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Further development of methods and automation of measuring parameters of thermoelectric semiconductor energy converters

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The concept and hardware and software tools for automated measurement of thermoelectric parameters of semiconductor energy converters have been developed. The system is built on the basis of advanced vacuum methods of direct measurements using a modern element base, high-precision analog-to-digital converters, which, in combination with software methods for processing the obtained experimental data, made it possible to increase the measurement accuracy. A precision software-controlled current stabilizer has been developed, which allows measuring the electrical characteristics of thermoelectric energy converters in a wide range of loads from units of microamperes to several amperes. The possibility of automated diagnostics and determination of operational parameters of semiconductor thermoelectric energy conversion modules, in particular thin-film ones, has been implemented, which makes it possible to reject defective modules and in general significantly increases the reliability of thermoelectric generators.

Experimental studies of a series of generator thermoelectric samples with known characteristics have been carried out and the effectiveness of the developed tools has been shown using the described methods for analyzing experimental data.

Keywords: thermoelectric converters, clean energy, automation, semiconductor structures, circuit design, microprocessor, computer modeling, 3D modeling, digital-to-analog converter.

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Introduction

Thermoelectric materials are important for both modern energy and electronics, as they allow direct conversion of thermal energy into electrical energy, and can also be used for cooling. This makes them promising for the construction of miniature generators and coolers for modern electronic systems, waste heat recovery, and the creation of autonomous power sources.

The efficiency of thermoelectric materials is determined by the dimensionless quantity ZT :

$$ZT = \frac{S^2 \sigma}{\kappa} T,$$

where S is the Seebeck coefficient, σ is the electrical conductivity, and κ is the thermal conductivity.

The accuracy of determining these parameters is

critically important for the development of new materials and increasing their efficiency. This is especially true for thin-film semiconductor materials, which are currently of considerable scientific interest due to the possibility of independently increasing the Seebeck coefficient (S) and decreasing the thermal conductivity (κ) [1]. Such studies require accurate measurements of the electrical conductivity, the Seebeck coefficient, and the thermal conductivity. However, such measurements are quite laborious and are complicated by the influence of contact resistances and heat losses, which requires improving methods and developing specialized installations. Also, an urgent task is the development of tools for automated diagnostics of thermoelectric energy conversion modules.

I. Analysis of the literature and formulation of the problem

Modern advances in the synthesis of semiconductor materials have led to an increase in the efficiency of converting thermal energy into electrical energy due to size effects and nanostructuring of materials, which has significantly expanded the scope of thermoelectric energy applications [2-4]. The most common are direct methods of thermoelectric measurements, in particular, the differential method, in which the voltage difference is measured at a given temperature gradient. In [5], a system for direct studies of thermoelectric characteristics by the differential method based on LabVIEW expansion boards, a precision electrometer, using a chromel-alumel thermocouple to measure the temperature difference created by a gradient heater is presented. The method uses thermocouples to determine temperature and is applicable in a wide temperature range. The method is simple to implement and provides high measurement accuracy under stable conditions, but is sensitive to the influence of parasitic thermo-EMF and inaccuracies in maintaining a given temperature gradient. Also, the geometry of the contacts significantly affects the accuracy of measurements, especially at high temperatures. These methods require a gradient heater and accurate maintenance and measurement of a small temperature gradient, and consideration of heat flows and losses, which can introduce a significant error in the assessment of the thermoelectric figure of merit. These problems of device design and analysis of experimental data are considered in [6-7], where it is shown that with a measurement error of the main thermoelectric parameters not exceeding 5%, the average error in the measurement of the figure of merit was about 20%.

Quasi-stationary methods make it possible to reduce measurement errors by smoothly changing the temperature gradient. It is used in modern installations for simultaneous measurement of several parameters. For example, automated systems allow determining the Seebeck coefficient and specific electrical conductivity simultaneously [8,9]. The limitation of this automated system is the measurement of only two main parameters, and despite a significant reduction in the measurement error, it still remains significant. The four-probe method is widely used for measuring electrical conductivity, since it eliminates the influence of contact resistance. In modern installations, it is combined with the measurement of the Seebeck coefficient to obtain complex characteristics of the material [10].

Also promising are methods for simultaneous measurement of several parameters, in particular, the Seebeck coefficient, specific electrical conductivity, thermal conductivity and other characteristics. In particular, in work [11], high-temperature installations were developed that allow simultaneous determination of several parameters up to 800 °C. As for the measurement of thermal conductivity, the most popular are stationary methods based on measuring the heat flux through the sample at a set temperature gradient. However, due to significant heat losses, such methods are difficult to apply to thin-film samples, where dynamic measurement

methods are preferred, for example, the laser flash method [12-14]. The latest methods allow simultaneous determination of thermal conductivity along with other parameters, which increases the efficiency of research.

When using existing tools for measuring thermoelectric parameters, many difficulties arise, in particular with the adaptation of measurement methods to given sample configurations and the integration of such tools into existing laboratory complexes. Therefore, further development of methods and development of specialized hardware and software tools for research and diagnostics of thermoelectric energy converters, as well as methods of processing, software filtering and data approximation to determine their operational characteristics remains relevant.

The aim of this work is to further develop research methods and design hardware and software tools for automating the measurement of thermoelectric parameters in semiconductor energy converters.

To achieve the set goal, the following tasks were solved:

- development of the concept and research methodology, as well as the design of the measuring cell;
- design of a precision software-controlled current stabilizer and algorithms for its operation and development of software and hardware measurement tools based on precision analog-digital converters for automated research of thermoelectric properties of energy converters;
- conduct research on thermoelectric materials and evaluate the effectiveness of the developed tools.

II. Further development of the research methodology and design of the measuring cell

Currently, the main efforts of scientists in the field of thermoelectrics are focused on finding materials with increased thermoelectric efficiency and expanding the operating temperature range (50 - 600 °C). The main parameters that determine the quality of a thermoelectric material are the specific electrical conductivity, the Seebeck coefficient, and thermal conductivity, in addition, for a thermoelectric energy converter, information about the internal resistance, generated currents, thermoelectric power, heat capacity, etc. is also necessary.

To measure all these parameters, direct methods were chosen, in which a heat flux is passed through the thermocouple due to a certain temperature gradient created between the heater and the cooler. This work is a further development of the direct measurement method described in [15], the main feature of the method is the use of two identical samples that are placed on both sides of the heater and cooled by identical water radiators (Fig. 1, b). The method is adapted for the study of thin-film thermoelectric materials and energy generation modules based on them. Thin films are clamped between a copper heater and water-cooled radiators through thin mica spacers (Fig. 1, c), which made it possible to maintain a stable temperature of the cold junctions. It is possible to study thin-film samples separately or to fix two samples

for the implementation of comparative methods.

A miniature low-power copper heater was made to heat small samples, and a tubular tantalum heat shield was used to reduce parasitic heat losses. The samples from the heater and radiators are separated by thin nickel plates that serve as measuring contacts, the thermocouples are loaded through the heater and radiators, which made it possible to implement a four-wire circuit. The general view of the measuring cell is shown in Fig. 1.

The system supports up to 5 thermocouples: one in the heater, one in each of the radiators, and two that can be drilled into the sample for additional monitoring of heat fluxes. A key improvement is the use of MCP9600 integrated thermocouple signal converters and an ADC, which are housed in a vacuum chamber and communicate with the central unit via an I2C interface. This has significantly reduced the length of wires susceptible to interference and induced currents, and allows temperature measurement with an accuracy of 0.05 °C.

The principle of determining the thermal conductivity coefficient using stationary methods is described in [15]. When implementing comparative methods, the amount of heat that has passed through the sample under study is determined from the known parameters of the reference sample, which is in similar conditions. The methods work quite well for different materials in a wide temperature range: from several degrees to 600 oC.

III. Concept, hardware and software for automated studies of thermoelectric properties

When developing the concept of the measuring system, special attention was paid to the versatility of solutions, which provided the possibility of studying both massive and film thermocouples, as well as diagnostics of thermoelectric modules, taking into account the experience of working on a previous installation, which had a number of limitations [15]. The structural diagram was designed with the possibility of autonomous operation with recording results on a memory card, and the possibility of control from a computer for performing non-standard experiments was also left.

The main idea is to use the maximum power point search algorithm (MPPT algorithm) for loading the thermocouple. To obtain maximum power, the external load must be equal to the internal resistance of the module at a given temperature. Two most popular algorithms are implemented. Perturb and Observe - the controller slightly changes the current and observes the change in power, if the power increases, it continues to change the parameters in the same direction. And the more accurate Incremental Conductance - the controller analyzes the derivative of power over voltage (dP/dV), and finds the point where $dP/dV = 0$.

For hardware implementation of the MPPT algorithms, a software-controlled current stabilizer built on an operational amplifier and a MOS-FET transistor is

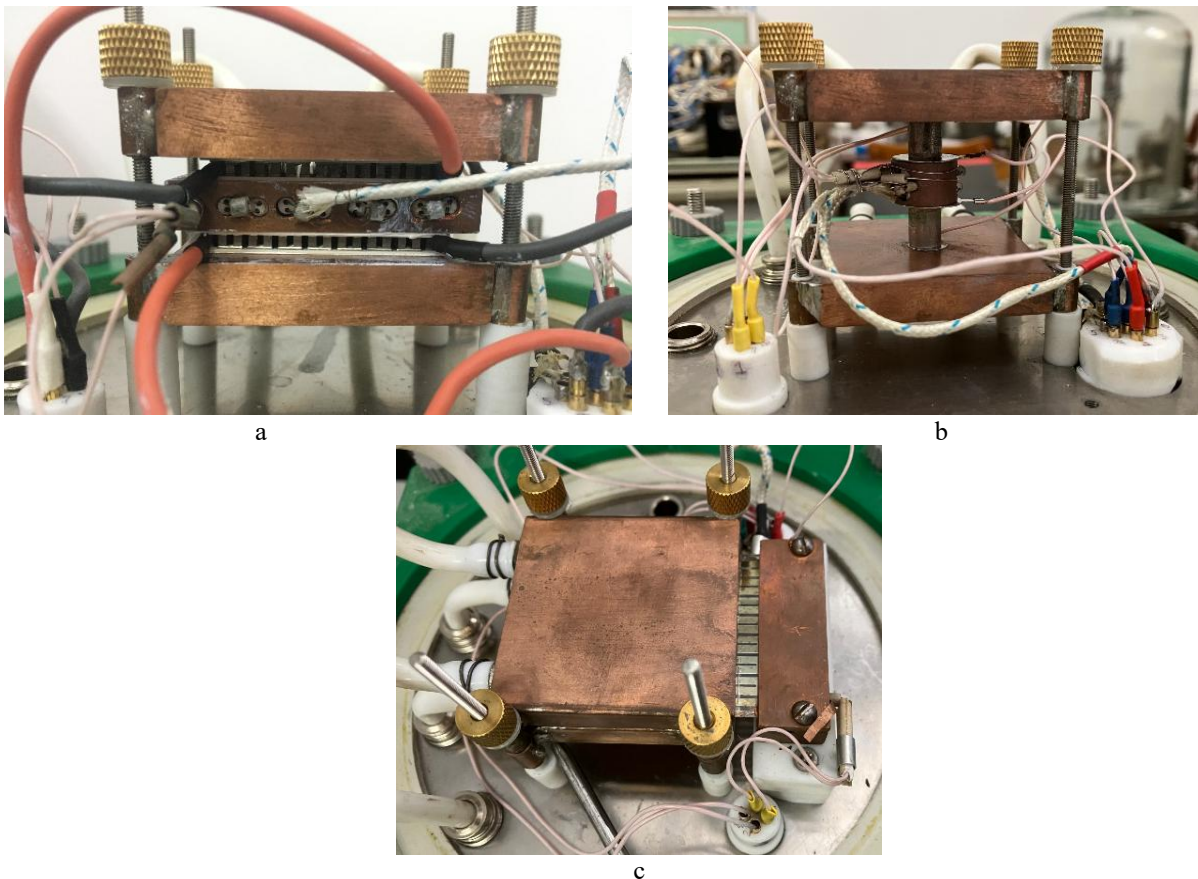


Fig. 1. General view of the measuring cell for the study of thermoelectric generators (a), cylindrical or rectangular thermoelectric samples (b) and thin-film thermoelectric converters (c).

used. The stabilization current is set by a digital-to-analog converter, and to ensure a wide range of operating currents from units of μA to several amperes, transistor switching of the shunt is implemented. The shunt is used both to ensure the feedback of the current stabilizer and to measure the current by the microcontroller's analog-to-digital converter.

The block diagram of the measuring system is shown in Fig. 2.

To ensure a stable temperature of the cold side, cold water is passed through two copper radiators, cooled by a refrigeration thermostat based on Peltier elements. It is also possible to use flowing tap water. The set temperature of the hot side is maintained by a microcontroller using a precision PID thermostat, the power of which is accurately measured and can be maintained constant, regardless of changes in external conditions or heater resistance.

A modern 32-bit microcontroller STM32G474 was chosen as the control device, which is designed for precise measurement purposes, the control program for the microcontroller is written in C.

To measure the generated voltage, high-precision 18-bit analog-to-digital converters MCP3421 with a built-in controlled amplifier were used, which were placed in the measuring chamber, which significantly reduced the noise level and increased the measurement accuracy. Measurements can also be carried out using the UNI-T UTM1805A certified industrial digital multimeter integrated into the measuring system, which supports data output to a computer and in the DC voltmeter mode provides a resolution of $1 \mu\text{V}$ with an accuracy of 0.015% and has an automatic measurement range selection mode.

Communication with the computer at the hardware level is provided by the hardware support of the USB microcontroller, and at the software level by means of a text command interpreter.

To determine the thermoelectric power and internal resistance, a stabilized load block was implemented, built on the basis of a software-controlled current stabilizer implemented on an operational amplifier (Fig. 3). The study of the influence of the Peltier effect under load

provides additional opportunities for studying heat transfer parameters, in particular the determination of thermal conductivity.

A four-wire measurement circuit has been implemented, with the possibility of changing the polarity on the relay. Power supply from the network through a transformer, the heater is powered by separate windings with the possibility of switching them, which provides additional control capabilities. The power is regulated by a triac using a PID algorithm with switching at the moment of transition through zero and period distribution according to the Bresenham algorithm.

3D modeling of the layout of elements in the housing has been carried out, taking into account their mutual influences. The electronic part of the installation is assembled in a compact housing, the boards are placed taking into account the minimization of mutual induction, the connection is made with a shielded wire, the housing is grounded (Fig. 4.)

The program allows you to conduct an experiment in offline mode, according to predefined scenarios, with recording of results on an SD card. When connected to a computer, it is possible to perform non-standard experiments with data registration not only from analog-to-digital converters, but also from a digital multimeter, perform pre-processing and visualization of data. With long-term regular operation, the installation has shown high reliability, stability and accuracy of results.

IV. Experimental studies and discussion of the results obtained

Let us consider the operation of the computer measurement system on the example of real studies of a series of samples consisting of hot-pressed cylindrical samples, the cold side of which consisted of Bi_2Te_3 and the hot side of GeTe.

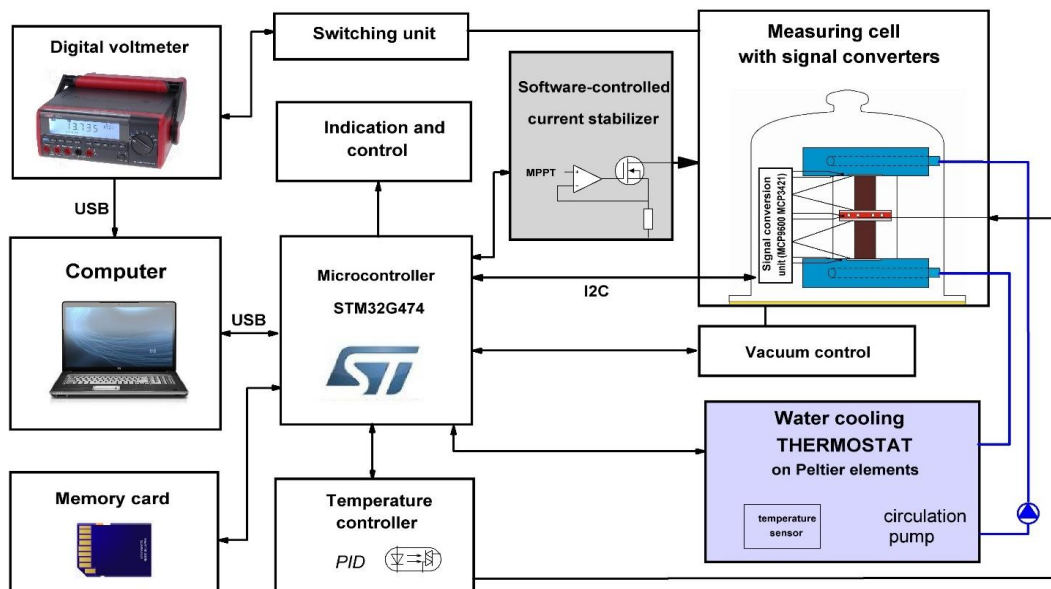


Fig. 2. Block diagram of the system for automated research of thermoelectric properties.

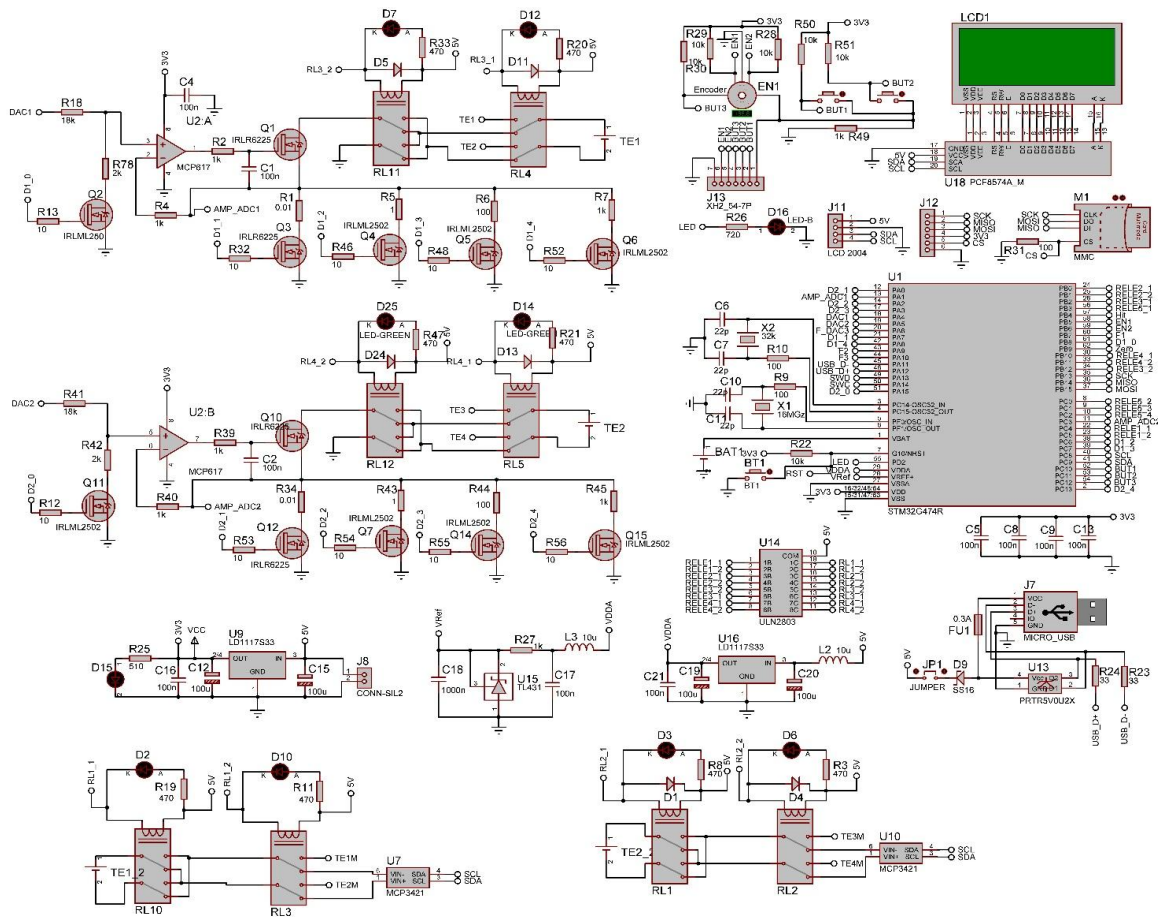
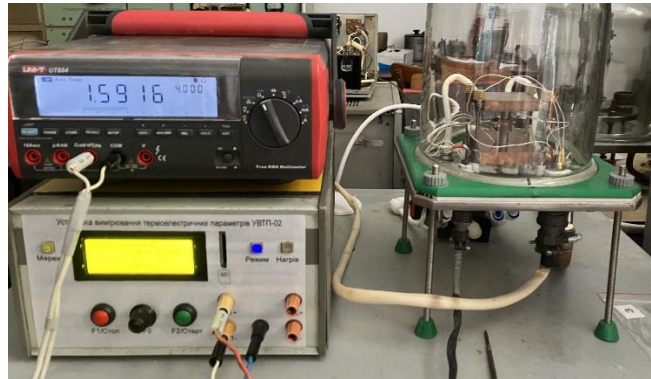


Fig. 3. Electrical schematic diagram of the system for automated research of thermoelectric properties.



a



b

Fig. 4. Control unit (a) and general view of the installation (b).

The cold junctions are cooled by flowing water and their temperature during the measurement is practically unchanged. The samples are clamped between copper radiators and the heater through thin 0.1 mm nickel plates which serve as measuring contacts, the load was applied through the connection to the copper base of the heater and radiators. The measurements were carried out in a vacuum of 1×10^{-1} Torr.

The parameters of the samples are given in Table 1,

and the results of the studies are shown in Fig. 5.

Table 1.

Parameters of Bi_2Te_3 GeTe samples.

Sample	Pressing temperature	Diameter	Length
DII	HP 450 °C	D=8,1 mm	L=11 mm
DIII	HP 500 °C	D=8,1 mm	L=9,5 mm

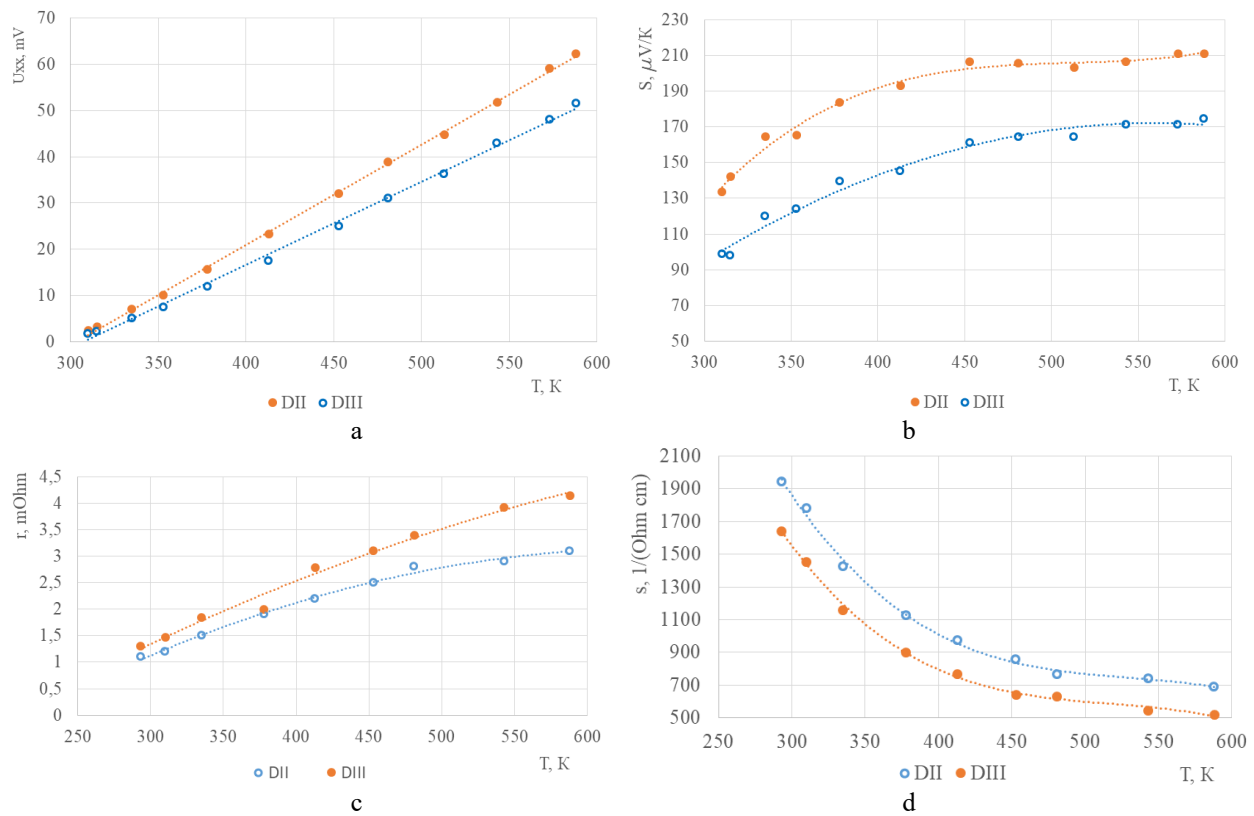


Fig. 5. Dependence of electrical parameters on the temperature of the hot side. Open circuit voltage (a), averaged thermo-EMF coefficient (b), internal resistance (c), specific electrical conductivity (d). Cold side temperature $T_{rad} = 19.5^\circ\text{C}$.

Analysis of the obtained results and comparison with the results obtained by similar methods in manual mode showed that the selected measurement methods and their hardware and software implementation showed high efficiency, made it possible to significantly reduce labor costs for conducting experimental studies and increased measurement accuracy. Visualization of the obtained results in the form of graphical dependencies makes it possible to detect errors and defective samples already during the measurement process.

Conclusions

1. A methodology has been developed, the design of the measuring cell has been improved, and the concept and implementation of hardware and software tools for automated measurement of thermoelectric parameters of semiconductor energy converters have been proposed.

2. A precision software-controlled current stabilizer has been developed, it has been shown that such circuitry allows for measurements of the electrical characteristics of thermoelectric energy converters in a wide range of loads from units of microamperes to several amperes. This made it possible to implement the idea of using the

maximum power point search algorithm for the thermocouple load.

3. Using the developed tools, experimental studies of thermoelectric composite high-temperature materials with known characteristics have been conducted and the high efficiency of the developed tools has been shown, which has significantly reduced the complexity of the process of studying the main electrical parameters of semiconductor materials and at the same time increased the accuracy of the results obtained.

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Подальший розвиток методів та автоматизація вимірювання параметрів термоелектричних напівпровідникових перетворювачів енергії

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Розроблено концепцію та апаратно програмні засоби автоматизованого вимірювання термоелектричних параметрів напівпровідникових перетворювачів енергії. Система побудована на основі вдосконалених вакуумних методів прямих вимірювань з застосуванням сучасної елементної бази, високоточних аналого-цифрових перетворювачів, що у поєднанні з програмними методами обробки отриманих експериментальних даних дало можливість підвищити точність вимірювання. Розроблено прецизійний програмно керований стабілізатор струму, який дозволяє проводити вимірювання електричних характеристик термоелектричних перетворювачів енергії в широкому діапазоні навантажень від одиниць мікроампер до декількох ампер. Реалізована можливість автоматизованої діагностики та визначення експлуатаційних параметрів напівпровідникових термоелектричних модулів перетворення енергії, зокрема тонкоплівкових, що дає можливість відбракувати дефектні модулі і в цілому значно підвищує надійність термоелектричних генераторів.

Проведено експериментальні дослідження серії генераторних термоелектричних зразків з відомими характеристиками та показано ефективність розроблених засобів із застосуванням описаних методик аналізу експериментальних даних.

Ключові слова: термоелектричні перетворювачі, чиста енергія, автоматизація, напівпровідникові структури, схемотехніка, мікропроцесор, комп'ютерне моделювання, 3D друк, 3D моделювання, цифро-аналоговий перетворювач.