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Thermoelectric properties of cold pressed samples of semiconductor $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions

K.V.Martynova, E.I.Rogacheva

National Technical University "Kharkiv Polytechnic Institute",
2 Kyrpychova Str., 61002 Kharkiv, Ukraine

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The composition dependences of thermoelectric (TE) properties of $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions ($0 < x < 1$) produced by cold pressing and subsequent annealing were investigated at room temperature. Samples were prepared from cast polycrystals, obtained by the cooling of melt down to room temperature in evacuated quartz ampoules and subsequent annealing. It was established that cast samples exhibited *p*-type conductivity in the entire composition range, and an increase in the Sb_2Te_3 content led to the growth of electrical conductivity and drop of the Seebeck coefficient. The change of the conductivity type from positive to negative in the composition range $x = 0 - 0.6$ took place after cold pressing and composition dependencies of the properties became more complex. The maximum figure of merit value ($Z_{max} = (3.1 \pm 0.4)\cdot 10^{-3}$ K^{-1}) that was achieved in cold-pressed annealed samples at $x = 0.8$ was comparable to the values of Z for single crystals of undoped $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions and for polycrystalline samples produced by other methods. It follows from the data obtained that the proposed method of preparing the samples of $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions by cold pressing and subsequent annealing may appear to be useful in thermoelectric devices.

Keywords: $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions, cold pressing, annealing, Seebeck coefficient, electrical conductivity, thermal conductivity, figure of merit.

При комнатной температуре исследованы зависимости термоэлектрических свойств от состава твердых растворов $(Bi_{1-x}Sb_x)_2Te_3$ ($0 < x < 1$), подвергнутых холодному прессованию и последующему отжигу. Образцы готовили из литых поликристаллов, полученных путем охлаждения расплава до комнатной температуры в вакуумированных кварцевых ампулах и последующего отжига. Установлено, что литые образцы имели *p*-тип проводимости во всем интервале концентраций, а увеличение содержания Sb_2Te_3 приводило к росту электропроводности и снижению коэффициента Зеебека. После холодного прессования наблюдалась смена типа проводимости с дырочного на электронный в интервале составов $x = 0 - 0,6$, а концентрационные зависимости свойств приобретали более сложный характер. Максимальное значение термоэлектрической добротности ($Z_{max} = (3.1\pm 0.4)\cdot 10^{-3}$ К$^{-1}$) в холоднопрессованных отожженных образцах наблюдалось вблизи состава $x = 0.8$ и равнимо по величине с известными из литературы максимальными значениями Z для монокристаллов и горячепрессованных образцов нелегированных твердых растворов $(Bi_{1-x}Sb_x)_2Te_3$. Из полученных данных следует, что предложенный метод приготовления образцов твердых растворов $(Bi_{1-x}Sb_x)_2Te_3$ путем холодного прессования с последующим отжигом может оказаться полезным для применения в термоэлектрических устройствах.

Термоелектричні властивості холоднопресованих зразків напівпровідникових твердих розчинів $(Bi_{1-x}Sb_x)_2Te_3$. *К.В.Мартинова, О.І.Рогачова.*

За кімнатної температури досліджено залежності термоелектричних властивостей від складу твердих розчинів $(Bi_{1-x}Sb_x)_2Te_3$ ($0 < x < 1$), підданих холодному пресуванню із наступним відпалом. Зразки виготовлено із литих полікристалів, які отримано охолодженням розплаву до кімнатної температури у вакуумованих кварцових ампулах і наступним відпалом. Встановлено, що литі зразки мали p-тип провідності в усьому інтервалі складів, а збільшення вмісту Sb_2Te_3 призводить до зростання електропровідності і зниження коефіцієнта Зеєбека. Після холодного пресування спостерігалася зміна тип провідності із діркового на електронний в інтервалі складів $x = 0 - 0.6$, а концентраційні залежності властивостей набували складнішого характеру. Максимальне значення термоелектричної добротності ($Z_{max} = (3.1\pm0.4)\cdot10^{-3}$ К$^{-1}$) у холоднопресованих відпалених зразках спостерігалося при $x = 0,8$, зрівняним із відомими з літератури значеннями Z для монокристалів і гарячепресованих полікристалів нелегованих твердих розчинів $(Bi_{1-x}Sb_x)_2Te_3$. Із отриманих даних випливає, що запропонований метод виготовлення зразків твердих розчинів $(Bi_{1-x}Sb_x)_2Te_3$ холодним пресуванням із наступним відпалом може бути корисним для застосування у термоелектричних пристроях.

1. Introduction

Nowadays, thermoelectric (TE) cooling devices, whose operation is based on the Peltier effect — one of the TE effects [1–3] are widely applied. The efficiency of a TE converter largely depends on the value of TE figure of merit Z of a material used, which is defined as $Z = S^2\sigma/\lambda$, where S is the Seebeck coefficient, σ is the electrical conductivity and λ is the thermal conductivity of the material. Therefore, the development of new materials with high Z values is an important practical task. "Solid solution method", first proposed by A.F.Ioffe is one of the common methods of increasing Z. It is based on the fact that during a solid solution formation, the atoms of introduced second component dissipate phonons more effectively than electrons leading to an increase of σ/λ ratio which in turn leads to an increase in Z [1–3].

Semiconductor V_2VI_3 compounds (Bi_2Te_3, Sb_2Te_3, and Bi_2Se_3) and their solid solutions belong to the best TE materials for cooling devices. The highest Z values ($Z > 3\cdot10^{-3}$ K^{-1}) were achieved near room temperature in $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions at $x \sim 0.75$ [1–3]. Although properties of $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions were studied in a number of works, only the composition range near $x = 0.75$ was studied in detail. A considerable amount of works were devoted to the study of single crystal samples [4–9]. It was found that undoped single crystals $(Bi_{1-x}Sb_x)_2Te_3$ had hole conductivity at room temperature throughout the whole range of compositions, and an increase in x led to a monotonic decrease in S and an increase in σ [4–6], while the thermal conductivity dependence on the composition was a curve with a minimum at $x = 0.5$ [5, 6, 8, 9]. The maximum figure of merit for undoped single crystals was $Z = 2.75\cdot10^{-3}$ K^{-1} at $x \sim 0.4$ [5, 6, 10]. Optimal doping (for example, by adding tellurium) makes it possible to achieve higher values of Z (up to $3.5\cdot10^{-3}$ K^{-1}) in single crystals $(Bi_{1-x}Sb_x)_2Te_3$ with compositions of approximately $x \sim 0.75$ [4, 11–13].

However, the use of $(Bi_{1-x}Sb_x)_2Te_3$ in the single crystal state is complicated because of the fragility of the samples, and also because of the complexity of the crystal growing process. This stimulates the use of polycrystalline materials. It should also be taken into account that in polycrystals the thermal conductivity can decrease in comparison with single crystals due to the scattering of phonons on the grain boundaries, which can lead to an increase in Z.

There are various methods for obtaining polycrystals, such as: cooling an ampoule with a melt at a certain rate to room temperature to obtain cast samples, pressing, pulse discharge and spark plasma powder sintering, extrusion, and others [2]. Although a number of studies have been devoted to the study of the TE properties of cast samples $(Bi_{1-x}Sb_x)_2Te_3$ [7, 14–20], they were mainly carried out on the samples within the ($x = 0.7$–0.8) composition range that corresponds to the maximum TE efficiency [7, 14–19]. Only in [20] were cast polycrystals obtained throughout the whole range of compositions by quenching the melt in the air and subsequent annealing at various temperatures, and then their phase composition and structure were studied. Based on these data, the phase diagram of the Bi_2Te_3–Sb_2Te_3 system was constructed,

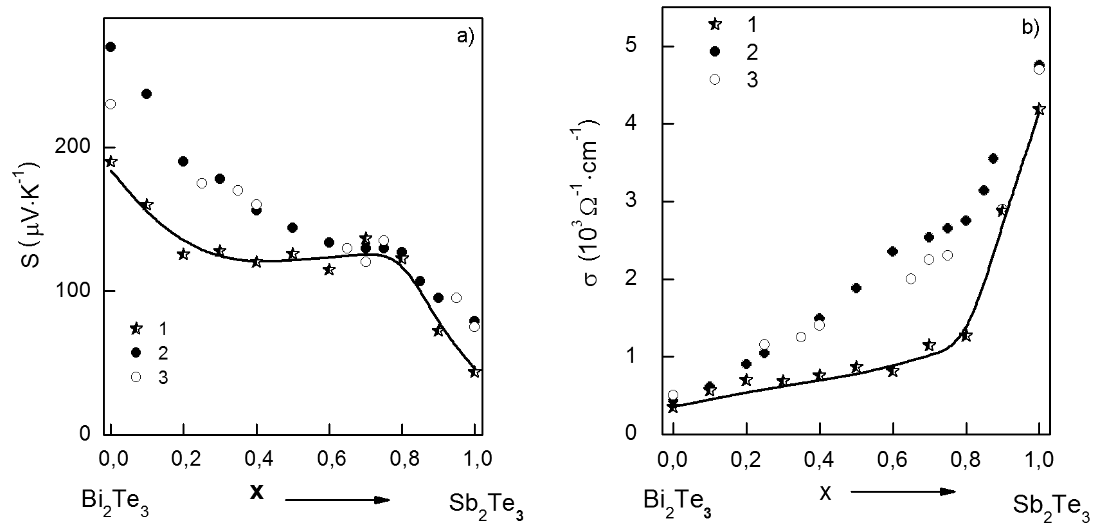

Fig. 1. Dependences of (a) — Seebeck coefficient S and (b) — electrical conductivity σ on the composition of $(Bi_{1-x}Sb_x)_2Te_3$ solid solution: *1* — this work (cast samples); *2* — single crystals [1], *3* — single crystals [5].

but the TE properties were not studied in this work [20]. To date, there are no studies on the TE properties of cast samples of undoped $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions over the whole range of compositions ($0 < x < 1$), although such studies were carried out for single crystals [1, 4–8].

As for the pressed samples, most often they are produced by hot pressing at temperatures of 450 – 550 K [6, 11, 21–25]. A characteristic feature of hot-pressed samples $(Bi_{1-x}Sb_x)_2Te_3$ [5, 7, 26], as well as of pulse discharge sintered samples [26], is a change in the conductivity type near $x = 0.6$. The samples exhibit electronic conductivity at concentrations lower than 0.6, and for larger x the hole conductivity takes place [6, 26]. Adding ~ 2.5 at. % excess Te into the single-crystal samples leads to the similar effect [4]. The change in the Seebeck coefficient sign in the region of inversion is accompanied by the appearance of a minimum in the $\sigma(x)$ dependence [6, 26]. To date, the reasons for the conductivity type inversion in pressed samples with a change of composition are not fully established. There were suggestions of a donor effect of oxidation [6] or mechanical deformation [27] that takes place during the producing of powders, possible evaporation of tellurium during heat treatment [28], and others. The following values of Z were reported: $Z = 3.07–3.17 \cdot 10^{-3}$ K^{-1} [6, 11, 21] for hot-pressed samples, and $Z = 2.85–3.0 \cdot 10^{-3}$ K^{-1} for pulse discharge sintered samples [22, 29] near $x = 0.75$. Along with hot pressing, the method of cold pressing followed by annealing can be used for TE purposes. This method was previously used to prepare samples only in the composition range near $x = 0.75$ [30–36], where values of $Z \sim 3.\cdot 10^{-3}$ K^{-1} were reported. There is no information available on the nature of the TE properties dependences on composition in the entire range of existence of $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions. Thus, the high values of Z observed for cold-pressed annealed samples for individual compositions draw attention to the study of the TE properties of samples prepared by this method over a wide range of compositions ($0 < x < 1$).

The purpose of this work is to investigate the dependences of TE properties (the Seebeck coefficient, electrical conductivity, thermal conductivity) of cold-pressed annealed samples of $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions on the composition over the entire concentration range ($0 < x < 1$).

2. Experimental

Cast samples of $(Bi_{1-x}Sb_x)_2Te_3$ ($0 < x < 1$) were prepared by fusion of elements (Bi, Sb, Te) of high purity (99.999 %) by the technique described in [37]. The synthesis and heat treatment of all 11 samples with different compositions were carried out simultaneously in the same furnace, that guaranteed the identity of the conditions for their production. To obtain the pressed samples, cast ingots were grounded in an agate mortar and sieved to obtain powders with a

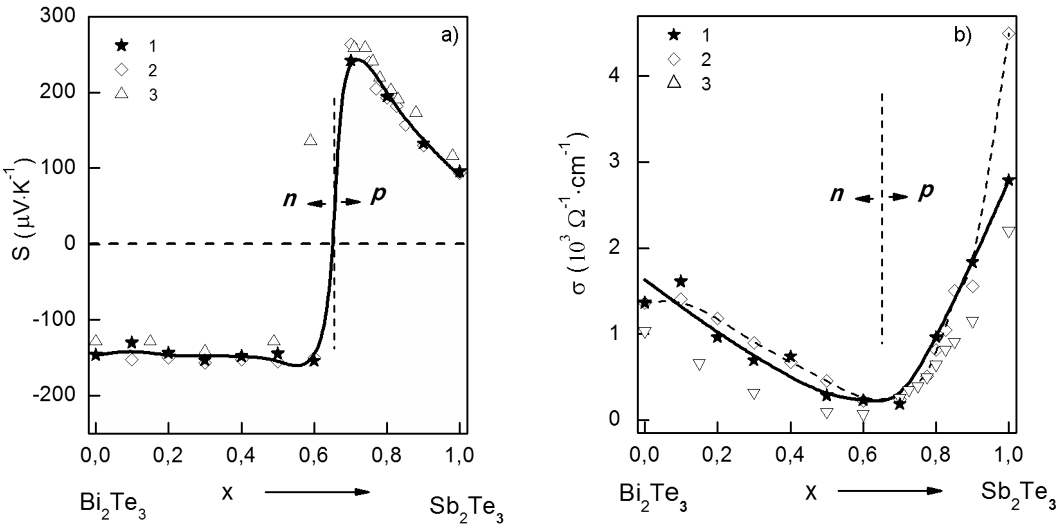

Fig. 2. Dependences of (a) — Seebeck coefficient S and (b) — electrical conductivity σ on the composition of $(Bi_{1-x}Sb_x)_2Te_3$ solid solution: *1* — this work (cold pressed samples); *2* — hot pressed samples [6]; *3* — pulse discharge sintered samples [26].

dispersion of ~ 200 μm. Tablet-shaped pressed samples with a $d = 15$ mm diameter and $h = 5$ mm height were produced at room temperature at a load of 69 MPa and one minute holding time under the press. Then the cold-pressed tablets were annealed in evacuated ampoules of heat-resistant glass for 300 h at a temperature of T_{ann} = 650 K, and then cooled to room temperature with the rate of switched off furnace. Microstructural analysis by optical microscope showed no inclusions of a second phase. Measurement of the Seebeck coefficient was carried by a compensation method in relation to copper electrodes, and σ was measured using the Van der Pau method. The errors of S and σ measurements were 3 % and 5 %, respectively. The conductivity type was determined by the sign of Seebeck coefficient. The thermal conductivity of the samples was measured by the dynamic λ-calorimeter method in the monotonic heating regime using the IT-λ-400 experimental facility with 5 % error. The homogeneity degree of the cast and pressed samples was controlled by measuring S and σ at different points of surfaces. The variation in values was within the error limits of the methods. All measurements were carried out at room temperature (T = 293 K).

3. Results and discussion

Fig. 1 shows the dependences of S and σ on the composition of $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions for cast polycrystals, together with literature data for single crystals [1, 5]. All cast samples had *p*-type conductivity, similar to the initial compounds.

One can see a general trend of decrease in S with increasing x for single crystals as well as for cast samples (Fig. 1a). In both cases, a bend was observed near $x = 0.7$, weakly expressed for the single crystals, but rather pronounced for the cast samples. In the cast samples, S decreases up to x ~ 0.3, then it does not actually change until x ~ 0.8, and after that decreases rapidly again. As a result, within $x = 0$–0.6 range of compositions, S is lower for the cast samples than for the single crystals with the same x, and in the interval $x = 0.7$–1, the values of S obtained in the present study for the cast samples coincide with the literature data for single crystals.

From Fig. 1b, it can be seen that the decrease in S is accompanied by a monotonous growth in electrical conductivity with increasing x in the entire range of $(Bi_{1-x}Sb_x)_2Te_3$ compositions, but close to $x = 0.8$ a sharp change in the slope of almost linear dependence is observed for the cast samples, whereas this change is hardly noticeable for the single crystals. As a result, the values of σ are lower than the literature data for single crystals samples [1, 5], except for the values for the initial compounds. The smaller values of σ for the cast samples in comparison with the σ values for the single crystals can be explained by the scattering of charge carriers by grain boundaries.

Fig. 2 presents the $S(x)$ and $\sigma(x)$ dependences for the cold-pressed annealed

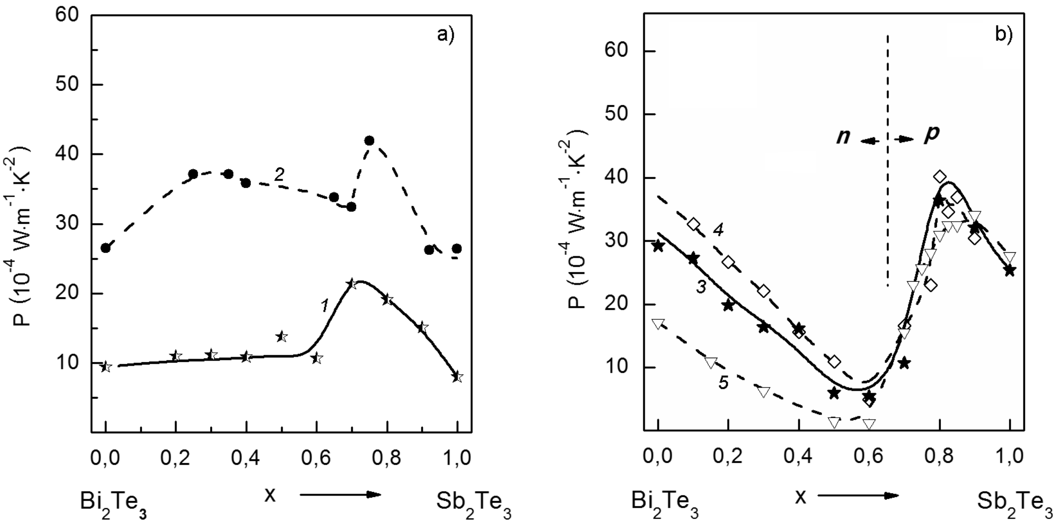

Fig. 3. Dependences of thermoelectric power factor $P = S^2\sigma$ on the composition of $(Bi_{1-x}Sb_x)_2Te_3$ solid solution: *1* — this work (cast samples), *2* — single crystals [5]; *3* — this work (cold pressed samples), *4* — hot pressed samples [6], *5* — pulse discharge sintered samples [26].

$(Bi_{1-x}Sb_x)_2Te_3$ samples, and also, for comparison, the data for hot-pressed and pulse discharge sintered samples reported in [6, 26]. First of all, a surprisingly good agreement should be noted between the obtained data and literature data [6, 26]. Figure 2a shows that in the interval $x = 0–0.6$ the Seebeck coefficient has constant negative values (S ~ −150 μV/K), which means that the conductivity type of the material changes as compared to the cast samples. In the vicinity of $x = 0.6$, the $n \to p$ inversion occurs, the Seebeck coefficient becomes positive, reaching a maximum value of S = 240 μV/K at $x = 0.7$, and then decreases. The $\sigma(x)$ dependence has a minimum at x ~ 0.65, which is the composition corresponding to the inversion of the conductivity sign (Fig. 2b).

Fig. 3 shows the composition dependences of TE power factor P ($P = S^2\sigma$) of the cast and cold pressed annealed samples (the present study), single crystals [5], hot pressed [6] and pulse discharge sintered [26] samples of $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions. According to Fig. 3, the $P(x)$ dependences for single crystals [5] and cast samples (the present study) have a similar character, but the values of P in the cast samples are lower than in the single crystal samples. It can also be seen that cold pressing followed by annealing leads to an increase in P compared to the cast samples. The maximum value of TE power factor (P_{max} = $37 \cdot 10^{-4}$ W/(m·K^2)) is observed at $x = 0.8$ for p-type samples.

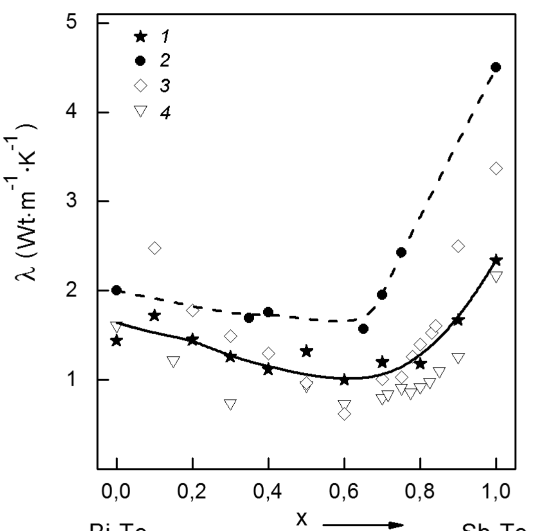

Fig. 4. Dependences of thermal conductivity λ on the composition of $(Bi_{1-x}Sb_x)_2Te_3$ solid solution: *1* — this work (cold pressed samples), *2* — single crystals [5]; *3* — hot pressed samples [6], *4* — pulse discharge sintered samples [26].

A similar $P(x)$ dependence and practically the same maximum values of P were observed by the authors of [6, 26] in hot-pressed polycrystals and pulse discharge sintered samples.

Fig. 4 shows the dependence of the thermal conductivity of cold-pressed annealed samples on the composition in comparison with the literature data for single crystals [5] and also samples prepared by hot pressing [6] and pulse discharge sintering [26].

All $\lambda(x)$ dependences are curves with a minimum at $x = 0.6–0.7$. A significant decrease in λ in the pressed samples in relation to single crystals is due to scattering of phonons by grain boundaries. It can be seen that the observed values of λ are close to the data for polycrystals [6, 26].

On the basis of the obtained data, the figure of merit of the cold-pressed annealed $(Bi_{1-x}Sb_x)_2Te_3$ samples was calculated. Fig. 5 shows the $Z(x)$ dependence in comparison with the literature data. It can be seen that the values of Z decrease with increasing x for the pressed samples in the n-type composition range ($x = 0–0.6$), while for p-type samples ($x = 0.7–1$) there is a maximum of Z at $x = 0.8$. The same character of dependence was observed for hot pressed [6, 11, 21], extruded [23], and pulse discharge sintered [26] samples. The obtained values of Z ($Z = 3.1 \cdot 10^{-3}$ K^{-1} at $x = 0.8$) are comparable to the maximum values of Z for other manufacturing methods ($Z = 3 \cdot 10^{-3}$ K^{-1} [23], $Z = 3.1 \cdot 10^{-3}$ K^{-1} [6, 11, 21]).

Thus, it follows from the data obtained in this work, that using cold pressing of cast polycrystals powders and subsequent annealing of compacted powders for manufacturing the samples of $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions, makes it possible to achieve the values of TE figure of merit, comparable to Z values obtained in single crystals, hot-pressed, pulse discharge sintered samples and for other manufacturing methods.

4. Conclusions

The dependences of TE properties of cast and cold-pressed annealed samples on the $(Bi_{1-x}Sb_x)_2Te_3$ solid solution composition were studied at room temperature and compared with the available literature data for single crystals, hot-pressed samples, and samples obtained by other methods.

It is shown that in the p-type cast samples, an increase in the Sb_2Te_3 content in the solid solution results in an increase in electrical conductivity of the material and a decrease in the Seebeck coefficient, with inflections in the $S(x)$ and $\sigma(x)$ dependences near $x = 0.8$, weakly expressed for single crystals. The obtained values of the TE power factor P are ~ 2.5–3.5 times lower than those reported in the literature for single crystals.

Cold pressing with subsequent annealing leads to a change in the conductivity type from hole to electronic one in the $x = 0–0.6$ concentration range. The $Z(x)$ dependence has a maximum at $x = 0.8$ ($Z_{max} = 3.1 \cdot 10^{-3}$ K^{-1}). The obtained values of Z exceed those known from the literature for undoped single crystals and are comparable to the values of Z for samples prepared by the hot pressing and other methods. This shows that the preparation of samples of $(Bi_{1-x}Sb_x)_2Te_3$ solid solutions by cold pressing with subsequent annealing can be used in certain cases for the manufacturing of materials for TE devices.

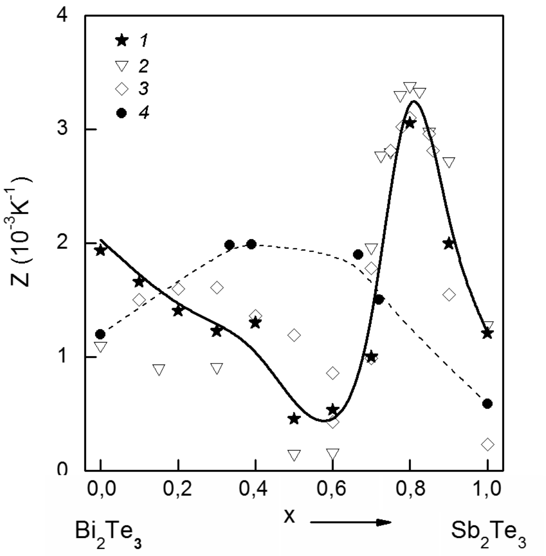

Fig. 5. Dependences of thermoelectric figure-of-merit $Z = S^2\sigma/\lambda$ on the composition of $(Bi_{1-x}Sb_x)_2Te_3$ solid solution: *1* — this work (cold pressed annealed samples), *2* — pulse discharge sintered samples [26], *3* — hot pressed samples [6], *4* — single crystals [5].

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