

Investigation of Temperature Dependencies of Seebeck Coefficient and Electrical Conductivity of $\text{Ge}_2\text{Sb}_2\text{Te}_5$ Thin Films

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At present, the chalcogenide semiconductors based on the Ge-Sb-Te system materials are actively used due to their successful application in the optical phase change memory and the perspectives of the application in the electric phase change memory.

The simultaneous study on the Seebeck coefficient and the electrical conductivity temperature dependencies for the $\text{Ge}_2\text{Sb}_2\text{Te}_5$ thin films, deposited by thermal evaporation in vacuum, has been performed. The structure and composition of the deposited thin films have been established. The Seebeck coefficient measurements point to the p-type conductivity of the layers over the entire temperature range. It has been determined that with the temperature increase from the room temperature to 135 °C the Seebeck coefficient and the resistivity have decreased from 450 to 360 $\mu\text{V/K}$ and from 30.9 to 0.47 Ωcm , respectively. In the temperature range from 135 up to 145 °C the drastic decrease of the parameters to 35 $\mu\text{V/K}$ and 0.0029 $\Omega\text{-cm}$ occurs, respectively, which is due to the crystallization process. The estimation of the activation energies for the Seebeck coefficient and the electrical conductivity indicates to a possible two-channel conduction model in the p-type amorphous $\text{Ge}_2\text{Sb}_2\text{Te}_5$ thin films.

The developed and manufactured software-hardware complex permits to simultaneously investigate the temperature dependencies of the Seebeck coefficient and electrical conductivity of the phase change memory materials thin films.

Keywords: phase change memory; thermal electricity; Seebeck coefficient; electric conductivity; thin films; $\text{Ge}_2\text{Sb}_2\text{Te}_5$.

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Исследование температурных зависимостей коэффициента термоЭДС и электропроводности тонких пленок материала фазовой памяти $\text{Ge}_2\text{Sb}_2\text{Te}_5$

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В настоящее время активно исследуются халькогенидные полупроводники на основе материалов системы Ge–Sb–Te в связи с их успешным применением в оптической фазовой памяти и перспективами применения в электрической фазовой памяти.

Проведено одновременное исследование температурных зависимостей коэффициента термоЭДС и электропроводности тонких пленок $\text{Ge}_2\text{Sb}_2\text{Te}_5$, осажденных вакуум-термическим испарением. Установлены структура и состав осажденных тонких пленок. Измерение термоЭДС указывает на *p*-тип проводимости слоев во всем температурном диапазоне. Установлено, что с увеличением температуры от комнатной до 135 °С коэффициент термоЭДС уменьшается от 450 до 360 мкВ/К, а удельное сопротивление – от 30,9 до 0,47 Ом·см. В диапазоне температур от 135 до 145 °С происходит резкое падение параметров – коэффициента термоЭДС до 35 мкВ/К, сопротивления до 0,0029 Ом·см, что обусловлено процессом кристаллизации. Оценка энергий активации коэффициента термоЭДС и электропроводности указывает на возможный двухканальный механизм проводимости в аморфных тонких пленках $\text{Ge}_2\text{Sb}_2\text{Te}_5$ *p*-типа.

Разработанный и изготовленный программно-аппаратный комплекс позволяет одновременно исследовать температурные зависимости коэффициента термоЭДС и электропроводности тонких пленок материалов фазовой памяти.

Ключевые слова: фазовая память; термоэлектричество; термоЭДС; коэффициент Зеебека; электропроводность; тонкие пленки; $\text{Ge}_2\text{Sb}_2\text{Te}_5$.

Для цитирования: Исследование температурных зависимостей коэффициента термо-ЭДС и электропроводности тонких пленок материала фазовой памяти $\text{Ge}_2\text{Sb}_2\text{Te}_5$ / Д.Ю. Терехов, П.И. Лазаренко, А.А. Шерченков и др. // Изв. вузов. Электроника. – 2017. – Т. 22. – № 6. – С. 518–527. DOI: 10.214151/1561-5405-2017-22-6-518-527

Introduction. Currently, chalcogenide semiconductors based on the materials of the Ge-Sb-Te system are actively investigated in connection with their successful commercial application in optical phase change memory (DVD-R / RW, DVD + R / RW and Blu-ray discs) and the prospects for use in electrical phase change memory [1]. The principle of phase change memory is based on a significant change in the electrical and / or optical properties of the chalcogenide semiconductor due to rapid phase transformations of the material occurring in the nanoscale volume under external low-energy effects (laser or current pulses). The current stage of the development of phase change memory technology is associated with the use of thin-film chalcogenide materials on the quasi-binary line GeTe-Sb₂Te₃, mainly Ge₂Sb₂Te₅.

Studies of thin Ge₂Sb₂Te₅ layers by X-ray diffraction showed that they crystallize into a metastable structure similar to the structure of common salt (fcc structure, symmetry group Fm3m). Te atoms occupy anionic positions in an elementary cell of NaCl type, and cationic ones are occupied at random by the atoms of germanium, antimony and vacancies (~20 %). The high content of vacancies in the crystalline phase, behaving like acceptors, leads to the fact that the crystalline phase of the NaCl type has a high p-type conductivity [2]. However, up to the present time, the electrophysical properties and mechanisms of charge carrier transport in thin films of materials of the Ge-Sb-Te system are not completely clear, which makes it difficult to target the improvement of phase change memory technology. Be noted that chalcogenides based on the ternary system Ge-Sb-Te belong to the class of telluride-containing materials widely used in thermoelectricity [2]. In [3, 4] is shown that volume materials on the quasibinary line GeTe-Sb₂Te₃, as well as thin films based on them in the amorphous and crystalline states, have high values of the Seebeck coefficient. In addition, they have very low thermal conductivity, which is due to the peculiarities of their structure, in particular, the presence of weakly bound atomic layers, formed by heavy elements with large atomic radii, and a large number of defects in the form of vacancies. These structural features can lead to a strong scattering of phonons, and, consequently, to a significant decrease in thermal conductivity.

Thus, the materials of the phase change memory, of the quasi-binary line GeTe-Sb₂Te₃, have high thermoelectric properties and are of interest for the use of thermoelectric devices. However, information on the thermoelectric properties of layers of PCM materials, including the effect of phase transitions on them, are few and noticeably different [4–7], which may be due to the difficulty of obtaining thin films of multicomponent materials of the Ge -Sb-Te system.

In [4–8] it is shown that thermoelectric effects can have a significant effect on the heat release in a phase change memory cell and, as a consequence, at the level of the programming current. Together with the decrease in the size of devices, the temperature gradient increases substantially, and thermoelectric phenomena begin to exert an increasing influence on the operation of PCM cells. Taking into account the influence of thermoelectric effects it will be possible to develop nanodimension cells of phase change memory with a reduced programming current and ultra-low energy consumption.

Thus, the simultaneous investigation of the temperature dependences of the Seebeck coefficient and the electrical conductivity of thin films of materials of the Ge-Sb-Te system will form the basis for further optimization of the design and technology of fabricating PCM cells with allowance of thermoelectric phenomena, and also to obtain additional information on the charge transport mechanisms. In connection with this, the purpose of this paper was the simultaneous measurement and subsequent analysis of the temperature dependences of the Seebeck coefficient and the electrical conductivity of thin Ge₂Sb₂Te₅ films.

Experiment. Measurement of temperature dependences of Seebeck coefficient and resistivity carried out using the developed software and hardware system (PAK). The basic electrical scheme of the PAK and 3D model of the sample holder is shown in Fig. 1. The sample holder designed on the ceramic base with a built-in heater (BH) with a power of 180 Watts, which ensures rapid heating of the internal volume of the measurement chamber. The temperature control of the ceramic base is carried out by two chromel-alumel thermocouples (type K). Gradient heaters (GH) with a power of 30 Watts, fixed to the ceramic substrate allows to create and control temperature gradient of the experimental samples. Herewith, one of the heaters is mounted on the guides, which makes it possible to vary the distance between them and to measure the samples with different geometric dimensions. Built-in platinum resistance thermometers PT1000 provide a high accuracy in temperature measurement of gradient heaters.

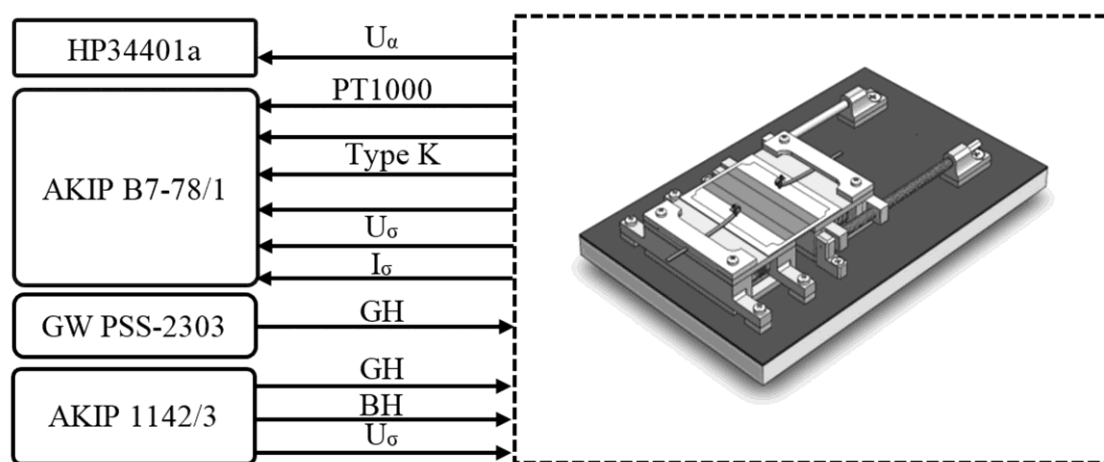


Fig.1. The principle electric scheme of the hardware and software set-up and the 3D model of the measuring station

The control of BH and GH is carried out by two laboratory power sources AKIP 1142/3 and GW Instek PSS-3203. The additional channel AKIP 1142/3 is used to form the voltage U_{σ} on the current-carrying tracks of the sample, intended for measuring the electrical conductivity. Using AKIP V7-78/1 in conjunction with a 10-channel scanner, the thermocouple voltage, the resistance of the PT1000 sensors and the current I_{σ} were measured. Measurement of the thermoelectric voltage U_{α} of the sample is carried out by a digital multimeter HP34401a with a high input resistance $> 10 \text{ G}\Omega$ and a long integration time of 100 PLCs. To connect the test equipment to the functional elements of the measuring chamber, a dedicated connector with a built-in PT1000 sensor is provided. Monitoring the temperature of the connector allows software to compensate for the cold ends of the thermocouples and the magnitude of the Seebeck coefficient that occurs in current-carrying contact conductors. To increase the accuracy of measurements, the following operations were performed:

- 1) Calibration of chromel-alumel thermocouples and platinum temperature sensors;
- 2) software development and adjustment of the system of simultaneous PID control of BH and GN;
- 3) Calibration measurements of the Seebeck coefficient using materials with standard Seebeck coefficient values (chromel, alumel, Pb, Bi).

The reliability of temperature measurement was achieved by conducting preliminary calibration of chromel-alumel thermocouples and platinum temperature sensors PT1000 used in

PAK. The calibration was carried out with the use of high-precision equipment of the collective center Electronic devices and equipment of National Research University of Electronic Technology.

The control of the measurement process in real time is provided by software developed in the LabView engineering and graphic programming environment and including the state machine. The software allows measurements in manual and automatic modes with control of the heating process by a finite state machine controlling a system of three PID regulators. One of the input parameters of the GN PID regulators is the temperature of the BN. Thus, in PID controllers, a monitoring mode of operation is provided and continuous maintenance of a predetermined temperature gradient over the entire measured temperature range. In the automatic mode of heating and maintaining the temperature, the maximum temperature in the measuring chamber, the heating step and the holding time are set as input parameters of the state machine.

The adjustment of the PID control system was carried out using a graphical method that enabled the adjustment of the PID coefficients. Fig. 2 represents the result of the PID control system maintaining the temperature gradient over the sample when the temperature of the BH is changed in the automatic mode.

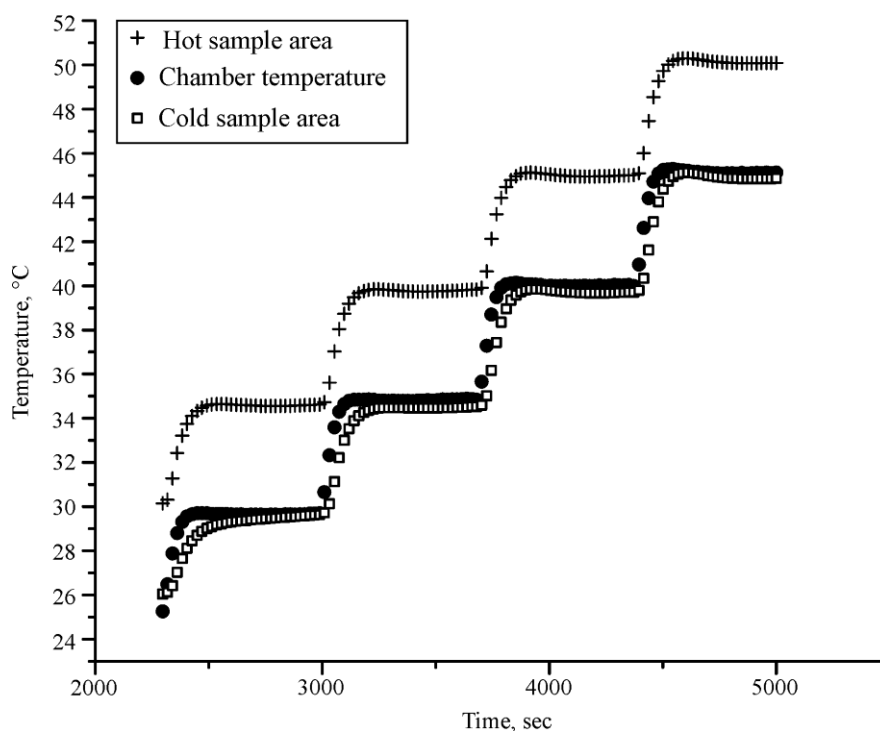


Fig.2. Maintaining of the temperature gradient along the sample by the PID control system with the temperature variation by the built-in heater

Calibration measurements of the Seebeck coefficient were carried out using as reference thermoelectric metals: bismuth (GOST 10928-90), lead (TU MHP 113-40), wires from alumel (MNMc 2-2-1) and chromel (HX 9, 5) alloys.

The measurements were carried out at room temperature, the temperature gradient was 10 °C, and the holding time for the regime was 120 min for all the investigated materials. The results of measurements of the Seebeck coefficients in comparison with the reference values [9] are shown in Fig. 3.

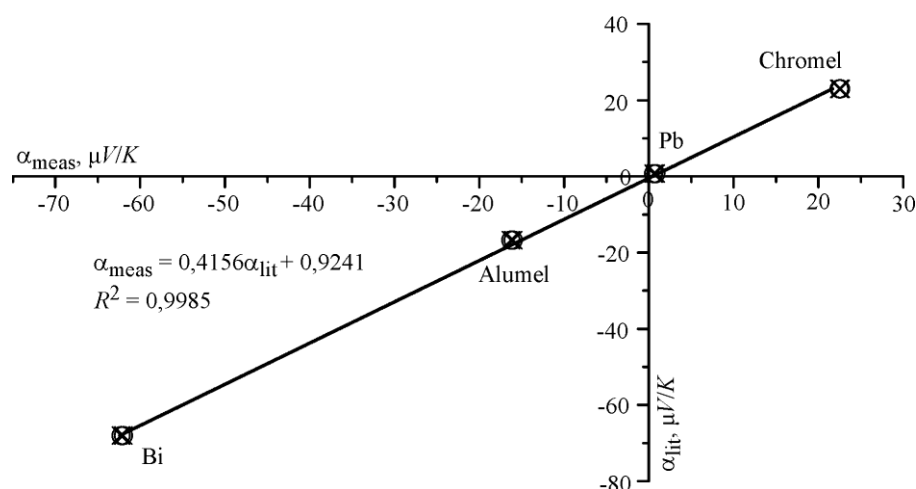


Fig.3. Interrelation of the experimentally obtained (α_{meas}) and reference (α_{lit}) values of Seebeck coefficients

The results of the calibration measurements showed that the deviation of the measured values of the of Seebeck coefficient for all materials is not more than 0.5 $\mu\text{V/K}$ from the reference.

The obtained correlation dependence of the experimental and reference data is well described by means of a linear equation with a high correlation coefficient (see Fig. 3). In the future, the obtained values of the Seebeck coefficients were programmed using the obtained equation.

The test samples were a polished, 60×47 mm² ceramic substrate on which W electrodes with a TiN sub layer were deposited first through a mask using magnetron sputtering, over which a Ge₂Sb₂Te₅ layer was deposited. The interelectrode distance was 10 mm, the thickness of the Ge₂Sb₂Te₅ layer was ~ 130 nm.

The deposition of thin Ge₂Sb₂Te₅ films by vacuum-thermal evaporation (VTE) was carried out on the UVN-2M-1 unit using the Knudsen cell, which is a quartz capacitance with resistive molybdenum heating.

As the evaporated material, polycrystalline Ge₂Sb₂Te₅ was synthesized at the Kurnakov Institute of General and Inorganic Chemistry, Russian Academy of Sciences by the method presented in [2]. The residual pressure in the chamber during the deposition of the Ge₂Sb₂Te₅ layer was approximately 2×10^{-5} mm Hg. The temperature of the substrate did not exceed 50 °C, which made it possible to obtain thin films in an amorphous state. To control the thickness of deposited films, a scanning atomic-force microscope NT-MDT Solver Pro was used. The elemental composition of the deposited films was investigated by X-ray spectral microanalysis (RSMA) using a scanning electron microscope JOEL JSM 7001F with an Inka Oxford elemental analysis system. The averaged values of the element concentrations for the initial amorphous films (Ge = 23.1 at.%, Sb = 23.2 at.%, Te = 53.7 at.%) were found to be close to the stoichiometric composition (Ge_{22,2}Sb_{22,2}Te_{55,6}) for Ge₂Sb₂Te₅.

To study the structure of thin films, we used a Rigaku Smart LAB X-ray diffractometer in the Center of Shared Facilities "MST & ECB. Fig. 4 (a) shows the diffractogram of the initial thin film Ge₂Sb₂Te₅, indicating the amorphous state of the layer material.

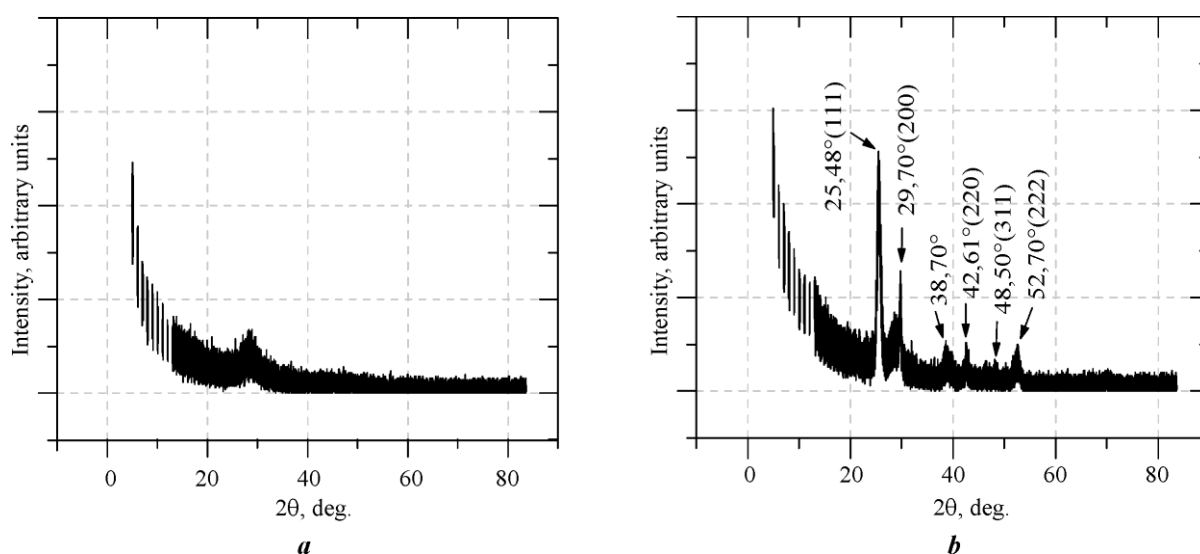


Fig.4. Diffractograms for the $\text{Ge}_2\text{Sb}_2\text{Te}_5$ thin films: *a* – without heat treatment, *b* – after heat treatment at 160°C

The heat treatment of this film at a temperature of 160°C for 15 minutes leads to the appearance of reflexes (Fig. 4 (b)), indicative of the crystallization of a thin film. The following reflexes were identified at $2\theta \sim 25.48, 29.7, 42.61, 48.50$ and 52.7° corresponding to the face-centered (111), (200), (220), (311), (222) cubic (fcc) lattice of the NaCl type. The position of these reflexes correlates with the position of the peaks presented in [10, 11]. However, their intensity is different, which may be due to the peculiarities of vacuum-thermal evaporation used in this work, and magnetron sputtering - in [10, 11].

Results and discussion. The temperature dependences of the Seebeck coefficient and resistivity were measured in the temperature range from room temperature up to 300°C with a heating step of 5°C and a time delay of 10 min. Fig. 5 shows the results of measuring the temperature dependence of the Seebeck coefficient and resistivity for thin films of $\text{Ge}_2\text{Sb}_2\text{Te}_5$. As can be seen from Fig. 5, with the increase of temperature from ambient to 135°C , a decrease of the Seebeck coefficient from 450 to $360\ \mu\text{V/K}$ and resistivity from 30.9 to $0.47\ \Omega\cdot\text{cm}$, which is typical for chalcogenide semiconductors in the amorphous state. In the temperature range from 135 to 145°C causes a sharp drop of the measured parameters up to $35\ \mu\text{V/K}$ and $0.0029\ \Omega\cdot\text{cm}$, respectively is observed. With a further increase in temperature, the Seebeck coefficient and the resistivity changes insignificantly. Thus, the character of the change in the Seebeck coefficient is in good agreement with the change in the resistivity of the material under study over the entire temperature range.

The sharp decrease in the Seebeck coefficient and resistivity of thin $\text{Ge}_2\text{Sb}_2\text{Te}_5$ films in the range from 135 to 145°C can be explained by the phase transition of the amorphous structure to the cubic type NaCl (fcc), which is a low-temperature metastable modification, and is confirmed by the results of XRD.

The results of the study of the Seebeck coefficient indicate the dominance of the p-type conductivity of the layers in the entire temperature range, both in the amorphous and in the crystalline states.

An analysis of the obtained data shows that in the range from room temperature to 135°C , an exponential temperature dependence of the electrical conductivity is observed, which indicates its activation nature for a thin film in an amorphous state.

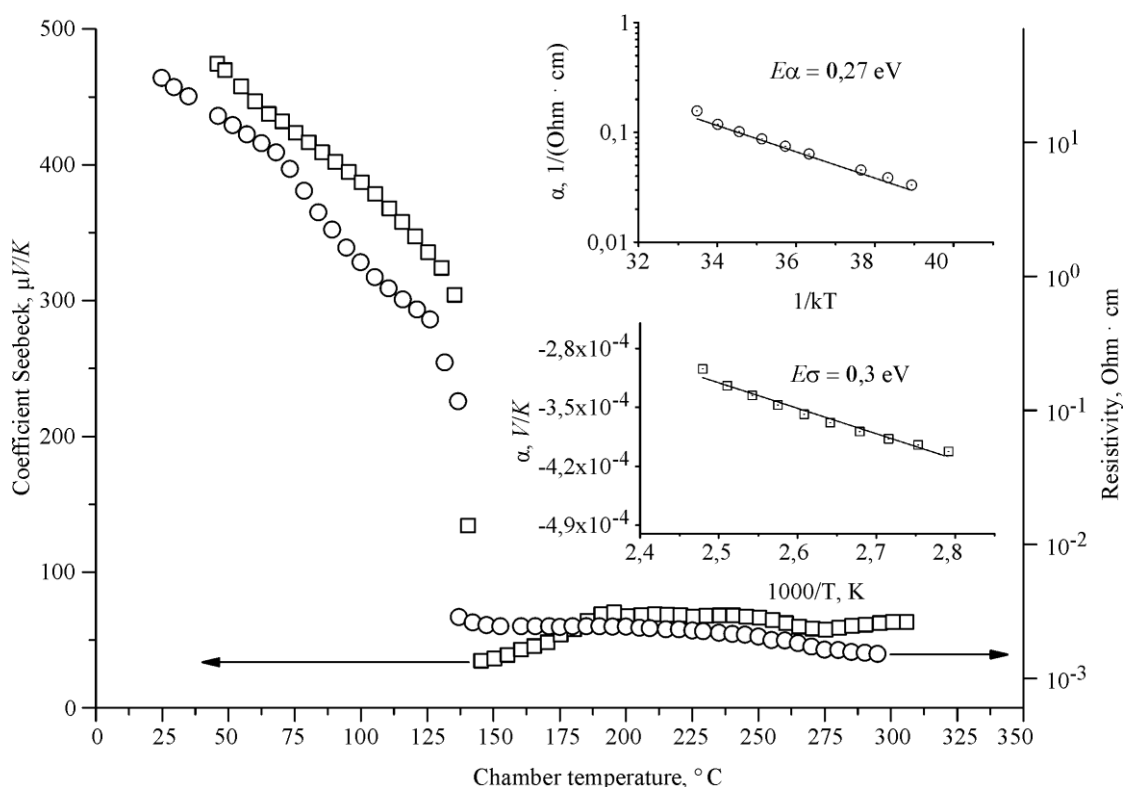


Fig.5. Temperature dependences of the Seebeck coefficient and resistivity for $\text{Ge}_2\text{Sb}_2\text{Te}_5$ thin films

At temperatures above room temperature, the conductivity of thin films of chalcogenide semiconductors is due to the mechanism of charge carrier transport through delocalized states of the allowed bands and is described by the following expression [12]:

$$\sigma = \sigma_0 \exp\left(-\frac{E_\sigma}{kT}\right),$$

where σ_0 – pre-exponential factor; E_σ – activation energy of conductivity; k – Boltzmann constant.

In this case, the Seebeck coefficient is [12]

$$\alpha = -\frac{k}{e} \left(\frac{E - E_F}{kT} + A \right),$$

where e – electron charge; E_F is Fermi level; $E = E_C$ and A is a factor determined by the scattering mechanism, if the conductivity is carried out by common conditions; $E = E_A$ (E_A is band tail edges of the allowed zone) and A is determined by the energy distribution of the density of states, if the conductivity is over localized states.

The inset to Fig. 5 shows the determination of the activation energies of the conductivity (E_σ) and the Seebeck coefficient (E_α) from the temperature dependences for amorphous $\text{Ge}_2\text{Sb}_2\text{Te}_5$ thin films. It turned out that $E_\sigma = 0.27$ eV, and $E_\alpha = 0.30$ eV.

The difference in the values of E_σ and E_α indicates that the mobility entering into the conduction expression has an activation character and charge transport is carried out over localized states at the tails of the zones [12].

The difference in the activation energies for the conductivity and Seebeck coefficient $E_\sigma - E_\alpha = 0.03$ eV determines the activation energy of the jump.

It was shown earlier, that deposited $\text{Ge}_2\text{Sb}_2\text{Te}_5$ thin films in the amorphous state have mobility gap width of 0.61 eV [13]. Taking into account the established p-type conductivity for the investigated layers, it can be assumed that the energy level controlling the activation character of the conductivity is located in the mobility gap 0.27 eV above the top of the valence band.

The results indicate a possible mechanism of two-channel conductivity in p-type amorphous thin films of $\text{Ge}_2\text{Sb}_2\text{Te}_5$, in which carriers introduced into localized states in the band tails and carriers in the extended states of the valence band contribute to the electrical conductivity.

Conclusion. Thus, a software and hardware system for simultaneous investigation of the temperature dependences of the Seebeck coefficient and the electrical conductivity of thin films of phase change memory materials was developed. The initial thin films deposited by vacuum-thermal evaporation are amorphous, and their composition is close to the stoichiometric $\text{Ge}_2\text{Sb}_2\text{Te}_5$. Heat treatment of the layers at a temperature of 160°C for 15 minutes leads to their crystallization with the formation of a cubic structure of the NaCl type.

An investigation of the temperature dependences of the Seebeck coefficient and the resistivity showed that with an increase in temperature from room temperature to 135 °C, the Seebeck coefficient decreases from 450 to 360 $\mu\text{V}/\text{K}$ and the resistivity decreases from 30.9 to 0.47 $\Omega\cdot\text{cm}$.

In the temperature range from 135 to 145°C, these parameters sharply drop to 35 $\mu\text{V}/\text{K}$ and 0.0029 $\Omega\cdot\text{cm}$, respectively, which is caused by the crystallization of thin films. An estimate of the activation energies of the Seebeck coefficient and the electrical conductivity indicates a possible two-channel conduction mechanism in p-type amorphous thin films of $\text{Ge}_2\text{Sb}_2\text{Te}_5$.

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