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MICROPROCESSOR SYSTEM FOR INVESTIGATION OF SENSOR CHARACTERISTICS ON THE BASIS OF POROUS SILICON

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Development of integrated sensors is a promising scientific and technical direction to create components for measurement and information systems. Among the different types of analyser one of the places are occupied by sensors of hydrogen containing compounds in gaseous and liquid condition (especially poisonous and carcinogenic). Fixing the presence of a substances in the gas or liquid environment is an actual task for scientific, industrial and medical application areas.

The main goal of this paper an inexpensive microprocessor system for measuring and analyzing electrical properties of nanocomposite sensing material such as porous silicon on the base of current and capacity-voltage characteristics and its hardware and software applying is represented.

The microprocessor system is designed for measuring sensors current-voltage and capacity-voltage characteristics based on porous silicon and nanocomposite structures and consists of the following units: power supply unit, DC voltage regulator, control and measurement module, RLC-meter E7-22, UART/USB and RS232/USB converters, measuring cell, PC. The software of the microprocessor system for measuring consists of the software for the control and measurement module implemented on the Atmega 128 microcontroller and the main device management software for the PC. The main program algorithm and software are implemented in the C# programming language in the Visual Studio 2017 environment

The results of the comparison allow us to conclude that the implemented modules of the microprocessor system for sensors I-V- and C-V characteristics testing can be used in the development of portable environmental monitoring devices, to study porous silicon sensors using them.

Keywords— porous silicon, nanocomposite; sensor, microcontroller, microprocessing system, current-voltage characteristic

Introduction

The rapid development of nanotechnologies and physics of nanoscale materials in the recent years leads to the creation of electronic devices that operate on new physical principles with new promising capabilities. And it allows to create defect-free materials with fundamentally new physic and chemical properties, as well as new classes of devices with a characteristic nano-dimensional mode.

Porous silicon (PS) is considered as a new class of nano-dimensional objects [1, 2]. The study of PS properties is an actual task as a result of the applying of this material for the creating of emitting and detecting electronics structures. One of the methods is the modification of the PS surface [3]. Nanostructured material ZnO with p-type conductivity on the PS surface PS-pSi by electrochemical method was obtained, without preliminary processing of the substrate. Also Au contact on the PS surface as a collateral surface coating was deposited.

The developed surface of PS is a good substrate for nanocomposite based on ZnO. Composite of zinc oxide - porous silicon (ZnO-PS) structure demonstrate a change in the spectrum of electromagnetic waves from 1.4 to 3.3 eV with a changing of electric properties. Different methods for producing nanoscale ZnO samples of various types are directed on the use of electronic, optical and piezoelectric properties [4].

Zinc oxide (ZnO) is a semiconductor material that is most intensively studied at present. Being transparent in a wide spectral region, ZnO is highly resistant to radiation and is relatively cheap, which makes it attractive for use in microelectronics. A great interest is also the integration of ZnO with silicon technology, which allows combining the unique functionality of these materials when creating photoconverters on silicon substrates and, in particular, in thin-film solids some elements [5]. On the other hand, porous silicon-ZnO composites have been used for white light emission and to tune ZnO grain size for possible sensing applications [6]. The tasks of micro-nanosensoric areat present time are to stimulate the study of thin films of zinc oxide. Increased interest in thin ZnO films is associated with the search for active materials for sensitive elements of nanosensors [7].

Development of integrated sensors is a promising scientific and technical direction to create components for measurement and information systems. Among the different types of analyser one of the places are occupied by sensors of hydrogen containing compounds in gaseous and liquid condition (especially poisonous and carcinogenic). Fixing the presence of a substance in the gas or liquid environment is an actual task for scientific, industrial and medical application areas [8].

Complex of obtained experimental data such as current-voltage characteristic, electrical conductivity, capacitance-voltage measurements on the ZnO(Au)-PS structures in the wide voltage range in the air and under adsorption of hydrogen containing compounds provided on microprocessor system can determine the mechanisms of conduction and sensory characteristics of the objects.

The main goal of this paper is represent an inexpensive microprocessor system for measuring and analyzing electrical properties of nanocomposite sensing material such as porous silicon on the base of current and capacity-voltage characteristics and its hardware and software applying. The results obtained in [9] are taken as a basis.

All this is comparing to high quality electronic complex for analyzing of semiconductor structure [10], Keithley 4200A-SCS Parameter Analyzer [11] and 4140A Agilent Semiconductor Parameter Analyzer [12].

Microprocessing System Hardware

The microprocessor system (MS) is designed for measuring sensors current-voltage and capacity-voltage characteristics (I-V and C-V) based on porous silicon and nanocomposite structures. The functional scheme of the microprocessor system is presented on Fig. 1.

The MS consists of the following units: power supply unit, DC voltage regulator, control and measurement module, RLC-meter E7-22, UART/USB and RS232/USB converters, measuring cell, PC. It is possible to connect the Keithley 2100 multimeter in current mode. In this case, the multimeter is connected to the X1, and the measuring R_m through the X2 is shortened. The multimeter control is programmed through the USB interface. If the Keithley 2100 multimeter is not used, then the X1 is shortened, and the X2 split is break.

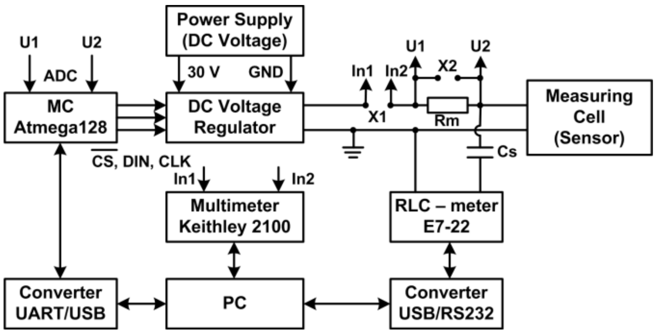

Fig. 1. MS functional scheme of semiconductor nanostructures I-V and C-V characteristics measurement

The control and measurement module is implemented on the basis of the Atmega128 microcontroller. The main functions of this module are: DC voltage source control, realized on the basis of DAC AD5541; voltage measurement U1 and U2, PC interaction via UART for receiving commands and data from a PC, and transmitting measurement results to PC.

Atmega128 is an 8-bit low-power AVR microcontroller with RISC architecture [13]. Its peripherals include, in particular, two 8-bit timers/counters, two 16-bit timers/counters, a real-time counter with a separate generator, two eight-bit PWM channels, six PWM channels with programmable bit (2 ... 16 digits), 8-channel 10-bit ADC, two programmable channels USART, 128 kB internal reprogrammable flash memory, 4 kB internal static RAM.

The DAC AD5541 connecting scheme to the microcontroller is shown in Fig. 2. The reference voltage Vref = 2.048 V for the DAC is formed using a reference voltage source (chip REF19x). Microchip AD5541 is a single-channel 16-bit DAC powered by 5 V [15]. The voltage at the DAC output is determined by the expression:

$$V_{Out} = \frac{V_{ref} \cdot Code}{2^N}, \qquad (1)$$

where Code is the decimal data word loaded in the DAC register, N - DAC bit. The AD5541 has a triple wired serial interface that is compatible with the standard SPI interface. The ADUM1300 Digital 3-Channel Insulator performs a solution between the microcontroller terminals and the DAC (CS, SCLK, DIN).

The DAC control is performed by a microcontroller, which generates a code within $0 \leq$ Code $\leq 2^N-1$. The range of this code change corresponds to the change in the output voltage DAC $0 \leq V_{Out} \leq V_{ref} * (2^N-1)/ 2^N$. Output voltage DAC is applied through the filter (R5C3) to the input of the voltage measurement circuit U1 and U2, shown in Fig. 2.

This voltage is applied to the non-inverting input of the operational amplifier U1:B (AD522), where it is multiplied by the magnitude of the gain K=(1+R6/R7), and then applied to the base of the transistor VT1 (BC547) to control it.

The OUT_ + and OUT_- terminals have a measuring cell with the structure under investigation. It allows to measure VA- and VF-characteristics for both positive and negative voltages with unipolar power supply.

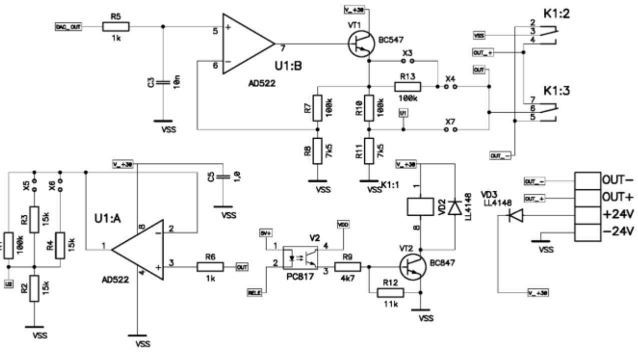

Fig. 2. Principal scheme of the voltages U1 and U2 measuring

Outputs U1 and U2 are connected to the ADC microcontroller Atmega128 inputs. Another measurement voltage OUT is applied to the non-inverting input of the U1: A operating amplifier, which acts as an emitter repeater. Its output is connected to the resistive divider R1R2R3R4, whose fission factor can be changed by the shortcut of the X5 and X8 jumpers. From the output of the divider, the voltage U2 is applied to another ADC input.

The power supply scheme of the voltage meter U1, U2 provides power to the measurement circuit via USB. This uses DC / DC converter, which converts the input voltage to 5 V into the output voltage of 24 V. Its power is 1 W.

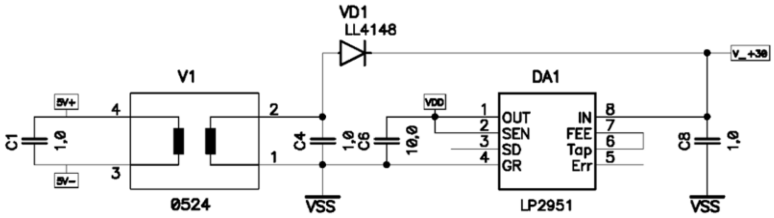

Fig. 3. The power supply scheme of the voltage meter U1, U2

The capacitance measurement of investigated structures at different bias voltage is performed using RLC-meter E7-22 [14], which RS232 output is connected through RS232/USB converter to the PC. Investigated structure through a C_S split capacitor is connected to RLC-meter input. RLC-meter E7-22 allows to measure alternating current at 120 Hz or 1 kHz. The main error of active resistance measuring is 0.5%, and the inductance L and capacity C - 0.7%. The smallest range of capacitance measurements is 2000 pF, and the largest one is 20 mF [14].

Microprocessing System Software

The software of the microprocessor system for measuring the VA- and VF-characteristics of the investigated structures consists of the software for the control and measurement module implemented on the Atmega 128 microcontroller and the main device management software for the PC. The software of the system module realizes a constant voltage control algorithm, fix the voltage on the measuring resistance R_m, transmits measurement data to the computer and accepts data and commands from the PC.

The algorithm for measuring the VA- and VF-characteristics consist in automatic voltage increase at the output of the DC regulator from the specified value of the voltage min U1 to the value max U1 with the step ΔU1. The variation of the input voltage U1 is 0 ... + 28 V. The minimum voltage step is 0.025 V.

Each of the characteristics measurement cycle includes the next procedures: - setting the voltage at the output of the regulator of constant voltage $U1_t$; - delay time 1 s before measuring voltage $U1_t$; - multiple measurements of the voltages $U1_t$ and $U2_t$ and the capacitance C_t (time delay between each measuring 0.1 s); - averaging of the measured values of $U1_{(m)t}$, $U2_{(m)t}$, $C_{(m)t}$ and their transfer to the PC.

The main program algorithm and software are implemented in the C# programming language in the Visual Studio 2017 environment. It allows to establish a connection between the PC and the microprocessor system; configure the virtual com-port settings; transfer control commands to the microprocessor system and RLC-meter E7-22; get the measurement results from them; store the received data in files and build graphics. The interface of the main program is presented in Fig. 4. It consists of a menu bar (File, Setup, Help) and several areas.

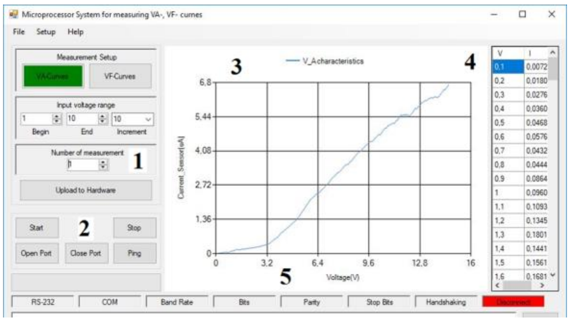

Fig. 4. View of the main program interface

In the 1st area (Measurement Setup) parameters are set: the measured characteristic (VA Curves or VF Curves); Input voltage range - minimum, maximum value, step; Number of measurement. All data for these parameters is selected from their windows. When you click the "Upload to Hardware" button, the values of all parameters will be transmitted through the virtual Com-port to the microcontroller. This requires that the Com-port be opened. To configure the parameters of the RS-232 interface, go to the Setup menu and in the window that opens (Fig. 4), select the RS232 submenu.

In the 2nd area there are the Start, Stop, Open Port, Close Port and Ping buttons. To activate the start of measuring it is necessary to click on the Start button of the program interface. Measurement can be stopped by pressing the Stop button.

The visualization window (area 3) allows displaying the measured or recorded data in the graphs mode I-V or C-V characteristics. In the 4 area are displayed measured values or recorded in the file. Fig. 4 shows the graph of measured I-V characteristics and their numerical values for PS-ZnO.

In the 5 area, RS-232 interface parameters are displayed, which are valid at the present time (the last parameters with which the program worked is stored).

Porous Silicon Sensor Preparation

Layers of PS were obtained by electrochemical etching of n-, p-types monocrystalline silicon in HF (48%)-ethanol solution. We used (111) Si wafers with thickness of about 400 μm and a diameter of 76 or 100 mm respectively. Specimens of n-type conductivity were irradiated by white light during electrochemical etching. Porosity of obtained layers with thickness of about 20-30 μm was equal to 40-80%. For measurement we have used samples of PS grown directly on silicon substrates p-type doping with a resistivity 10 Ω • cm (KDB-10). Both the surface of single-crystal wafers was mirror polished.

A Teflon chamber is the laboratory electrolytic cell to form a PS. Anode is a monocrystalline silicon plate and cathode - grid of platinum or other resistant to hydrofluoric acid conductive material. As the electrolyte was used ethanol solution of hydrofluoric acid with the ratio of components C_2H_5OH: HF = 1: 1.

For sensory study of PS electrochemically etched PS-pSi (p-type silicon substrate KDB-10 j = 5 mA/cm^2, t = 10 min) sample was selected.

Etched substrate was separated on two single samples of 1 cm^2 square approximately. Then two investigated specimens were selected to forming two sensors: nanocomposite PS-ZnO and PS-Au.

Nanostructured material ZnO with p-type conductivity on the PS surface was deposited by electrochemical method without preliminary processing of the substrate. The method of obtaining a nanostructured ZnO material with p-type conductivity led to an attempt to grow electrochemically nanostructure in electrochemical cell with electrodes from a solution of reagents, where voltage is applied to the working electrode. As an acting electrode-cathode porous silicon was used, and as the second electrode-anode - a plate of graphite. The nanowires were tried to obtain by deposition in water equimolar solution of $Zn(NO_3)_2 \cdot 6H_2O$ and hexamine ($C_6H_{12}N_4$) at a concentration of 10-30 mM, pH 7 ± 0,1 and 0,1 M KCl [16].

For another sensor Au thin films on PS surface were obtained by thermal precipitation. After precipitation semi-transparent Au conductive film was annealed at 550° C.

Contact to the porous layer about 2 mm in diameter was obtained by electrically conductive graphite glue. A lateral scheme of the electrodes configurations for current-voltage (I-V) and capacitance-voltage characterizations was applied.

PS surface morphology after Au thin film deposition with the help of a scanning electron microscope REEMA-102-02 are shown on Fig.5. Metallic surface after Au deposition cross-section scan by compo self-emitted electron image confirm the edge of etched Au contact.

Microstructure analyses of PS-ZnO samples were carried out using a scanning electron microscope (SEM) JEOL JSM 7800F equipped with an integrated EDXS spectrometer (Bruker Quantax 400, XFlash 6|30 silicon drift detector). The mineralogical characterization was performed with a SEM Quanta 600 (FEI Company) equipped with parallel EDXS spectrometers (Bruker Quantax 200 with two Dual XFlash 5030 EDX detectors). The element content on the studied surfaces was obtained after SEM-based image processing - Mineral Liberation Analysis (MLA) [17] employing the MLA-software package (JKTech) [18].

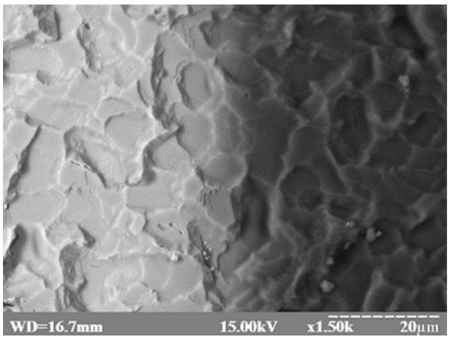

Fig. 5. PS-Au scanning electron surface image

The results of studying the PS surface morphology before and after ZnO film deposition with the help of a scanning electron microscope, as well as component-analysis results, resulted in a presence of ZnO compound on the surface (PS-pSi - 30.5%) are shown on Fig. 6. After electrochemical etching of a crystalline silicon surface, PS is matte and brown and there is clearly triangle-like structured surface. Color surface after ZnO deposition by electrochemical etching is more matte and cross-section scan electron image confirm that the PS-ZnO nanocomposite has disordered covered by ZnO pores.

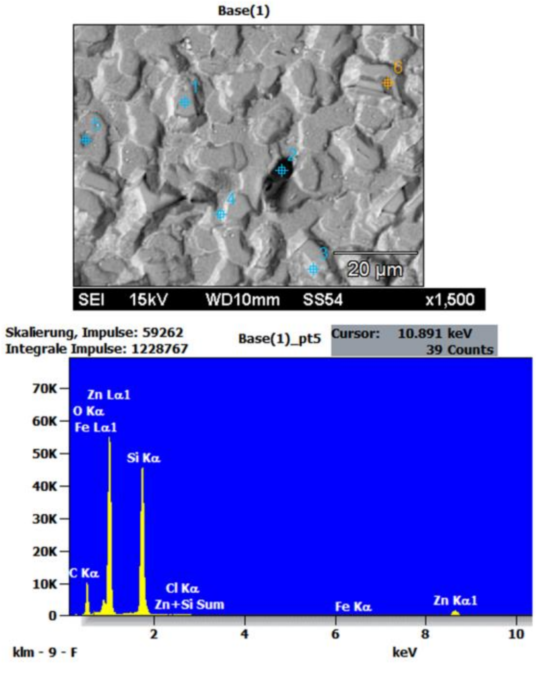

Fig. 6. ZnO-PS nanocomposite scanning electron surface image and mass-analysis result

Emissions of gases such as methane and propane led to a deviation from the example of the studied structure as a metal-semiconductor-dielectric. And the adsorption effects may be due to the lower polarization of gas molecules when interacting with the PS developed surface.

For the structure of PS-Au (Fig. 7, a), there is capillary adsorption in the pores of the PS, which leads to a change in the dielectric constant of the porous layer. PS can be considered as a complex dielectric material, where its dielectric constant depends on various components such as air, water and hydrocarbon vapours seeping into the pores. As a result, the capacity of such a sensory structure leads to a change in the C-V curves under the action of external adsorbents, which indicates a change in the dielectric constant of the porous structure based on the size and molecular weight of the absorbed molecules.

For PS-ZnO we observe different characteristics (fig. 7, b), which is due to the varizone structure of the sensor and differences in the effect of hydrogen-containing gases.

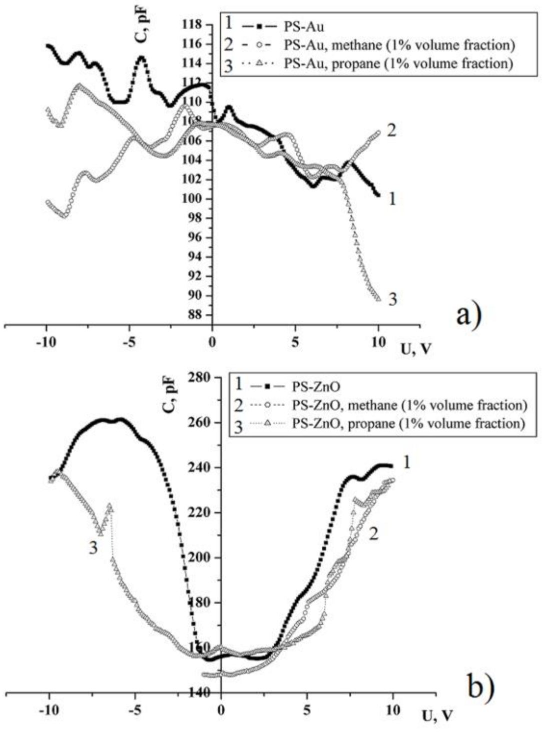

Fig. 7. C-V characteristics measurement

Therefore, the investigated material such as PS-Au and PS-ZnO, especially that modified by ZnO clusters (fig. 8, b), represent itself as a hybrid nanocomposite material, the properties of which can be controlled, in particular the nature and size of the structure.

Establishing the interaction of components of hybrid materials on the basis of changes in the resulting influence of surrounding components allows to expand the general characteristics of objects with the next application as new sensible sensors.

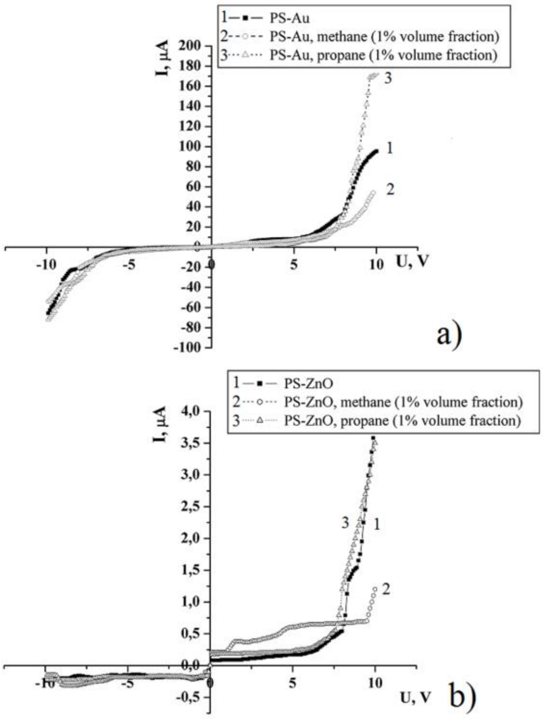

Fig. 8. I-V characteristic testing results of measurements

Conclusion

The microprocessor system functional scheme for I-V and C-V characteristic measurements (DC power supply, voltage regulator, control and measurement module, RLC-meter E7-22, measured cell with sensor) of sensor based on porous silicon and semiconductor nanostructures is developed. The functional scheme is constructed in the form of a sample-layout.

An algorithm for sensors electrical properties measuring has been developed and software has been implemented. That allows to measure the I-V and C-V characteristics of these sensors, visualize their dependencies, and store the measurement results in the files.

Microprocessor system testing using the PS-nSi sensor and comparing the results with the results of constant voltage and current measurements in manual mode using Keithley 2100 devices was provided. The results of the comparison allow us to conclude that the implemented modules of the microprocessor system for sensors I-V- and C-V characteristics testing can be used in the development of portable environmental monitoring devices, to study porous silicon sensors using them.

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МІКРОПРОЦЕСОРНА СИСТЕМА ДЛЯ ДОСЛІДЖЕННЯ ХАРАКТЕРИСТИК ДАВАЧІВ НА ОСНОВІ ПОРУВАТОГО КРЕМНІЮ

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Розробка інтегральних давачів є перспективним науково-технічним напрямом створення компонентів вимірювально-інформаційних систем. Серед різних типів аналізаторів важливе місце займають давачі водневомісних сполук у газоподібному та рідкому стані (особливо токсичних і канцерогенних). Фіксація присутності речовини в газоподібному або рідкому середовищі є актуальним завданням для наукових, промислових і медичних застосувань.

Основною метою даної роботи є створення недорогої мікропроцесорної системи для вимірювання та дослідження електричних властивостей нанокомпозитного чутливого матеріалу, такого як поруватий кремній, на основі вольт-амперних та вольт-фарадних характеристик, та їх застосування як давачів для моніторингу навколишнього середовища.

Мікропроцесорна система призначена для вимірювання характеристик давачів на основі поруватого кремнію та нанокомпозитних структур і складається з таких блоків: блок живлення, регулятор постійної напруги, контрольно-вимірювальний модуль, RLC-метр E7-22, перетворювачі UART/USB і RS232/USB, вимірювальна комірка, комп’ютер. Реалізована принципова схема контрольно-вимірювального модуля дозволяє вимірювати вольт-амперні і вольт-фарадні характеристики для додатних та від’ємних напруг при однополярному джерелі живлення.

Програмне забезпечення мікропроцесорної системи складається з програмного забезпечення для контрольно-вимірювального модуля, реалізованого на мікроконтролері Atmega 128, та програмного забезпечення для персонального комп'ютера, яке керує вимірювальною системою та обробляє отримані дані. Програмне забезпечення для комп’ютера реалізовані мовою програмування C# у Visual Studio 2017.

Отримані результати дозволяють зробити висновок, що реалізоване апаратне і програмне забезпечення мікропроцесорної системи вимірювання параметрів давачів може бути використано при дослідженні нових поруватих кремнієвих давачів з подальшим їх використанням в портативних пристроях моніторингу навколишнього середовища.

Ключові слова: поруватий кремній, нанокомпозит, давач, мікроконтролер, вольт-амперні характеристики, вольт-фарадні характеристики

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