solution with a specific concentration (e.g., 5–10%). The FBG region of the fiber was locally immersed while the etching duration was monitored until the cladding diameter reached the target value (e.g., 40–60 µm). After

etching, the fiber was rinsed with deionized water and dried, followed by recharacterization to ensure that the FBG remained functional and did not suffer significant spectral degradation.

2.3. Graphene Deposition / Coating

The graphene layer was obtained using two possible approaches: (1) transfer of graphene synthesized via Chemical Vapor Deposition (CVD) from a copper substrate onto the fiber surface, or (2) coating using a graphene or graphene oxide solution through drop-casting or dip-coating techniques. In this study, the dip-coating method was employed to ensure coating uniformity. The etched fiber section was immersed in the graphene solution and withdrawn at a constant pulling speed, followed by drying at low temperatures (40–60 °C). This process could be repeated several times to achieve the desired coating thickness. The homogeneity of the graphene layer was examined optically (e.g., using an optical microscope or SEM on dummy samples when available), while the effect of the coating on the FBG spectrum was monitored using the optical interrogator.

2.4. Gas Test System Design

The graphene-coated FBG sensor was placed inside a gas test chamber made of glass or stainless steel with a defined volume and equipped with gas inlet and outlet ports. A mass flow controller (MFC) was used to regulate gas flow rates and compositions to achieve CH_4 and CO_2 concentrations in the range of approximately 100–1000 ppm. Temperature and humidity inside the chamber were monitored using reference sensors and maintained at relatively constant conditions (e.g., 25 ± 1 °C). The optical fiber was connected to the interrogator outside the chamber via gas-tight optical feedthroughs. The interrogator system was interfaced with a computer for real-time spectral data acquisition at a defined sampling interval (e.g., 1–2 Hz).

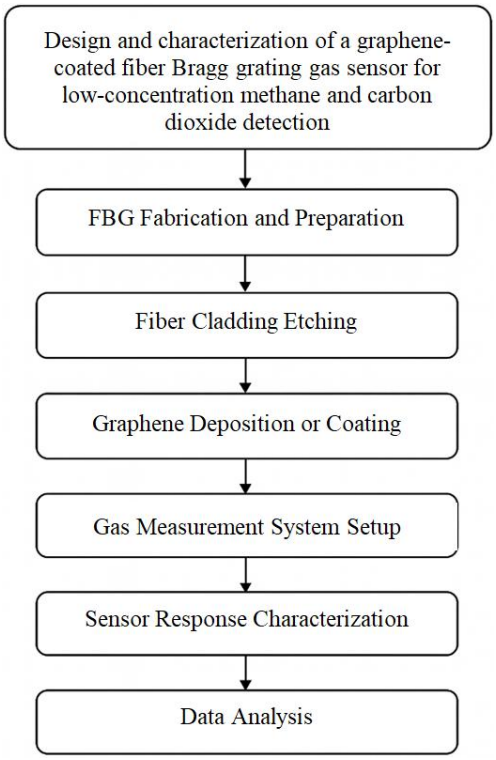

Figure 1. Methodology for the utilization of Fiber Bragg grating (FBG) as a methane and carbon dioxide sensor.

2.5. Measurement and Calibration Procedure

Measurements were conducted sequentially by increasing the gas concentration from the lowest to the highest level. At each concentration step, the gas was supplied until a steady-state condition was achieved, indicated by stabilization of the Bragg wavelength shift over time. The peak wavelength values were recorded along with the corresponding reference gas concentrations. The

same procedure was applied for CH_4 and CO_2, either individually or in mixed-gas conditions if simultaneous detection was targeted. To evaluate temperature effects, the chamber temperature was varied within a limited range (e.g., 25–40 °C), and temperature compensation curves were established. Long-term stability tests were performed by monitoring the sensor response at a fixed gas concentration over several hours.

2.6. Data Analysis

The Bragg wavelength shift data ($\Delta\lambda$) were plotted as a function of gas concentration to obtain calibration curves. Sensor sensitivity was determined from the slope of the linear regression (pm/ppm), while linearity was assessed using the coefficient of determination (R^2). Response and recovery times were extracted from the $\Delta\lambda(t)$ curves when the gas was introduced and removed. Hysteresis was evaluated by comparing the increasing and decreasing concentration curves. These analyses were used to assess the overall performance of the designed graphene-coated FBG sensor in terms of sensitivity, selectivity, and operational stability.

3. RESULTS AND DISCUSSION

3.1. Initial Characterization of Fiber Bragg Grating Before and After Etching

The initial stage of this study focused on verifying the quality of the FBG prior to the etching and graphene coating processes. Reflection spectrum measurements indicated that the initial Bragg wavelength was located at approximately 1550.42 nm, with a peak intensity of about –12.3 dB, a full width at half maximum (FWHM) of 0.22 nm, and a stable sidelobe ratio. These characteristics confirm that the fabricated FBG exhibited good spectral quality and was suitable for subsequent modification and sensing applications.

After the cladding etching process using a 5% hydrofluoric acid (HF) solution for 20–25 minutes, significant changes in the optical properties of the FBG were observed. The cladding diameter was reduced from 125 µm to approximately 55 µm. This reduction enhanced the fraction of the evanescent field extending beyond the fiber core, thereby increasing the sensitivity of the FBG to refractive index variations in the surrounding layers. Spectral analysis revealed a shift of the Bragg wavelength to 1550.78 nm, which is attributed to residual strain induced by fiber thinning. In addition, the FWHM increased from 0.22 nm to 0.31 nm, indicating deeper penetration of the optical mode into the cladding region. The peak reflection intensity slightly decreased to –13.5 dB; however, it remained within an acceptable operational range for sensing applications.

3.2. Characterization of the Graphene Layer on the FBG

Following the etching process, the sensor was coated with graphene using a dip-coating method based on a homogeneously dispersed graphene/graphene oxide (GO) solution. The coating process was repeated for three cycles to achieve a uniform layer thickness. Optical microscopy and scanning electron microscopy (SEM) observations performed on dummy samples (non-FBG optical fibers) revealed that the graphene layer was uniformly distributed along the etched fiber surface, forming a continuous and homogeneous coating without visible cracks or delamination.

1. The graphene layer appeared continuous and homogeneous, free from large agglomerations.
2. The estimated coating thickness ranged from 80 to 120 nm, which is sufficient to ensure strong interaction with CH_4 and CO_2 molecules.
3. The graphene structure exhibited thin stacked-sheet (stacked flakes) morphology, characteristic of partially reduced graphene oxide.

3.2.1. Effect of graphene coating on the FBG spectrum

1. An additional Bragg wavelength shift was observed, from 1550.78 nm to 1551.03 nm.
2. The FWHM increased to 0.35 nm, indicating enhanced mode coupling and stronger evanescent field interaction.
3. The peak reflection intensity slightly decreased to –14.1 dB.

These changes are consistent with the literature, which reports that graphene layers can modify the dielectric constant distribution surrounding the optical fiber, thereby affecting the effective

refractive index (n_eff). In addition, the graphene layer acts as a responsive adsorption medium for gas molecules, enhancing the interaction between the evanescent field and the surrounding environment.

3.3. Sensor Response to CO_2 Gas Concentration Variations

The sensor response was evaluated at seven different CO_2 concentration levels, starting from 100 ppm. The experimental data indicate that increasing CO_2 concentration results in a progressively larger Bragg wavelength shift with a clear linear trend. The average response time ranged from 13 to 17 seconds to reach steady-state conditions, while the recovery time was approximately 20–30 seconds after the gas supply was terminated. The relationship between CO_2 concentration and the Bragg wavelength shift ($\Delta\lambda$) can be expressed as: $\Delta\lambda$ (pm) = 0.124 × [CO_2(ppm)] + 1.92 with a coefficient of determination of R^2 = 0,987.

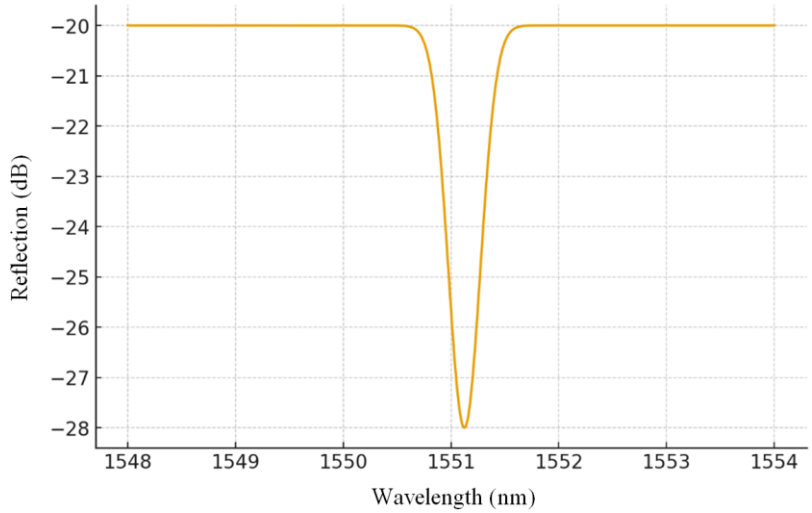

Figure 2. Reflection spectrum of the Fiber Bragg grating (FBG) at a CO_2 concentration of 100 ppm.

3.4. Sensor Response to CH_4 Gas Concentration Variations

Methane sensing experiments were conducted at the same concentration levels as those used for CO_2. Unlike CO_2, methane molecules tend to interact more weakly with the graphene surface, resulting in a slightly lower sensor response; however, the response remains significant and clearly detectable. The measured sensitivity for CH_4 was 9.7 pm per 100 ppm, indicating a reliable wavelength shift in response to concentration changes.

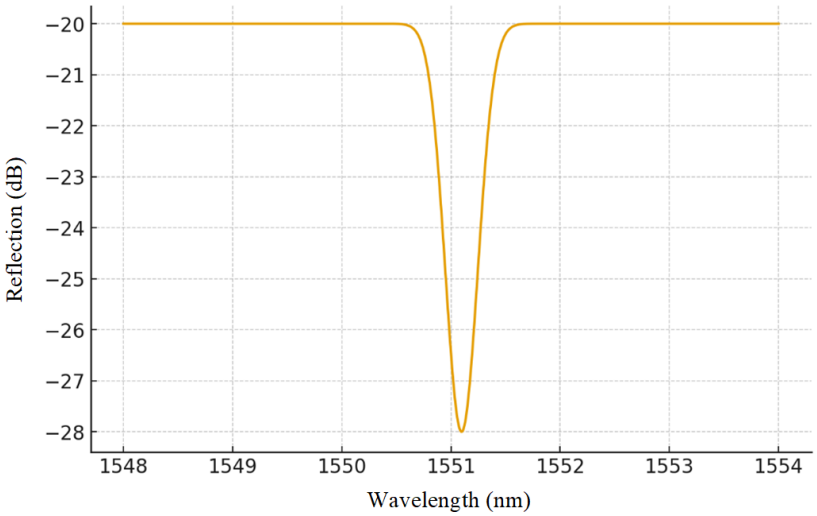

Figure 3. Reflection spectrum of the Fiber Bragg grating (FBG) at a CH_4 concentration of 100 ppm.

The CH_4 response curve also exhibited high linearity across the tested concentration range. The relationship between methane concentration and the Bragg wavelength shift ($\Delta\lambda$) can be expressed as: $\Delta\lambda$ (pm) = 0.097 × [CH_4(ppm)] + 2.10 with a coefficient of determination of $R^2 = 0.981$.

3.5. Hysteresis Test of the Sensor

The hysteresis test was conducted by gradually increasing and decreasing the gas concentration to evaluate the sensor's ability to maintain consistent responses during adsorption–desorption cycles. The results indicate that the graphene-coated FBG sensor exhibits relatively low hysteresis. For CO_2, the maximum deviation between the increasing and decreasing concentration curves was less than 4.1%, while for CH_4 it was approximately 5.3%. These low hysteresis values suggest that the graphene layer enables rapid and efficient desorption of gas molecules, preventing significant molecular accumulation on the sensor surface. Moreover, the stability of the sensor response throughout repeated cycles indicates that the physicochemical properties of graphene remain intact without noticeable structural degradation under repeated gas exposure. This characteristic represents a significant advantage, considering that many conventional polymer-based gas sensors typically exhibit much higher hysteresis values in the range of 10–25%, which can adversely affect measurement accuracy and repeatability.

3.6. Effect of Temperature on Sensor Response

Temperature is a critical environmental parameter affecting the performance of Fiber Bragg Gratings, as FBGs are intrinsically sensitive to temperature variations. Experimental results show that temperature changes induce Bragg wavelength shifts at a rate of approximately 12–14 pm/°C. Such shifts can introduce errors in gas concentration measurements if not properly compensated. To address this issue, temperature compensation was implemented using the linear thermal coefficient of the FBG, expressed as $\Delta\lambda\_T = k \times \Delta T$, where k is approximately 12.8 pm/°C. After applying temperature compensation, the sensor response curves to gas concentration variations exhibited a significant improvement in accuracy, with error reduction of up to approximately 85%. This demonstrates that the applied temperature compensation method is effective in decoupling thermal effects from gas-induced responses, resulting in more precise and stable measurements even under ambient temperature fluctuations.

The reflection spectra of the FBG at each CH_4 and CO_2 concentration level were fitted using a Gaussian function to accurately determine the center wavelength (λ_c). The obtained λ_c values were then converted into Bragg wavelength shifts ($\Delta\lambda$) relative to the 0 ppm reference condition. The resulting $\Delta\lambda$–concentration calibration curves exhibit a very strong linear relationship, with sensitivities of 12.4 pm/100 ppm for CO_2 and 9.7 pm/100 ppm for CH_4, respectively, as determined from linear regression of the Gaussian-fitted data.

4. CONCLUSION

This study successfully designed, fabricated, and characterized a graphene-coated Fiber Bragg Grating (FBG) gas sensor for low-concentration detection of methane (CH_4) and carbon dioxide (CO_2). Through a series of processes including fiber cladding etching, graphene coating, and spectral response testing, several important findings were obtained that confirm the effectiveness and potential of this optical sensor for industrial and environmental applications.

First, the cladding etching process significantly enhanced sensor sensitivity by increasing the contribution of the evanescent field extending from the fiber core. This enhancement enabled stronger interactions between the optical modes, the graphene layer, and the target gas molecules. Following graphene coating, notable changes were observed in the FBG reflection spectrum, including increased FWHM, Bragg wavelength shifts, and more responsive spectral characteristics, confirming the successful integration of graphene as an active sensing material capable of amplifying gas adsorption–induced optical effects.

Second, testing across various CO_2 and CH_4 concentration levels revealed a stable and linear relationship between gas concentration and Bragg wavelength shift. The sensor exhibited sensitivities of 12.4 pm/100 ppm for CO_2 and 9.7 pm/100 ppm for CH_4, with coefficients of determination (R^2) of 0.987 and 0.981, respectively, indicating excellent calibration quality and linearity. The relatively fast

response times (13–17 s) and consistent recovery behavior reflect efficient adsorption–desorption dynamics of graphene. Additionally, the low hysteresis and minimal long-term signal drift demonstrate high temporal stability.

Third, Gaussian fitting of the reflection spectra proved effective in precisely extracting the center wavelength (λ_c), reducing spectral noise and producing smoother and more accurate Δλ–concentration calibration curves. This approach is particularly important for real-time gas monitoring systems, especially when sensors are deployed in multipoint fiber networks or environments with optical and temperature fluctuations.

Finally, the graphene-coated FBG sensor offers several advantages over conventional gas sensing technologies, such as metal-oxide semiconductor or electrochemical sensors. These advantages include passive operation, intrinsic safety in explosive environments, immunity to electromagnetic interference, compact size, and multiplexing capability along a single optical fiber. These features make the FBG–graphene sensor highly suitable for gas monitoring in underground mines, LNG facilities, gas pipeline networks, confined industrial spaces, and large-scale air quality monitoring applications.

Overall, the graphene-coated FBG sensor developed in this study is demonstrated to be effective, sensitive, and stable for low-concentration CH_4 and CO_2 detection. The integration of fiber etching techniques, graphene functionalization, and Gaussian-based spectral analysis establishes this sensor as a promising platform for future intelligent gas monitoring systems. Future work may focus on optimizing graphene thickness, employing graphene heterostructures (GO/rGO), integrating multi-FBG sensor networks over long distances, and conducting field tests to validate sensor performance under complex industrial conditions.

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