Laser optoacoustic leak detector

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An optoacoustic (OA) leak detector based on a commercially available CO_2 laser is proposed to be used for testing leak-proofness of vacuum vessels. The leak detector with a differential Helmholtz resonator was applied to detection of SF_6 admixtures introduced into weak gas flows. The dependence on the flow rate of a gas sample in the detector was analyzed. The minimum detectable flow rate was estimated to be $\sim 1 \cdot 10^{-9}$ Pa·m^3/s, which is comparable with the sensitivity of commercial He leak detectors.

Introduction

Commercial leak detectors have two operating modes: the mode of evacuation of a tested vessel (helium leak detectors) and the mode of excess pressure in a vessel (helium and halogen detectors). The leak detection in the excess pressure mode is based on analysis of air nearby a supposed leak for a known substance (gas marker). The gas marker is mixed with air prior to analysis, and the tested vessel is filled with this mixture under the pressure exceeding the atmospheric one. In the case of a defect, the mixture leaks from the vessel, and the leak detector detects marker molecules in the adjacent air.

For more intense operation of leak detectors, they are usually equipped with a pump, which draws in air with the working substance from the place of a possible leak. Helium, halogen-containing gases (freon and others), sulfur hexafluoride (SF_6), and other substances can be used as gas markers. Helium leak detectors are very expensive including significant costs associated with consumption of expensive helium gas during operation. Halogen leak detectors are less sensitive than helium ones.

Sulfur hexafluoride leak detectors are characterized by rather low operating costs because they require low concentrations of SF_6 in air (tenths of percent). These leak detectors can be of *contact type* (equipped with a specialized probe connected with a pump[1]) and of *remote type*. In detectors of the former type, the probe is used to approach the place of a possible leak; and detectors of the latter type are capable of detecting SF_6 in air at the distance from few meters to several tens of meters.[2–3]

Nowadays the optoacoustic (OA) method is widely used for detection of the trace amounts of pollutants,[4] especially, in local monitoring of air pollution due to human activity and in analysis of multicomponent gas mixtures produced in various technological processes. In both of the cases, the measurement procedure involves alternation of gas samples in the measurement cell of the OA detector (OAD). This is usually accomplished in two ways: either the pre-evacuated cell is periodically filled with the analyzed mixture or the mixture is continuously pumped through the cell. It is just the second way that increases the efficiency of the OA analysis in timely detection of leaks of toxic and explosive substances, study of the kinetics of chemical reactions, etc. It is implemented using OADs with acoustically resonant cells.[5] Among them, cells constructed following the scheme of the differential Helmholtz resonator (DHR) have obvious advantages (simple design, low resonance frequency, possibility of increasing significantly the signal-to-noise ratio with the aid of differential schemes).[6]

This paper presents the results of development and test of a laser OA leak detector with a waveguide CO_2 laser.

Design of an OA detector with DHR

Figure 1 shows the block-diagram of an OAD with DHR. The detector consists of two identical cells $2a = 7.2$ mm in diameter and $L = 150$ mm in length. The cells are connected by the same capillaries $2r = 5$ mm in diameter and $l = 100$ mm in length. Windows from BaF_2 or ZnSe were set vacuum-tight at the cell end faces. Nipples located at the centers of the capillaries were used to fill and evacuate OAD. Knowless EK 3027 electret-foil microphones with the sensitivity $R_m = 20$ mV/Pa were set in the walls of the OAD cells. The detailed description of the design of such an OAD can be found in Ref. 7.

Figure 2 depicts the normalized calculated and experimental frequency dependences of the sensitivity of the OA detector. In the frequency region of 200–2300 Hz two resonances were observed. The most intense resonance at the frequency of 627 Hz had FWHM of 60 Hz; another resonance at 2020 Hz had

FWHM of about 120 Hz and three times lower intensity.

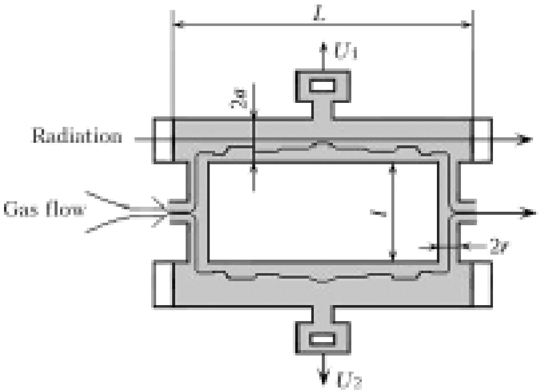

Fig. 1. Schematic diagram of the optoacoustic detector with the differential Helmholtz resonator.

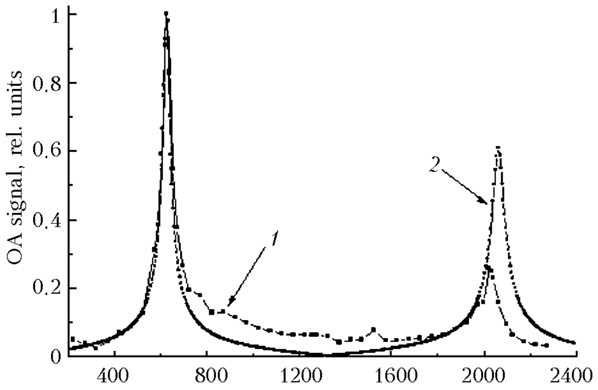

Fig. 2. Normalized dependence of the sensitivity of the OA detector with DHR on modulation frequency of laser radiation: experiment (*1*) and calculation (*2*).

The amplitudes of the electrical signals U_1 and U_2 at the output of each of OAD microphones are described by the equation:

$$U_1 = -U_2 = R_m P_1 = R_m R_{cell} \sigma c W_0 , \qquad (1)$$

where P_1 is the amplitude of acoustic vibrations; R_m, R_{cell} are the sensitivities of the microphones and the cell; σ is the absorption cross section of a gas; c is the gas concentration; W_0 is the laser output power.

The concentration sensitivity of DHR of the OA detector is determined by the equation:

$$C_{min} = \Lambda \, \Delta f^{1/2} / W_0 \sigma, \qquad (2)$$

where $\Lambda = (U_n^2)^{1/2}/R$, in $W \cdot cm^{-1} \cdot Hz^{-1/2}$; $(U_n^2)^{1/2}$ is the rms value of the noise voltage, in V; $R = R_m R_{cell}$ is the detector sensitivity, in $V \cdot W^{-1} \cdot cm$; Δf is the width of the transmission band, in Hz.

The threshold sensitivity of OAD described above amounts to $3 \cdot 10^{-9}\ W \cdot cm^{-1} \cdot Hz^{-1/2}$. For gas media absorbing CO_2 laser radiation, such as ethylene, ammonia, and SF_6, this sensitivity ensures measurement of the absorption coefficients from 10^{-10} to $10^{-12}\ cm^{-1}$, which corresponds to the concentrations of absorbing molecules from 10^{-2} to 10^{-3} ppb.

Selection of a gas marker

The versatility is the characteristic feature of laser OA gas analysis. From the viewpoint of detection of one or another gaseous substance with an OA system it is versatile, since the main parameters (kind and number of substances to be detected, minimum detectable concentrations of these substances) are largely determined by the characteristics of the laser used.

To conduct experiments on detection of admixtures in weak gas flows, sulfur hexafluoride (SF_6) was selected as a gas marker because SF_6 has a strong absorption band in the spectral region of 10.5–10.6 μm (Refs. 8, 9), which includes emission lines of the CO_2 laser. In this spectral region, SF_6 has the maximum absorption coefficient of 0.85 $(cm \cdot Torr)^{-1}$ at the 10P(16) CO_2 laser line (wave number of 947.75 cm^{-1}) (Ref. 9). The SF_6 absorption coefficients at the 10P(18), 10P(20), and 10P(22) CO_2 laser lines are equal, respectively, to 0.60, 0.53, and 0.40 $(cm \cdot Torr)^{-1}$, that is, 1.5–2 times lower than that at the 10P(16) line.

To detect a trace concentration of SF_6 in air, it is sufficient to use a wave-guide CO_2 laser in the free lasing mode without a selective cavity. In this case, at normal cooling, the laser operates mainly at the 10P(20) line, and the output power is twice as high as that of a laser with a diffraction grating at the 10P(16) line. Thus, the advantage of the higher absorption coefficient of SF_6 at the 10P(16) CO_2 laser line is virtually compensated for by the higher output power of the laser without a diffraction grating.

Design of the leak detector and test results

Figure 3 depicts the schematic diagram of the experimental setup for detection of admixtures in weak gas flows. The experiment consisted in detection of the gas marker in the case of small leak of a gas mixture from a vessel.

The experimental setup comprised of a wave-guide CO_2 laser with HF pumping, an OA detector with DHR, a power meter (calorimeter), a pulsed oscillator, a frequency meter, a selective microvoltmeter, and a synchronous detector. The CO_2 laser operated in the repetitively pulsed mode. The pulse repetition frequency was 627 Hz, the duty cycle was 50% (meander), the mean output power was 1.4 W, and the most probable lasing line was 10P(20). The laser radiation passed through one arm of the resonance OA detector. A sampler probe 1.5 m in length and 3 mm in diameter was connected to OAD.

Air samples were pumped through OAD by an electric pneumatic pump with the rate of 0.5–1 liter/min, which was controlled by a serial gas flow controller (rotameter). If the gas marker was present in the air sample, the laser radiation was

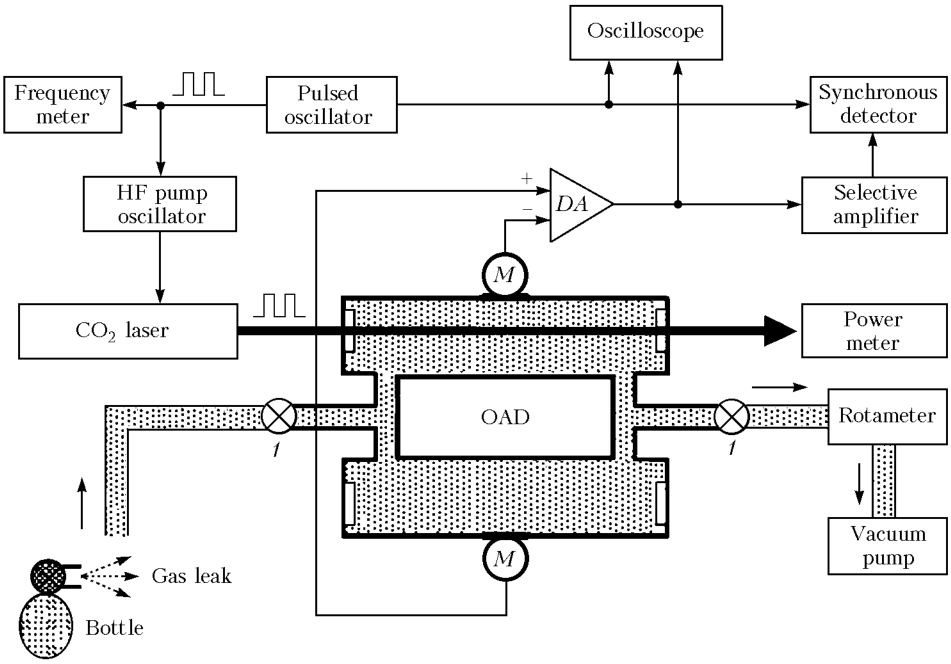

Fig. 3. Schematic diagram of the experimental setup for optoacoustic detection of admixtures in weak gas flows: valves *1*, optoacoustic detector OAD, microphone *M*, and differential amplifier *DA*.

absorbed in OAD and an acoustic wave was generated at the pulse repetition frequency. The acoustic signals were recorded with two microphones, whose voltage was fed to a differential amplifier and then to a selective amplifier and a synchronous detector.

The effect of the flow rate of the gas sample in OAD has been studied. Figure 4 (curve *1*) depicts the experimental dependence of noise measured with the selective microvoltmeter on the flow rate at the laser turned off. At the flow rate from 0 to 0.5 liter/min, the measured noise remained unchanged and was determined by the noise of the electronic system. As the flow rate varied from 0.5 to 0.75 liter/min, the noise level increased by 2—2.5 times. The further increase of the flow rate up to 1 liter/min resulted in a drastic increase of the noise, roughly by 30 times.

It was also found that the sensitivity of detection of the gas mixture decreased with the increasing flow rate. Figure 4 (curve *2*) shows the experimental dependence of the OA signal measured by the selective microvoltmeter on the flow rate in OAD when detecting acetone vapor over an open bottle. Acetone has the absorption band at the wavelength of 10.6 μm. As can be seen from Fig. 4, the sensitivity of the OA detector drops down with the increase of the flow rate. This can be explained by dilution of the gas sample by air at the increased flow rate.

Thus, in this OAD design, the optimal flow rate of the gas sample at the maximum signal-to-noise ratio is ~0.5 liter/min.

In the experiments on detection of admixtures in weak gas flows, a source of SF_6 leakage was modeled using a 2.7 liter bottle filled, up to the pressure of 1.5 bar, with nitrogen and 0.023% SF_6 (230 ppm) admixture. The leakage occurred through the loosely tighten valve. The measured leakage of the gas mixture amounted to $(0.7–1.0) \cdot 10^{-3}$ Pa · m^3/s. Under these conditions, the leakage of SF_6 was detected reliably with SNR ≈ 150 at the flow rate of 0.5 liter/min. The delay in obtaining of a OA signal from SF_6 was from 1 to 3 s depending on the length of the sampler hose and the flow rate of the air sample in OAD.

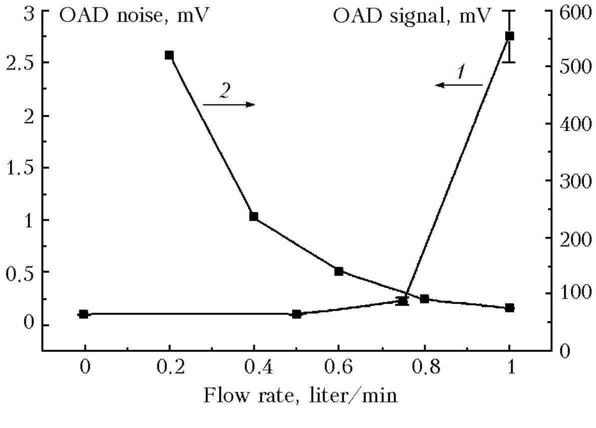

Fig. 4. Dependence of noise (*1*) and OAD sensitivity (*2*) on the flow rate of the gas sample in the detector.

The ultimate sensitivity of the laser OA leak detector for pure SF_6 was estimated as $\sim 1 \cdot 10^{-9}$ Pa·m^3/s. The sensitivity of a commercial TI-14M leak detector in the mode of excess He pressure in the tested vessel air sampling from its surface is $\sim 1 \cdot 10^{-9}$ Pa·m^3/s. Thus, the tested laser OA leak detector does not yield to the traditional detectors of

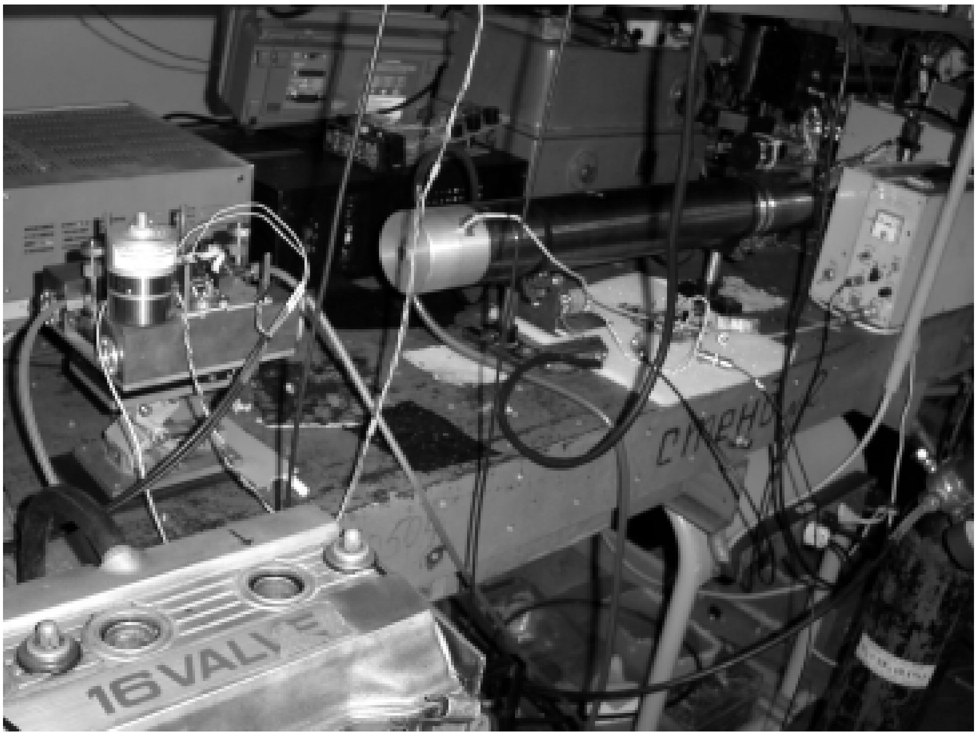

Fig. 5. External view of the pilot laser optoacoustic leak detector.

weak gas flows in sensitivity. If the flow-through OAD detector will be placed inside the laser cavity, this could improve the sensitivity by an order of magnitude.

The above results were put in the foundation for the development of a pilot device, which is shown in Fig. 5.

Conclusion

The capabilities of using a detector with the differential Helmholtz resonator in a laser OA leak detector have been studied experimentally. The laser OA leak detector was intended for detecting admixtures due to leaks from sealed vessels in weak gas flows. Sulfur hexafluoride (SF_6), which has a strong absorption band in the spectral region of 10.5–10.6 μm, was selected as a gas marker. The obtained experimental data have suggested that for detection of SF_6 admixtures it is sufficient to use a continuous-wave CO_2 laser operating in a free lasing mode without a selective cavity. In this case, the advantage of higher SF_6 absorption coefficient at the $10P(16)$ CO_2 laser line is virtually compensated for by the higher output power of a laser without a diffraction grating.

The effect of the flow rate of the gas sample in OAD has been analyzed. It has been shown that the increase of the flow rate results in the increase of the noise level, as well as the decrease of the OAD sensitivity due to dilution of the gas sample by the ambient air. In the OAD design studied, the optimal flow rate of the gas sample at the maximum signal-to-noise ratio amounted to ~0.5 liter/min.

The reliable detection of the leakage of the gas mixture containing 0.023% SF_6 (230 ppm) has been achieved at the leakage rate of $(0.7–1.0)\cdot 10^{-3}$ Pa · m^3/s.

The delay in obtaining the OA signal from SF_6 varied from 1 to 3 s depending on the length of the sampler hose and the flow rate of the air sample in the OAD. The ultimate sensitivity of the laser OA leak detector for pure SF_6 was estimated as $\sim 1\cdot 10^{-9}$ Pa · m^3/s, which is comparable with the sensitivity of the serial TI-14M helium leak detector operated in the mode of the excess pressure in the tested vessel and air sampling from its surface.

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