Further progress in acoustoelectronic componentry utilizing BAWs and its adoption require the continuation of the search for solutions to the following basic problems:

— development of a new design philosophy of high-Q thin-film acoustic resonators and microwave filters, as well as elaboration of a theory describing their operation;

— the search for new acoustic materials and structures characterized by low losses in the propagation of acoustic microwave waves for resonators and filters;

— development of the physical principles of acoustoelectronic methods of generating acoustic and electromagnetic microwave oscillations in piezoelectric semiconductor films, and, in particular, investigation into acoustoelectronic nonlinearity and determination of the conditions for narrow spectral line generation in resonators on the basis of thin semiconductor films;

— elaboration of the growth technologies of piezoelectric, dielectric, and metallic films with specified electrophysical and acoustic properties, and

— elaboration and employment of new techniques of investigating the physical properties of thin layers and films along with development of the methods for controlling the acoustic parameters of thin layers and films.

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Multimode acoustic sensors and systems

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1. Introduction

Heightened terrorist threats, environmental contamination, and the spread of new diseases have set new urgent tasks before the developers of electronic sensors. One of them consists in creating miniature devices intended for the detection of hazardous substances which may be present at low concentration in gaseous and liquid mixtures. The solution to this complicated and multidisciplinary problem is being pursued along several alternative directions in many laboratories all the world over.

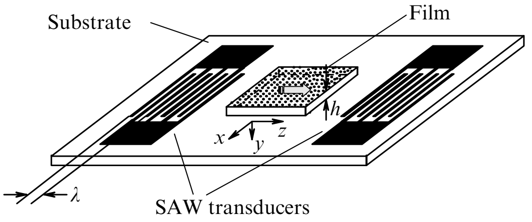

Figure 1. Schematic of a one-channel gas sensor utilizing surface acoustic waves.

Among acoustic sensors, devices which utilize Rayleigh surface acoustic waves (SAWs) have gained the widest acceptance (Fig. 1). A small localization depth (~ 10 μm) makes these waves especially sensitive to the mass load of the surface. That is why a variation in the properties of film that lies in the path of a wave is responsible for variations in its velocity v, phase φ, and amplitude A, which are recorded at the device output as frequency or voltage variations. The frequency response of acoustic sensors advantageously sets them apart from devices of other types because it furnishes a high measurement accuracy and simple compatibility with digital information processing systems. However, none of the present-day sensors alone, including acoustic ones, is capable of ensuring selective detection of a given gas because attempts to make a chemical coating that reacts to a single gas component and not to all the rest have not met with success.

The solution to this problem was found by producing 'electronic nose' and 'electronic tongue' instruments similar to the olfactory and gustatory organs of animals and humans [1–3].

The main constituent of these devices is the so-called sensor array — a set of several elements with differing sensitivity to the same gas components. The sensors are made different by combining devices of different types into the array (for instance, electrical resistors, capacitors, field-effect transistors, quartz microbalances, SAW-based devices) and using a specific chemical coating for each sensor. The set of responses read out from the sensors is subjected to special mathematical processing which enables determination of the entire composition of the mixture being tested or its correspondence to one of those belonging to the database. Under this approach, the sensor array becomes the key element of the entire instrument, which determines its accuracy, resolving power, and stability. In this case, there emerges a fundamental contradiction: on the one hand, the greater the number of sensors contained in the array and the greater their diversity, the more perfect the operation of the electronic nose as a whole; on the other hand, the greater the number of sensors and hence the number of gas-sensitive coatings, the less stable the operation of the instrument as a whole due to the aging of the films. This contradiction brings up the question: Is it possible to develop arrays containing a large number of different sensors and, at the same time, a small number of chemical coatings for them?

Pursuing this line of inquiry, the staff members of the IRE RAS (Moscow) proposed and elaborated a new, purely acoustic approach. Its key idea consists in harnessing, instead

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[1] Perished tragically in the terrorist attack in the center of Moscow on 9 December 2003.

of one sensor property and one type of wave (as was previously the case, see Fig. 1), *all properties and all acoustic vibrations* which may exist in solids. Indeed, since up to 10 waves of different types can propagate through piezocrystals, each of the waves exhibiting its own reaction to external actions, by selecting the types, quantity, and properties of the waves that probe the medium adjacent to an acoustic line it is possible to form the proper set of responses even without recourse to gas-sensitive coatings.

Within similar approach it is possible to solve another applied problem — to simultaneously measure several characteristics of the environment or a chemical – biological process. In this case, advantage is taken of several probing waves, their characteristics are measured, and a system of equations is constructed to relate the wave characteristics to the medium's parameters. When the number of equations is equal to the number of unknowns, the solution of the system is unambiguous.

We demonstrate the working capacity of the new approach by specific examples.

2. Integral SAW-sensor array. Analyzer of adsorption from the gas phase

The device operation relies on the dependence of relative SAW-velocity variation due to adsorption (the SAW response $\Delta v/v$) both on the properties of adsorbent film (variations in the density $\Delta\rho/\rho$, elastic moduli $\Delta c_{ij}/c_{ij}$, conductivity $\Delta\sigma/\sigma$, and temperature ΔT) and on the direction of wave propagation on an anisotropic substrate [4]. This dependence permits combination of several SAW sensors on a common substrate and employment of a common adsorbent film for them (Fig. 2a). The difference between the sensors in the integral array (Fig. 2b) is ensured by the anisotropy of the five probing waves, which differently 'read out' the variations in film properties due to the adsorption. The array becomes more compact and manufacturable, while the drift of its responses with time ('aging') is corrected by way of experimental measurements of the time variations of the parameters $\Delta\rho/\rho$, $\Delta c_{ij}/c_{ij}$, $\Delta\sigma/\sigma$, and ΔT (see below).

The measurement of film characteristics in adsorption is performed with the same five waves which probe the film (Fig. 2a). Solving the system of equations which relate the wave responses $\Delta v/v$ to the film characteristics ($\Delta\rho/\rho$, $\Delta c_{11}/c_{11}$, $\Delta c_{44}/c_{44}$, $\Delta\sigma/\sigma$, ΔT) yields the temporal variations of these characteristics upon feeding a gas and cutting off its supply (Fig. 2c), their equilibrium values, and the variations in the equilibrium values with time ('aging'). The measurement accuracy is 0.001 °C for temperature drops, and 0.1% for the remaining quantities. The time resolution is 1 s.

The application of the integral array is limited to gaseous analyts because surface waves vanish in contact with a liquid. The greatest number of sensors in the array is limited to five and cannot be increased owing to the overlap of neighboring channels and a lowering of the diversity of responses. To overcome these limitations, Ivan Anisimkin et al. [5] came up with the proposal to replace Rayleigh SAWs with normal modes in thin piezoelectric plates.

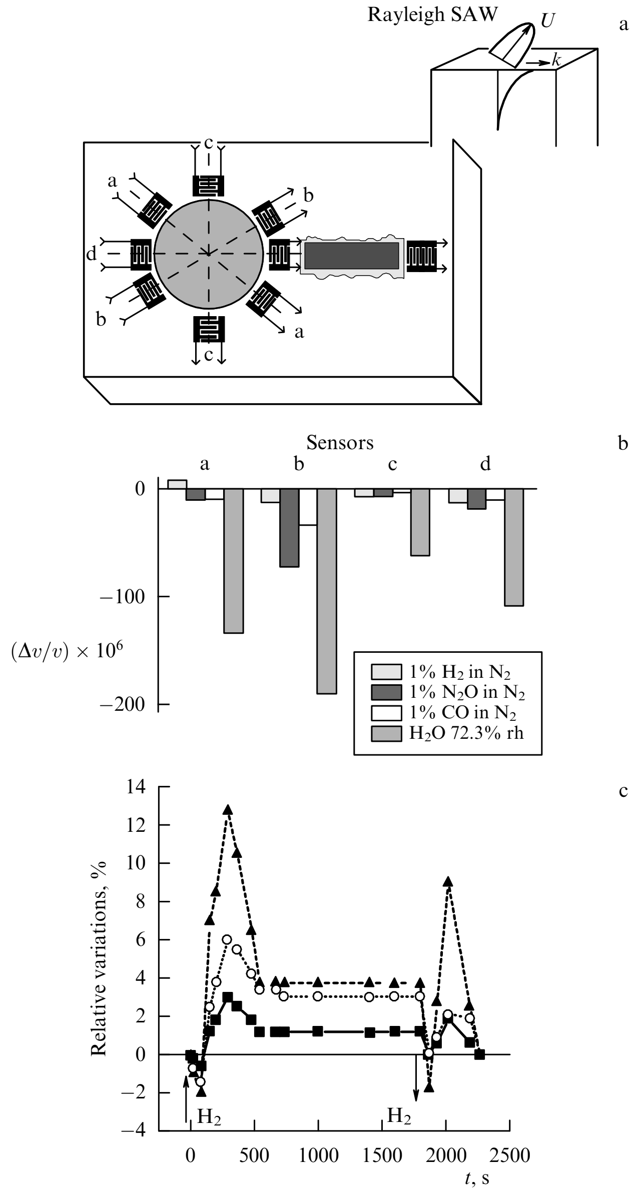

Figure 2. Multichannel SAW-based gas sensor array (adsorption analyzer): (a) schematic diagram of the array; in the inset — a Rayleigh SAW; (b) responses of the SAWs in different acoustic channels (sensors) to gases, and (c) temporal variations of the density $\Delta\rho/\rho$ (■) and the elastic moduli $\Delta c_{11}/c_{11}$ (▲), $\Delta c_{44}/c_{44}$ (○) of a $Pd_{0.97}Ni_{0.03}$ film in the adsorption of the 1% $H_2 + N_2$ gas mixture (the film thickness is 300 nm, and the temperature is 20 °C).

3. Acoustic sensors on the basis of normal modes in thin piezoelectric plates

For a long time, normal modes in plates found no practical implementation because it was believed that their excitation necessitated unreasonably thick samples with $h/\lambda \gg 1$ (h is the plate thickness, and λ is the wavelength). However, recent research [5 – 11] has changed this notion by demonstrating that this is true only of isotropic and weakly anisotropic materials, whereas the number of modes in strongly anisotropic piezoelectric crystals may amount to several dozen even for $h/\lambda \sim 1$ (Fig. 3). Moreover, the normal modes of piezoelectric plates turned out to be more diversified than in isotropic bodies and, since their energy is distributed over the entire plate thickness, they are only slightly absorbed by a liquid and may be employed for its detection via the upper and lower surfaces. In short, this type of acoustic waves has unexpectedly advanced to the forefront of the most attractive waves for use in acoustic sensors intended for various purposes.

Figure 4a provides an example of the application of normal modes for detecting liquids of equal mass of 378 mg

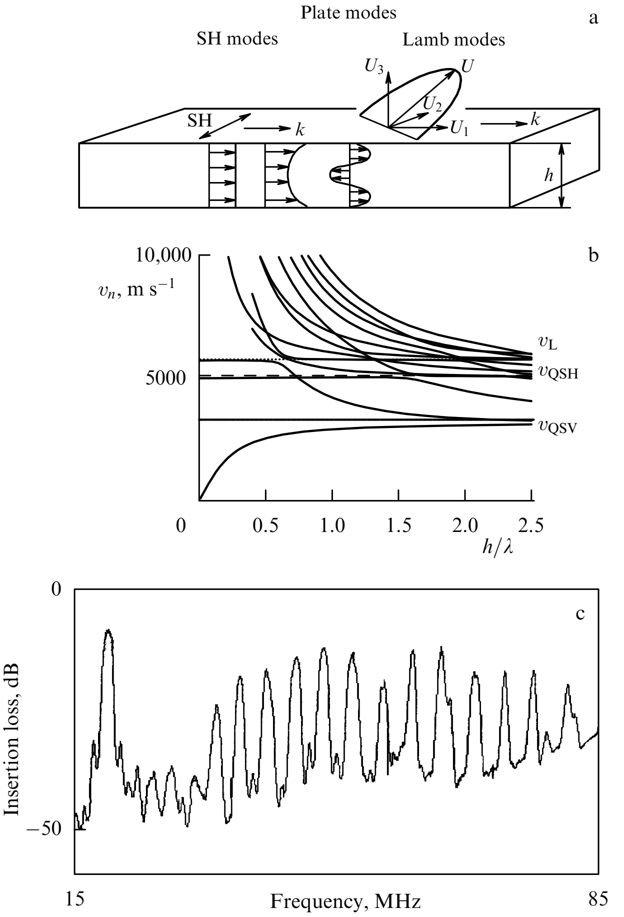

Figure 3. Main characteristics of normal modes in thin piezoelectric plates: (a) mode types: SH — shear horizontal modes, U_1, U_2, U_3 — Lamb elliptic modes; in isotropic plates $U_2 = 0$; (b) typical variations of mode velocity v_n (dispersion) as a function of the normalized plate thickness h/λ, n is the order of the mode, v_L, v_{QSH}, v_{QSV} — velocities of bulk waves propagating in the same direction in an infinite piezoelectric crystal (ST, x-quartz), and (c) frequency responses for normal modes in the plate 128°yx-LiNbO$_3$ with $h/\lambda = 2.475$.

with a different shear viscosity η: water ($\eta = 1.003$ mPa s) and glycerol ($\eta = 1450$ mPa s). The magnitudes of response (insertion loss variation, ΔIL) depend strongly on the serial number n of the mode, this magnitude being different for various liquids. By switching from one mode to another (from one sensor to another) it is therefore possible to form the proper set of output signals without resorting to sensitive coatings and without changing the direction of wave propagation. The number of responses in such an array is equal to the number of excited modes and is, as may be seen from a comparison with Fig. 2b, an order of magnitude greater than the number obtained by utilizing SAWs.

The same device is capable of detecting small compositional variations in a liquid from its viscosity. By utilizing the mode that exhibits the greatest difference of the responses to water and glycerol ($n = 22$ in Fig. 4a) it is possible to 'sense' less than 2 μl of glycerol in 760 μl of water, as well as to follow the kinetics of the dissolution of one liquid in the other (Fig. 4b).

4. Acoustic analyzer of liquids and thermal processes

The fourth example of employment of the multimode acoustic approach is provided by the analyzer shown in Fig. 5a. It utilizes both SAWs and bulk acoustic waves (BAWs), which probe a rod *1* with thermally insulated side faces at different distances d from the working surface. By the example of vaporization of the droplets of volatile compounds, the analyzer operation consists of the following steps [12]. When a droplet *2* is deposited on the working surface of the rod it begins to evaporate and cool. Inside the rod there forms a temperature profile $\Delta T(d)$ which is measured by the family of BAWs (Fig. 5b). Determined from the $\Delta T(d)$ profile are the temporal variations ΔT of the droplet temperature, the temperature gradient $(\Delta T/\Delta d)_{d=0}$ at the droplet–rod boundary, the amount of absorbed heat $Q = \varkappa S\tau\,(\Delta T/\Delta d)_{d=0}$, and the specific heat of evaporation $q = Q/m$ ($\varkappa$ is the heat conductivity coefficient, τ is the evaporation time, and m is the droplet mass). The time τ is measured from the disappearance (on deposition) and emergence (upon evaporation) of a surface wave propagating through the droplet. The evaporation rate is defined as

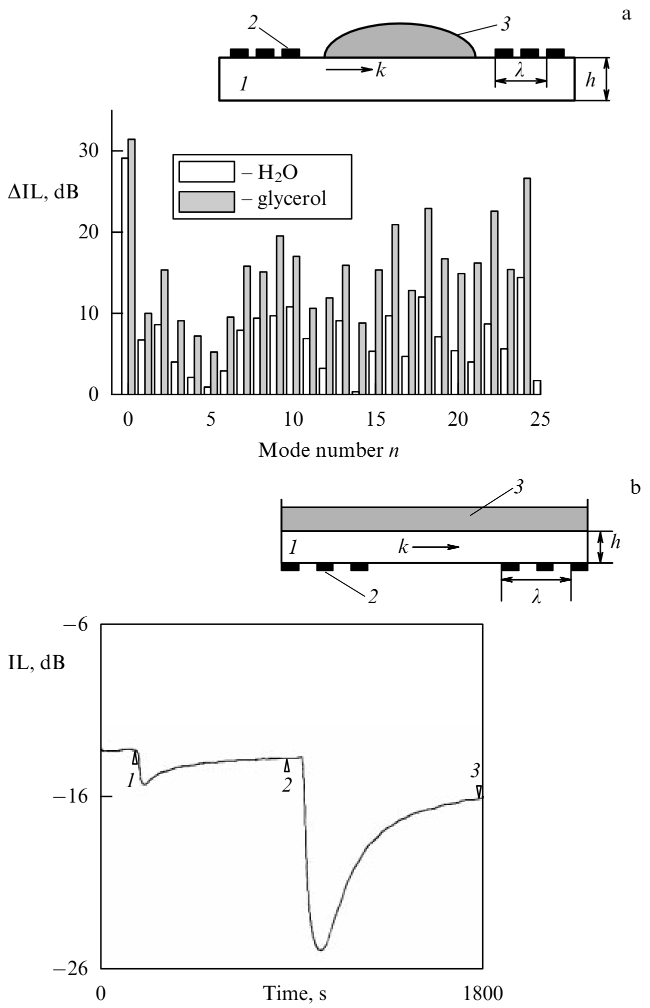

Figure 4. Multimode acoustic liquid sensor (viscosity meter): (a) normal mode responses to the action of liquids with different shear viscosities (a 128°yx-LiNbO$_3$ plate, $h/\lambda = 2.475$); (b) kinetics of glycerol dissolving in water (the $n = 22$ mode): mark *1* — injection of 2 μl of glycerol into 760 μl of water (the insertion loss variation for the mode is ΔIL$_{12} = 0.5$ dB), mark *2* — additional injection of 10 μl of glycerol (ΔIL$_{13} = 2.9$ dB). Shown on the right-hand side of the drawing are experimental setups: *1* — piezoelectric plate, *2* — transducers for the excitation and reception of normal modes, *3* — in Fig. 4a — a droplet on the working surface; in Fig. 4b — a pool with the liquid on the opposite surface.

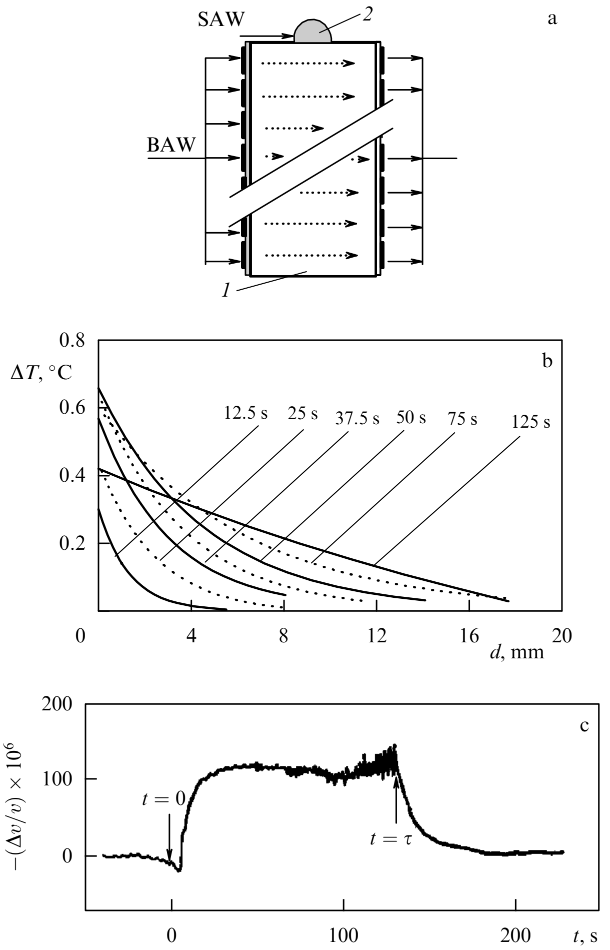

Figure 5. Acoustic analyzer of liquids and thermal processes: (a) schematic diagram of the analyzer: *1* — solid rod, *2* — microdroplet of the liquid being tested; (b) profiles of cooling during (< 60 s) and after (> 60 s) evaporation (the rod is a fused SiO_2, the droplet is ethanol 5 μl in volume), and (c) typical form of microdroplet evaporation kinetics: $t = 0$ — beginning of evaporation, $t = \tau$ — end of evaporation (the rod is an Si single crystal, the droplet is ethanol 8.6 μl in volume).

$u_{\text{evap}} = m/t_{\text{evap}}$, and the kinetics of the process is determined from the temporal dependence of the acoustic response $\Delta v/v$ (Fig. 5c).

As a result, the acoustic analyzer simultaneously measures four characteristics of the thermal process: its kinetics (with a resolution of 1 s), the temperature variation ΔT (with an accuracy of 0.01 °C), the released (absorbed) amount of heat Q (the threshold is 0.05 J or 30 J g^{-1}), and the evaporation rate u_{evap} (accurate to 0.5 mg s^{-1}). The minimal sample volume comprises 0.5 μl.

5. Acoustic sensor system for closed-space control

Controlling banks, museums, nuclear power stations, and housing is required to ensure their security. The internal conditions in the premises change owing to respiration, motion, and human perspiration, and special sensors acquire information about this.

Proceeding from the multimode approach, we developed a laboratory prototype of a control system consisting exclusively of acoustic sensors. These sensors register the temperature, relative humidity (rh), airflow velocity, and composition of the internal atmosphere from its thermal conductivity. By way of selecting the type of acoustic wave, each sensor is made sensitive to only a single parameter under monitoring. The output sensor signals are recorded by conventional receiving equipment. The signals do not require mathematical processing.

The temperature sensor selectivity is provided by utilizing the 15th mode of a yz-$LiNbO_3$ plate with a thickness $h/\lambda = 1.67$. This mode detects temperature variations at a level of 0.001 °C, but it is 'insensitive' to the mass load of the surface, the humidity, gas composition, and transport velocity of the ambient air.

For thermal conductivity ($\varkappa$) and air flow velocity (U) sensors, advantage is taken of Rayleigh SAWs in yz-$LiNbO_3$ with a small localization depth (0.7λ) and a high thermal sensitivity (10^{-4} °C^{-1}) at $T \approx 100$ °C — the small localization depth raises the speeds of sensor response, and the high thermal sensitivity permits us to apply the thermoconductometric principle of operation [13] and to eliminate gas-sensitive coatings. Maintenance of the sensors at a constant high temperature (100 °C) prevents water vapor from being adsorbed on the substrate and makes the sensors humidity- and temperature-insensitive.

The humidity sensor utilizes a thermally stable SAW in an acoustic line produced from ST, x-quartz with a gyroscopic PVA polymer film. To eliminate the sensor actuation under changes in $\varkappa$ and U, it is kept at a constant room temperature whereby the thermoconductometric effect is absent.

The sensor system characteristics are collected in Table 1.

Table 1. Characteristics of the acoustic sensor system.

Parameter	Range	Resolution	Stability/year
Temperature, °C	5–90	±0.05	±3%
Humidity, % rh	1–50	±0.5	±5%
	50–90	±1	±10%
Air flow rate, ml min^{-1}	10–300	±10	±5%
Thermal conductivity, mW mK^{-1}	5–45	±0.1	±5%

6. Conclusions

The implementation of high-sensitive acoustic waves in the analysis of liquid and gaseous media is a new promising avenue of acoustoelectronics, which is radically different from all traditional applications relied upon highly stable oscillations and substrates.

The broadening of the utilized-wave spectrum has consequently led to an increase in the number of measurable parameters, the circle of processes being investigated, and the stability of sensor arrays. At the same time, it has brought up new questions whose solution underlies the compatibility of the approach: it is necessary to find a way to improve the measured accuracy and spatial resolution of the acoustoelectronic sensors in view of the fact that their responses are formed throughout the whole path of wave propagation between the radiating and receiving transducers; it would be expedient to derive the criteria for a better optimized probing-wave selection in each specific case, considering that the set of crystallographic orientations and directions of wave propagation in piezoelectric crystals is unlimited, and how to weaken the unwanted effect of the vibrating surface of the acoustic sensors on the properties of the media under investigation and how to broaden the range of parameters being measured, which now is limited to mass, elasticity, viscosity, electrical conductivity, and temperature, need to be ascertained. We had intended to devote our further investigations to solving these questions, but fate decreed differently... .

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