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A STUDY OF HYGROMETER BASED ON THE METHOD OF INTRACAVITY LASER ABSORPTION

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Abstract: *The article presents the results of experimental studies of a fiber-optic humidity sensor, the principle of operation of which is based on the method of intracavity laser absorption. A method of intracavity laser absorption is described, which makes it possible to dramatically increase the sensitivity of the fiber humidity sensor by switching to it. A schematic diagram of a fiber optical humidity sensor using a differential detection scheme and a design of an active ring laser fiber consisting of a diode pump laser, a fiber pump beam splitter, two directional couplers, a measuring ring laser, a ring comparison laser, two pump radiation filters and germanium photodetectors are developed. Graphical results of experimental studies of a fiber-optic sensor using a differential registration scheme are demonstrated. Measurements are shown in the range of 1800-1940 nm with a center of ~ 1872 nm, where the absorption of water vapor located in the cuvette is recorded, and from 1940 to 2090 nm - carbon dioxide located in the monochromator and having three absorption bands in this spectral region, according to which the frequency scale of the spectrophotometric hygrometer is calibrated. Comparison of the experimental data with the calculated values showed their satisfactory agreement.*

Key words: *resonator, sensor, condensate, optical fiber, laser, diode, photodetector, spectrum.*

Аннотация: *Оптик толали намлик сенсорининг тажрибавий тадқиқот натижалари келтирилган, унинг ишлаш тамойили ички резонаторли лазерли ютилиш усулига асосланган. Ички резонаторли лазерни ютиш усули тавсифланган бўлиб, унга ўтиш орқали толали намлик сенсорининг сезгирлигини тубдан оширишга имкон беради. Дифференциал қайд этиш схемасидан фойдаланган ҳолда оптик толали намлик сенсорининг принципиал схемаси ва қўзғатувчи диод лазеридан, қўзғатувчи толали нурни ажратувчи, иккита йўналтиргичдан, ҳалқали ўлчаш лазеридан, таққослаш риштасидан иборат ҳалқа лазери, иккита қўзғатувчи нурланиш фильтрлари ва германий фотодетекторларидан иборат фаол толали конструкцияси ишлаб чиқилган. Дифференциал қайд этиш схемасидан фойдаланган ҳолда оптик толали сенсорни тажрибавий тадқиқ этишнинг график натижалари кўрсатилган. Ўлчашлар 1800-1940 нм оралиғида марказда ~ 1872 нм, бу ерда кюветадаги сув буғининг ютилиши қайд этилган ва 1940 дан 2090 нм гача бўлган карбонат ангидрид монохроматорда ва учта ютилиш йўлига эга спектрал соҳада кўрсатилган, бу орқали спектрофотометрик гигрометрнинг частота шкаласини калибрлаш олиб борилган. Тажриба маълумотларини ҳисобланган қийматлар билан таққосланиши уларнинг мос келиши қониқарли эканлигини кўрсатди.*

Таянч сўзлар: *резонатор, датчик, конденсат, оптик тола, лазер, диод, фотодетектор, спектр.*

Аннотация: *Представлены результаты экспериментальных исследований волоконно-оптического датчика влажности, принцип действия которого основан на методе внутрирезонаторного лазерного поглощения. Описан метод внутрирезонаторного лазерного поглощения, путем перехода на который дает возможность кардинально повысить чувствительность волоконного датчика влажности. Разработаны принципиальная схема волоконного оптического датчика влажности с использованием дифференциальной схемы регистрации и конструкция активного волокна кольцевого лазера, состоящего из диодного лазера накачки, волоконного светоделителя накачки, двух направленных ответвителей, измерительного кольцевого лазера, кольцевого лазера сравнения, двух фильтров излучения накачки и германиевых фотодетекторов. Продемонстрированы графические результаты экспериментальных исследований волоконно-оптического датчика с использованием* *дифференциальной схемы регистрации. Проведены измерения в диапазоне 1800–1940 нм с центром ~ 1872 нм, где регистрируется поглощение паров воды, находящихся в кювете, а с 1940 до 2090 нм - углекислого газа, находящегося в монохроматоре и имеющего в этой спектральной области три полосы поглощения, по которым производится калибровка частотной шкалы спектрофотометрического гигрометра. Сравнение экспериментальных данных с расчётными значениями показало их удовлетворительное совпадение.*

Ключевые слова: *резонатор, датчик, конденсат, оптическое волокно, лазер, диод, фотодетектор, спектр.*

Introduction

At present, the manufacture of optical fibers is carried out mainly by chemical methods. The main disadvantage of chemical vapor-phase methods for the manufacture of optical fibers is the high content of hydroxyl groups – OH. The presence of OH groups in the fiber layer of a silica fiber leads to an increase in optical losses, in particular, in the wavelength range of 1.35-1.6 μm, there is resonant absorption of radiation by OH hydroxyl groups. To get rid of them, annealing and reflow of the porous workpiece is carried out in a diffusion furnace in the presence of chlorine for 24 hours. To reduce optical losses in optical fibers, a high degree of purification of the starting materials from contaminants is required. Therefore, it is necessary to control oxygen and hydrogen-containing impurities during the formation of the fiber layer [1].

Infrared absorption is photonic absorption caused by the resonance of interatomic bonds under the action of radiation with a given wavelength. The main maximum of the absorption band for SiO_2 is observed at a wavelength of 9.1 μm with an "exponential tail" and harmonics at a wavelength of 1.7 μm. This significantly limits the long-wavelength region of the quartz glass transmission window to 1.6 μm. The introduction of alloying additives to change the refractive index shifts the absorption edge, in particular, the B_2O_3 additive leads to an increase in losses, starting with a wave of 1.1 μm, while GeO_2 and F additives do not affect the increase in losses in the long-wavelength region [2].

It is much more difficult to remove impurities of OH groups, since the hydrogen ion has a very high affinity for oxygen. The main peak of the resonance absorption band of the OH group is observed at a wavelength of 2.73 μm, the first harmonic at 1.38 μm, and the combination band at 1.24 μm. The last two effects strongly affect losses in the operating wavelength range of 1.25 - 1.55 μm. With the content of OH groups 10^{-6} of the mass, the losses at a wavelength of 1.38 μm reach 50 dB / km, and by 1.24 μm they decrease to 2.5 dB / km. The ratio of the magnitude of these two maxima is influenced by the presence of a P_2O_2 additive. The resonance of the P-OH bond is observed at 1.6 μm. Thus, high phosphorus concentrations have a strong effect on losses at 1.55 μm (compared to 1.3 μm). The maximum for OH groups observed at a wavelength of 1.38 μm mainly affects the losses in the operating range of semiconductor LEDs and 1.3 μm lasers [3, 4].

In the case of using an optical fiber as a sensor for adsorbed OH groups, an erbium fiber laser with a generation wavelength of about 1.55 μm can be used as a radiation source. The relatively small absorption of OH groups at this wavelength can be increased by adding phosphorus to the sensor fiber.

In the spectrophotometric method for determining the moisture content of gas, in contrast to the condensation method, even when hydrate formation inhibitors are added to natural gas [3], a value proportional to the total content of water vapor in the volume is measured and calculated from the absorption spectra. In the case when there is water in the gas in the vapor and droplet phases, it becomes necessary to use other physical methods for measuring the concentration of water.

Research Methods and the Received Results

With the development of computer technology and intensive computerization of scientific and industrial equipment, a situation arises when computer automation of spectrophotometers makes it possible to fundamentally improve their characteristics. For quantitative gas analysis, the most attention is attracted by the spectral regions in which the registered gases have maximum or significant absorption coefficients, and these, as a rule, are the ranges in which the spectral information is collected in databases and is currently being refined and expanded. The intensities of the strongest molecular lines and bands are described with high accuracy, which is quite sufficient for solving a wide range of applied problems. In this regard, there has always been a great interest in the use of this information for calibration and directly in the operation of spectral instruments for various purposes [4]. The spectral bases HITRAN [5], GEISA and HITEMP already contain several million lines and describe the IR spectrum range quite fully and accurately. They include fairly reliable experimental and calculated information on the magnitude of the absorption, broadening, and shift coefficients for molecules. The list of these molecules includes a significant portion of gases, including methane and water.

The instrument data bank of samples of experimental and model spectra of water and methane vapors was formed for different pressures and ratios of their content in the mixture. If it is known that the test substance is a combination of several known and entered in the database of calibration mixtures, and its spectrum is close to a linear combination of the initial spectra, then the characteristics of the substance can be calculated through the concentrations of the initial components. In the proposed algorithm, the concentrations of the basic components are calculated from a system of equations including the absorption coefficients of the spectra at S points located in the selected spectral range:

$$\sum_{j=1}^{N}(k_j \times T_{ij}) = I(v_i),\ i = \overline{1,S}, \tag{1}$$

where T_{ij} is an element of the matrix $\{T_{ij}\}$ ($i = \overline{1,S}$; $j = \overline{1,N}$, $S > N$, $S = 100$ was used, and N was from 2 to several tens) of the absorption coefficients at point i for the base spectrum; k_j - the required concentration; $I\ (v_i)$ - absorption coefficients at point v_i for the spectrum of the investigated substance; N is the number of experimental and model spectra in the instrument data bank.

To obtain physically meaningful solutions of the system containing only positive values of k_j, for the first time to solve such a spectroscopic problem, the regularization method [6] is used in the form of additional equations-inequalities

$$k_j \geq 0,\ j = \overline{1,N}. \tag{2}$$

And the regularizing term is taken in the form

$$J(\bar{k}) = \sum_{i=1}^{S}\left\{I\left(v_i\right) - \sum_{j=1}^{N} I_{ij}\left(v_i\right) \times k_j\right\}^2 + \sum_{j=1}^{N}\left\{A \times \xi\left(k_j\right)^2\right\}, \tag{3}$$

where $I(v_i)$ absorption coefficients of the analyzed spectrum at informative frequencies v_i; $\tilde{I}_{ij}\ (v_i)$ are the absorption coefficients at frequencies v_i for the experimental or calculated spectrum numbered j with a known value of the parameter or characteristics of the substance (concentration, percentages, temperature, etc.). The function $\xi\ (k_j)$ is responsible for limiting in the negative range of k_j values:

$$\xi(k_j) = \begin{cases} 0,\ \text{if } k_j \geq 0, \\ k_j,\ \text{if } k_j \leq 0. \end{cases} \tag{4}$$

The regularization coefficient A is chosen so that the values of k_j that are negative do not exceed the modulus of the required concentration error. For $A = 0$, the solution to this problem will coincide with the solution by the least squares method.

In the future, the sought value of the characteristic f^* for the analyzed sample is determined by substituting the found concentration coefficients k_j of the control gas mixtures and their experimentally observed characteristics $\tilde{f}_j$ of spectra numbered j:

$$f^* = \sum_{j=0}^{N} k_j \times \tilde{f}_j. \tag{5}$$

In the above method of positive concentrations, the choice of the tuning parameter of regularization A depends on the specific problem, the optical parameters of the spectrum analyzer, and other features that arise when finding the characteristics of interest for the substance under study. In the future, it is possible to search for functional dependencies using data banks, taking into account the parameters that combine experimental and model spectra [6].

The absorption index decreases with decreasing wavelength [7].

The optimal wavelength for practical use is 1.94 µm. Since the absorption of water in this range has the property of induced polarization, characteristic of atomic vibrations, this phenomenon is considered atomic polarization. Water hitting a substance changes its spectrum. If the controlled object is irradiated with infrared radiation at this wavelength and the power of the transmitted or reflected radiation flux is measured, then it changes depending on the humidity.

The wavelengths of the measured and basic streams can be optimally selected taking into account the spectrum of the measured substance, the measuring range of humidity and other requirements.

When developing a rational design of a highly sensitive fiber sensor, one should take into account the need to introduce an absorbing medium into the laser cavity. In this case, the use of any additional elements in the form of absorbing cells leads to additional reflections in the resonator and, ultimately, to a decrease in the potential sensitivity.

The ideal solution is to completely eliminate external elements and use the active gain medium of a fiber laser as a sensor directly.

In conventional designs of fiber lasers, the central core of the fiber is covered with a SiO_2 shell, which does not sufficiently transmit water vapor; therefore, the response time of the fiber absorption sensor is very low (response time is hours and days), and the sensitivity is unsatisfactory [8] – [13].

The use of ion-beam or plasma doping methods makes it possible to easily create an erbium-doped central core of a fiber laser without an outer SiO_2 shell, which opens up wide possibilities for its use as a highly sensitive and fast-acting sensor of water adsorbed on the fiber surface [14].

Water adsorbed on the outer surface of the fiber causes strong absorption, despite the fact that the main beam of laser radiation propagates in the bulk of the fiber. This is due to the fact that in a single-mode fiber, the maximum of the energy density of optical vibrations is much more displaced towards the fiber surface than in multimode fibers. With a certain diameter of the central vein, the maximum density can be found even outside the central vein, in the outer shell. By using a sufficiently thin SiO_2 shell, it is even possible to combine the maximum density with the shell surface.

However, this option is very difficult for practical implementation of a laser and has no advantages in the long-term stability of the sensor. This is because the entire length of the fiber laser is sensitive to water vapor.

With this design, the fiber laser can be made as a ring laser based on a single-mode fiber, which can significantly simplify and thin out the longitudinal mode structure of the fiber laser. This will make it possible to abandon the unstable process of competition between modes in the case of intraresonant absorption of the ILA and to carry out predominantly "narrowband" ILA. Although this will result in a loss in sensitivity and spectral selectivity, the stability of the sensor will increase dramatically, and the radiation registration system is greatly simplified. The need for narrow-band spectral selection using a Fabry-Perot interferometer is completely eliminated, and a simple differential amplitude registration becomes possible.

The sensor design based on these principles is shown in Fig. 1. The sensor has two differentially switched fiber sensing elements with ILA, which are ring fiber lasers with a ring length of no more than 0.5 m, with a common pumping system from a laser diode using a highly stable fiber beam splitter of pumping power. The unified pumping system makes it possible to significantly reduce the pumping-induced instability of the output power of ring lasers and to further increase the stability. The use of two identical sensitive elements allows not only to increase the stability of operation, but also, if necessary, to make relative or compensation measurements of humidity, which expands the functionality of the device.

Characteristics of optical fiber laser absorption sensor:

1. Gas pressure range, MPa 2.5-7.5;

2. Type of flow hygrometer;
3. Measured water vapor concentration, mg/ m^3 30-40 000;
4. Calculated from the concentration of the dew point temperature of water in natural gas, °C -35...+15;
5. Absolute error in determining the dew point, °C 1;
6. Operating temperature of the medium, °C -20...+35;
7. The time of one measurement cycle is not more than, min 8;
8. The time of entering the mode is not more than, min 15.

The developed sensor for monitoring water vapor with a differential signal recording circuit has the following parameters: the operating wavelength of an annular laser on an erbium fiber λ=1.55 μm; wavelength and pumping power, respectively λ=980 nm and P_{pump} =100 mW; water steam sensitivity is 10^{-5}-10^{-6} %.

When converting the absolute water vapor content in natural gas to the water dew point temperature, the values of the natural gas moisture dew point temperature, reduced to a pressure of 3.92 MPa and the water vapor concentration, were used. The range of measured concentrations of water content at the dew point was from -40 to +8°C. At the same time, the measurement error was less than 1°C, and the reproducibility was not worse than 0.25°C.

Under the action of water vapor in the working cell, the emission power of the annular laser decreases, which leads to the appearance of a difference signal from two germanium photosensors. To extend the dynamic range and increase the sensitivity of the photodetectors, filtering of pumping radiation is required, which can be carried out either with a light filter or with a specially designed fiber absorber. Both lasers and photosensors are placed in a passive thermostat to equalize their temperatures. Temperature equalization is necessary to prevent a temperature shift in the frequency of generation of the fiber laser, which can change the absorption coefficient of the fiber.

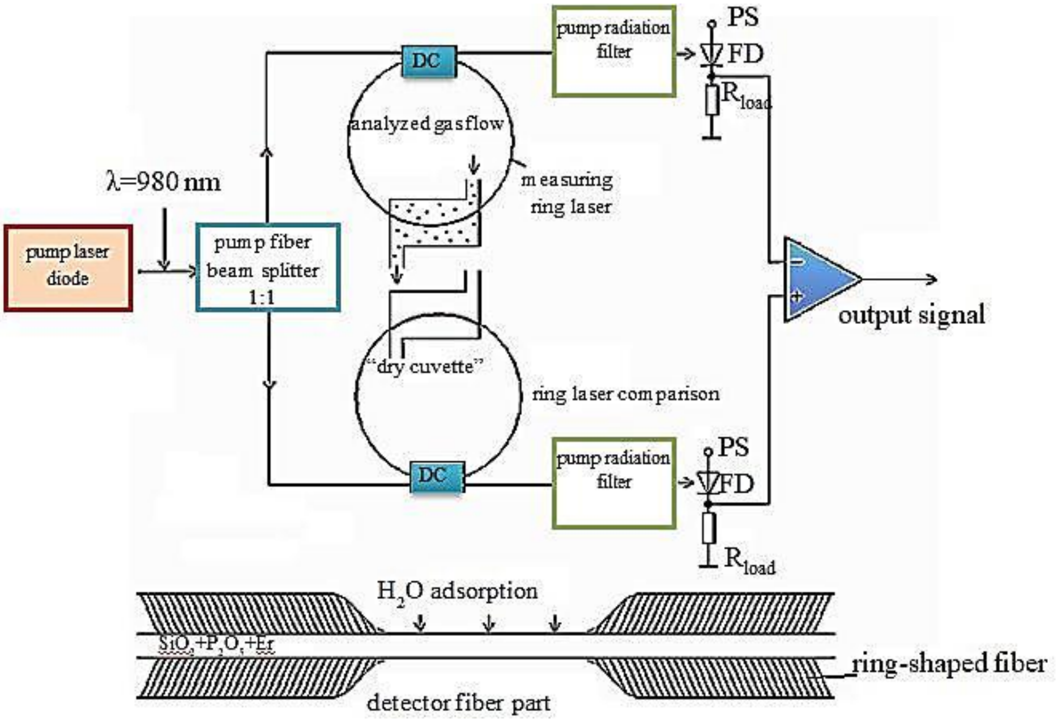

Fig. 1. ***Block diagram of a fiber optic sensor using a differential registration circuit and design of an active fiber of a ring laser***

The 4th curve in Fig. 2 is the combined absorption spectrum of water vapor (concentration at a pressure of 10 atm in an air cell of ~ 5500 mg/m^3) and carbon dioxide (in a monochromator at a pressure of 1 atm). After self-adjustment, filtration and logarithmization [6], this spectrum takes the form of a 2-curve line, which allows the gas humidity sensor to operate with high accuracy in an automatic continuous mode.

In the range of 1800-1940 nm with a center of ~ 1940 nm, it was noted that the water vapor in the cell and the absorption of carbon dioxide from 1940 to 2090 nm in the monochromator, as well as three absorption poles in the same region of the spectrum, on their basis, the frequency scale of the spectrophotometric hygrometer.

To quantify the content of water vapor in a gas, it is necessary to solve the problem of determining the concentration for multicomponent mixtures with overlapping a large range of measured absorption coefficients. This problem was solved by spectroscopic methods of analysis and mathematical algorithms for automatic search in the spectra of informative points [6] in combination with a model of positive concentrations using regularization.

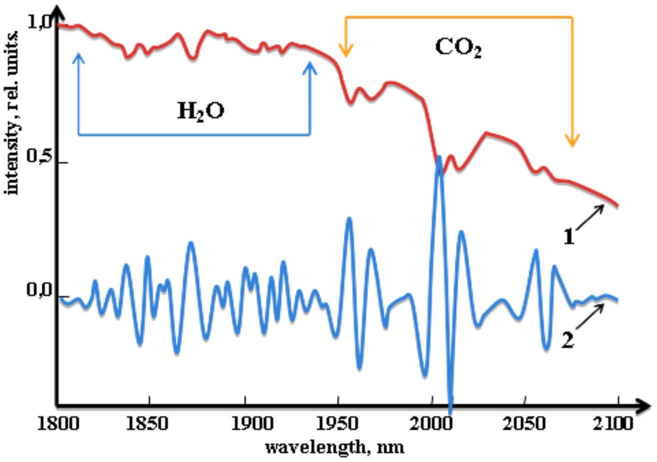

Fig. 2. ***Combined absorption spectrum of water vapor at a pressure of 10 atm in an air cell at a concentration of ~ 5500 mg / m^3 and carbon dioxide at a pressure of 1 atm in a monochromator.***
1-recorded spectrum; 2-the same spectrum after self-regulation, filtering and logarithm.

The original software allows statistical collection, determination of water vapor concentration from experimental and model spectra recorded in the data bank. Information processing is carried out using computer programs based on original algorithms and methods of the theory of appearance detection [6, 15].

Fig. 3 shows a graph of the change in water vapor concentration during one of the periods of experimental results. The range of water concentration measurement was from ~ 100 mg/m^3 to ~ 11000 mg/m^3 when checking the results of the experiment in various modes. By converting the amount of absolute water vapor in the natural gas to the dew point temperature, the moisture content of the natural gas was reduced to a pressure of 3.92 MPa and a water vapor concentration using the dew point temperature. The range of the measured water concentration at the dew point is from –10 to + 40 °C. At the same time, the measurement error was less than 1 °C, and the reflection coefficient was not less than 0.25 °C.

Using the mathematical package Curve Fitting Toolbox for the MATLAB program, we will determine the type of functional dependence, its coefficients, and calculate the criteria for the suitability of the functional dependence based on experimental data:

$$f(x) = a_0 + \sum_{i=1}^{5}\left[a_i \sin(wx) + b_i \cos(wx)\right] \tag{6}$$

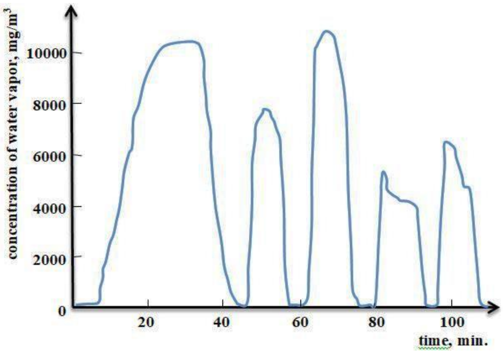

Fig. 3. ***Graph of change of water vapour concentration measured by differential signal recording sensor.***

In Table 1, this dependence is compared with the fourth-degree polynomial, which is often used in practice as a calibration function of humidity sensors.

Table 1.

The values of the fitness criteria of the two calibration functions

f(x)	R-square	Adjusted R-square	SSE	RMSE
$12.15-5.47\cos(0.051x)-51.97\sin(0.051x)-$ $-5.648\cos(2\cdot0.051x)-18.83\sin(2\cdot0.051x)-$ $-4.404\cos(3\cdot0.051x)-11.34\sin(3\cdot0.051x)-$ $-4.044\cos(4\cdot0.051x)-5.367\sin(4\cdot0.051x)-$ $-3.561\cos(5\cdot0.051x)-2.555\sin(5\cdot0.051x)$	-0.998	0.997	76.79	2.263
$6.621\cdot10^{-8}x^4-0.0005x^3+1.431x^2-1726x+7.637\cdot10^5$	0.995	0.993	188.584	2.928

Conclusions

The spectroscopic method allows determining the concentration of water vapor in natural gas in the region of 1872 nm from absorption spectra in a continuous mode. High sensitivity and selectivity to gas components allowed covering a wide range of water concentrations (2.5-40,000 mg/m^3) measured at a pressure of 30-40,000 MPa.

Possible areas of application of the spectrophotometric hygrometer are gas, oil and chemical industries and other sectors of the national economy, where it is necessary to control the concentration of water vapor in technological processes. The spectrophotometer can be of considerable interest in carrying out scientific research in order to study the absorption spectra of gases at high pressures, as well as as an aid for the experimental refinement of formulas and tables for recalculating moisture indicators for various pressures.

Thus, the combination of the capabilities of spectroscopy and mathematical methods of data processing makes it possible to create automated devices that can significantly increase the efficiency of spectroscopic methods and greatly facilitate the complex work associated with analyzing spectra when monitoring the parameters of natural gas, both during its production and during transportation and processing.

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