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The influence of geometry on the sensitivity of a carbon molecular electron converting element

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The technology developed by the authors for creating carbon converting elements for molecular electronic motion sensors using screen printing and laser micro-processing is presented. A batch of samples of converting elements with flat and cylindrical channels with different values of the thickness of the working channel: from 36 to 400 μm and a different distance between the electrodes from 55 to 270 μm was made. Seismic sensors were manufactured using manufactured samples, studies of their frequency characteristics were carried out and configurations were established to ensure the maximum value of the conversion coefficient. For the studied geometric range, the conversion coefficient turns out to be higher, the thinner the channel in which the signal is converted, while for the distance between the electrodes there is an optimal value corresponding to the highest value of the conversion coefficient.

Keywords: seismic sensors, electrochemical systems, microfluidics, microstructures, carbon electrodes.

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Introduction

A technology of measurement of motion parameters based on principles of molecular electron transfer has been recently applied when creating broadband seismometers, accelerometers and angular velocity sensors [1–10]. The principal advantage of instruments that are manufactured based on this technology is that it is possible to record weak nanometer-level movements in the infra-low-frequency portion of the spectrum up to hundredths and thousandths of a hertz. Consequently, the application field is related to creating high-sensitivity seismometers and accelerometers for studying a natural seismic field of Earth [2,3,11–17]. The technology can get a significant commercial potential provided that it is widely introduced into seismic surveying, where yearly demand is several hundred thousand or even millions of the sensors per year. The test works done in recent years have shown that the instruments based on molecular electron transfer have a significant advantage both in sensitivity and the frequency range as compared to the electrodynamic geophones that are the most common in seismic surveying [18,19].

The primary converting element of the sensors based on the principles of molecular electron transfer is a miniature electrochemical cell. This cell is a system of electrodes with typical sizes of several tens of microns, which is immersed into a highly concentrated electrolyte solution. When switching on an external power supply, there is a difference of potentials between the electrodes, which initializes electrochemical reactions with participation of the solution components, thereby resulting in electrical current between the electrodes. A principle of measurement of the external signal is that in motion of a frame of the sensor that contains such a cell, the liquid is affected by inertia forces and it starts moving in relation to the electrodes. At the same time, the liquid flow affects a speed of supply of reagents of the solution to the electrodes to result in variations of the interelectrode current, which are a primary output signal of the cell.

Considerable efforts of researchers from a number of laboratories in Russia, China and USA are aimed at searching new technological solutions to create the converting electrochemical cell, which can decrease a spread of parameters of the sensitive elements during their manufacturing and reduce the prime cost [18,20–31]. As a result, approximately, over the last five years there were dozens of various designs of the electrochemical cells that are characterized by high output parameters and good repeatability of the characteristics.

At the same time, one of the main limitations for extending the application field of this technology is a cost. The main reason of high cost of the modern molecular electron seismic sensors is use of platinum for creating microelectrodes of the electrochemical cell. Platinum is a quite expensive material and technological processes that are used when creating the electrochemical cells based thereon are also characterized by the high cost. As an alternative to platinum, carbon can be used, so can its low-dimensional modifications in the future. Application of the carbon material makes it possible to multiply reduce the cost of the converting elements. At the same time, as shown in the paper [32], the characteristics of the converting elements of the electrochemical motion sensors with platinum and carbon electrodes are quite close to each other provided that geometrical characteristics of the converting elements are similar to each other.

The aim of research taken by the authors in the present study was to determine geometrical characteristics of the converting elements with the carbon electrodes, which ensured the highest conversion coefficient. For this purpose, the authors have developed the technological processes that are based on screen printing and laser micro-processing and allow varying the geometrical parameters of the converting elements in a quite wide range. Based on the developed technological processes, samples of the converting elements with flat and cylindrical channels were manufactured, in which the liquid flow is converted into an output electric signal and which have a different thickness of the working channel and a different distance between the electrodes. For the manufactured cells, frequency characteristics of their response to external mechanical effects are studied and sets of the geometrical characteristics, at which the conversion coefficient gets to the maximum, are specified.

1. Configurations of the converting elements used in the study

1.1. Design of the electrochemical cell

The research object in the present study is a 4-electrode electrochemical cell of a planar type. It is 4 parallel electrodes arranged on walls of the nonconducting channel in the order A–K–K–A. We note that a height of the electrode is small as compared to a width of the channel, in which the working liquid flows under impact of the inertia forces that are caused by the external mechanical signal. In this condition, the cell properties slightly depend on a form of the channel section and according to the common terminology the converting elements belongs to the planar type. The cell is designed to ensure a flow of the liquid between the electrode as they are arranged in the channel, i.e. at each moment of time the liquid flows from the anode to the cathode for one anode-cathode pair and from the cathode to the anode for another pair. The working liquid is an aqueous solution of the MeI electrolyte, where Me is a metal ion (most often, it is K^+ or Li^+), with addition of a small amount of molecular iodine I_2.

In the solution, the molecular iodine is in the form of positive triiodide ions. In reactions initiated by the external difference of potentials, ions of atomic iodine are formed from the triiodide ions at the cathode, while the reactive is reverse at the anodes. Since the total number of the ions is constant, the occurring reaction and transfer of charges can last for an arbitrarily long time without varying the compositing of the working liquid. Each elementary reaction includes transmission of two electrons through the surface of the electrodes and the interelectrode current flows in the system. In turn, the current value depends on the inertia forces that create the liquid flow and provide transfer of the ions between the electrodes.

Mathematically, transfer of active ions in the instruments of this type obeys the equation of convective diffusion of the ions:

$$\frac{\partial c}{\partial t} = D\Delta c + (\mathbf{v}, \boldsymbol{\nabla})c, \quad (1)$$

where v — the velocity of the electrolyte, c — the concentration of the current carriers (triiodide ions), D — the coefficient of diffusion of these ions.

The equation makes it clear that sensitivity of the instrument (the value of changes of the concentration in time and variations of the current related to these changes) mainly depends on the velocity of the working liquid, which can be varied by changing hydrodynamic resistance of the channel, and on a concentration gradient as well. In turn, the hydrodynamic resistance depends on the channel thickness, while the concentration gradient is determined by a distance between the electrodes. Influence of the said geometrical parameters on the conversion coefficient of the electrochemical cell is investigated in the present study.

1.2. Form of the electrochemical cell and the ranges of study of the geometrical parameters.

The most previously studied samples manufactured using the platinum electrodes are either various options of the planar system, in which the liquid moves in the flat channel, whose walls have the electrodes formed [24,27,31–33], or configurations manufactured by creating through holes in dielectric substrates, whose surface have the electrodes manufactured [21–23,26,29,30], which is related to simpleness of technologies required for their implementation. The known options that can be manufactured with greater difficulties provide only small quantitative improvement of the output characteristics. Therefore, in the present study we also choose the two above-mentioned widespread configurations.

In addition to technological limitations, in order to determine the range of study of the geometrical characteristics, it is necessary to take into account a number of factors related to physics of signal conversion. First of all, we note that the influence of the liquid flow on distribution of the active ions is mathematically described by the second summand on the right-hand side of the equation of convective diffusion (1). It follows from the form of this summand that the necessary condition for obtaining the high conversion coefficient is coincidence of areas, in which the liquid predominantly flows and there is a gradient in distribution of the concentration of the active triiodide ions. In turn, for each pair of the anode-cathode electrodes, a typical size of the area, in which the ions diffuse, is determined by the distance between these electrodes. Therefore, in particular, if the considered electrodes are on one of the channel walls, then its width shall not greatly exceed the distance between the electrodes. Otherwise, the liquid in a central part of the channel is practically not involved in signal conversion. On the other hand, a very thin channel makes great resistance to the liquid flow, thereby reducing an absolute value of the hydrodynamic velocity. Therefore, when creating experimental samples, the geometry parameters were selected so that the distance between the electrodes and the sizes of the liquid flow channel were of the same order, i.e. differed in no more than 10 times. As for the absolute values of the sizes, it is known than in the electrochemical systems, at the distances of more than 500 μm from a solid surface the liquid in a gravity field becomes unstable, thereby resulting in low-frequency noises of the interelectrode currents [34]. On the other hand, very small sizes of the channels make very high resistance to the liquid flow, which can be compensated only by significantly increasing their number or decreasing their length. It is quite difficult to provide both technologically. Based on the said considerations, the present study included selection of a range of change both of the channel size and the interelectrode distance from several tens to several hundred micrometers. We note that this range is quite typical for the electrochemical systems of a similar purpose irrespective of a method of their creation.

1.3. Electrochemical cell with the carbon electrodes manufactured on the walls of the flat channel

The electrochemical cell of Fig. 1 with the flat channel, in which the signal is converted, is designed with two parallel nonconducting plates, which are separated by a likewise nonconducting gasket of a pre-defined thickness with a hole that encompasses the entire electrode system both on one plate and on the other. Each plate has the electrodes that are manufactured as parallel conducting tracks which are internally located and have a gap pre-defined during manufacturing. The plates also have holes for access of the working liquid to the electrodes, while the holes in the upper and the lower plates are displaced from each other. In this case, the inertia forces acting on the working liquid in a direction that is perpendicular to a plane of the said plates form the liquid flow through the converting electrochemical structure. At the same time, in space between the plates the liquid flow turns out to be directed predominantly along the plates surface and perpendicular to the conducting tracks. Thus, the liquid flow is formed to ensure transfer of the ions between the electrodes of the said converting structure. This design was first proposed for the platinum electrodes in the study [35] and investigated for a small set of the geometrical characteristics of the converting system.

The main parameters, whose influence on the conversion coefficient of the electrochemical cell is investigated in the present study, include the distance between the electrodes, which is pre-defined by a laser machine cut width, and the channel width that is determined by the thickness of the used gasket.

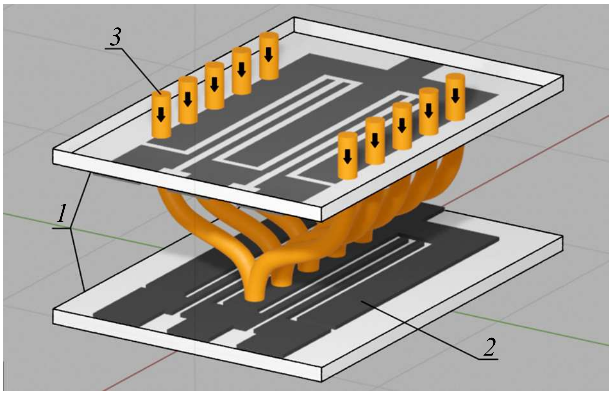

Figure 1. Diagram of the sensitive element with the flat channel: *1* — the plate with the holes, *2* —the electrodes, *3* — the liquid flow lines.

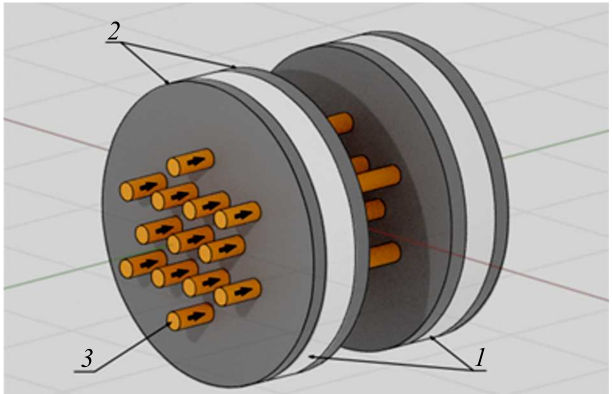

Figure 2. Diagram of the sensitive element with the cylindrical channel: *1* — the plate, *2* —the electrodes, *3* — the liquid flow lines.

1.4. Electrochemical cell with the carbon electrodes manufactured on the walls of the cylindrical channel

The similar converting element consists of two parallel dielectric plates that are separated by the gasket with the hole that encompasses all the electrodes of the electrochemical cell (Fig. 2). Both the sides of each plate has the electrodes in the form of the conducting carbon layer that covers the entire area. A dielectric coating is additionally manufactured above the conducting layer at the side of the cathode. The plates also have round holes designed to transmit the working liquid through the converting element. Each hole can be presented as a cylindrical channel, in which the liquid transfers the ions between the electrodes of different polarities.

It should be noted that the holes practically manufactured on the plates have noticeable taper, which is related to use of a laser beam when manufacturing. At the same time, a diameter of the hole at the beam inlet will exceed the output one. The laser beam mills at an external side of the cell, therefore, the input holes will be visible on an anode layer, so will the output holes on a cathode layer. For correct operation of the device, it is important that the diameter of the output hole does not exceed a certain value, thereby maintaining an allowable level of the reaction taking place at the cathode.

Further study will include investigation of the influence of the distance between the electrodes and the width of the working channel on sensitivity of the instrument, wherein in case of the cell with the cylindrical channel the said geometrical parameters are equal to the thickness of the dielectric plate and the diameter of the holes at exit of the laser beam, respectively.

2. Manufacturing of the samples and a procedure of experimental studies

2.1. Method of manufacturing of the samples with the flat channel

A sheet of fiberglass FR-4 of the thickness of 0.5 mm is cut by the laser marker G-MARK 100 [36] to produce a square blanket of the size 45×45 mm. This blank is placed in a fixture and using the laser engraving complex „UniMarker UV Mini“ [37] both sides of the plate are engraved to obtain locations for applying a carbon resistive polymer paste PURP-0.05 [38]. Then, the blanks are processed with alcohol solution. The paste is applied to the processed blanks by screen printing and it is solidified in a drying oven (Fig. 3, *a*). The laser marker is used to process a past surface according to the drawing (Fig. 3, *c, d*). Each required value of the gap between the electrodes is taken to design an individual drawing and to form a software program to control the laser machine. The blanks are again processed with alcohol solution. In the final operation, contact areas of the solidified paste, which are formed at the sample edges, are additionally coated by the carbon paste, terminals of insulated copper wire of the diameter of 0.1 mm are glued to the paste to be followed by solidification in the drying oven. An epoxy two-component glue is applied to the terminal glueing location.

The finished elements (Fig. 3, *b*) are subjected to check measurements of the gaps and the resistance. The quality of the created samples was controlled by means of a microscope that can make images of the sensitive elements with magnification of 10x (Fig. 3, *e–i*). Besides, a real distance between the electrodes was measured using the microscope. The results have shown that intervals between the electrodes were 275, 170, 105, 55 μm for four types of the samples. The spread of the distances between the electrodes was below 5 μm within one type of the samples. Additionally, the resistance of the electrodes of the conducting paste was measured, and the samples with the resistance between extreme points of the electrodes of at most 400 Ω were taken for the tests.

2.2. Method of manufacturing of the samples with the cylindrical channel

A sheet of the fiberglass FR-4 of the pre-defined thickness was cut by the laser marker to produce a square blanket of the size 45×45 mm. The blanks are processed with alcohol solution. The paste is applied to the blanket by screen printing and it is solidified in the drying oven. The dielectric paste is applied to the cathode layer by screen printing and it is solidified in the drying oven. The laser marker is used to create a fine grid of 2500 holes. For this, each pre-defined diameter of the channels is taken to make the drawing (Fig. 4, *a*) and to create the software program for operation of the laser machine. The blanks are processed with alcohol solution. The contact areas are filled with the carbon paste, the terminals of insulated copper wire are glued to be followed by solidification in the drying oven. An epoxy two-component glue is applied to the terminal glueing location. The finished elements (Fig. 4, *b*) are subjected to check measurements of the channel diameters and the resistance.

2.3. Procedure for investigating the dependence of the conversion coefficient on the geometrical parameters of the electrochemical cell

For analysis of the characteristics, the finished samples of the electrochemical cells were assembled in a single layout of Fig. 5 irrespective of their specific type. The sealing gasket *1* was placed between the fiberglass plates with the electrodes *2* arranged thereon. The rubber membranes *3* and the stop rings *4* were installed at both sides of these plates. The structure was completed with the metal flanges *5* with a thread for fixing screws.

The sensor elements assembled in this way were fixed in a vise to ensure leak-tightness and were additionally fixed with the screws. When tightening the screws, the sealing gasket and the O-rings of the membranes were tightly pressed to the composite epoxy plates, thereby providing leak-tightness of an internal cavity of the sensor. Upon completion of the assembly, the sensors were filled with the electrolyte *6* — the aqueous solution LiI 4M, via a narrow hole in the stop ring, with addition of molecular iodine in the concentration of 0.03 M, by means of a syringe. It was followed by degasation in a vacuum chamber.

The assembled sensor was equipped with an electromagnetic actuator: the magnet *7* was attached to one of the membranes and there was the coil *8* nearby, which was fixed on an immovable part of the structure. This actuator converts current oscillations in the coil into mechanical oscillations of the electrolyte in the converting elements, thereby making it possible to model effects of the inertia forces with seismic signals in the pre-defined frequency range [39].

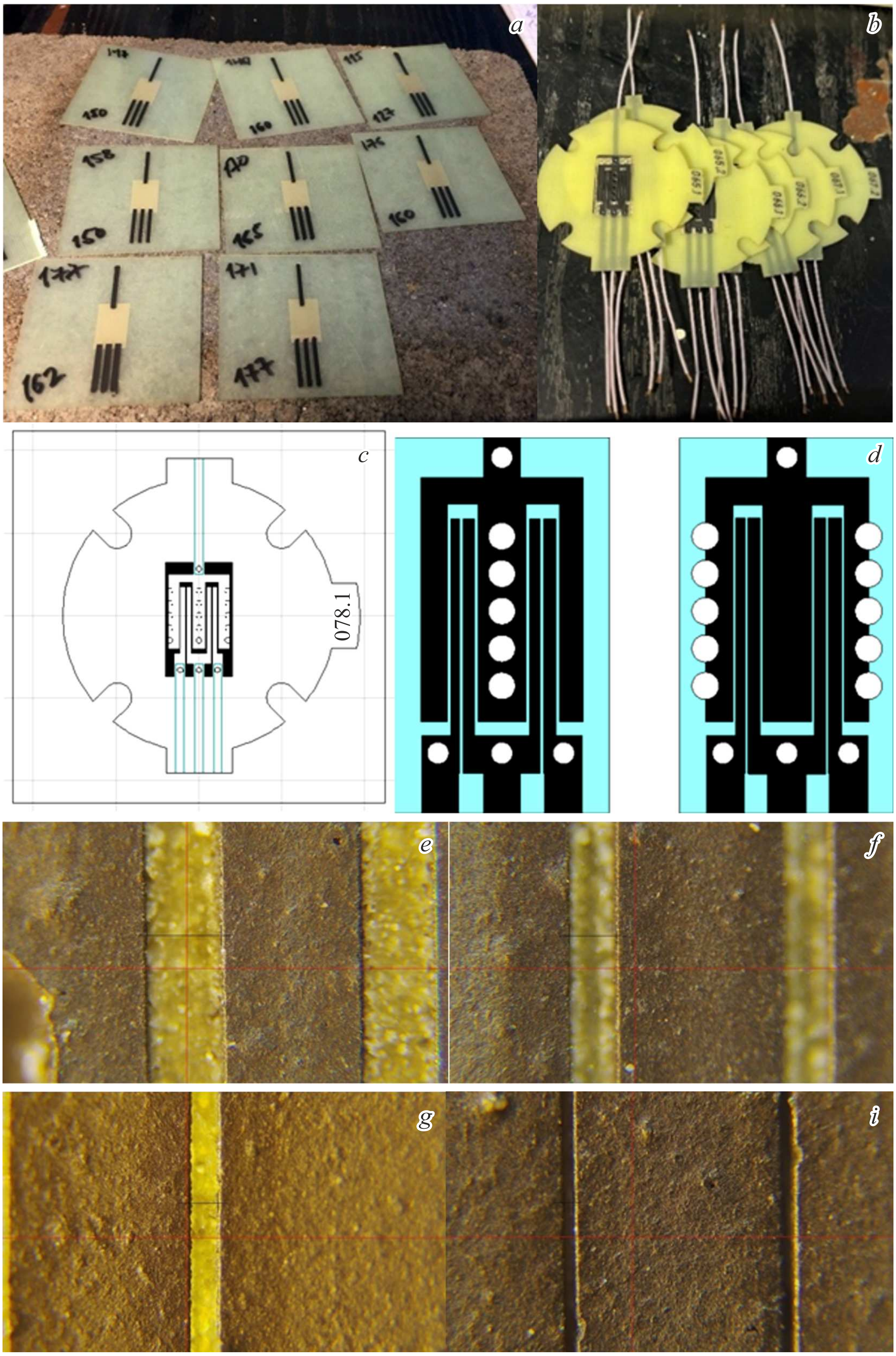

Figure 3. Cell with the flat channel: *a* — the blanks; *b* — the finished samples; *c* — the drawing of the blank; *d* — the drawing of the electrodes and the holes; *e–i* — the samples with the gaps 275, 170, 105 and 55 μm, respectively.

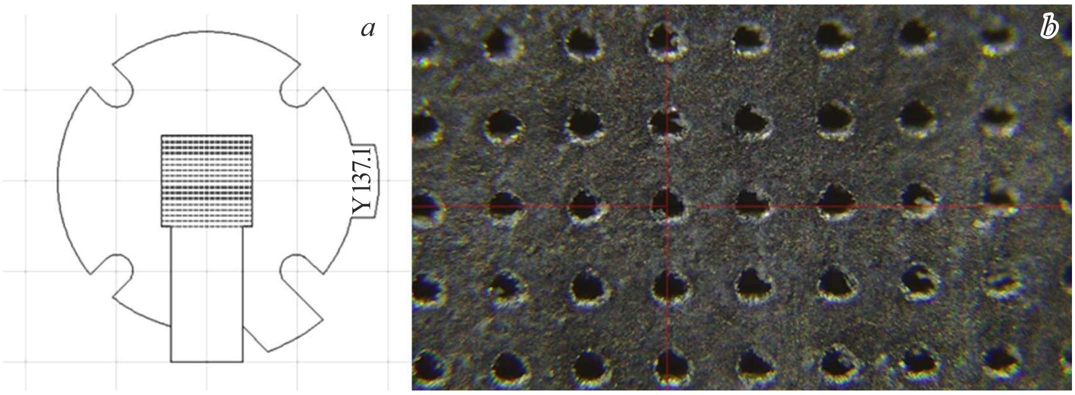

Figure 4. Cell with the cylindrical channel: *a* — the drawing of the blank; *b* — the photo of the finished sample of the sensitive element.

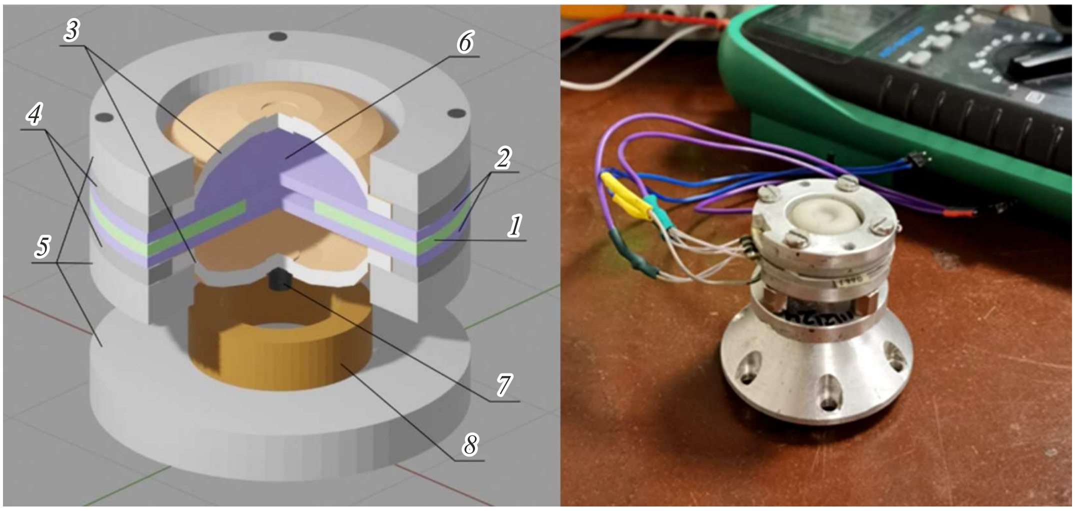

Figure 5. Diagram of the assembled sensor (on the left): *1* — the gasket, *2* — the plates with the electrodes, *3* — the membranes, *4* — the stop rings, *5* — the flanges and the holder of the coil, *6* — the electrolyte solution, *7* — the magnet, *8* — the coil; the photo of the assembled sensor (on the right).

2.4. Manufacturing of a mockup of the sensor based on the studied electrochemical cells

The study included specification of the dependence of sensor on the value of the gap between the electrodes and the thickness of the working channel. The test samples with the flat channel had 4 option of the distances between the electrodes: 275, 170, 105, 55 μm. The thickness of the working channel for the samples with the flat channel was assumed to be 0.4, 0,2, 0.1 mm. 3 samples of the sensitive elements were manufactured for each option of the distances.

3 options of the thickness of the fiberglass plates were selected for the cells with the cylindrical channel. 200, 100, 60 μm. An allowable diameter of the grid holes, which is often used when operating the cells of this type, has sizes of about several tens of microns. In the present study, the diameters of the grid holes 36, 40, 44, 48 μm have been pre-defined for the software program of the laser installation. During the measurements, the actual sizes were about 55, 60, 65, 70 μm. 3 samples of the converting elements were manufactured for each option as well.

During the measurements, according to the diagram of Fig. 6, a sinusoidal signal was supplied from a DAC output to the electromagnetic actuator's coil with the resistance of 150 Ω, the signal amplitude varied within the range 30–200 mV, the frequency varied within the range 0.05–100 Hz. The actuator converted the current oscillations in the coil into mechanical motion of the electrolyte in the sensor. The signal was read out from the sensor by the 16-digit ADC (produced by National instruments, the model NI USB-6215) [40]. The obtained data were processed in the DADiSP software to obtain an amplitude-frequency response of the instrument.

3. Results

The dependence of sensitivity (a ratio of the signal current obtained from the sample to the coil current) of

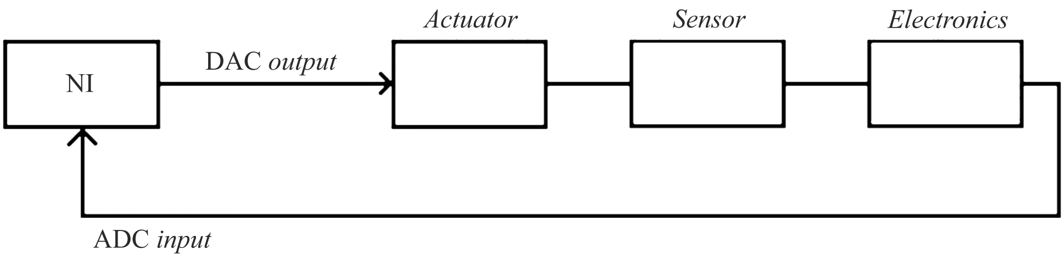

Figure 6. Schematic diagram of the installation.

the samples with the flat channel on the DAC output's signal frequency is shown in Fig. 7. Fig. 7, *a* illustrates the characteristics of the cells with the fixed channel width, which are averaged across several samples of the identical geometry, and the different colors belong to the different interelectrode distances. Fig. 7, *b* shows the characteristics of the samples with the fixed distance between the electrodes, which are averaged across several samples of the identical geometry, the different colors belong to the different values of the channel width. Within the present study, each averaging of a group of prototypes of the same geometry was taken to manufacture a small number of the samples. Therefore, no problem of finding dispersion was set. Let us take the obtained data to compare the characteristics of the samples with the flat channel with the various configurations of the converting element.

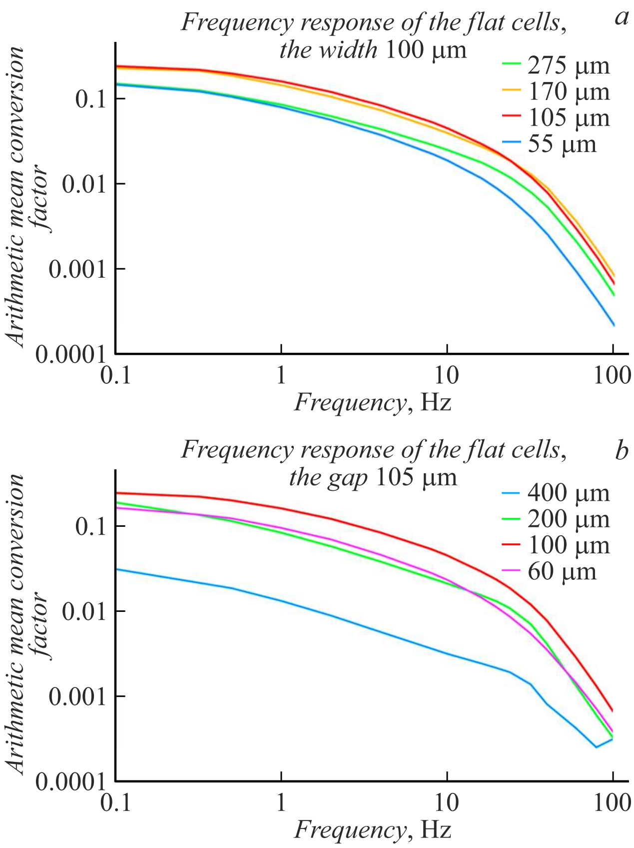

Figure 7. Frequency characteristics of the samples with the flat channel: *a* — with the various values of the gap between the electrodes; *b* — with the various values of the width of the working channel.

The natural result (Fig. 7, *a*) is low sensitivity of the samples with the distance between the electrodes $275\,\mu$m as compared to the parameters 170 and $105\,\mu$m: this gap significantly reduces the concentration gradient and, according to the equation of convective diffusion, the lesser the gradient, the lower sensitivity of the instrument. A possible reason for low sensitivity of the samples with the gap of $55\,\mu$m is demonstrated in Fig. 8: the value of the gap is comparable with the height of the electrodes above the plate, thereby making an initial assumption about planarity of the structure incorrect. A hydrodynamic shadow is formed in an area between the electrodes, i.e. a portion of the working liquid stops its participation in conversion of the external signal. The data allow unambiguously concluding: there is an optimal distance between the electrodes for the cells with the flat channel in the selected range of the parameters.

Let us analyze the characteristics of Fig. 7, *b*. One can notice relatively low sensitivity of the samples with the channel width of 0.4 mm and the samples with the width of 0.06 mm against the background of the parameters 0.2 and 0.1 mm. The result for the samples with the channel width of 0.4 mm is predictable: with increase of the channel width its hydrodynamic resistance decreases and, consequently, the velocity of the flow of the working liquid decreases and, according to the equation of convective diffusion, sensitivity of the instrument decreases. The samples with the channel width of 0.06 mm are subject to another mechanism: with significant decrease of the channel width a portion of solution molecules involved in boundary sticking to the

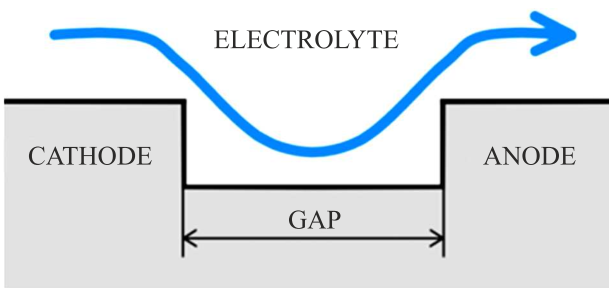

Figure 8. Hydrodynamic shadow.

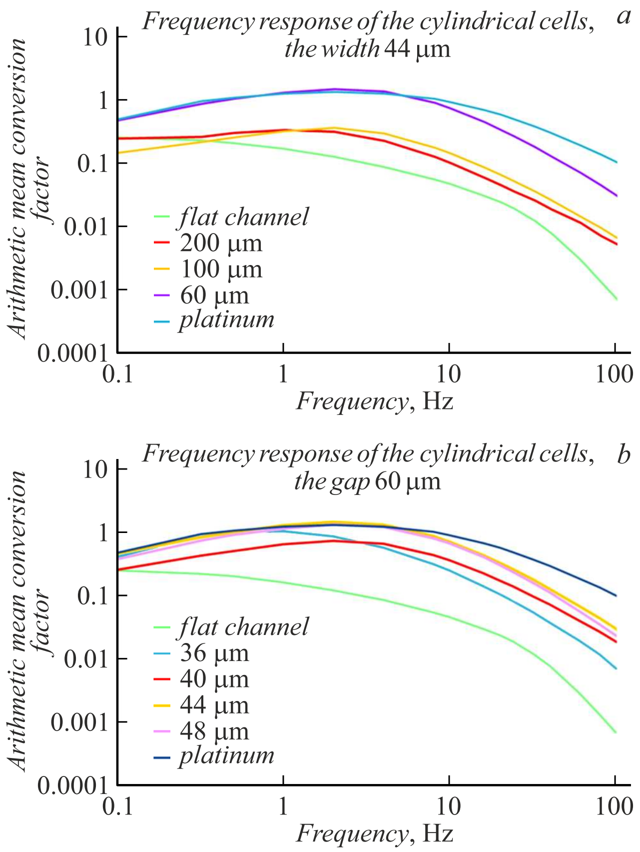

Figure 9. Frequency characteristics of the samples with the cylindrical channel: *a* — with the various values of the gap between the electrodes; *b* — with the various values of the width of the working channel.

channel walls increases, thereby also decreasing the effective velocity of the flow of the electrolyte liquid and, therefore, decreasing sensitivity of the instrument. Thus, it was found that there is the optimal value of the width of the working channel for the cells with the flat channel.

The dependence of the conversion coefficient on the signal frequency for the cells with the cylindrical channel is shown in Fig. 9. Fig. 9, *a* illustrates the characteristics of the cells with the fixed channel width, which are averaged across several samples of the identical geometry, and the different colors belong to the different interelectrode distances. Fig. 9, *b* shows the characteristics of the samples with the fixed distance between the electrodes, which are averaged across several samples of the identical geometry, the different colors belong to the different values of the channel width. As before, due to a small amount of the samples for averaging the characteristics an issue of dispersion calculation was not investigated. Besides, for comparison the graphs were supplemented with characteristics of the best sample with the flat channel, which are obtained in the present study (the distance between the electrodes is 105 μm, the channel thickness is 0.1 mm), and of the commercial converting sensitive element based on a platinum mesh, which is produced by „R-sensors" and designed as described in the study [41].

Let us take the obtained data to compare the characteristics of the samples with the flat channel with the various configurations of the converting element. In Fig. 9, *a*, the green curves correspond to the samples with the flat channel, the red curves correspond to the cells with the cylindrical channel on the composite epoxy substrates of the thickness of 200 μm, the orange curves correspond to the cylindrical channel on the substrates of the thickness of 100 μm, the violet curves correspond to the cylindrical channel on the substrates of the thickness of 60 μm, the blue curves are the samples with the platinum mesh. In Fig. 9, *b*, the light blue curves correspond to the samples with the channel width of 36 μm, the orange curves correspond to the width of 40 μm, the red curves correspond to the width of 44 μm, the violet curves correspond to the width of 48 μm. One can judge the characteristic by how big is the spectrum band, in which it prevails over the other characteristic. Thus, a simple monotonic dependence was detected within the pre-defined rant of the thickness of the composite epoxy plates: the smaller the distance between the electrodes, the higher conversion coefficient of the sample. The obtained result complies with the theoretically expected one according to qualitative analysis of the equation of convective diffusion (1) as well as more detailed analysis given in the study [42]. The mechanisms that describe the behavior of the characteristics with the different values of the width of the working channel are similar to those described for the cells with the flat channel: with the channel width of 36 μm a portion of molecules of the working liquid, which are involved in the boundary sticking processes is great and, in turn, it means that its respective characteristic is below in relation to the others. The characteristic of the samples that have the width of 48 μm is slightly inferior to the curve with the width of 44 μm: the wide channel creates low hydrodynamic resistance and, consequently, the low velocity of flow of the solution. It has been found that there was the optimal value of the width of the working channel for the cell with the cylindrical channel as well.

Conclusion

Thus, the present study presents the technological process of manufacturing the converting elements for the molecular electron motion sensors based on the carbon materials using screen printing and laser processing of the materials, which allows implementing a wide range of the geometrical characteristics of the converting electrochemical cells. The performed experimental studies of the manufactured samples could reveal a width of the working channel and the interelectrode distance, at which the conversion coefficient reached its greatest value for the considered configurations of the planar type and turned out to be quite conformable to the converting elements made of the platinum mesh. At the same time, the cost of the initial materials and the technological process turns out multiply lower.

These circumstances allow expecting wide application of the carbon materials when creating the molecular electron sensors for the most large-scale application fields, first of all, for seismic surveying.

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Conflict of interest

The authors declare that they have no conflict of interest.

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