

Sensor for Measurement of Hydrocarbons Concentration Based on Optic Fiber

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ABSTRACT

Measuring system based on Photonic Bandgap Fiber (PBGF), which will be able to detect, distinguish and measure the concentration of various aliphatic hydrocarbons is proposed. Such a system can be applied on each stage of fuel evaluation, production, distribution and storage of petroleum products. Aliphatic hydrocarbons absorption spectra show several peaks around 1400-1600 nm wavelength. Thus, tunable lasers with 20 micron core photonic bandgap fiber matching above wavelengths were chosen for the optical measurement system. Measurements were performed at a low pressure to eliminate pressure broadening effect and to sharpen the absorption peaks. The sensitivity of measurement increases with increasing of the fiber's length. Length of fiber was adjusted using several methods. The best results were obtained using argon ion beam cutting. The angled cut was performed to improve signal to noise ratio.

Keywords: Photonic bandgap fiber, Gas sensor, Hydrocarbons measurement, Methane

1. INTRODUCTION

Fast development of geomonitoring systems, biotechnology and nanotechnology branches constantly motivates various research groups to design faster and more sensitive measurement tools and technologies^[1, 2]. Optic fibers are already broadly used as sensors and crucial elements of sensing systems. Fiber probes coated with sensitive layers and employing various physical and chemical phenomena were used for detection of carbon dioxide, dissolved oxygen, hydrogen peroxide, piridyne, red blood cells, bacteria, etc.^[3-9]. Detections of acetylene, hydrogen, methane and biocompounds using new generation of optic fibers: photonic crystal fibers with solid core surrounded by air holes cladding structure were reported^[10-12]. Photonic bandgap fibers of a honeycomb structure with a hollow core surrounded by the highly regular microstructured cladding and light propagating in the core due to photonic bandgap phenomenon were employed for sensing of acetylene and methane^[13-16]. The most of above measurements were performed in uncontrolled flow conditions.

Nowadays, when the petroleum becomes scarce resource the efficient and fast measurement system is required on each stage of fuel production:

- on the stage of exploration and evaluation of quality of oil and gas resources,
- during the oil recovery methods from oil and gas production fields,
- on various steps of petroleum refining,
- during the distribution and storage petroleum products, to maintain their quality.

In all mentioned cases, not only the system's sensitivity but also its endurance, compactness and portability are important features. This paper describes the development of compact set-up for measurement of concentration of aliphatic hydrocarbons in the continuous flow regime. Proposed device allowed to reduce the total size of the set-up (portability) and the necessary minimum volume of gas sample even to 0.01 cc. In the case of fibers' mass production, presented research project will result in low cost, high-sensitivity, real-time measurement system. Huge possibilities of

proposed in-situ measurement method were revealed during preliminary research using tunable laser and glass or metal tanks^[17] and later with the perforated optical fibers^[18] as the gas cells.

PBGFs employed as gas cells are light and durable. Differing from traditional gas cells, they can be rolled to save space and make the sensor compact. Furthermore, considerable lengths (limited only by the gas flow properties, thus required measuring time) can be used to further enhance sensitivity. Sensing aliphatic hydrocarbons at low pressure and in narrowband of IR spectrum using PBGF has not been investigated yet by any research group. The length of hydrocarbon's molecule chain might be evaluated basing on a small shift of single peak during the measurement of vibrational spectrum in the range assigned to the certain, chosen chemical bond.

2. EXPERIMENTAL SET-UP

2.1. Gas dosing sub-system

Methane was diluted with high purity nitrogen to required concentration and such a sample was dosed into the PBGF via the gas-cell-type mechanical splice of special construction. Sample passed throughout the core of optical fiber for proper measurement of light absorption. "Fed-in" and "continuous flow of gas" dosing modes were tested. Splices on the both ends of the PBGF were connected to the rotary pump to acquire required low pressure (0.1-0.01 atm). In the case of continuous flow mode, the slight pressure difference was kept between splices to assure the gas flow. The gas-providing system is presented in Fig. 1.

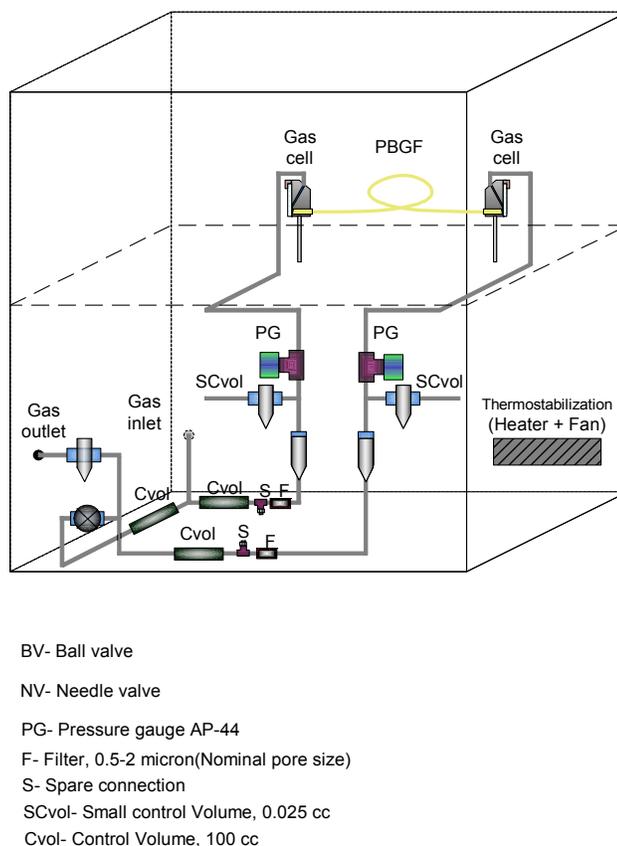


Fig. 1. Gas providing system.

2.2. Optical sub-system

Light emitted by tunable laser (Agilent, 1550 nm center emission wavelength) passed via Single Mode Fiber (SMF), PBGF and then again via SMF reaching the power meter. All fibers were coupled using mentioned gas-cell-type mechanical splices. Splices allowed the gap between fibers ranging from 0 to 20 microns. The schema of experimental set-up is depicted in Fig. 2. Proper measurement in proposed system highly depended of the loss-less light coupling.

Wavelength range of absorbance peaks of the sample matched the laser emission wavelength and the band-gap of used fiber. Measurements were performed in low pressure to eliminate pressure broadening effect and to sharpen the absorption peaks.

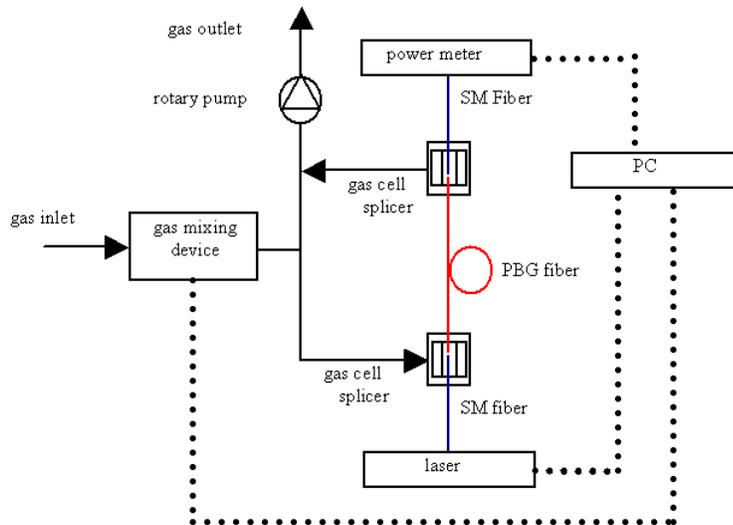


Fig. 2. Schema of experimental set-up.

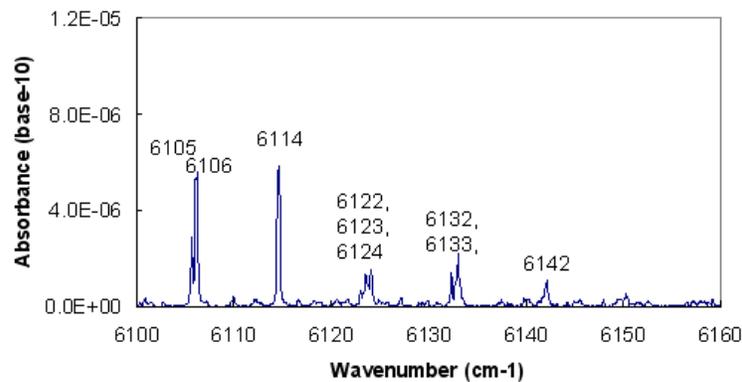


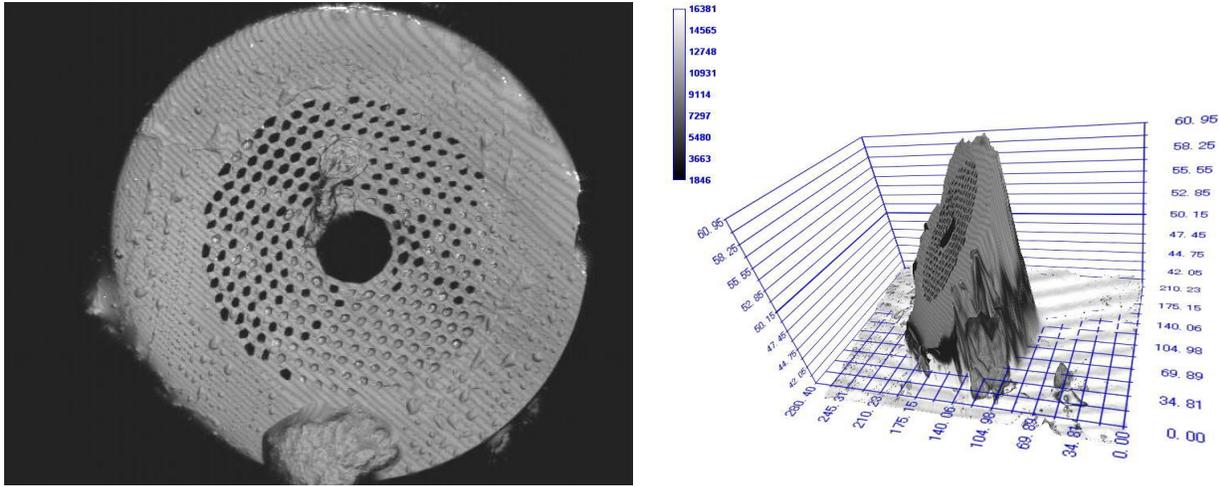
Fig. 3. Methane IR absorption spectrum, PNNL^[20].

2.3. Optic fiber

20 micron core (Blaze Photonics) photonic bandgap fiber was used as a gas cell. Light ($6536\div 6211\text{ cm}^{-1}$) was mostly guided in a hollow, circular core surrounded by a microstructured cladding formed by a periodic arrangement of

air holes in undoped silica. Up to 65 % of the fiber cross-section was composed of solid silica but less than 5 % of light propagated in glass. More than 90% of optical power was located in the hollow core or in the holes of cladding. Holes of core and cladding were filled with the tested gas. Fiber was coated by single acrylate layer. To avoid troublesome aligning both ends of photonic bandgap fiber were spliced to single mode optic fibers using customized splices. Splices were perforated and placed in small shielded gas cells to enable gas flow inside the PBGF's core. The simulations of absorption peaks for the methane gas were performed using HITRAN [19] software and data were compared to Pacific Northwest National Laboratory database [20]. The IR absorption spectrum of methane according to PNNL database is shown in Fig. 3.

A



B

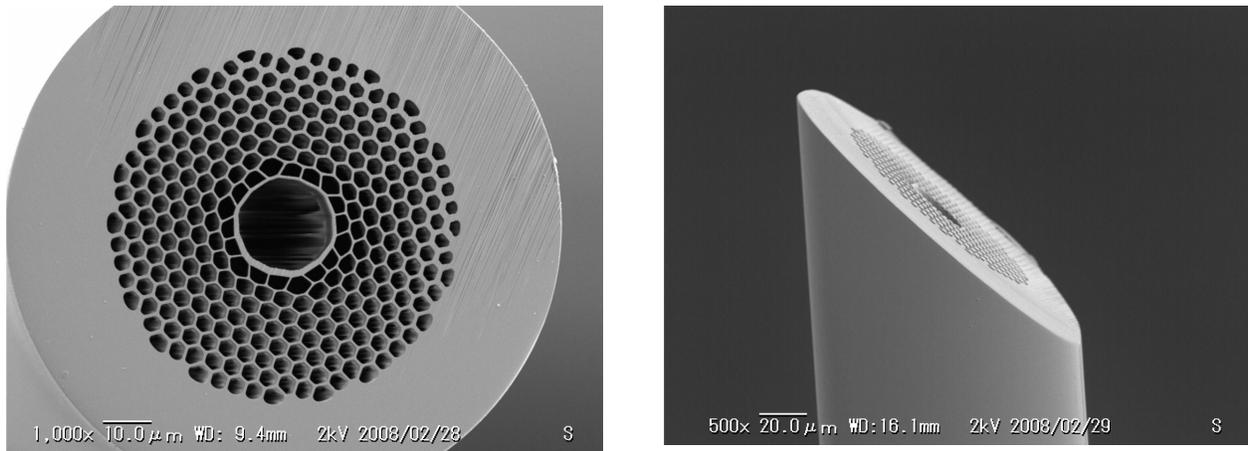


Fig. 4. PBGF cutting using CSP, (laser microscope), (A) and using FIB, (SEM), (B).

Elongating, mechanical and chemical processing of the fiber's tappers and side surfaces in various ways to match sensing goals, simplify the connecting process, introduce sample to the core of the fiber and to minimize transmission losses were broadly investigated [21-23]. In presented experiments to avoid pollution and to properly cut the tip of PBGF length of fiber was adjusted using several methods such as ordinary cutter, cutting the samples frozen in liquid nitrogen, using of fiber cleaver. The best results were obtained using Cross Section Polisher, SM-09010 (Nihon Denshi) using argon ion beam and Focused Ion Beam [24-27]. This procedure assured shaping of the end to required angle

and minimized destruction of surface part of cladding. Traditional fiber cleaver besides lower precision allowed for only one cutting angle. The laser microscope photographs of cutting results using CSP and SEM photos of FIB cut are presented in Fig. 4. Last two methods could be especially advantageous for precise input of the light into the fiber's core and for preventing the surface reflections. In this case, the only disadvantage was relatively long cutting time.

3. RESULTS AND DISCUSSION

Microcapillary gas flow simulations were performed employing the standard mathematical software. All presented results were obtained assuming gas flow within the core and 20°C temperature. Velocity of the gas was calculated from the pressure difference $\Delta p = p_1 - p_2$. The known flow equation of Darcy-Weisbach (1), widely used in sanitary engineering was applied to experimental conditions:

$$\Delta p = \lambda (l / d) (\rho v^2 / 2) \tag{1}$$

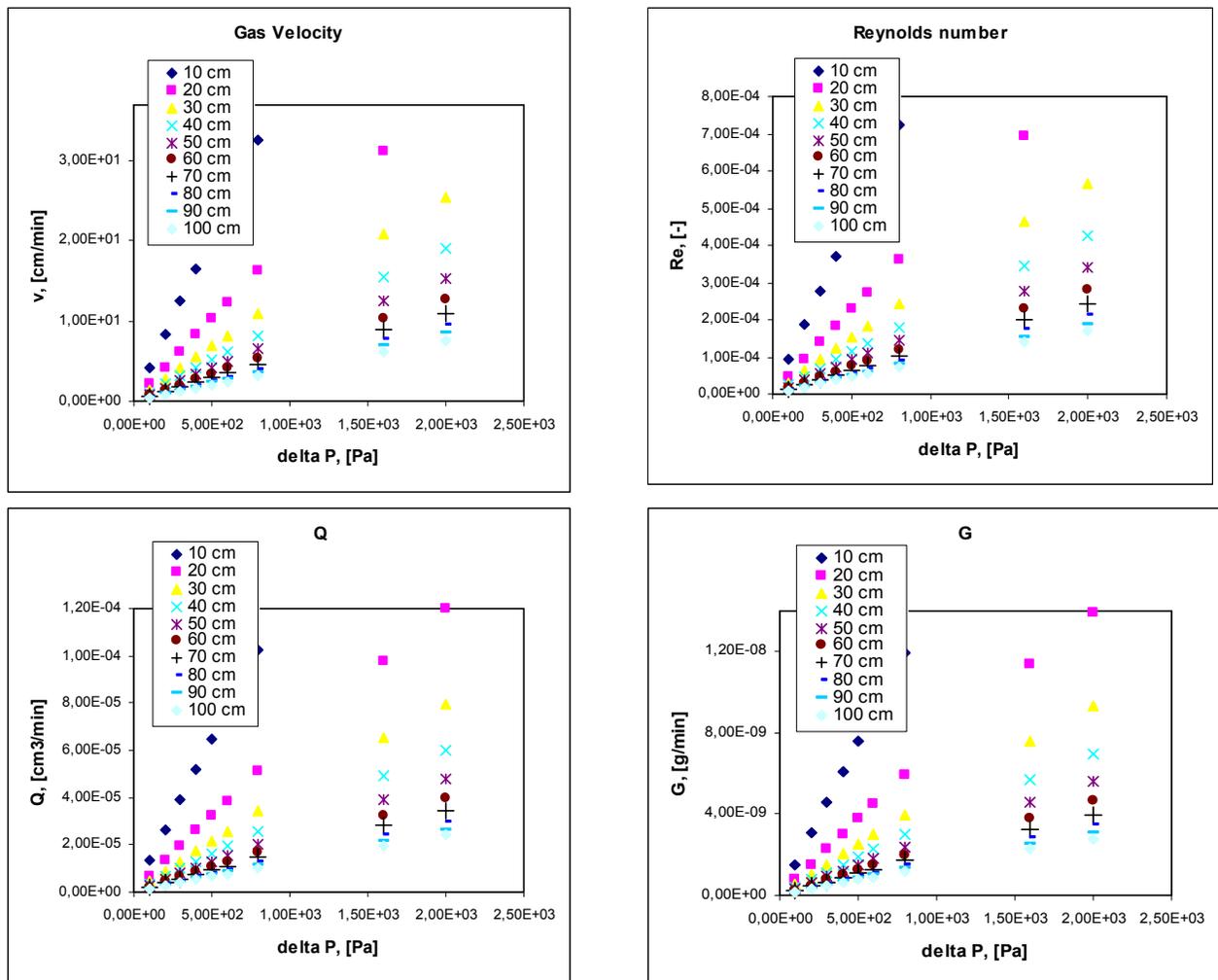


Fig. 5. Simulations of gas velocity, Reynolds number, volume and mass flows for 20 micron core fiber (compressible flow regime).

Δp is pressure difference on both ends of the optical fiber, l - length of the fiber, d - core diameter, ρ - gas density at certain pressure, v - gas velocity, λ - coefficient, which depends on Reynolds number, for laminar flow ($Re < 2300$):

$$\lambda = 64/Re \tag{2}$$

Darcy-Weisbach equation does not take the compressibility into an account and obtained values for gas are different from the real case. Therefore, quasi-Panhandle equation (3), for modeling of the compressible fluid flow was also taken into account.

$$p_1^2 - p_2^2 = G^2 RT/F^2 (\lambda (l/d) + 2 \log_e (v_2/v_1)) \tag{3}$$

The results of modeling process are shown in Fig. 5. Velocity was calculated from the equation (3) for various lengths and pressure differences of 20 micron core PBGF.

Methane absorption measurement results using 1 m PBGF of 20 micron core and tunable laser are shown in Fig. 6. Experiment was performed at 0.1 atm pressure. Obtained spectra are in good accordance with the reference data.

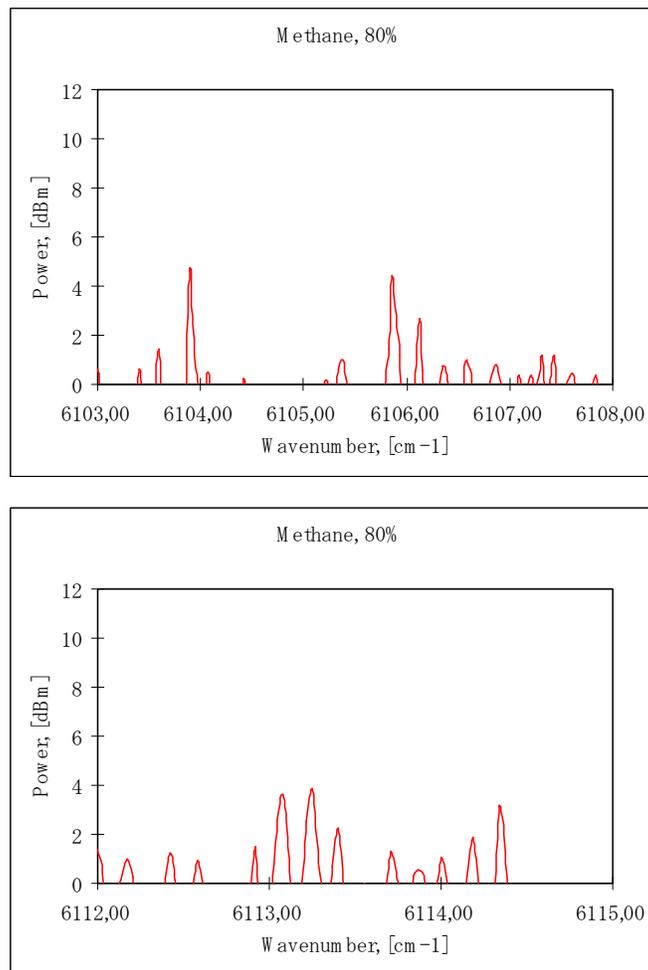


Fig. 6. Measured absorption peaks of methane.

4. CONCLUSIONS

Method of aliphatic hydrocarbons concentration measurement using PBG fiber as a gas cell was proposed.

Several methods of cutting and shaping the ends of the PBG fiber were tested. Cross Section Polisher and Focused Ion Beam were considered as the most precise and perspective.

Simulations of flow rate of gas and of gas velocity in dependence on applied pressure were performed and the absorption peaks of methane were obtained.

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