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TECHNOLOGICAL MODES OF MANUFACTURING MEDICAL PURPOSE THERMOELECTRIC SENSORS

This work presents technological modes of manufacturing thermoelectric heat flux converters. It has been established that the optimal thermoelectric material for a thermopile is low-temperature materials based on Bi_2Te_3. The effectiveness of using such technological modes for manufacturing thermoelectric microthermopiles capable of recording laser radiation with an improved conversion coefficient of 1 – 1.5 orders of magnitude compared to existing measuring transducers has been experimentally confirmed. The specified technological modes significantly simplify and mechanize the method of manufacturing medical purpose thermoelectric heat flux sensors and microgenerators for powering low-power medical equipment.

Key words: technological mode, thermoelectric converter, medical purpose thermoelectric heat flux sensor.

Introduction

General characterization of the problem. Thermoelectric heat flux sensors are widely used in medicine due to their ability to accurately measure changes in the heat release of the human body [1 – 20]. This allows them to be used for diagnostics and monitoring of the condition of patients, especially in cases where it is important to detect local changes in body temperature [21 – 40]. The main aspects of thermoelectric sensors [1 – 40]:

1. Operating principle:

- is based on the thermoelectric Seebeck effect, where a temperature difference between two points causes an electric voltage to arise;
- the sensor responds to the heat flux that occurs due to the temperature difference between the two sides of the sensor.

2. Medical applications:

- blood flow distribution: heat flux measurement can be used to assess blood microcirculation, which is useful in diagnosing diabetic foot or for studying skin pathologies;

- monitoring of wound conditions: in wounds or postoperative sutures, changes in heat flux may indicate inflammation or healing;
- tumor diagnosis: tumors are usually accompanied by a local increase in heat flux due to intense blood circulation in the affected area;
- neurophysiology: sensors help in measuring thermal changes caused by neural activity.

3. Sensor types:

- disposable or reusable devices;
- high-sensitivity sensors for localized measurement, for example in dermatology.

4. Advantages:

- high accuracy;
- small size, which allows them to be integrated into wearable devices;
- rapid response to changes in heat flux.

5. Production technologies:

- use of biocompatible materials;
- micromechanical design (MEMS) to ensure compactness and accuracy.

The prospects for the development of such sensors in medicine include their integration into multifunctional diagnostic and rehabilitation systems, which contributes to more accurate and comfortable monitoring of patients' health.

The purpose of this work is to develop special technological modes for the production of improved thermoelectric microthermopiles for medical purpose heat flux sensors.

1. Selection of thermoelectric material for manufacturing microthermopile

Thermoelectric semiconductor materials (TEM) must satisfy a number of requirements: maintain a high figure of merit in a wide temperature range, have significant mechanical strength, be easy to process when manufacturing samples of the required sizes, not be subject to the oxidizing action of the atmosphere, not sublimate or decompose at elevated temperatures, etc. The most important of these requirements is to achieve high values of the thermoelectric figure of merit, on which in most cases the possibility of using a thermoelectric material depends [1 – 3].

Thermoelectric materials are classified according to their operating temperature range: low-, medium-, and high-temperature. Low-temperature materials are semiconductors with the operating temperature of 0 – 350 °C, medium-temperature materials are semiconductors with the operating temperature of 350 °C – 650 °C, and high-temperature materials are semiconductors with the operating temperature of 700 °C – 1000 °C.

In this case, low-temperature materials are used to manufacture thermoelectric sensors.

As mentioned, the TEM is characterized by the figure of merit:

$$Z = \frac{\alpha^2 \sigma}{\chi} \tag{1}$$

where α is the Seebeck coefficient, σ is electrical conductivity, χ is the thermal conductivity.

$$\alpha = \frac{E}{\Delta T}, \tag{2}$$

$$\sigma = \frac{Il}{Us}, \tag{3}$$

$$\chi = \frac{I^2 Rl}{\Delta Ts}\ . \tag{4}$$

The most effective low-temperature materials are solid solutions based on Bi_2Te_3.

In the process of selecting the material for manufacturing the thermopile legs, low-temperature materials based on Bi_2Te_3 obtained by different methods were tested: single crystals (the Bridgman, Czochralski, zone melting), extrusion and pressing. Fig. 1 shows samples of n- and p-type thermoelectric material obtained by extrusion.

Fig. 1. Extruded bars of thermoelectric material of n- and p-type conductivity.

In the process of manufacturing a thermopile, such bars are cut into thin plates (Fig. 2), which are then glued together in pairs.

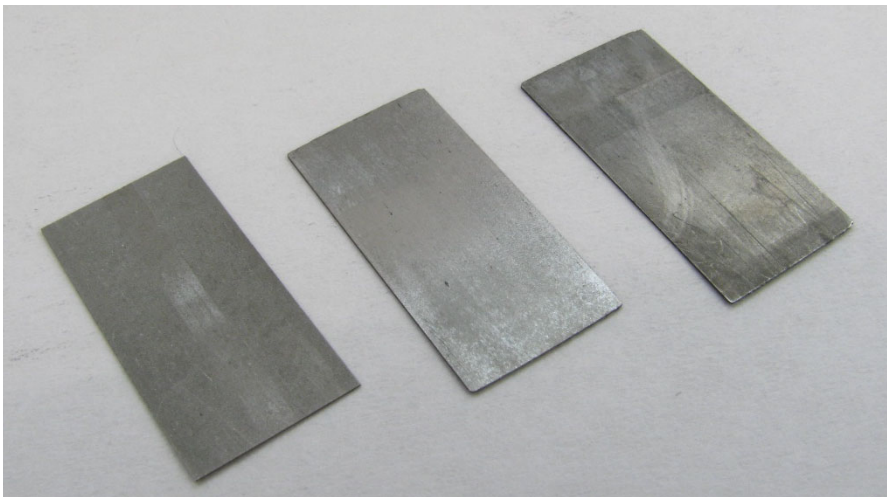

Fig. 2. Plates of thermoelectric material of n- and p-type conductivity.

Unlike single crystals grown by the Bridgman, Czochralski, and zone melting methods, the extruded material has high mechanical and technological properties; uniformity along the length of the rods, high mechanical strength, which is especially important in the manufacture of small-sized legs [1 – 3].

In the process of obtaining thermoelectric material, internal stress occurs as a result of various thermal and mechanical loads. The presence of final stresses, reaching ultimate strength values, can lead to the occurrence of micro- and macrocracks, which significantly weaken the strength of the material and can worsen its thermoelectric properties. The best strength is demonstrated by samples obtained by extrusion. The high mechanical strength of the extruded material is explained by its relatively high homogeneity.

The thermopile legs were made of semiconductor material obtained by the extrusion method.

Of great importance for the materials used in thermoelements is the degree of their homogeneity. Inhomogeneities create closed thermoelectric currents in the middle of the leg, which reduce the efficiency of thermoelements.

TEMs obtained from powders by hot or cold pressing are currently the most widely used due to their technological simplicity and the possibility of manufacturing thermoelements of the required sizes [1 – 3].

In the simplest case, when the material of all grains is homogeneous and isotropic, the contact between grains is ideal and the influence of various microscopic defects (cracks, holes, various types of inclusions and cavities) is so small that it can be neglected, the expressions for describing the electrical and thermal conductivity of pistons are isomorphic, therefore, regardless of the configuration of the grains, their sizes, mutual orientation and contact area, the ratio σ/χ should remain constant. The Seebeck coefficient should also not depend on the listed factors, so the values of powder and single-crystal materials should coincide. In most cases, the figure of merit of powder materials is somewhat lower than that of single crystals, due to additional scattering of phonons and current carriers at grain boundaries, dislocations, vacancies, microcracks; due to the presence of oxide films, etc.

When using powders from anisotropic materials, the figure of merit of pressed powders may be significantly lower than that of single crystals. The anisotropy of thermoelectric power during grain misorientation leads to the appearance of eddy currents, which also deteriorates the properties of the material.

To achieve maximum figure of merit, it is necessary to perform grain orientation. In layered materials (in low-temperature TEMs operating in the temperature range of 0 ÷ 300 °C), represented by Bi_2Te_3-based alloys, such orientation is achieved during hot pressing [1 – 3] – the grains are arranged with cleavage planes perpendicular to the pressing direction. Orientation is also achieved when manufacturing samples by extrusion. When using such techniques, the powder material becomes anisotropic and approaches single crystals in the figure of merit.

2. Design of a thermoelectric microthermopile

The disadvantage of the above-mentioned technology for manufacturing thermoelectric microthermopiles is a significant percentage of defective plates of thermoelectric material due to microcracks that may occur during their grinding.

The operating principle of a semiconductor thermopile is based on the direct thermal into electrical energy conversion in accordance with the Seebeck effect, which states that in a closed circuit consisting of two dissimilar conductors, the junctions of which are at different temperatures, a thermoelectric power arises.

The thermopile design is a monolithic block with dimensions not exceeding (22 × 22 × 20) mm^3. The block consists of 1600 *p*- and *n*-type semiconductor legs of square cross-section and insulated from each other. The *p*- and *n*-type legs are connected in pairs in a series electrical circuit (Fig. 3) and are arranged parallel to the heat flux, i.e. the heat flux passes along the thermopile legs (along the height). The extreme *p*- and *n*-type legs in the electrical circuit are connected to nickel buses, which are glued to two opposite side surfaces of the semiconductor thermopile [1 – 3].

The dimensions of the manufactured thermocouple leg are: (20 × 0.5 × 0.5) mm^3. The thermopile legs are made of extruded thermoelectric semiconductor material based on Bi_2Te_3 alloys. The average values of the electrical parameters of this material are: α_{av} = 190 μV K^{-1}, σ_{av} = 900 Ohm^{-1}cm^{-1}.

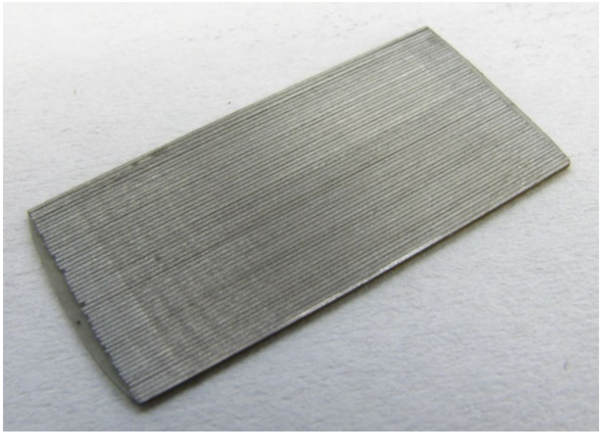

Fig. 3. Glued thermopile legs made of extruded thermoelectric material.

3. Technological modes of manufacturing thermoelectric microthermopile

Micromodules are, as a rule, single-stage thermopiles made of low-temperature thermoelectric materials based on ternary alloys of bismuth telluride [1 – 3], which acquire *p*- or *n*-type conductivity depending on their doping. The process flow chart of micromodules includes the following stages: synthesis of low-temperature thermoelectric alloys of *p*- and *n*-type conductivity; crushing of alloy ingots into powder with the required grain size with its subsequent extrusion through dies measuring 7×7 mm^2 or 5×10 mm^2. The obtained thermoelectric legs with the appropriate cross-section are characterized by high parameter values in the temperature range of 200 ÷ 600 K and good mechanical properties, which allows them to be used in production conditions. From these legs, bars of the required length and an average value of 180 ÷ 200 μV/K and 900 ÷ 1200 Ohm^{-1}cm^{-1} are cut with a diamond disk. Then the bars are cut using a mechanical wire cutting into 0.7 mm thick plates and after appropriate chemical treatment, the plates are glued together, maintaining the sequence of alternating *p*- and *n*-conductivity. The electrical insulation between the plates is a polyamide film with a thickness of 10 microns, the adhesive chosen is epoxy compound K-400 with a plasticizer. The blocks obtained after drying were cut into rows of legs of the required thickness (in our case – 0.7 mm). After chemical etching, the ends of the rows of glued legs were covered with an anti-diffusion layer, followed by the creation of interconnect coating. After these operations, we obtain the required number of micromodule units, each of which contains a selected number of half-elements (legs) connected electrochemically.

The structure of micromodule fragment is shown in Fig. 4.

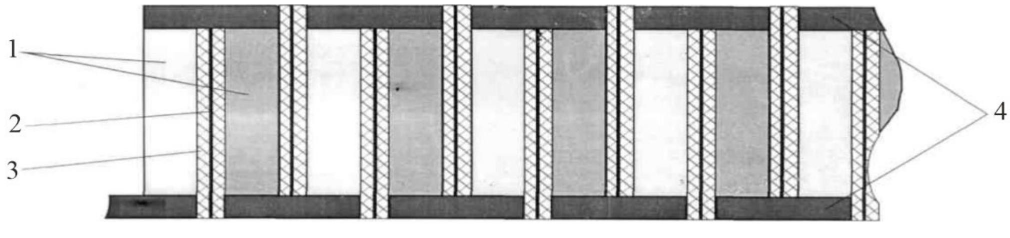

Fig. 4. Fragment of thermopile micromodule: 1 – half-elements of p- and n- type conductivity, 2 – polyamide film, 3 – epoxide compound, 4 – interconnects.

According to the above-mentioned process flow chart, 4 units of micromodules were assembled, which were glued into a block through a polyamide film and connected to each other from the heat-generating side. Fig. 5 shows the assembled and connected micromodule.

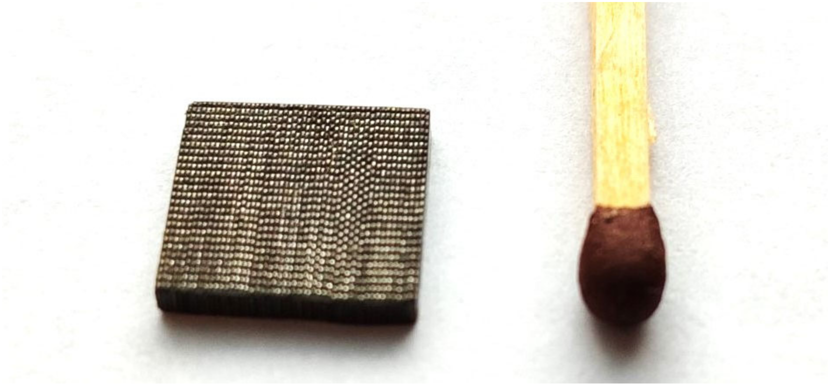

Fig. 5. Assembled and connected thermopile micromodule.

Electrical leads were soldered to the micromodule block, which is actually a thermopile, and its heat-exchange sides were covered with a heat-conducting protective layer containing boron nitrite.

In general, the technological chain for manufacturing a thermopile can be reduced to the following diagram (Fig. 6):

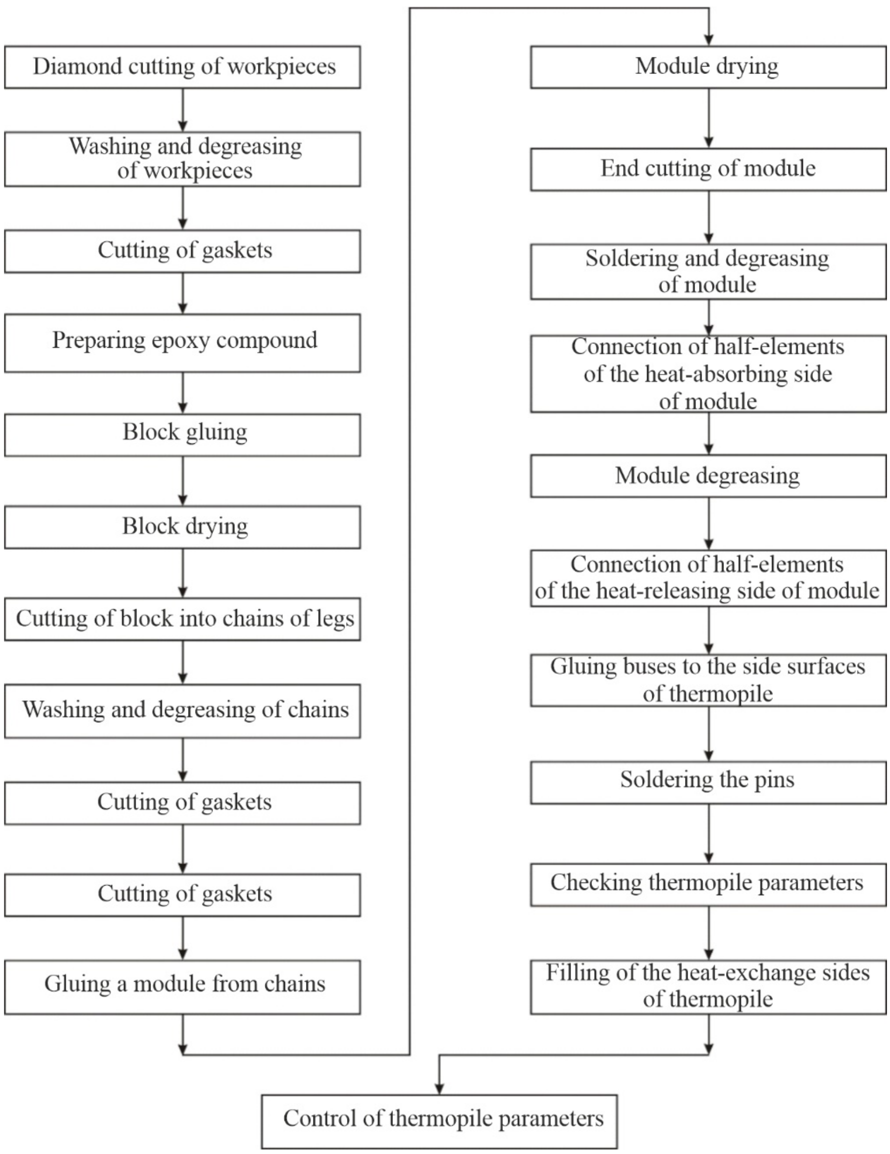

Fig. 6. Process flow chart for the production of thermopile.

3.1. Anti-diffusion layers and interconnects of thermoelectric micromodules

To create thermopiles, highly efficient low-temperature thermoelectric materials are used, which are ternary alloys based on bismuth telluride Bi_2Te_3 [1 – 3] and which acquire *p*- or *n*-type conductivity depending on their doping.

The relatively high operating temperature of the hot junctions of the micromodule half-elements requires the use of anti-diffusion layers on the surfaces of the contact half-elements. Various technologies and methods for sputtering anti-diffusion metal layers are known: chemical, electrochemical, melt immersion, mechanical deposition, vacuum sputtering, vapor phase deposition, cathodic sputtering, plasma spraying. Each of the above methods has negative and positive features. Cathode sputtering provides the best results. However, the extremely slow deposition rate and high energy consumption of the process do not allow this method to be used. Plasma spraying allows for a significant increase in the rate of coating deposition, but requires the use of rather complex and energy-intensive technological equipment. At the same time, galvanic methods are not characterized by low cost of the technological process, relative simplicity of equipment and sufficiently high quality of metal layers. The choice of materials for galvanic anti-diffusion layers is quite limited.The point is that it is necessary to ensure, in addition to significant adhesion, also a small contact resistance r_0, which significantly affects the thermoelectric figure of merit Z_{TE} of the thermoelement [1 – 3]:

$$Z_{\mathrm{TE}} = Z_{\mathrm{TEM}} \frac{1}{1+\frac{2r_0}{r}}, \tag{5}$$

where Z_{TEM} is the figure of merit of thermoelectric material, *r* is half-element resistance.

Nickel *Ni* is considered one of the most suitable for creating anti-diffusion contact layers. It provides contact resistance at the level of $1 \div 5 \cdot 10^{-6}$ Ohm·cm^2.

The reliability of *Ni* antidiffusion layers is determined by the existence of internal stresses in the metal coatings. Classical Watts sulfuric acid electrolytes are characterized by rather large values of internal stresses, therefore they cannot be used to create antidiffusion layers and interconnects of highly reliable thermoelectric microbatteries.

Ni coatings with zero internal stress values can be obtained using modern electrolytes: fluoroborate, sulfanate, sulfamate, fluoroborate, and sulfamate sulfate.

The most promising is the sulfamic acid electrolyte, which allows, by changing the electrodeposition modes, to regulate the values of stresses from tensile to compressive. The work investigated the influence of the concentration of *Ni* sulfamic acid on the values of stresses in the deposited layers obtained in electrolytes of the following composition

1. Nickel sulfamic acid, g/l	100 – 800
2. Nickel chloride, g/l	20
3. Boric acid, g/l	30
pH	4.0
t_e, °C	40 – 60
I_K, A/dm^2	3.10

The obtained results indicate that with an increase in the concentration of *Ni* sulfamic acid from 100 to 800 g/l, the tensile stress in the deposited layers decreases and reaches zero values at 650 g/l (t_e = 40 °C). If the concentration of *Ni* sulfamic acid is further increased, the tensile stresses are transformed into compressive stresses.

For the application of anti-diffusion layers and interconnects in micromodules, nickel sulfamic acid electrolyte was used. The thickness of the *Ni* layer was 100 – 120 µm. To ensure minimal internal stress values, deposition was carried out at reduced cathodic current density values. Also, in order to reduce stresses, immediately after the nickel plating, the thermopile was annealed at a temperature of 150 °C for 4 hours. As a result, a high-quality protective interconnect coating with adhesion of at least 120 kg/cm^2 and high cyclic stability was obtained.

3.2. Improving the reliability of thermoelectric microthermopile

One of the effective methods of increasing the reliability of multi-element systems is the use of redundant elements, which, in the event of a system element failure, take over the functions of the failed element in whole or in part. In this work, the so-called passive redundancy method was used. It consists of the fact that the legs of a multi-element thermopile are shunted by passive resistors, which, in the event of a leg failure, prevent the opening of the thermopile electrical circuit, and therefore the failure only entails a decrease in the thermopile power, but not a complete failure of the thermopile (Fig. 7).

This method is economical and effective with the optimal choice of the ratio of resistances of the redundant legs and redundant shunts, which depends on the failure criterion of the thermopile. Such a criterion for passive redundancy is the permissible percentage of decrease in the output power of the thermopile for the guaranteed operating time.

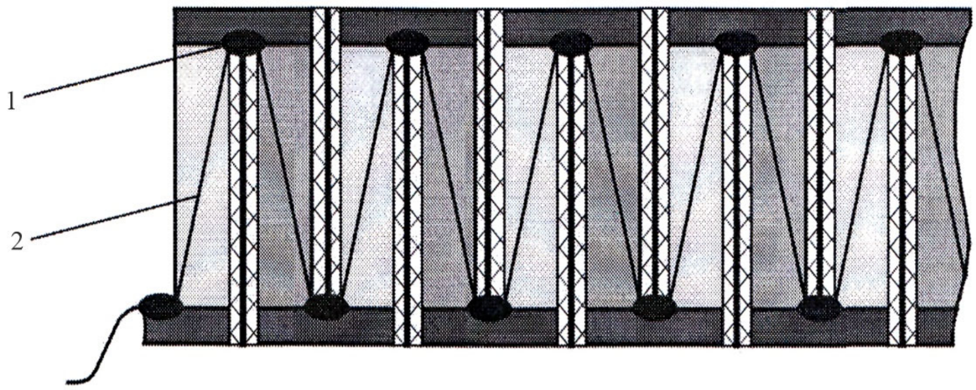

Fig. 7. Fragment of thermopile micromodule with passive resistors:
1 – soldering of passive resistor, 2 – passive resistor.

To calculate the reliability of the thermopile and optimize the ratio of the thermopile resistances and redundant elements, methods and computer programs developed at the Institute of Thermoelectricity of the National Academy of Sciences and the Ministry of Education and Science of Ukraine were used. The calculation results are given in Table 1.

Table 1

Dependence of thermopile parameters on the number of broken legs.

T_c – 50 °C T_h – 250 °C						
Number of broken legs	*U*, B	%	*W*	%	η,%	%
0	6.75	0	0.180	0	2.95	0
1	6.61	2.0	0.172	4.1	2.85	3.5
2	6.53	3.2	0.168	6.9	2.77	6.0
3	6.45	4.4	0.164	9.0	2.65	10.0
4	6.36	5.8	0.159	11.7	2.57	13.0
5	6.27	7.1	0.155	13.8	2.45	17.0

Experimental studies on the dependence of the parameters of a micro thermopile on the number of broken legs showed good agreement and coincidence with the calculation results given in Table 1.

Conclusions

1. Special technological modes for manufacturing thermoelectric micro thermopiles with an increased density of elements (up to several thousand) based on highly efficient semiconductor materials have been developed, which significantly simplifies and mechanizes the method of manufacturing thermoelectric medical purpose heat flux sensors and microgenerators for powering low-power medical equipment.
2. The dependences of the parameters of thermoelectric microthermopiles on the number of broken legs and methods for increasing the failure rate of such microthermopiles by optimally selecting the ratio of the resistances of the redundant legs and redundant shunts are established, thereby achieving an acceptable percentage of reduction in the output power of the thermoelectric thermopile for the guaranteed operating time.
3. It has been established that thermoelectric heat flux sensors are promising for monitoring and early diagnosis of inflammatory processes and cancer. The introduction of such sensors into medical practice will become an effective means of diagnosing various human diseases.

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ТЕХНОЛОГІЧНІ РЕЖИМИ ВИГОТОВЛЕННЯ ТЕРМОЕЛЕКТРИЧНИХ СЕНСОРІВ МЕДИЧНОГО ПРИЗНАЧЕННЯ

У роботі наведено технологічні режими виготовлення термоелектричних перетворювачів теплового потоку. Встановлено, що оптимальним термоелектричним матеріалом для термобатареї є низькотемпературні матеріали на основі Bi_2Te_3. Експериментально підтверджено ефективність використання таких технологічних режимів для виготовлення термоелектричних мікробатарей, здатних реєструвати лазерне випромінювання з покращеним коефіцієнтом перетворення в 1 – 1.5 порядки у порівнянні з існуючими вимірювальними перетворювачами. Вказані технологічні режими значно спрощують та механізують методику виготовлення термоелектричних сенсорів теплового потоку медичного призначення та мікрогенераторів для живлення малопотужної медичної апаратури.

Ключові слова: технологічний режим, термоелектричний перетворювач, термоелектричний сенсор теплового потоку медичного призначення.

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