FEATURES FOR THE DESIGN OF A SPECIALIZED INFORMATION-MEASURING SYSTEM FOR THE STUDY OF THERMOELECTRIC PROPERTIES OF SEMICONDUCTORS

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Methods for studying thermoelectric parameters of semiconductors that are optimal for the implementation of software and hardware have been analyzed and selected. It is based on the Harman method and its modifications, adapted for pulse measurements, which are convenient to implement on a modern element base. An important advantage of these methods is the absence of the need for accurate measurements of heat fluxes, which greatly simplifies and reduces the time for conducting experimental research.

The required operating ranges for the voltage $10\mu V$–1 V, for the current $10\mu A$–300 mA and the element base performance at the processing level of 40–200 million samples per second have been determined. Structural and electrical circuits, as well as software for a specialized computer system for studying thermoelectric parameters of both bulk and thin-film thermoelectric materials, and express analysis of the operational characteristics of finished modules have been developed. It has been shown that the proposed scheme copes well with the task. And the use of FPGA and 32-bit microcontrollers provide sufficient processing speed up to 200 MSPS and the necessary synchronization modes for the implementation of the Harman pulse method even when studying films of nanometer thickness.

Experimental studies of both bulk thermoelectric modules based on Bi$_2$Te$_3$ and thin-film thermoelectric material based on PbTe have been carried out. The effectiveness of the developed tools and techniques has been shown, which made it possible to more than halve the time for sample preparation and experiment. Based on the presented models, all the main thermoelectric and operational parameters have been determined, in particular, electrical conductivity, Seebeck coefficient, thermal conductivity, thermoelectric figure of merit.

As a result of the development of specialized computer tools, it was possible to reduce the labor intensity of the process of measuring the main electrical and operational parameters of semiconductor thermoelectric materials and energy conversion modules based on them, as well as to automate the process of defects identification of thermoelectric modules. The labor intensity of the research process has decreased not only due to the automation of the measurement process, but also due to an optimized technique that allows research on a sample of one configuration, since the manufacture and preparation of samples are the most laborious.

Keywords: computer tools, information-measuring systems, signal processing, microcontroller systems, circuit design, speed, thermoelectric properties, defects identification

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1. Introduction

The aggravation of environmental problems has led to the constantly growing interest of the scientific community in alternative energy, in particular, in thermoelectric semiconductor energy converters. Semiconductor thin films and nanomaterials are of great interest for practical applications, due to the possibility of an independent increase in the Seebeck
coefficient (S) and a decrease in thermal conductivity (κ) using nanostructuring of the material. Such studies require the measurement of electrical conductivity, Seebeck coefficient, thermal conductivity, which is a rather laborious task when using classical methods, since the required samples are of various configurations. The rapid development of computer technology and microelectronics has led to the emergence of a large number of automated and computerized laboratory instruments for scientific experiments. A large number of universal tools for laboratory research have been developed, but due to the narrow specialization of thermoelectric research, it is not always possible to use them effectively. An urgent task is the adaptation of methods and the development of means, both for studying the main thermoelectric parameters of semiconductor materials and express methods for determining the operational characteristics of thermoelectric energy conversion modules.

2. Literature review and problem statement

Thermoelectric materials have been used for many years as a reliable solid-state technology for applications ranging from local cooling to waste heat utilization [1]. The achievements of recent years in the synthesis of semiconductor materials have led to an improvement in the conversion of thermal energy into electrical energy due to the nanostructuring of materials, and have expanded the application boundaries of thermoelectric energy [2, 3]. In addition, a time-Table nanostructured material with high electrical conductivity has been obtained by doping [4]. Direct measurement methods are often used to study thermoelectric properties requiring a gradient heater [5] and accurate maintenance and measurement of a small temperature gradient, which makes a significant error in estimating the thermoelectric figure of merit. These problems of designing devices and analyzing experimental data are considered in [6, 7]. With an error in measuring individual thermoelectric parameters of about 5%, the average error in measuring the figure of merit is 20%. Also, the problems of increasing the accuracy of sensors for measuring physical quantities at low temperatures are described by the authors of [8]. The authors of [9] described in sufficient detail the technique and means of high-temperature (1,000 K) thermoelectric measurements. Despite the simplicity of the measurement methods, their practical implementation is rather difficult, especially at high temperatures. This leads to large errors in determining the properties, complicates the comparison of the results obtained by different groups and the comparison of the efficiency of new thermoelectric materials. The work [10] presents a system for direct studies of thermoelectric characteristics by a differential method based on LabVIEW expansion cards (National Instruments, USA), precision electrometer, and using chromel-alumel thermocouples for measuring the temperature difference created by a gradient heater.

The authors of [1, 11] have shown that studies of thermoelectric parameters based on the Harman method and its modifications are promising, significantly allowing obtaining all the main characteristics on one sample, do not cause degradation of the material under study. These methods require the development of high-speed research tools and processing of the results obtained. The authors of [11] developed a system for measuring thermoelectric figure of merit based on a high-speed digital oscilloscope with the ability to transfer data to a computer for further processing.

Based on the analysis of existing methods and means, it can be concluded that each of them solves a specific problem in thermoelectricity, but does not provide an opportunity to conveniently and effectively investigate the entire complex of thermoelectric characteristics. And the need to prepare samples of various configurations significantly increases the complexity of research. Taking into account the shortcomings of the existing means, the developed software and hardware means make it possible to obtain all the necessary parameters of the test sample in one technological cycle using non-destructive methods, in particular, electrical conductivity, Seebeck coefficient, thermal conductivity coefficient, time constant and thermoelectric figure of merit, as well as carry out express quality control of finished thermoelectric modules according to the specified parameters.

3. The aim and objectives of the study

The aim of the work is to develop software and hardware for automated research of thermoelectric parameters of semiconductor materials, which will allow automated quality control of finished thermoelectric energy conversion modules.

To achieve this aim, the following objectives have been set:
- to carry out the selection and adaptation of methods for measuring thermoelectric quantities, to determine the necessary operating ranges, resources and speed of the element base for the implementation of the measuring system;
- to develop structural and electrical circuits and to select the element base of the information-measuring system for the study of thermoelectric properties of semiconductor materials;
- to develop algorithms and software for quality control of finished thermoelectric energy conversion modules;
- to study thermoelectric properties of semiconductor materials using the developed means.

4. Methods and models for the study of thermoelectric parameters and resources required for the implementation of the information-measuring system

To measure all the main thermoelectric parameters, the Harman method [12] and its modifications [11, 13] have been taken as a basis. This method belongs to indirect measurement methods, has a short experiment time, and does not require complex and laborious measurements of heat fluxes through the sample. In addition, the method makes it possible to minimize the laboriousness and cost of research, since all measurements can be carried out on a sample of one configuration.

When passing a direct current I through the sample (Fig. 1), a temperature difference ΔT between its ends is established. When alternating current of sufficient frequency is passed through the sample, the heat transfer caused by the Peltier effect does not occur and a temperature difference is not established.

Electrical conductivity is measured on alternating current as:

$$\sigma = \frac{I}{U} \cdot \frac{l}{S}.$$  
(1)

Here I, U are current and voltage at the AC measurements.
If the DC and AC forces are equal and small enough to neglect the release of Joule heat, and the exchange of heat with the environment is small enough, the Seebeck coefficient is determined by the formula:

$$\alpha = \frac{U_{\text{res}} - U}{\Delta T}. \quad (2)$$

The thermal conductivity coefficient is defined as:

$$\kappa = \frac{\alpha I T}{\Delta T S}. \quad (3)$$

This method makes it possible to directly determine the dimensionless figure of merit of the material:

$$ZT = \frac{U_{\text{res}} - U}{\alpha}. \quad (4)$$

The main point to focus on is the difficulty of measuring extremely small temperature gradients $\Delta T$. This is due to the difficulty of providing good thermal contact with the sample and the temperature sensor without causing significant heat loss through the sensor probes. One of the options for the optimal connection of the sample is the use of a four-point probe circuit, two electrodes made of different metals (for example, copper-constantan or chromel-alumel) on each side of the sample, and transmitting and reading the potential difference in pairs. In some cases, to improve the accuracy, measurements are taken in two directions of the current and averaged. For thin films, it is also convenient to control the temperature difference by optical methods described in [14].

In the case of analysis of the transverse properties of thin films and express quality control of finished thermoelectric modules, it is advisable to study the time dependence of the voltage caused by the Peltier effect $U_{\text{res}}$ at a constant current through the sample, which is approximated by the expression:

$$U_{\text{res}}(t) = U_{\text{res}} \left(1 - e^{-t/\tau}\right). \quad (5)$$

Here $U_{\text{res}} = U - U_{\text{res}}$ is voltage caused by the Peltier effect in stationary mode, $\tau$ is time constant, which is determined by the thermal conductivity and heat capacity of the elements of the thermoelectric module [15]. For a single-stage thermoelectric module, the cold and hot heat transitions of which are in heat exchange with the environment, the time constant can be represented as [15]:

$$\tau = \frac{C l}{2\kappa N S}. \quad (6)$$

Here $C$ is heat capacity of elements on cold and hot heat transitions, respectively, $N$ is number of legs in a module, $S$ is leg cross-sectional area, $l$ is length.

The time parameter determines the thermal characteristics of the module and, along with the electrical conductivity and thermoelectric figure of merit, makes it possible to quickly determine the deviations of the quality parameters of thermoelements. In particular, when diagnosing thermoelectric energy conversion modules, the following deviations of quality parameters are possible (Table 1).

### Table 1

| Defect                                    | R     | Z     | $\tau$ |
|--------------------------------------------|-------|-------|--------|
| The polarity of the thermoelectric module  | Does not change | Decreases | Increases |
| legs is reversed                           |       |       |        |
| Detachment of interconnected plates from   | Does not change | Does not change | Increases |
| ceramics                                   |       |       |        |
| Thermal and electrical contacts of legs    | Decreases | Does not change | Decreases |
| of a module with a solder meniscus          |       |       |        |
| Short-circuiting legs in a module          | Decreases | Decreases | Decreases |
| Degradation of the thermoelectric material | Increases | Decreases | Does not change |
| of the module                              |       |       |        |

From the analysis of the dependence $U_{\text{res}}(t)$ (5), it is possible to estimate the speed that a specialized computer system should have. If the time between measurements is comparable to the time parameter $\tau$, this will allow obtaining about 5 measurements during the time of reaching the stationary mode, although given the low signal levels and the presence of noise, it is desirable to have a slightly higher sampling rate. In [11], the authors investigated Tl-doped PbTe films, and at a film thickness of 500 nm, the time parameter was 10 $\mu$s at an optimal current of 10–20 $\mu$A. Experimental studies for semiconductor PbTe films of micrometer thickness (1–1.5 $\mu$m), obtained by the method of vacuum evaporation, gave the time parameter $\tau \approx 3–6 \mu$s, and for similar films of nanometer (10–100 nm) thickness, the value is $\tau \approx 50–100 \mu$s. Bulk thermoelectric modules are characterized by a time $\tau$ of several seconds and there are no special requirements for speed. The studies were carried out both on a prototype of the device developed on the basis of a 32-bit STM microcontroller and using a modern Tectronix digital oscilloscope with a bandwidth of 100 MHz. To ensure a wide range of thicknesses of investigated films, it is optimal...
to choose an analog-to-digital converter (ADC) with a sampling rate of 40-200 million samples per second. However, even the use of STM32F303 with built-in digital-to-analog and analog-to-digital converter for 5 MSPS made it possible to build a speed measuring system, which is sufficient for studying both bulk thermoelectric converters and thin films with a thickness of up to 100 nm.

When measuring on alternating current, the period of alternating current should also not exceed the time parameter \(\tau\), which means that it can be limited to the switching frequency of 20 MHz. It is necessary to ensure the choice of the current depending on the operating current of the thermoelement in a wide range from 10 \(\mu\)A to 500 mA.

The absolute values of the voltage across the sample significantly depend on the thermo-EMF of the material, the resistance of the sample, the selected current through the sample; and for different materials, the shape and thickness of the sample, it can range from 10 \(\mu\)V to 1 V [11, 13]. Therefore, to bring the voltage to the operating range of the ADC, an instrumental operational amplifier with a software-controlled gain is required, and the ADC itself must have a capacity of at least 12 bits to ensure sufficient resolution.

The system must have sufficient memory to store the digitized experimental data. The sampling frequency of the ADC must be selected depending on the time parameter \(\tau\) of the process so that the data fit into the memory and the number of points is sufficient for further software processing.

Other units, such as external heating, data exchange with external devices, do not have special requirements for speed and can be guided by the microcontroller in the main cycle.

5. Features of the development of hardware for the information-measuring system

The rate of physical processes in the studied objects determines the minimum period of time \(T_0\). During this time, all the processes of converting information in the sensors during \(T_s\) and processing information according to the algorithm of the information-processing device during \(T_p\) should be completed. That is, \(T_s + T_p \leq T_0\).

If the \(T_0\) value is large enough, for example, a few milliseconds or more, and the number of sensors is small, then the information-measuring system can be implemented on a single personal computer or smartphone. It is also possible to implement a system based on a microcontroller or microcomputer. In any case, there are no fundamental problems in the design process of such systems.

If the \(T_0\) value is small enough, for example, a few nanoseconds or less, or the number of sensors is hundreds or more, then such an information-measuring system is rather difficult to build as a system on a single crystal or on a single personal computer. Here, quite often, a personal computer serves as an output device; a cluster system, a specialized multiprocessor system with a matrix topology, hardware implementation on FPGA, etc. can serve as an information-processing device.

A block diagram of a specialized software and hardware system for the study of thermoelectric properties and express diagnostics of thermoelectric modules for energy conversion is shown in Fig. 2. Fig. 3 shows the developed block diagram of the measurement unit.

![Fig. 2. Block diagram of a specialized hardware and software system for the study of thermoelectric properties and express diagnostics of thermoelectric modules. Arrows shaded in black are informational, and unshaded arrows are control](image)

![Fig. 3. Block diagram of the measurement unit](image)
computer system based on a high-speed FPGA. A Nexys A7 debug board with Artix-7 and 128 MiB DDR (USA) memory has been used for prototyping. The main task of fast digitizing of an analog signal is solved using a high-speed analog-to-digital converter AD9643 (Analog Devices, USA), which provides 14-bit accuracy and guaranteed no data loss over the entire operating temperature range at a conversion rate of up to 250 MSPS. The ADC output data are output directly to the external 14-bit LVDS output port. Reading, accumulation, filtering and mathematical processing of data are implemented on FPGA, and communication with a computer, signal generation, switching control and high-speed current source control are implemented on a 32-bit STM32F303 microcontroller.

Fig. 4 shows the block diagram of receiving and processing data from ADC chip according to the LVDS standard, developed on the basis of the manufacturer’s recommendations [16, 17] with modifications for the selected element base.

It includes an input receiver (INBuf), a data packer (Repack), a data stream synchronizer (FIFO), an ADC chip control unit (SPI). In addition to these nodes, a frequency synthesis module (MMCM) is used at the top level. Multi-channel ADC systems should be clocked from a low-jitter reference generator, a Si571 chip, passing its output clock signal through a multiplier corresponding to the number of ADC channels. In this case, with accurate alignment of the data lines and clock signals of the ADC channels on the printed circuit board, we will get a coherent system on the receiving side of the FPFA and complete in-phase between the channels. When using a single ADC channel, the circuit can be simplified using a standard FPFA frequency synthesis module based on the Mixed-Mode Clock Manager (MMCM).

The input receiver contains the primary connections between the external printed circuit board signals and the FPFA logic. Depending on the data transmission interface, the buffer can be differential or unipolar. Following the buffer, a node for regulating the delay of the signal passage IODelay is installed to align the edges of the parallel data bus and for the initial coordination of multichannel nodes for connecting the ADC to the FPFA. Further, it is possible to set the FD trigger if the data transfer mode is «SDR». In the «DDR» mode, the trigger is replaced by the IDDR that has one data input and two outputs, from which signals are output on the same clock signal edge, and the module itself is clocked by the ADC clock frequency. From the FD/IDDR output, the data goes to the data packer, the main purpose of which is to pack the data into a convenient internal bus format that is used in FPFA. After that, the data goes to the data flow synchronizer, which is built according to the FIFO scheme and converts the data flow from the sampling frequency of the ADC to the frequency of the internal bus of the FPFA device. Further, the data is transmitted for processing; it can be either a digital signal-processing unit inside the FPFA, or an external interface. The analog module is controlled via SPI interfaces, the implementation of which on FPFA is standard.

A circuit diagram of a voltage-controlled DC stabilizer has been developed, which is implemented on a high-speed operational amplifier (Fig. 5); the current sensor is a shunt, which is selected by the reference resistor-switching unit depending on the current value. The voltage polarity reversal is implemented by synchronous switching using an H-bridge based on opto-isolated MOSFET transistors. A high-speed ADC carries out the voltage measurement on the sample; the unit for bringing the voltage within the range of the ADC operation is implemented on a high-speed, precision, instrumental operational amplifier with a software-controlled gain of 1, 10, 100, and 1000.

If it is necessary to increase the switching frequency, the integrated optodrivers can be replaced with high-frequency field-effect transistors with a corresponding correction of the control circuit. The proposed scheme with correct synchronization has shown high stability and low noise.
6. Diagnostic algorithm and software for its implementation

The program for the STM32F303 microcontroller is written in C and provides hardware operation, self-diagnostics of the main units, generation of control signals, reception and preprocessing of data.

The developed algorithm for the implementation of fast diagnostics of thermoelectric modules based on time measurements is schematically shown in Fig. 6.

This algorithm provides both diagnostics of contact reliability and complete diagnostics of the thermoelectric element. Electrical conductivity and internal resistance are calculated by expression (1), and thermoelectric figure of merit – by formula (4). The constant $\tau$ is determined from the approximation of the time dependence of the voltage $U_\alpha(t)$, which is carried out in the program by the least-squares method using the algorithm for minimizing functions of many variables by the Nelder-Mead deformed polyhedron method. Although this technique is quite simple, its implementation has shown good efficiency – finding the minimum takes 80–100 iterations and does not slow down the program execution.

Setting the parameters of the experiment, as well as further processing, visualization and storage of data, is carried out using the written control program, the main window of which is shown in Fig. 7.

A two-way exchange of information is carried out between the controller and the computer via the USB interface; the unit is controlled and data is requested by sending commands and receiving a response after their execution.

The program has a graphical interface with tabs, which group the main stages of the experiment. The Manual Control and Experiment Settings tab contains the main controls and presets. In manual mode, the program allows you to control individual functional units independently, which allows you to debug and conduct non-standard experiments.

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**Fig. 6. Possible implementation of the algorithm for fast diagnostics of thermoelectric modules based on time measurements**
The next tab allows you to measure thermoelectric properties at different temperatures (Fig. 7). A separate tab for automatic measurement of temperature dependences and a tab for express diagnostics of finished thermoelements are also implemented.

The measurement results for each sample are saved in a separate file with the possibility of further expanding the experiment. The ability to select specific data for export to MS Excel for their further processing has been implemented.

In general, the hardware and software complex provides automated modes of operation required to measure all the main temperature parameters. In particular, the temperature dependences of the AC resistivity, the DC Seebeck coefficient, the temperature gradient in a stationary mode, and the thermoelectric figure of merit. In addition, the complex provides the measurements of the time dependence of the voltage when entering the stationary mode and software processing of the obtained data.

7. Results of experimental studies of thermoelectric properties obtained using the developed means

Fig. 8. a shows the obtained timing diagram of the voltage when entering the stationary mode for the industrial thermoelectric module SP1848-27145 (China) based on Bi$_2$Te$_3$. Fig. 8. b shows a similar dependence for a PbTe thin film obtained by vacuum evaporation on a mica substrate. The film thickness was 1215 nm.

The program in the microcontroller processes the array of received data.

Fig. 8. Time dependences of DC voltage: a — for thermoelectric module SP1848-27145; b — for a PbTe thin film on a mica substrate
In the program analysis of these dependences, first, according to the first measurement, before the appearance of a temperature difference on the sample, the voltage \( U = 33 \text{ mV} \) was determined, which was subtracted from the data obtained for their approximation by expression (5). The approximation was carried out by the least-squares method. The approximation results are shown in Fig. 8 as a solid line.

For the SP1848-27145 thermoelectric module, the values \( U_{\text{st}} = 48.2 \text{ mV}, \tau = 6.3 \text{ s} \) were obtained, which makes it possible to find the thermal resistance of the module \( R = 0.33 \text{ Ohm} \) and its thermoelectric figure of merit, which is \( ZT = 4.6 \) at 300 K. The obtained results quite accurately correspond to the data specified in the documentation for the module. For a film sample with a size of 0.5×0.5 mm, \( U = 112 \mu \text{V}, U_{\text{st}} = 35.8 \mu \text{V}, \tau = 4.3 \mu \text{s} \), which makes it possible to find sample parameters given in Table 2.

| Parameter                              | Value     |
|----------------------------------------|-----------|
| Material                               | PbTe      |
| Electrical conductivity, Ohm·cm\(^{-1}\) | 134.4     |
| Seebeck coefficient, \( \mu \text{V/K} \) | 249.3     |
| Coefficient of thermal conductivity, \( \text{W/cm K} \) | 7.5 \ \times \ 10^{-3} |
| Thermoelectric figure of merit \( ZT \) at 300 K | 0.32      |

8. Discussion of research results

The obtained results (Fig. 8) are in good agreement with the data given in the technical documentation for the industrial thermoelectric modules and with previous studies by direct measurements on samples obtained from the same material under identical deposition conditions for thin films [18]. The discrepancies did not exceed 3–5%.

The selected methods and models (5), (6) made it possible to implement the entire complex of studies on a sample of one configuration (Fig. 1), which significantly reduced the labor intensity and time for preparing the experiment in comparison with classical methods [1, 9, 10]. In addition, the described research methods and tools are non-destructive and do not require, for example, drilling a sample to determine thermal conductivity, as in the radial flow method. Due to the fact that these methods do not require measurement of heat fluxes through the sample, one of the largest sources of error in classical methods is eliminated [5].

The developed circuit solutions (Fig. 3, 5) made it possible to create a system that allows one to obtain all the necessary parameters of the test sample in one technological cycle using non-destructive methods. And the applied computer program provides convenient control of the measurement process for a series of samples of the same type, carrying out automated analysis according to the diagnostic matrix of defects (Table 1) to quickly identify defective specimens and the probable type of defect.

Harman method and its modifications, despite the simplicity of implementation and efficiency, have a number of significant disadvantages and limitations in comparison with direct measurement methods. In particular, for thin films, this method is difficult to separate the contribution of the substrate to the thermal conductivity of the film material; here, whenever possible, one should give preference to the more accurate but much more complex laser flash methods described in [19]. The advantage of this technique is the ability to quickly assess all the main parameters and provide defects identification of finished thermoelectric modules, including thin-film ones.

The prospects for further development of the research are integration with other devices for studying the parameters of semiconductors into a single database, in particular, with a device developed for studying thermoelectric parameters by impedance spectroscopy methods [17].

9. Conclusions

1. The analysis has been carried out, the methods of studying the thermoelectric properties of semiconductors have been selected and optimized, which makes it possible to carry out studies on a sample of one configuration. The necessary operating ranges and hardware resources for the construction of a modern information and measurement system have been determined. It has been shown that for the implementation of such a system, it is necessary to provide operating voltage range from 10 \( \mu \text{V} \) to 1 \text{V}, current range from 10 \( \mu \text{A} \) to 500 \text{mA}, and ADC speed at the level of 40–200 MSPS with a resolution of 12 bits.

2. The structural and electrical diagram of the hardware-software complex for the study of thermoelectric properties of semiconductor materials has been developed that makes it possible to flexibly configure the system depending on the selected experimental parameters using modern programmable element base. It has been shown that the proposed alternating current stabilizer circuit copes well with the task, and the use of FPGA and microcontroller software provide sufficient speed and the necessary synchronization modes for implementing the Harman pulse method.

3. An operating algorithm has been developed, and a specialized computer system has been created that makes it possible to obtain all the necessary parameters of the test sample in one technological cycle on a sample of one configuration by non-destructive methods. It also provides automation of measurements, recording, processing and visualization of data, and quick diagnostics of a series of similar thermoelectric modules with the determination of the probable type of defect.

4. A working prototype of the device has been created, and experimental studies of the properties of thin-film thermoelectric materials and energy conversion modules have been carried out, which showed good agreement with the results obtained on similar samples by direct methods, and discrepancies did not exceed 3–5% at less than half the time for sample preparation and experiment.

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