ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 3, Issue 8 , August 2016

The fiber-optical sensor for chromatographic measurements

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ABSTRACT: The authors present the detector for chromatographic measurements having two sensitive elements, i.e. fiber ring lasers. Influence of water vapor results in the fall of capacity of radiation of one laser. Therefore, differential signal is generated out of two photo detectors**.**

KEYWORDS: optical cable; fibere-optical line; fiber optic sensor; photo detector; photo sensor.

I. INTRODUCTION

Nowadays, there is a growing need for sensors due to the rapid development of the process control and regulation systems, implementation of novel engineering procedures, and transition to flexible automated production processes. In addition to high metrological characteristics, sensors shall be characterized by high reliability, durability, stability, small dimensions, weight and energy consumption, compatibility with microelectronic data processing devices. At the same time, they must be characterized by ease of manufacturing and relatively low cost. Fiber-optical sensors meet the above requirements to the large extent.

In fiber-optical sensors optical fiber can be implemented either as a transmission line or as the most sensitive element of the probe [sensor]. In this case, such parameter of fiber as sensitivity to electric field, magnetic field, vibration, temperature, pressure, deformations (for example, bending) can be used for practical purposes. Plethora of these effects in optical communication systems are regarded as shortcomings, whereas in sensors these effects are considered rather an advantage which could be availed of.

Currently, production of optical fibers is generally performed by chemical methods. The basic shortcoming of chemical vapor-phase method of production of optical fibers is the high content of hydroxyl groups - OH. Presence of OH groups in a light-conducting layer of quartz fiber leads to the increase in optical losses, in particular, in the range of wavelengths of 1,35÷1,6 microns there is resonant absorption of radiation by hydroxyl groups.

II. STATEMENT OF THE PROBLEM

In case of implementation of optical fiber as a sensor of the adsorbed OH groups, it is possible to use fiber erbium laser as the radiation source generating wavelength close 1,55 micron. Rather small absorption of OH groups across this wavelength can be increased, using phosphorus additives in the sensor fiber.

We investigate the possibility of huge increase in sensitivity of the fiber sensor by transition to the technique of intra-resonator laser absorption. Emergence and development of laser equipment has led to creation of new version of the absorbing analysis –intra-resonator laser absorption (ILA). The preliminary studies explain occurrence of specific range of generation in optical quantum generators by the availability of selective absorption by a medium in the resonator. These studies date back to the early 70th [1]. Reverse peaks in the range of generation of the laser allows identifying them with certain nature of absorbing atoms or molecules, i.e. to carry out high-quality analysis of the gas resonator

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placed inside. Measurement of size and a shape of reverse peaksallowseventually to determine quantity of the absorbing particles. The room in the laser resonator filled with gas, leads to occurrence of lines, the absorption area of which falls under the generation diapason, which changes the nature of the spectrum or integrated intensity of radiation of the laser.

Nowadays, the most essential limiting factor in the introduction of analytical practice of this method is the complexity of processing of an analytical signal when receiving result of definition.

Accuracy of registration of location and shape of reverse peak in the generation range in many respects depends on the character of a spectral contour of radiation of the laser. In this regard, special attention has to be paid to elimination of parasitic selection of the performance of the resonator leading to the emergence of liner structure in the range of generation of the laser. The main reason for emergence of it is the return to the channel of generation of the part of the radiation disseminated or reflected by various elements of laser system: end faces of active cores, windows ditch, surfaces of prisms, plates, resonator mirrors. This phenomenon on canbe partly resolved by applying direct methods as well as by means of various reduction techniques of their influence.

Direct weakening of reflection and dispersion is carried out, first of all, by installing whenever possible of all intra-resonator surfaces at Brewster angle towards the direction of the generated radiation, by thickening of the used plane-parallel plates (windows of a ditch, etc.), use of wedge-shaped substrates for resonator mirrors, as well as by improving the quality of production and processing of all details of the laser system. Reduction of influence of the disseminated and reflected radiation is reached, first, due to increase in the length of the resonator of the laser; secondly, by the shift of structure of a range of radiation during impulse generation [2] .One more important parameter is the time of continuous generation. As it was specified earlier, occurrence of reverse peaks in the range of generation happens during the time of stable generation in the vicinity of the line of absorption of the investigated gas. As a rule, this time is much less than the time of complete generation and at best reaches 2-3 ms. As shown in work [3], determination of coefficients of absorption at the level of 10^{-9} -$10^{-10}$$cm^{-1}$requires achievement of time of continuous generation not worse than 1-10 ms.

In case of selection of the laser for the solution certain analytical task, optimal are conditions when the area of generation of the laser matches the strongest lines of absorption of the investigated gas. Currently, the most widely used laser types in spectrometers are used ruby lasers (λ = 694 nanometers); neodymium (λ = 1,06 microns); LK-type laser (λ= 600 nanometers); helium - neon (λ = 3,39 microns) lasers.

It is possible to increase sensitivity of a method by changing laser oscillation frequency, both in pulse, and from pulse to pulse. One of the ways of extending the dynamic diapason of the defined concentration of gases is the change of time of generation and duty factor of the laser resonator. Different versions of diagrams of resonators and elements of laser spectrometers can be found, for example, in the research work [3].

For decomposition of radiation of the laser across the range, a spectrograph is usually used or, in need of achieving of good performance (to one thousands of cm^{-1}), Fabri-Perot's interferometers. Distribution of intensity in a range of radiation of the laser is registered by means of photographic or multichannel photo-electric receivers. The system of transformation and registration of radiation of the laser in many respects determines sensitivity, spectral permission, and also a possibility of use of instant or integrated methods of measurement and calculation of coefficients of absorption.

In order to achieve the maximum sensitivity of a spectrometer in case of the choice of a certain spectral device it is necessary to minimize influence of hardware distortions in the spectrum of laser radiation. The influence of hardware on a reverse peak shape in the range of generation of the laser can be neglected if width of lines of absorption exceeds 10-30 times the width of hardware function of a spectral device.

The main requirements to photo-detectors are, firstly, uniform spectral sensitivity in the operating area of wavelengths of generation; secondly, big dynamic range on intensity of the registered radiation; thirdly, good spatial resolution.

III.THE CONCEPT OF THE PROBLEM DECISION

In quantitative chromatographic measurements the use of equal sensitive sensorsplays an important role. Equal sensitive sensors, as a rule, arebuilt on the principle of detecting of gas carrier [4].

At an exit from a column of the analyzed substance concentration of gas carrier decreases, as forms an output signal of the sensor.

At such creation of the chromatograph absolute calibration of the device on different substances isn't required; it is enough to execute her once with any standard.

Wide use of such sensors is interfered by the fact that some physical or chemical parameter of gas carrier has to differ very significantly from all other substances, and owing to this fact the choice of gases carriers is strongly limited. On heat conductivity and molecular weight hydrogen and helium, on electronegativity – inert gases, on radioactivity – radioactive isotopes strongly differ. Use of these gases carriers for ordinary measurements is irrational because of the high cost of pure gases or danger of application.

We suggest to use as a gas carrier, clear water vapors which can be received easily, for example, from some crystalline hydrates of salts heating up or pure evaporation. The further task consists in development of the selective sensor of vapors of water with the minimum working volume and high high-speed performance. The optical fiber sensor developed earlier, possessing high sensitivity, rather slow, also has large working volume. During creation of optical fiber sensors the total failure from the external absorbing elements (basins) and use as the sensor directly of gain medium of the fiber laser would be the ideal decision.

However, for creation of the high-speed detector this approachisnot applicable.

For preserving high sensitivity it is necessary to keep a intra-resonator method of measurement of absorption, and for reduction of working amount - to change a form of the absorbing cell and whenever possible to increase intensity of light of the fiber laser. The design of the absorbing cell is shown in figure 1.

The absorbing cell has a substrate from chemically resistant ceramics with small TKLR (polikor) with the flute about 14 microns wide executed by a scrapping a diamond cutter. On the center of a substrate the opening with a diameter of 0,8 mm through which there passes the flow from a column is drilled. Two single-mode ends of fiber of the fiber ring laser keep within a flute; the gap between end faces of fibers is regulated with high precision in case of production. Fiber of the laser was made by method of an ion-beam alloying of multimode Tm fiber. Now production of procurements of optical fibers is performed by chemical vapor-phase methods, and receipt of fibers - a pulling. A lack of similar methods is the high content of hydroxyl groups in the received fibers that reduces potential sensitivity of the sensor.

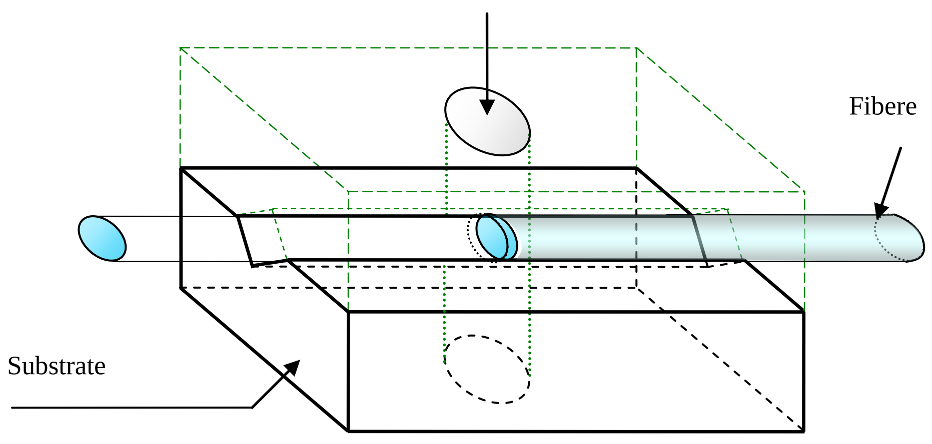

Fig. 1 Design of the absorbing detector cell

Creation of an intensifying layer in light guides by methods of ion-plasma technology (by an alloying their rare-earth metals REM impurity) allows to reduce concentration of hydroxyl groups. The basis of technology is made by implantation of impurity in fiber ionic bombing by the directed bunches or ion-plasma processing in the gas category. An ion-beam alloying of Tm fiber it was carried out at the energy levels of 50-150 keV with a possibility of electromechanical scanning of an ionic bunch [5].

After implantation fiber became covered by SiO_2 layer method of high-frequency spraying. The received procurement baked at a temperature over 1100 degrees in the electric furnace by a continuous method and at once became covered in vacuum by a protective layer of Al.

Single-mode sections of fiber were produced by an extracting of multimode fiber in the continuous way in a torch flame. The extracting accounted 500 – 600%.

The fine-tuned gap between end faces of fibers forms the resonator of Fabri-Perot by means of which setup of operating frequency of the fiber ring laser is made. Besides, in a gap of the resonator field amplitude increases that

allows to increase sensitivity. The substrate with the set fibers is closed by a ceramic cover with a similar hole. Connection of parts can be realized by glues or fusible glass (glass quick solder). In a prototype, the soldering was made by AgCl melting.
The right choice of operating wavelength is necessary for receipt of the maximum selectivity of the sensor to water vapors. Vapors of water have in visible and near infrared area several strong lines of absorption 0,9 micron, 1,14, wide lines around 1,44 and 1,85 and a strong strip of absorption in the range 2,55 - 2,8micron.
The absorption diapason in the field of 2,7 microns the strongest also provides the highest sensitivity, however in this range absorption of quartz fiber largely interferes with receipt of laser generation. The range of a wavelengths of 1,8-1,9 microns is more convenient as it corresponds to the range of generation of ions of Tm.

IV. IMPLEMENTATION OF THE CONCEPT

When developing a rational design of the highly sensitive fiber sensor it is necessary to consider the need of introducing of the absorbing environment in the laser resonator. At the same time, the use of any additional elements in the form of the absorbing cells leads to additional reflections in the resonator and, finally – to decrease of potential sensitivity.
The ideal decision is full refusal of external elements and use as the sensor of directly active strengthening environment of the fiber laser. In this regard we have developed the fiber-optical sensor of control of water with use of the differential scheme of registration of a signal.
In regular designs of fiber lasers the central duct of fiber is covered with a layer of SiO_2 which does not pass sufficiently water vapors therefore the speed of fiber sensor of absorption is not enough (response time hours and days), and sensitivity is unsatisfactory.
Use of methods of an ion-beam or plasma alloying allows creating easily the central duct of the fiber laser alloyed by erbium without external cover of SiO2 that opens ample opportunities for use her as the highly sensitive and high-speed sensor adsorbed on a water fiber surface.
The water adsorbed on an external surface of fiber causes strong absorption in spite of the fact that the main flow of radiation of the laser extends in volume of fiber. This result from the fact that in single-mode fiber of at most density of energy of optical fluctuations it is much stronger displaced to a fiber surface, than in multimode fibers. With a certain diameter of the central duct the higher density can be even out of the central duct, in an external cover. Applying rather thin cover from SiO_2 it is possible even to combine density maximum with a cover surface.
However, this option is very difficult in case of practical sale of the laser and has no advantages in long-term stability of the sensor. It is caused by the fact that all length of the fiber laser turns out sensitive to water vapors.
From the point of view of increasing long-term stability it is necessary to limit sensitive area of the sensor of long about several centimeters, to place this area in flowing to a ditch, and to make other part of the laser tolerant.
In case of such design the fiber laser can be executed ring-like, on single-mode fiber that can significantly simplify and resolve longitudinal mode structure of the fiber laser. It will allow to refuse unstable process of the competition of fashions in case of inside resonant absorption of FRP and to perform mainly "narrow-band" FRP. Though at the same time loss in sensitivity and spectral selectivity will turn out, however stability of the sensor will sharply increase, and also the system of registration of radiation strongly becomes simpler. There is virtually no need for narrow-band spectral selection by means of Fabri-Perot's interferometer, and the possibility of simple differential amplitude registration appears.
The design of the detector is shown in figure 2. The sensor has two differentially connected fiber sensitive elements from VRP representing ring lasers with the general system of a rating with use of a highly stable fiber beam splitter of a rating. The uniform system of a rating allows weakening significantly the instability of output power of ring lasers determined by a rating.
Under the influence of water vapors in operating ditch, emissive power of the ring laser is reduced that leads to appearance of the difference signal of two photo-sensors. Filtering radiation of pump is necessary for extending dynamic diapason and improving sensitivity of photo-sensors that can be realized by a plate of a semiconductor light filter.
Both lasers and photo-sensors are located in the passive thermostat for balancing their temperatures. Alignment of temperature prevents temperature frequency displacement of generation of the fiber laser that can lead to frequency change of setup of a gap between fibers and to sharp falling of sensitivity.

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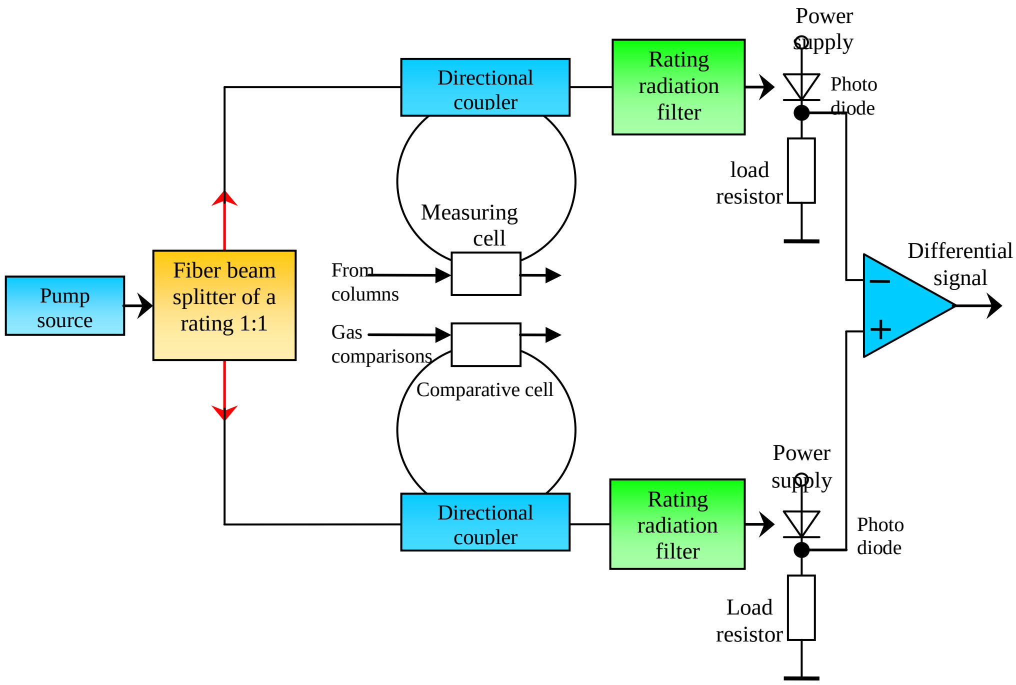

Fig. 2. The block diagram of the fiber sensor (the differential scheme of registration is implemented).

V. CONCLUSION

The water vapor sensor with differential diagram of registration of the signal has sensitivity at the level of 10^{-2} %, thus, as a result of ion-plasma alloying of optical fibers it is possible to receive rather high concentration of the embedded impurity in a near-surface layer up to 100 nanometers thick for creation on their basis of active sensor elements.

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ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 3, Issue 8 , August 2016

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