SENSITIVE ELEMENT OF AN INTELLECTUAL SENSOR (FOR MEASURING PHYSICAL AND CHEMICAL CHARACTERISTICS)

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The work is devoted to the development of a sensing element of an intellectual sensor for the creation of an analyser of meteorological characteristics. The use of such a sensor gives new opportunities for creating devices for portable gadgets controlled by a microcontroller. The practical application of such devices opens the possibility of correct analysis of air pressure, temperature, and humidity data in real time.

Keywords: sensitive element, intellectual sensor, control, properties, meteorological characteristics.

Received 11.11.2022; Received in revised form 14.12.2022; Accepted 23.12.2022

1. Introduction

Today, scientific and technical research in the field of obtaining, processing, and transmitting applied information is becoming extremely relevant. The implementation of start-ups of this type contributes to a deeper understanding of natural phenomena, the development of correct methods and effective devices for conducting scientific research in general, and meteorological measurements of physical and chemical characteristics of the environment in particular [1, 2]. New developments contribute to the expansion of the range of solved practical problems related to the implementation of a fairly modern area of computer technology – the creation of components of information and measurement systems (IMS) based on new modern technologies [3, 4].

The aim of the work is to develop a sensing element of an intelligent sensor for creating a sensor of pressure and other meteorological characteristics. The use of such an element will make it possible to create a device for portable gadgets controlled by a microcontroller. Such devices will allow to analyze data on air pressure, temperature, and humidity in real time.

The subject of the study is the components of the sensing element of an intelligent sensor.

The object of the study was the process of obtaining an extremely sensitive layer of an intelligent sensor element.

The problems of the work were:

a) to develop a methodology for manufacturing a sensitive element of an intellectual sensor to create a pressure sensor and other meteorological devices;

b) to provide practical recommendations for creating an intelligent sensor that has a sensitive element.

2. Metrological compatibility of the measurement object

It is known that the system-wide conditions for assessing the accuracy of many information and measurement systems (IMS) are related to the issues of metrological compatibility of the system and determining the overall accuracy of the measurement result [5]. Metrological compatibility is the coordination of metrological characteristics of all functional elements that form a roadmap for the process of measuring, converting, transmitting, and processing information. The metrological compatibility of functional elements is achieved by establishing a single set of metrological characteristics, uniform forms of their representation, and methods of normalization, evaluation and control methods.

The main condition for the metrological compatibility of functional elements is to ensure the balance of errors of the methodological, algorithmic, hardware and software tools of the system, which is on the maximum permissible error level of the task result. For standard and smart sensors, only the sensing element is common. The sensing element provides information about the physical and chemical characteristics of the sensor.

Note that in addition to the sensing element, an intelligent sensor contains (Fig. 1): a sampling circuit that converts an analogue signal into a digital signal; a computing element that analyses measurement data obtained with the help of a sensing element; an interface with the external environment that allows the device to exchange information with other components in information and measurement systems.

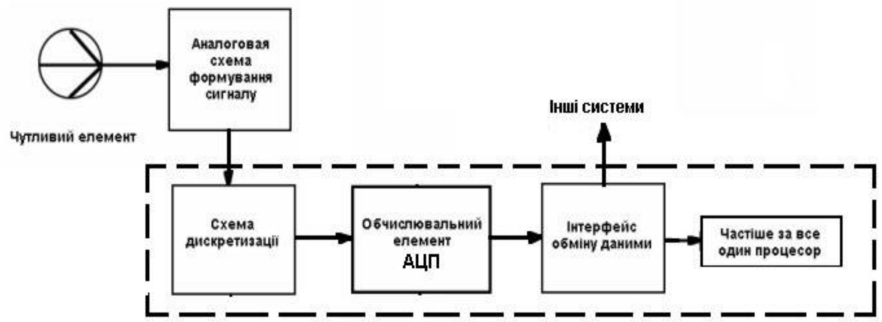

Fig. 1. Diagram of an intellectual sensor.

When solving a real-world problem related to the processing of an intellectual sensor signal, some components are necessary. Such as: physical characteristic to be measured; the relationship between the measured physical characteristic and a fixed parameter value; expected frequency spectrum of the signal and probable sources of environmental noise; physical characteristics of the working environment; conditions of occurrence of errors and appropriate methods of their prevention; calibration requirements; requirements for the system interface; maintenance requirements.

An intellectual sensor should reliably detect typical errors, process and eliminate deficiencies; predict dangerous conditions and deficiencies, and ensure correct system operation, as well as record everything in protocols.

Figure 2 shows an example of a block diagram of an intelligent sensor that can be used to measure temperature.

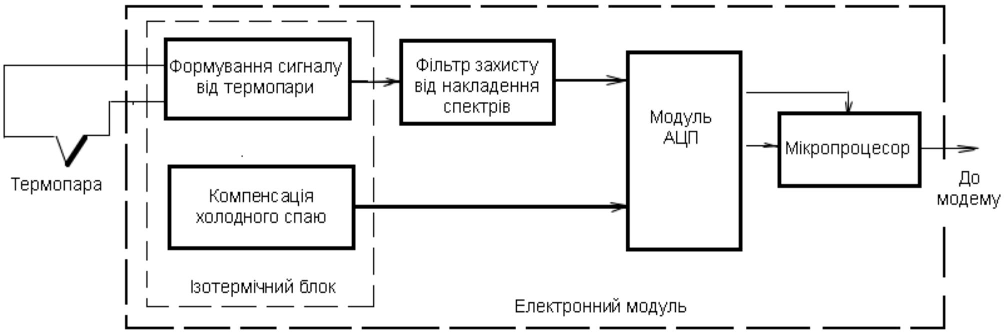

Fig. 2. Functional diagram of an intellectual sensor with a sensitive element (like thermocouple).

In this example (Fig. 2), three main requirements for creating an intellectual sensor into account are taken: minimizing power consumption to reduce self-heating; a high-quality and compact printed circuit board, where interference and electrical noise are minimized; and high-quality power supply decoupling to reduce electrical noise.

3. Experimental methodology

A large number of scientific papers are devoted to the methods of management, diagnostics, and control of physical and chemical parameters; in particular, environmental characteristics (see, for example, [6-8]). Among the described methods, temperature and radiation methods are distinguished. Today, the most promising approaches are those that involve the use of the latest technologies - nanotechnology. In particular, we are talking about devices that exploit quantum-dimensional effects [1].

In this context, based on a unified approach that classifies various manifestations of quantum-dimensional (in particular, clustered [6]) phases of matter, we have obtained the results of studying not only the mechanisms of formation of a film clustered hetero structure (FCHS), which is the main component of an intellectual sensor, but also the use of the latter in sensors and devices (based on them) for measuring the physical and chemical properties of the environment. For this purpose, we proposed an IMS for determining the morphological parameters (mass, layer thickness, geometry) of the components of the smart sensor – PCGS. Software tools and an interface for the IMS operation were developed to perform quantitative assessments.

The material selection and thickness of the films were chosen taking into account the physical and chemical properties required for the measurements. Preference was given to those films that have a band structure commensurate with the base crystal substrate and appropriate radiation and temperature resistance. The technology of manufacturing such films is a complex of complicated technological processes, one of the stages of which is measuring film thickness and determining its morphology – micron and submicron structure.

We have studied the formation of silicon-based PCGFs synthesized from the vapor phase on a single-crystal Si wafer substrate. The single crystals with an area of 0.28 cm^2 and a thickness of several microns were used as a substrate. The substrate was applied to the surface of Ag electrodes of crystalline quartz, which is the basis of the piezoelectric resonator. The crystal structure of the FCHS corresponded to a "highly blurred texture" (defined by us as a cluster raster on Si). The studied samples of the nanocluster subsystem were synthesized by the method of open evaporation of silicon powder in a vacuum. Their thickness was controlled using a micro interferometer type MI-4 [1].

Electron microscopic studies of the film morphology, i.e., its submicron structure, were performed using an atomic force microscope.

The process of forming FCHS was carried out using a piezoelectric quartz sensor with a constant frequency of 1.67 MHz/mm. The sensor was connected to a standard quartz film thickness gauge (QFTG-5, developed at the Institute of Semiconductor Physics of the National Academy of Sciences of Ukraine named after Prof. V. E. Lashkarev) with a pre-programmed scale (for a single silicon crystal). The graduation chart was designed in such a way that it could be used to measure the small thickness of the silicon deposited on the substrate. Operational control of the FCHS thickness was carried out using the quartz resonator method with elements of the technique described in detail in [1]. The principle of operation of the QFTG -5 is based on measuring the shift in the resonant frequency of the quartz sensor depending on the material layer deposited on it. The system allows measuring the thickness of a vacuum-coated coating with an accuracy of 0.1 nm.

The purpose of the QFTG-5 is: a) quartz control of the physical thickness of the material in the process of vacuum coating of a film structure on a substrate;

b) the measurement based on the effect of shifting the resonant frequency of a quartz crystal when its mass changes. The mass of the crystal is increased by applying a material to its surface. KVTP-5 is a programming device. The appearance of QFTG-5 is shown in Fig. 3. The device allows controlling the process of sputtering the material onto the substrate surface using software based on the SciLab package. The QFTG-5 is equipped with a keyboard and a multifunctional display for visualising operating modes and programming the parameters of a thin film deposition process.

The procedure for sputtering a layer of material onto a substrate was based on an analytical relationship that corresponds to the mathematical model of the film texture. In this way, complex, multilayer films can be specified. Each layer is characterised by a specific thickness and type of coating, the density of the material to be sputtered, and the evaporation coefficient in the chamber. Based on these parameters and the relative change in the frequency of the quartz sensor, the film thickness is calculated.

The BF-100 quartz crystal generator is designed to extend the service life of industrial-standard quartz crystals with operating frequencies of 5 and 6 MHz. The generator ensures the operation of the quartz crystal, generates and/or amplifies the signal for its transmission to the QFTG-5. The BF-100 generator operates with a cable at a distance of 1 meter from the crystal. The cable connecting the generator and the controller can be up to 4 meters long. The BF-100 oscillator has a modification of the housing with holes for mounting on a flat substrate surface. A water-cooled crystal holder of the BFAG-140 type is used as part of a quartz coating thickness control system. The sensor is designed in such a way that the replacement of the quartz crystal is as simple as possible. Standard quartz crystals with a geometric dimension of 0.55 mm are installed in the BFAG-140 holder. The BFAG-140 is equipped with a water cooling system, which allows controlling the application of the film coating at temperatures up to 300°C.

Key features of the QFTG-5: a) diameter of water cooling pipes up to 5 mm; b) length of pipes up to 500 mm; c) housing material – stainless steel; e) heat-resistant coaxial cable. The crystal holder is installed in the vacuum chamber through a sealed inlet. Two inlets for tubes with a diameter of 5 mm and one electrical inlet are connected to the BFAG-140. The water-cooling tubes can be bent. This allows you to orientate the position of the crystal in the chamber. On the outside of the chamber, a generator is connected to the electrical output to generate and amplify the signals coming from the quartz crystal.

Fig. 3. Quartz film thickness gauge (five version) QFTG-5.

The signal from the generator is fed to a measuring device that calculates the thickness of the applied film by changing the oscillation frequency of the quartz crystal. The holder can be optionally equipped with a pneumatic flap to prevent dust from entering the crystal during different coating modes. A sealed inlet is necessary for the introduction of tubes that provide water cooling of the quartz holder inside the chamber. The QFTG-5 is supplied with a SciLab-based software package. The automated system of QFTG-5 operation is provided with a protocol. The SciLab-based service program for a personal computer allows deposition of films of more complex morphology. The software allows to create an unlimited number of layers and films using any materials. It provides storage of all configuration data and its editing. Graphs allow you to visually illustrate and track the coating process.

In the chamber where a vacuum of ~ 1-10^{-6} Torr is maintained, there are plates of a quartz gauge on a sital base, a "boat" made of tantalum foil, where the evaporating silicon is located. A voltage is applied to the tantalum through the contacts, which heats the Si to the evaporation temperature. The current varies between 10 and 15 A. By setting the current strength, it is possible to obtain films of different thicknesses and morphological structures. Another possibility for heating the tantalum "shuttle" is to apply voltage pulses to the electrodes through the contacts, the duration of pulses is set by a timer. The frequency of the pulse sequence varied from 0.1 to 0.01 Hz. The frequency of the pulses was such as to ensure the restoration of vacuum conditions in the vacuum chamber between discharges. The energy emitted during discharge varied from pulse to pulse. To control the discharge parameters, a scheme was developed to measure the energy released at the electrodes during one pulse. A micrograph of the film (index 7 in Fig. 4) was obtained using a tunneling microscope. The cluster raster of the film on a silicon substrate allows us to estimate the geometric dimensions of deep submicron Si formations (with dimensions of 15÷20 nm) – nanoclusters that were shaded by carbon. To obtain particularly thin films with a thickness of ~25-50 nm, the energy transfer emitted when applying current to tantalum must be sufficient to keep the flux density of evaporated Si atoms constant.

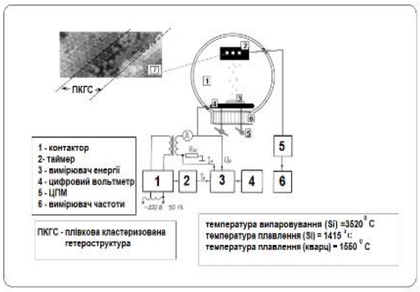

Fig. 4. Scheme of the experimental setup for obtaining a film as a super-sensitive element.

4. Methodology for measuring the thickness of the sensing element of an intelligent sensor

As noted above, the main component of the QFTG-5 is a quartz resonator plate that determines the frequency of a stable oscillator. Since the generated frequency depends on the mass of the quartz resonator, if the generated frequency deviates, taking into account the calibration, it is possible to determine the thickness of the film deposited on the quartz substrate, and hence on samples under the same conditions. The mass of the layer deposited per unit surface area of quartz Δm and the change in the frequency of the quartz resonator Δf are related by the simple relation:

$$\Delta m = \frac{m_g}{f_g} \Delta f \tag{1}$$

where m_g is the mass of the quartz substrate of a unit area; f_g is the resonant frequency.

The sensitivity of the quartz resonator method is quite high. For example, for a crystal with a resonant frequency f = 5 MHz and a mass m_g = 100 mg, the sensitivity is equal to

$$\frac{m_g}{f_g} = 0.02\frac{\text{mcg}}{\text{Hz}}$$

That is, by recording a 1 Hz shift in the frequency of the quartz resonator, it is possible to control the increase in film mass in a hundredth of a microgram. It should be noted that the quartz resonator is quite sensitive to temperature changes. To take into account the resonant frequency shift with temperature in our measurements, we used quartz resonators that were selected based on the results of measurements of the temperature dependence of the resonant frequency. We have tested more than 40 resonators of the RK170 and RK171 brands with a natural resonance frequency of 5...10 MHz. According to our temperature dependences, five percent of the resonators had a weak frequency dependence on temperature.

Approximately 75% of the resonators were characterized by a decreasing dependence. In addition, we identified (rejected) approximately 20% of the resonators that were characterized by broken dependencies with a clearly defined extremum. In addition to all the above, taking into account that the accuracy of the method of measuring the mass of a deposited film layer on a substrate using a quartz resonator is determined by the stability of the oscillation frequency, we have proposed an electrical circuit of a stable quartz oscillator. The latter was designed to operate in the frequency range from 1 to 100 MHz and was assembled on a high-quality, ultra-sensitive, high-frequency amplifier with a quartz resonator. One of the transistors is an emitter buffer repeater, which reduces the influence of the load on the parameters of the generated signal. The oscillator amplifier is designed as follows: common collector – common base – common collector. To neutralize the capacitance of the quartz holder, another transistor was used, which was connected to the negative feedback network. By the way, negative feedback stabilizes the output oscillation amplitude. The correction capacitance increased the stability of the generation at high frequencies. The generator circuit achieved a wide bandwidth of the amplifier signal. That's why the amplifier had an incredibly small additional phase roll-off up to frequencies of about 100 MHz. And this, in turn, resulted in high stability of the generation frequency. The sum of the input and output impedances of the amplifier is less than the equivalent dissipation resistance of a quartz resonator.

The generator was mounted on a small board and placed in a vacuum chamber. The resulting signal is powered and sampled through a vacuum connector. This arrangement made it possible to significantly reduce the length of the conductors connecting the quartz resonator and reduce noise crosstalk. When silicon evaporates, energy is released, some of which occurs in the form of radiation. The latter can heat up the quartz resonator plate. Our measurements have shown that the temperature of the substrate increases slightly during the session. Recovery takes place in 2-3 minutes. In order to avoid the parasitic effect of the temperature drop of the frequency on the measurement results, we used exactly those resonators that had a weak temperature dependence.

The relative instability of the oscillator frequency is a tenth of a μHz.

Fig. 5 shows a dependence illustrating the kinetics of the resonance frequency of a quartz crystal after film sputtering. The frequency drift after the coating is due to the adsorption of the residual gas by the deposited silicon layer. By using crystals with a weak temperature dependence, we were able to bring the accuracy of the frequency drift from the resonant value Δf to 1 Hz. The error of the method is due to the accuracy corresponding to the deviation from the resonant frequency Δf and the accuracy in determining the mass of the crystal m_g. In our case, the error in measuring the thickness of the coating corresponded to 5%. The quartz resonator method allowed us to develop a technique for manufacturing films with a controlled and predetermined thickness.

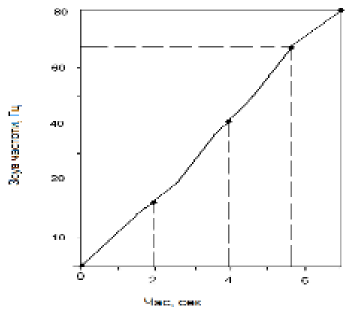

Fig. 5. Kinetic curves of the synthesis of films on a silicon substrate obtained using a piezoelectric resonance sensor.

Table 1 shows the results of measuring the film thickness using the quartz resonator method.

Table 1

Comparison of silicon film thickness measurement results

Sample number	Quartz Δ *f*, Hz	Film thickness according to quartz data, mcm
1	80	1,034
2	105	1,368
3	120	1,519
4	136	1,748
5	156	2,006
6	200	2,584
7	320	4,134

5. Conclusions

Thus, the method described in this paper allowed us to measure the thickness of the substance (silicon) layer during the deposition process in its pure form, i.e. without taking into account the mass of adsorbed gas. This is very important for determining the target thickness in ion flow experiments.

The above methodology for controlling the process of forming a certain film thickness with a deep submicron morphology as the basis of a component of the sensing element of an intelligent sensor opens up new opportunities for creating high-performance devices of a new generation. The practical implementation of this technology makes it possible to highlight the success of the technology for producing ultrasensitive films and devices based on them.

According to the method described above, a sensing element for an intelligent sensor was created and used to measure meteorological characteristics, the mathematical and computer model of which will be presented in the following papers. In the process of implementing the described method of creating a sensing element for an intelligent sensor, the following should be taken into account.

First. The elastic properties of the sprayed substance differ from the elastic characteristics of the substrate.

Second. The density of the sputtered substance is less than the density of the same substance in a massive sample.

Third. The shift in the resonance frequency is caused not only by the mass of the deposited layer, but also by the stresses that occur at the interface between the substrate and the film material.

The practical significance of the research results is that the proposed integrated approach allows using this method as a method of non-destructive testing of the surface of an intelligent sensor with nanocluster morphology.

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