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Thermoelectric thin film energy generators based on lead telluride compounds

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The work presents the results of experimental studies of thin film thermoelectric generators based on quaternary compounds PbSnAgTe and doped PbTe:Sb in the temperature range of 300–450 K. It is shown that the selected chemical composition $Pb_{14}Sn_4Ag_2Te_{20}$/PbTe:Sb and the technological processing parameters make it possible to obtain 5-μm thin films with low internal resistance and a high Seebeck coefficient, which ensures high thermoelectric power and long-term stability. The conversion efficiency of the studied thin film thermoelectric generator with 3.5 pairs of $Pb_{14}Sn_4Ag_2Te_{20}$/PbTe:Sb at a temperature difference of 100 K was 4.7%.

Keywords: thermoelectric converters, clean energy, lead telluride, doping, temperature dependencies, thickness.

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Introduction

Thermoelectric materials are widely used as energy sources [1], coolers [2], and sensors [3,4]. Lead telluride is an efficient thermoelectric material across a wide temperature range [5]. The long-standing interest in its study is driven both by its unique physicochemical properties and the relatively simple technology required to obtain high-quality crystals and films. LAST compounds based on PbTe are high-performance thermoelectric materials [6]. The thermoelectric properties of these compounds are highly sensitive to chemical composition [7]. LAST materials of both n-type and p-type can be obtained by adjusting the chemical composition, which makes the system particularly promising for power-generation applications [8]. Therefore, optimizing the material composition is important to improve thermoelectric properties.

Thin film thermoelectric miniature energy generators have broad prospects for practical application, particularly in wearable microelectronic devices [9]. Wearable electronic devices are among the most important emerging technologies for end users [10], especially for biomedical applications such as continuous real-time monitoring of daily physical and biological activity, navigation, and a range of other functions [11].

Ultralight, flexible, and shape-adaptive thermoelectric systems powered by human body heat are the subject of current research [9,12]. Removing the device for recharging or battery replacement is inconvenient and leads to loss of data-collection time for users. Therefore, there is an urgent need to meet market demands for lightweight and durable autonomous power sources for wearable electronic devices [9]. Thin film thermoelectric generators can efficiently convert thermal energy directly into electrical energy [13]. However, existing thermoelectric generators exhibit low efficiency and a rigid, brittle structure, which limits their contact area with surfaces and thus reduces heat-recovery efficiency.

The purpose of this study is to refine the fabrication technology and determine materials that can boost the performance of thin film thermoelectric generators.

I. Experimental Method

Based on the conducted studies of various base materials for fabricating a thermoelectric module,

quaternary PbSnAgTe compounds were selected for the p-type legs, while doped PbTe:Sb was chosen for the n-type legs.

n-type films were obtained from a SnTe:Sb compound containing 0.3 at.% Sb at an evaporation temperature of T_v = (870 ± 10) K, with deposition onto freshly cleaved (0001) muscovite mica substrates heated to T_p = (470 ± 10) K. The deposition time was varied within τ = (35–300) s to obtain condensate films with a thickness of d = (170–6000) nm.

p-type films were produced by depositing vapor from $Pb_{14}Sn_4Ag_2Te_{20}$ and $Pb_{16}Sn_2Ag_2Te_{20}$ sources onto freshly cleaved (0001) muscovite mica substrates. The evaporator temperature was T_v = 870 K, and the substrate temperature was T_p = 470 K. The thickness of the films was controlled by the deposition time in the range of 60–200 s.

The film thickness was measured using an MII-4 microinterferometer with digital image-processing techniques.

For the fabrication of the thin film thermoelectric energy generator, the dimensions of the p- and n-type legs were 10 mm (length) and 2.5 mm (width), with a thickness of 1–6 μm. The microgenerators consisted of 3.5 (Short) and 7.5 (Long) p/n pairs (Fig. 1).

Kinetic parameters were measured using Hall-effect methods on custom-built equipment described in [14]. The impedance-measurement technique and the corresponding calculation models are described in [15].

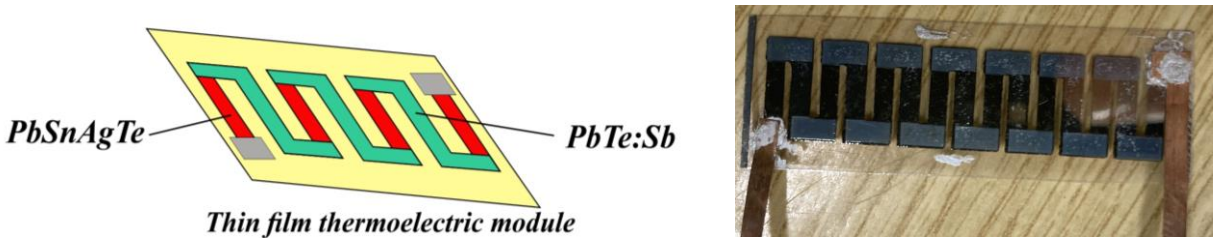
The thermoelectric parameters of the thin film energy converters were measured using a custom automated setup, whose block diagram is shown in Fig. 2.

The setup provided a temperature difference ΔT between the hot and cold junctions of the thin film thermoelectric generators, while the temperature of the cold end was maintained constant using water cooling, which significantly increases the accuracy of thermoelectric measurements.

The setup includes analog units for temperature measurement and stabilization and provides loading of the thermoelement over a wide current range from 10 μA to 100 mA. A separate galvanically isolated precision voltmeter UTM1805A with computer data output was used to measure the electrical parameters of the sample. This implementation enabled automated selection of the optimal load, measurements across wide ranges, and preliminary automated processing of the experimental results.

II. Results and Discussion

Figure 3 shows the dependences of the electrical conductivity (a), Hall carrier concentration (b), carrier mobility (c), and the Seebeck coefficient on the thickness of $Pb_{14}Sn_4Ag_2Te_{20}$ and $Pb_{16}Sn_2Ag_2Te_{20}$ films. The studied compositions exhibit p-type conductivity. Silver as a dopant in lead telluride shows a weak acceptor effect. With the introduction of Sn, p-type conductivity is obtained with relatively high hole concentrations on the order of 10^{18}–10^{19} cm^{-3}. Moreover, as the tin content



a b

Fig. 1. Configuration and photograph of the fabricated thin film generators.

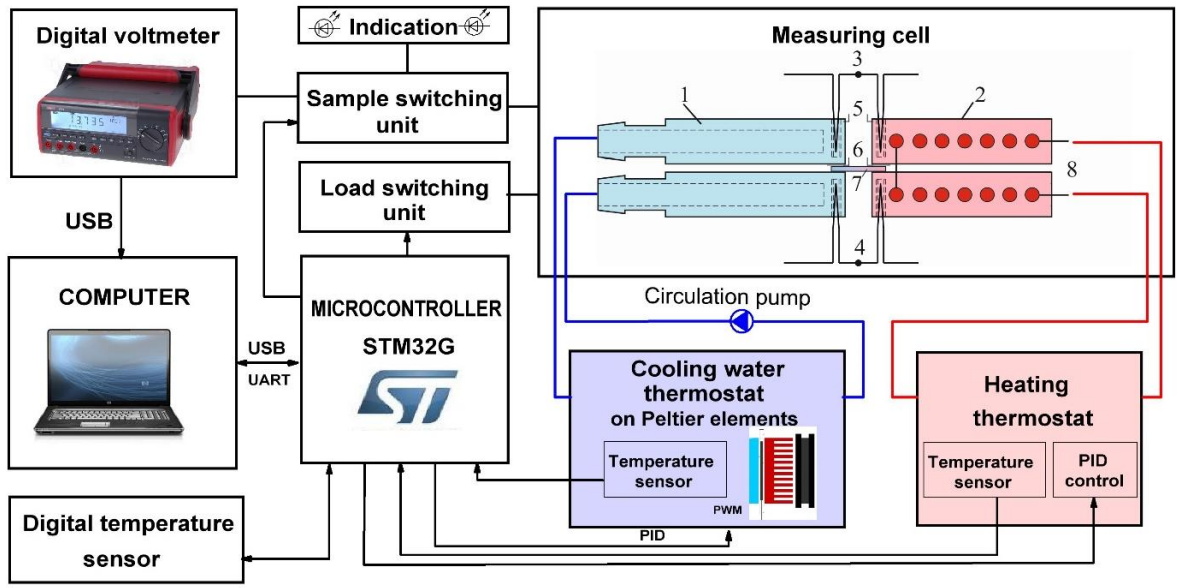


Fig. 2. Block diagram of the automated setup for measuring thermoelectric parameters of films. 1 – water radiator, 2 – electric heater, 3,4 – thermocouples, 5 – load contacts, 6 – measurement contacts, 7 – thin film sample.

increases, the carrier concentration increases (Fig. 3b, curves 1 and 2).

As the film thickness decreases, the hole concentration increases several times. This change in carrier concentration is associated with the acceptor action of oxygen. It should be noted that the contribution of the surface-oxidized layer depends on the film thickness: the thicker the film, the smaller this contribution is. The charge carrier mobility in the obtained films is quite high (Fig. 3c) and decreases significantly with decreasing thickness due to enhanced surface scattering.

The Seebeck coefficient of the obtained films is quite high and weakly dependent on their thickness, especially for films thicker than 500 nm (Fig. 3d). For $Pb_{14}Sn_4Ag_2Te_{20}$ films, the Seebeck coefficient is significantly higher. It should be noted that bulk samples made of pressed $Pb_{14}Sn_4Ag_2Te_{20}$ material exhibited the lowest thermal conductivity. This allows us to assume that films based on this chemical composition will be characterized by a sufficiently high thermoelectric figure of merit ($ZT = S^2\sigma/\kappa$). Therefore, they can be used as p-type legs for high-efficiency thin film thermoelectric energy converters.

Figure 4 shows the dependences of electrical conductivity (a), Hall carrier concentration (b), carrier mobility (c), and the Seebeck coefficient on the thickness of PbTe:Sb (0.3 at.%) films. The studied films exhibit n-type conductivity. Analysis of the obtained results indicates a donor doping effect of Sb atoms, which form an n-type material with a high carrier concentration of $(10^{19}–10^{20})$ cm^{-3}.

This carrier-concentration behavior is related to the defect subsystem of the crystal structure, particularly the type of point defects and their charge states [16]. Antimony in the PbTe crystal lattice can occupy either Pb sites or Te sites, which can be described by a disproportionation of charge states. When substituting Pb in its sublattice, Sb acts as a donor.

As the film thickness decreases, the electron concentration also decreases significantly. This change is associated with the acceptor behavior of oxygen; as the film thickness increases, the oxygen-related effects weaken considerably and become negligible for films thicker than 4–5 μm.

The charge carrier mobility in the obtained films is much lower than in PbSnAgTe samples (Fig. 4c) and decreases significantly with decreasing thickness due to enhanced surface scattering.

The Seebeck coefficient of the obtained PbTe:Sb films is comparable to that of the PbSnAgTe samples (Fig. 4c) and is practically independent of their thickness, especially for films thicker than 2 μm. One notable difference is a significant increase in the Seebeck coefficient in the region of small thicknesses; however, the low electrical conductivity in this region does not lead to an increase in thermoelectric power for films thinner than 1 μm.

The study of the thermoelectric parameters of thin film microgenerators is presented in Fig. 5 and Fig. 6. Figure 5a shows the open-circuit voltage of thin film thermoelectric generators as a function of temperature difference for film elements of various thicknesses consisting of 7.5 p/n pairs of $Pb_{16}Sn_2Ag_2Te_{20}$ (p-leg) and PbTe:Sb (0.3 at.%) (n-leg). The output voltage of all obtained samples is proportional to the temperature difference. The Seebeck coefficient is practically independent of temperature and is seven times higher for films of smaller thickness (up to 1 μm) compared to films with a thickness of 2–5 μm (Fig. 5b).

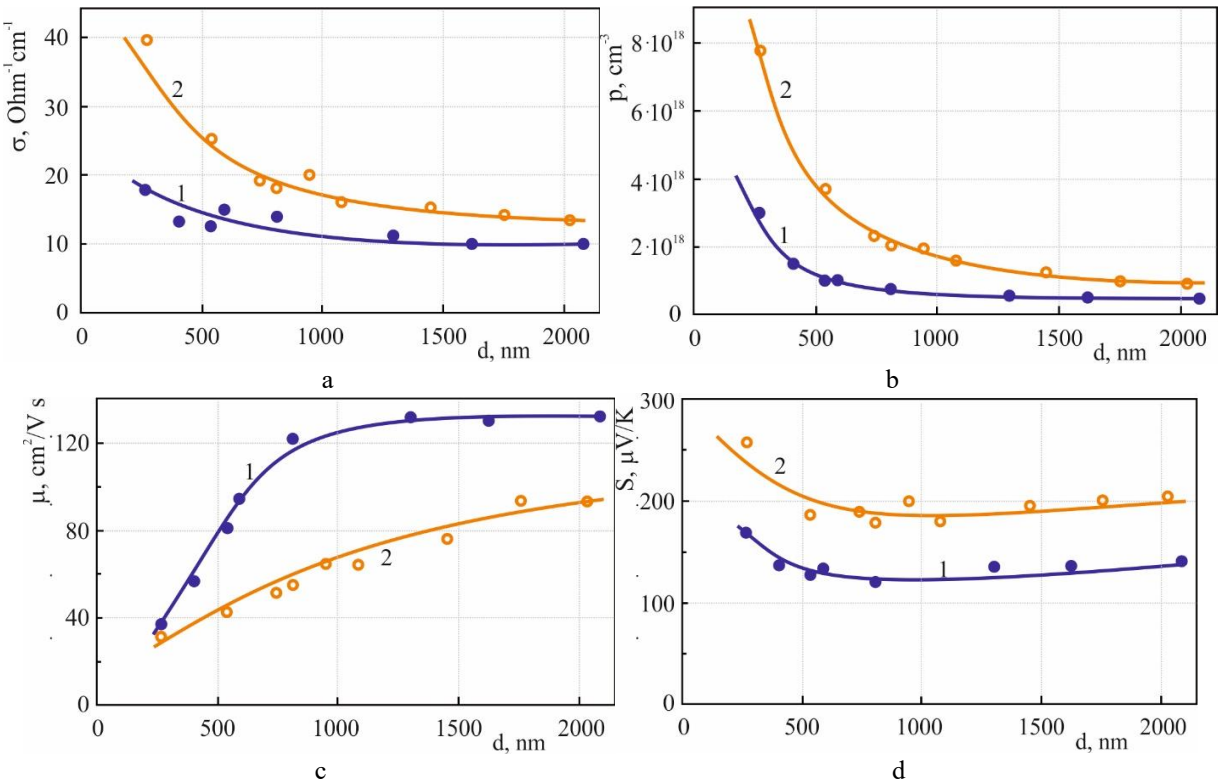


Fig. 3. Dependence of electrical conductivity σ (a), Hall carrier concentration (b), Hall mobility of charge carriers (c), and the Seebeck coefficient on the film thickness: ●, 1 – $Pb_{16}Sn_2Ag_2Te_{20}$; ○, 2 – $Pb_{14}Sn_4Ag_2Te_{20}$.

The maximum output power of thin film thermoelectric microgenerators is calculated from the internal resistance and the generated potential difference. The output power can be calculated as $P = R_{load}\,(\Delta U / (R_{load} + r))^2$, where R_{load} is the electrical load resistance, ΔU is the open-circuit voltage, and r is the internal resistance of the generator. Assuming that maximum power is achieved when $R_{load} = r$, then $P_{max} = \Delta U^2/(4r)$.

The internal resistance of the thin film thermoelectric generators was measured at such a load current that the load resistance was close to the internal resistance of the

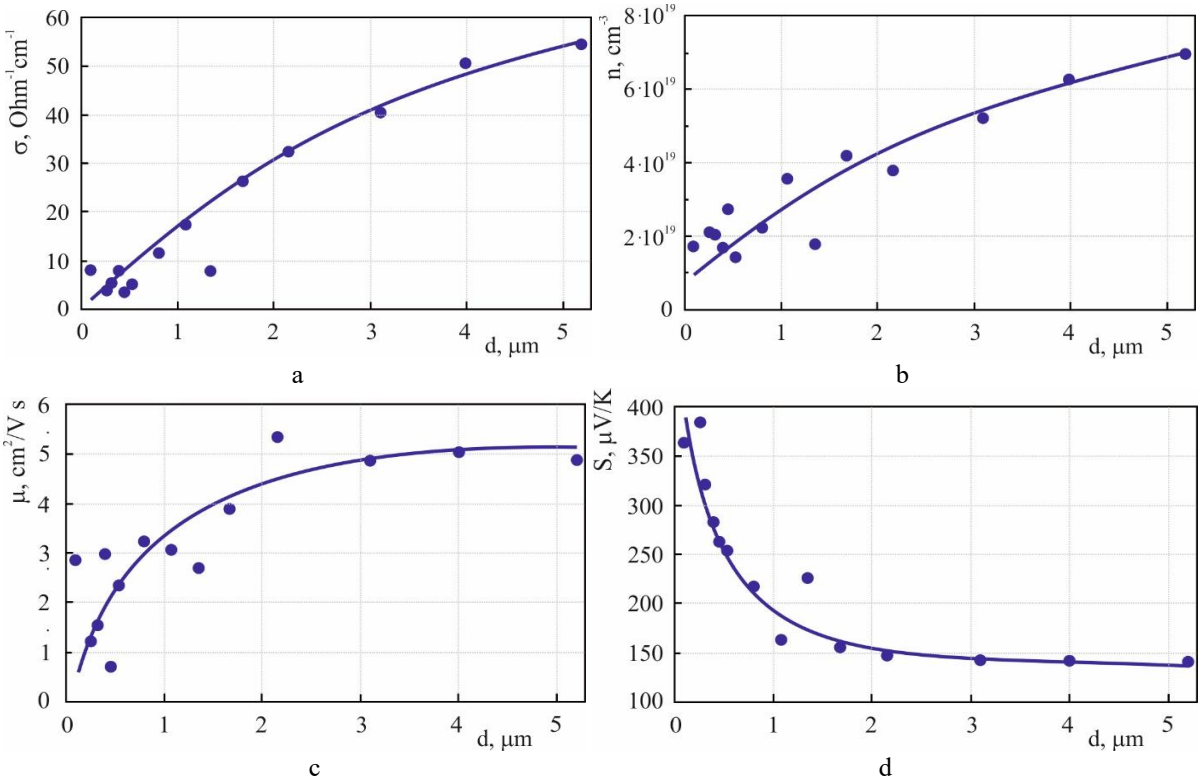


Fig. 4. Dependence of electrical conductivity σ (a), Hall carrier concentration (b), Hall mobility of charge carriers (c), and the Seebeck coefficient (d) on the thickness of PbTe:Sb (0.3 at.%) films.

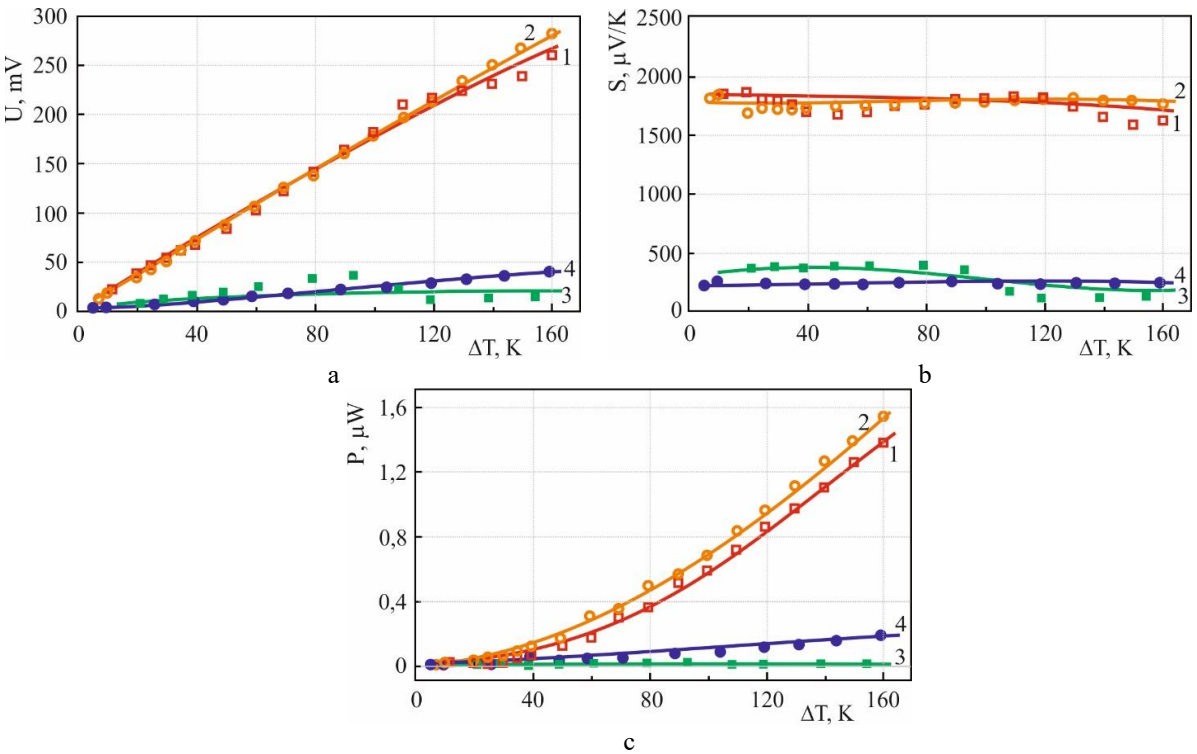


Fig. 5. Dependence of the generated potential difference U (a), Seebeck coefficient S (b), and maximum power P (c) on the temperature difference for thermoelectric micromodules based on $Pb_{16}Sn_2Ag_2Te_{20}$ (p-leg) and PbTe:Sb (0.3 t.%) (n-leg) of various thicknesses (7.5 p/n pairs). Film thickness:
□, 1 – 0.27 μm; ○, 2 – 1 μm; ■, 3 – 2.8 μm; ●, 4 – 5.2 μm.

source, and for these generators it was 9–11 kΩ.

The highest obtained thermoelectric power for the generator consisting of 7.5 pairs of $Pb_{16}Sn_2Ag_2Te_{20}$ (p-leg) and PbTe:Sb (0.3 at.%) (n-leg) was 1.6 μW at a temperature difference of 160 K for samples with a thickness of about 1 μm (Fig. 5c). This may be explained by an optimal thickness ratio for these compositions, since thicker $Pb_{16}Sn_2Ag_2Te_{20}$ films were less structurally perfect, which significantly reduced their electrical conductivity and increased the internal resistance of the generator. This indicates the need for additional studies on the influence of fabrication technological factors on generator parameters to produce devices with lower internal resistance.

In Fig. 6a, the open-circuit output voltage of thin film thermoelectric generators is shown as a function of temperature difference for film elements with a thickness of about 2.5 and 5 μm, consisting of 3.5 p/n pairs of $Pb_{14}Sn_4Ag_2Te_{20}$ (p-leg) and PbTe:Sb (0.3 at.%) (n-leg). The output voltage of all obtained samples is also proportional to the temperature difference. The Seebeck coefficient exhibits a stronger temperature dependence and is approximately seven times higher than that of films of similar thickness with p-legs based on $Pb_{16}Sn_2Ag_2Te_{20}$ (Fig. 5b), despite these thermogenerators having two times fewer thermoelectric pairs.

The internal resistance of thin film thermoelectric generators with a p-leg based on $Pb_{14}Sn_4Ag_2Te_{20}$ and a thickness of 5 μm was about 2 kΩ.

The highest obtained thermoelectric power for the generator consisting of 3.5 pairs of $Pb_{14}Sn_4Ag_2Te_{20}$ (p-leg) and PbTe:Sb (0.3 at.%) (n-leg) was 4.3 μW at a temperature difference of 160 K for samples with a thickness of about 5 μm (Fig. 5c). Under the selected technological fabrication conditions, thin films with a thickness of 5 μm based on $Pb_{14}Sn_4Ag_2Te_{20}$ / PbTe:Sb demonstrate low internal resistance and a high Seebeck coefficient, which ensures high thermoelectric power and long-term stability.

Impedance spectroscopy is a powerful method for studying power-generating devices. This technique makes it possible to separate the frequency regions of most processes that affect device performance. However, interpreting the equivalent circuit requires models that adequately describe the underlying physical processes. In the case of thermoelectric converters, several physical models enable partial interpretation of the information obtained from impedance spectra, including those described in [17, 18].

An important property of a thermoelectric energy converter is its ability to operate as an electrical power generator under an applied temperature gradient, as well as to transfer heat and create a temperature gradient when an electric current flows. When an alternating current passes through a thermoelement, it behaves like a capacitor, storing part of the energy in thermal form and vice versa. An equivalent electrical circuit (Fig. 7) can be used to represent this behavior when interpreting the impedance spectra.

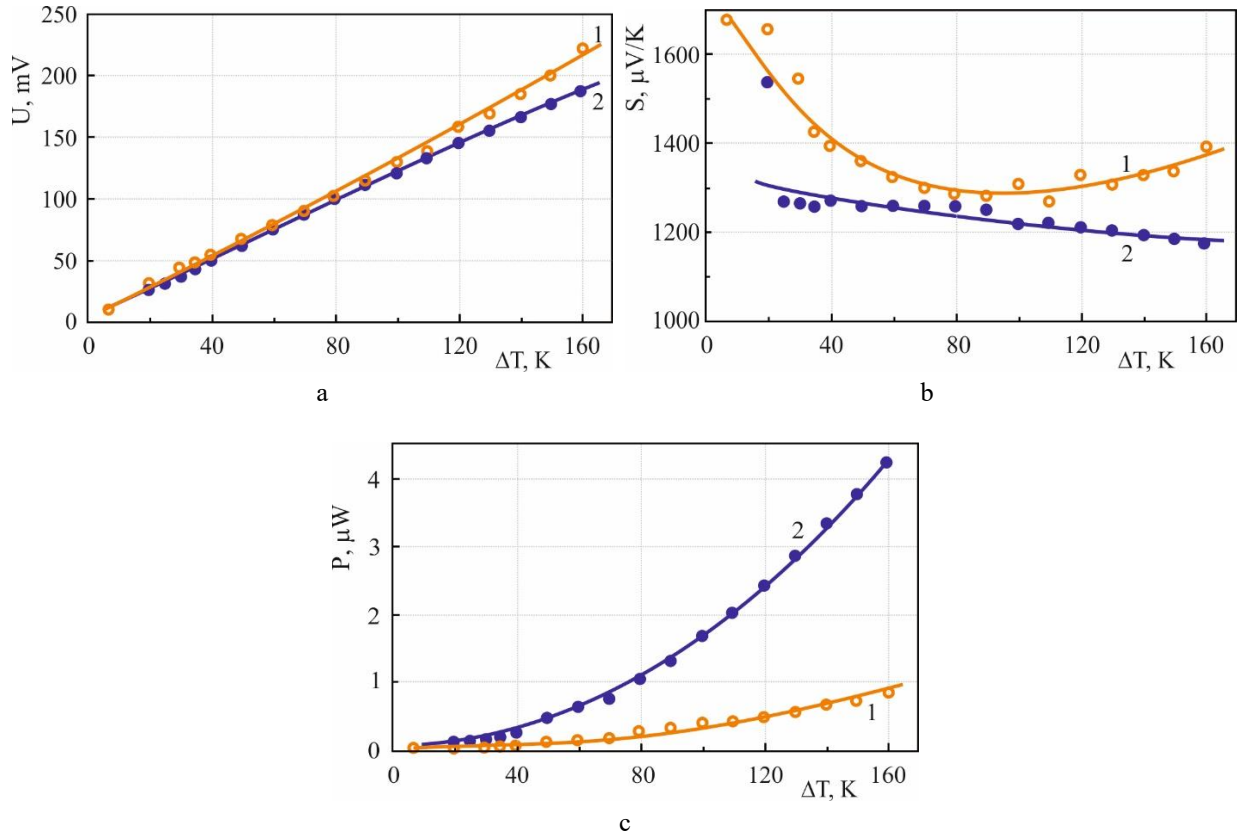


Fig.6. Dependence of the generated potential difference U (a), Seebeck coefficient S (b), and maximum output power P (c) on the temperature difference for thermoelectric micromodules based on $Pb_{14}Sn_4Ag_2Te_{20}$ (p-leg) and PbTe:Sb (0.3 at.%) (n-leg) of various thicknesses (3.5 p/n pairs). Film thickness: ○, 1 – 2.5 μm; ●, 2 – 5.0 μm.

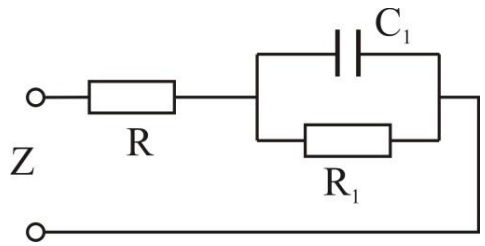


Fig. 7. Equivalent circuit of a thermoelectric element.

Within the framework of the electrothermal model [17], the total impedance Z of a thermoelectric element consists of the internal electrical resistance R and the impedance $Z_T=S\Delta T/i$ (S is the Seebeck coefficient, ΔT is the temperature difference, i is the current) caused by thermoelectric heat transport. The impedance Z_T must correspond to the internal resistance of the voltage source originating from thermoelectric effects and is determined by the thermal capacitance C_T and the thermal resistance R_T which in turn are related to R_1 and C_1 as described in [17]:

$$C_T = S^2 T C_1, \quad (1)$$

$$R_T = \frac{R_1}{S^2 T}, \quad (2)$$

where T is the temperature.

The thermoelectric figure of merit can be determined without performing direct thermal measurements by analysing the zeros $\omega_z=(R_1+R)/(R_1C_1R)$ and poles $\omega_p=1/(R_1C_1)$ of the function Z(s) [17], according to $ZT=(\omega_z/\omega_p)-1$.

Knowing the geometric dimensions l, s, and the number of elements $n$in the thermoelectric module, one can determine its thermal conductivity $\kappa=l/(nsR_T)$ and electrical conductivity $\sigma=nl/(sR)$ (Table 1).

Table 1.
Thermoelectric parameters of the investigated modules.

Parameter	T=300 K	T=323 K	T=373 K
Seebeck coefficient, μV/K	1660	1760	1873
Number of elements	3,5		
Seebeck coefficient per element, μV/K	474,3	502,8	535,1
Thermoelectric figure of merit ZT	0,34	0,92	1,25
Element length, mm	10		
Cross-sectional area, m²	0,00014		
Electrical conductivity, $\Omega^{-1}\cdot cm^{-1}$	18,8	29,4	35,6
Thermal conductivity, W/(cm·K)	0,037	0,026	0,030

Thermoelectric materials require a specific combination of properties: high carrier mobility, low lattice thermal conductivity, and high dielectric permittivity. The maximum conversion efficiency for a thin film module can be expressed as

$$\eta_{max} = \frac{\Delta T}{T_H} \frac{\sqrt{1+ZT}-1}{\sqrt{1+ZT}+\frac{T_C}{T_H}},$$

where T_H, T_C are the temperatures of the hot and cold sides, $\Delta T = T_H - T_C$ is the temperature difference between them, and ZT is the thermoelectric figure of merit.

Based on the calculated ZT values obtained from impedance measurements, the conversion efficiency of the investigated thin film modules was estimated for a temperature difference of 100 K (Fig. 8). The obtained efficiency values are comparable to those of similar modules based on IV–VI compounds [19].

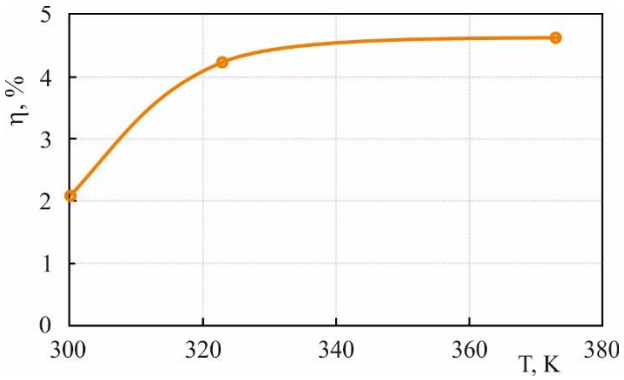


Fig. 8. Thermoelectric efficiency of a thin film module based on PbSnAgTe compounds and doped PbTe:Sb.

Thus, the obtained thin film elements demonstrate high thermoelectric figure of merit and and advance the prospects for integrating thermoelectric energy converters into scientific and technological systems.

Conclusions

Experimental studies were carried out to investigate the influence of film thickness and chemical composition on the thermoelectric properties of thin films of $Pb_{14}Sn_4Ag_2Te_{20}$, $Pb_{16}Sn_2Ag_2Te_{20}$, and PbTe:Sb. It was shown that with decreasing thickness of PbSnAgTe films, the hole concentration increases several times, whereas for PbTe:Sb films, the carrier concentration significantly decreases. This variation in carrier concentration is associated with the acceptor effect of oxygen.

Experimental investigations of thin film thermoelectric generators based on four-component PbSnAgTe compounds and doped PbTe:Sb were conducted in the temperature range of 300–450 K. It was found that the chosen chemical composition $Pb_{14}Sn_4Ag_2Te_{20}$/PbTe:Sb and the fabrication conditions make it possible to obtain films with a thickness of 5 μm, low internal resistance, and high Seebeck coefficient, ensuring high thermoelectric power and temporal stability.

Based on mathematical modeling, the theoretical conversion efficiency of the investigated thin film thermoelectric generator with 3.5 p/n pairs of $Pb_{14}Sn_4Ag_2Te_{20}$/PbTe:Sb at a temperature difference of 100 K was estimated to be 4.7 %, making these materials promising for use in high-efficiency thin film thermoelectric energy converters.

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Термоелектричні тонкоплівкові генератори енергії на основі сполук телуриду свинцю

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У роботі представлено результати експериментальних досліджень тонкоплівкових термоелектричних генераторів на основі чотирьохкомпонентних сполук PbSnAgTe та легованого PbTe:Sb в діапазоні температур 300-450 К.

Показано, що вибрані хімічний склад Pb14Sn4Ag2Te20/PbTe:Sb та технологічні фактори отримання дають можливість отримати плівки товщиною 5 мкм з низьким внутрішнім опором та високим значенням коефіцієнта Зеєбека, що і забезпечує високу термоелектричну потужність та стабільність у часі. Величина ефективності перетворення для досліджуваного тонкоплівкового термоелектричного генератора 3,5 пар Pb14Sn4Ag2Te20/PbTe:Sb при різниці температур 100К становила 4,7 %.

Ключові слова: термоелектричні перетворювачі, чиста енергія, телурид свинцю, легування, температурні залежності, товщина.