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Investigation of $Bi_2Te_{2.88}Se_{0.12}$ Bulk Single Crystal Produced Using Bridgman Method

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Abstract:
As part of research on the influence of selenium as dopant on bismuth telluride, a successful synthesis of single crystal was carried out. Single crystal of p type conductivity, with the given compound formula, $Bi_2Te_{2.88}Se_{0.12}$, *was obtained by the Bridgman process. The obtained empirical formula does not deviate from the given compound formula.*
Single crystal was characterized by Hall Effect system based on the Van der Pauw method. Also, bulk sample was characterized by Seebeck coefficient (S), thermal conductivity (κ) and electrical resistivity (ρ) measurements, as a function of temperature in the range of 40 - 320°C by a home made impedance meter.
The prepared single crystal has a figure of merit (Z) of $2.16 \times 10^{-3}\ K^{-1}$ *at 40°C. Values of ZT are about 1.0 at 27°C for commercialized p and n type of bismuth telluride ingots. T is absolute temperature.*

Keywords: *Bulk single crystal, Bridgman method, Hall and Van der Pauw method, Thermoelectric properties, Doping*

1. Introduction

Clean and renewable energy materials have become a worldwide research point to deal with the energy and environmental crisis. To effectively combat the energy crisis, we must increase usage renewable sources of energy. Renewable energies which come from wind or sunlight are not depleted when used. One of the most common usages of renewable energies is the direct conversion of sunlight into electricity.

The main goal of using thermoelectric materials is energy conversion. But they are not widely used due to poor utilization of the conversion of electrical energy into thermal energy, and vice versa. In order to evaluate the performance of thermoelectric materials, the energy conversion efficiency of thermoelectric devices is estimated by the quality factor $ZT= S^2T\sigma/\kappa$, where S is the Seebeck coefficient, σ is electrical conductivity, and κ is thermal conductivity. It is obvious that the better the electrical conductivity and the worse the thermal conductivity, the higher the ZT value will be [1]. A value of ZT=1 is considered good, but it is still far below the value that would be good enough, for example, to convert unused thermal energy into electricity in thermal power plants. The use of thermoelectric materials in thermal power plants would reduce fuel oil consumption, and therefore CO_2 emissions. It is believed that the ZT factor should be increased to about 3.5 in order to start thinking about commercially viable energy conversion systems based on the Seebeck effect. Thermoelectric effect refers to phenomena in which a temperature difference creates an electric potential or an electric potential creates a temperature difference.

The term refers to three interrelated effects: the Peltier effect, the Seebeck effect and the Thomson effect [1].

In practice, massive semiconductor materials from bismuth telluride, Bi_2Te_3 (doped with Sb, Se, etc.) with a value of $ZT\approx1$, are now most widely used. An important subject in the field of materials science and technology is to find out the mechanism of improving the properties of thermoelectric materials.

1.1 Thermoelectric Materials – Example of Bismuth Telluride (Bi_2Te_3)

Bi_2Te_3 is a well-known binary chalcogenous semiconductor compound used in thermoelectric devices operating at room temperature. It is mainly used in refrigerators and generators. It also has applications in optoelectronic and electrochemical devices such as heat pumps, infrared sensors, and high efficiency photovoltaic cells [2]. The thermoelectric properties of Bi_2Te_3 are characterized by the dimensionless quality factor *ZT*.

Bismuth telluride is widely studied as a thermoelectric material, especially in the temperature range around 300 K. Thermoelectric materials based on bismuth telluride are mainly used for the production of electricity from waste heat or in the production of thermoelectric coolers. Bi_2Te_3-Bi_2Se_3 and Bi_2Te_3-Sb_2Te_3 solid solutions are used as the basic material for thermoelectric cooling devices and thermogenerators operating at temperatures of 300 - 350°C. Among the chalcogenides that occur as components of these solid solutions, bismuth telluride has been studied in the most detail. The field of research of bismuth telluride and solid solutions based on it began on the initiative and under the leadership of Academician A.F. Ioffe in the Laboratory of Semiconductors and then supported by the Institute of Semiconductors of the Academy of Sciences, AN USSR. 1952 A. F. Ioffe expressed the assumption that the most efficient thermoelectric materials could be solid solutions of compounds. Since 1954. intensive research into the physical properties of bismuth telluride begins in England, America, Germany, Japan and France. Bismuth telluride itself has sufficiently high thermoelectric parameters. By alloying bismuth telluride, n and p type can be obtained. Because of its crystal structure, bismuth telluride is highly anisotropic. Its electrical resistivity is about four times greater parallel to the crystal growth axis than normal to the growth axis. Thermal conductivity, on the other hand, is about twice as high parallel to the crystal growth axis as normal to the growth axis. Since the anisotropic behavior of the resistivity is greater than the thermal conductivity, the maximum value of the quality factor (Z) occurs in the parallel orientation. Because of this anisotropy, the thermoelectric elements must be mounted in the cooling module so that the crystal growth axis is parallel to the length or height of each element and normal to the ceramic substrate. When bismuth telluride is produced by directed crystallization from a melt, it is usually obtained in the form of ingots. Bismuth telluride forms continuous isomorphous solid solutions with Bi_2Te_3 and Sb_2Te_3 [3]. In the field of crystal chemistry and geochemistry, isomorphism means the ability to form solid solutions by replacing certain substances in the crystal lattices of one particle (atom, ion or molecule) with another.

1) Elements can replace each other in the nodes of the crystal lattice when building the same type of lattice (only ions of the same sign can be replaced).

2) Goldschmidt's rule: when the radii of their ions, i.e. atoms, are approximate (for isomorphic replacement, the radii can differ by a maximum of 15%, calculated on less than two ions or atoms that replace each other with perfect isomorphism, and 15-25% in case of imperfect isomorphism).

3) Ions of the same or similar polarity can be replaced.

4) Isomorphism can be realized if the isomorphic substitution does not violate the electroneutrality of the crystal.

The most effective composition of n-type low-temperature thermoelectric materials used in thermoelectric coolers and generators in the Bi_2Te_3-Bi_2Se_3 system was found in the interval up to 33.3% mol Bi_2Se_3 [3,4]. Compounds of bismuth and tellurium have a narrow energy gap, which at room temperature is Eg= 0.15 eV.

Compounds based on bismuth telluride are very important materials for thermoelectric refrigerators and devices for electricity production.

Research on thermoelectric materials is being carried out in the Serbia and in the World [5-25].

2. Materials and Experimental Procedures

Single crystal doped with Se was synthesized by Bridgman method [1,26-31]. The high purity bismuth (Sigma – Aldrich, 99.999%), tellurium (Sigma – Aldrich, 99.999%) and selenium (Alfa Aesar, 99.999%), were prepared and merged in a stoichiometric relationship 2:3.

The crystal growth was achieved in a closed quartz ampoule under a pressure of 10^{-5} Pa. Before the synthesis beginning, it was necessary to prepare a quartz ampoule. One end of the quartz ampoule is heated to obtain a semi-open tube. The ampoule is coated on the inside with a thin film to eliminate wetting of the ampoule with the material in the molten state. A thin film on the inside of the ampoule prevented the batch in the molten state from chemically reacting with the material from which the ampoule was made in which the single crystal solidified. On the underside, the ampoule was conical in shape for easier germ formation. In this way, only a small part of the melt was subcooled at the top of the conical capillary. During growth through the capillary, one germ with the most favorable orientation overcame the others. A single crystal formed in a capillary was used as a seed for crystallization.The ampoule was heated to the synthesis temperature for 3 days. Then the ampoule was on a stable temperature gradient for 14 days. It was then cooled naturally to room temperature. For the synthesis of 1 mole of a single crystal of bismuth telluride doped with selenium with the given empirical formula $Bi_2Te_{2.88}Se_{0.12}$, the following was required: Bi=10.515732 g; Te=9.245874 g and Se=0.238393 g. The calculation was made for a 20 g batch.

The obtained single crystal of p tipe of conductivity was characterized by the Hall measurements (mobility (μ), bulk carrier concentration (nb), sheet carrier concentration (nS), resistivity (ρ), conductivity(σ), Hall coefficient (RH)).

Hall measurements were performed on a Hall Effect measurement system (Ecopia, HMS-3000) at room temperature with four ohmic contacts at different electric currents for an applied magnetic field strength of 0.37 T. Software for Hall Effect measurement system (Ecopia, HMS-3000) automatically calculated bulk and sheet carrier concentration, resistivity, conductivity and Hall coefficient. Calculations were done on the basis of voltage obtained by Van der Pauw laws and input data was entered into the software (sample thickness D, current intensity I, the magnetic induction of permanent magnet B). Samples cleaved parallel (||) to the plane of crystallisation were of square cross-section. The measured samples were cleaned in acetone before they are used for measurements.

Measurements of thermoelectric properties of single crystal of p tipe of conductivity was carried out by home-made impedance meter based on the ″Lagre ΔT method″ [32] with temperature range from 40 to 320°C. Home-made impedance meter simultaneously measured all three thermoelectric parameters, Seebeck coefficient, conductivity and resistivity. The measurement process was performed in vacuum of 10^{-2} to 10^{-3} Pa on sample with a height of about 11.72 mm and adiameter of 9.15 mm and took at least 15 hours. TE characteristics of sample, resistivity (ρ), Seebeck coefficient (S) and conductivity(κ) were used to determine figure of merit, ZT. Figure of merit, ZT, was calculated according to the expression:

$$ZT=S^2\cdot T/\rho\cdot\kappa \qquad (1)$$

Thermoelectric measurements were taken for decreasing temperature.

3. Results and discussions

Bismuth, tellurium and selenium are merged in a stoichiometric relationship 2:3, namely with given compound formula of $Bi_2Te_{2.88}Se_{0.12}$.

From the empirical formula of the compound, $Bi_2Te_{2.88}Se_{0.12}$, the percentage of elements was determined as follows: Bi=52.75%; Te=46.23% and Se=1.19%.

Fig. 1.

Figure 1 displayed the look of the p type $Bi_2Te_{2.88}Se_{0.12}$ single crystal obtained. The obtained crystal could be easily cleaved along the (001) plane and it has a shiny surface.

3.1 Hall Effect measurements of p type bulk single crystal, $Bi_2Te_{2.88}Se_{0.12}$, at room temperature

Hall Effect measurements at room temperature with four ohmic contacts for the single crystal of p type, $Bi_2Te_{2.88}Se_{0.12}$, was carried out by passing a 0.05, 0.1, 0.5, 1 and 5 mA current through the sample under magnetic field of 0.370 T. The obtained single crystal was characterized by the conductivity (σ), resistivity (ρ), mobility (μ), bulk carrier concentration (n_b), sheet carrier concentration (n_S) and Hall coefficient (R_H). The measured samples were cleaved from different regions of single crystal with cutting route which was for cleaved samples parallel (||) to the plane of crystallisation. In the following text these samples will be marked as 7/4 (||) and 7/7 (||), respectively. All calculated data from Hall measurements for samples are presented in Table I and Table II.

Tab. I

Tab. II

Sheet carrier concentrationis was found to be from 10^{16} to 10^{18} cm^{-2} for both samples, 7/4 (II) and 7/7 (II).

The variation of the Hall coefficient R_H at room temperature with current is shown in Table I and II.

Values of Hall coefficient is negative for sample 7/4 (||) at 0.1 and 0.5 mA and for sample 7/7(||) at 1 mA which can be contributed to interval time between measurements or deviation from stoichiometry. The positive value of the Hall coefficient points out that BiTe single crystal doped with Se is p type and the majority charge carriers are holes.

3.2 Thermoelectric properties of p type bulk single crystal, $Bi_2Te_{2.88}Se_{0.12}$

Results of thermoelectric properties in the temperature interval from 40°C to 320°C for p type bulk single crystal, $Bi_2Te_{2.88}Se_{0.12}$, are given in Figure 2. Figure shows the temperature dependence of electrical resistivity (ρ), Seebeck coefficient (S), thermal conductivity (k) and figure of merit (ZT) of sample. A TE characteristic was measured to define quality. These properties strongly depend on carrier concentration, mobility, crystal structure and defects in the crystal structure.

a)

b)

c)

d)

Fig. 2. a) to d)

Electrical resistivity (ρ) as a function of temperature (T) is displayed in Figure 2a. The R-T curve demonstrates electrical resistivity increase as temperatures increase. This trend has been kept to about 150°C. In the temperature interval from 150°C to 320°C is fairly constant.

Temperature dependence of Seebeck coefficient (S) in temperature range from 40°C to approximately 300°C is shown in Figure 2b. The sample showed value of Seebeck coefficient of 225 $\mu V°C^{-1}$ at 40°C to 50 $\mu V°C^{-1}$ at 292°C. Seebeck coefficient had a positive sign over the whole investigated temperature range which indicates p type of conduction (major conductivity carriers are holes) which is in agreement with the Hall coefficient. The variation in thermal conductivity (κ) with temperature of studied sample is shown in Figure 2c. The analyzed sample had thermal conductivity of 1.17 $Wm^{-1}K^{-1}$ approximately. Figure of merit was calculated using measured values for electrical resistivity, Seebeck coefficient and thermal conductivity. Its temperature dependence is presented in Figure 2d. The ZT peak in Figure 2d is about 2.16 at 40°C, which is significantly greater than Bi_2Te_3 based alloys. The ZT value of the Bi_2Te_3based alloys starts to drop above 75°C and is below 0.25 at 250°C [33].

4. Conclusions

The possibility of producing high quality $Bi_2Te_{2.88}Se_{0.12}$ single crystal using the Bridgman method was examined. Single crystal was successfully prepared.

The compositions of commercialized p type and n type ingots are near $Bi_{0.5}Sb_{1.5}Te_3$ and $Bi_2Te_{2.7}Se_{0.3}$ (which is optimum composition for thermoelectric cooling devices), respectively, and their ZT values are about 1.0 at 27°C [33-35]. Our investigation showed that a ZT peak of 2.16 at 40°C can be achieved in a p type $Bi_2Te_{2.88}Se_{0.12}$ bulk single crystal obtained by the Bridgman process.

Conventional Bi_2Te_3 based materials have a peak ZT of about 0.25 at 250°C [33]. Obtained crystal ingot of 80 mm in length and of 9.2 mm in diameter cleaved easily along the (00l) planes. Measurements of the Hall effect and thermoelectric properties indicate that bulk simple was p type conduction, suggesting that major conductivity carriers were holes. The electrical resistivity (ρ), thermal conductivity (k) and Seebeck coefficient (S) were measured simultaneously in temperature range from 40°C to 320°C. The thermal conductivity was observed to be 1.17 $Wm^{-1}K^{-1}$ approximately.

The results obtained in our researches provide additional useful information related to the issue of the thermoelectric materials development, which is conditional on finding materials with a high-quality factor, Z [K^{-1}]. The combination of material properties required for thermoelectric materials to have quality and usable properties is a challenge for scientists. The basic in the research of thermoelectric materials is to satisfy the properties that are in opposition to each other. In order to increase the quality factor as much as possible, that is, to obtain a higher thermal current, which is the absolute value of the Seebeck coefficient, we must have a high electrical conductivity and a low thermal conductivity.

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Садржај:

У оквиру истраживања утицаја селена као допанта на бизмут телурид, извршена је успешна синтеза монокристала. Монокристал n типа проводљивости, са датом формулом једињења, $Bi_2Te_{2.88}Se_{0.12}$*, добијен је Брџмановим поступком. Добијена емпиријска формула није одступала од дате формуле једињења.*
Монокристал је окарактерисан системом Холовог ефекта заснованом на Ван дер Пауовој методи.
Такође, масовни узорак је окарактерисан Зебековим коефицијентом (S), мерењима топлотне проводљивости (κ) и електричне отпорности (ρ) у функцији температуре у опсегу од 40 - 320°C мерачем импедансе домаће израде.
Припремљени монокристал има висок фактор квалитета (Z) од 2.16 x 10^{-3} K^{-1} *на 40°C.*
За комерцијализоване инготе бизмут телурида n и н типа вредности ZT су око 1.0 на 27°C. T је апсолутна температура.

Кључне речи: *Масивни монокристал, Брицман метода, Холова и Ван дер Паува метода, термоелектрична својства, допирање*

FIGURE LEGENDS

Fig. 1. Single crystal of p type, $Bi_2Te_{2.88}Se_{0.12}$

Fig. 2 Temperature dependence of electrical resistivity (*a*), Seebeck coefficient (*b*), thermal conductivity (*c)* and figure of merit (*d)* for p type bulk single crystal, $Bi_2Te_{2.88}Se_{0.12}$

TABLE LEGENDS

Table I Hall measurements for sample 7/4 (II) of square cross-section with thickness of 1.23 mm

Table II Hall measurements for sample 7/7 (II) of square cross-section with thickness of 2.09 mm

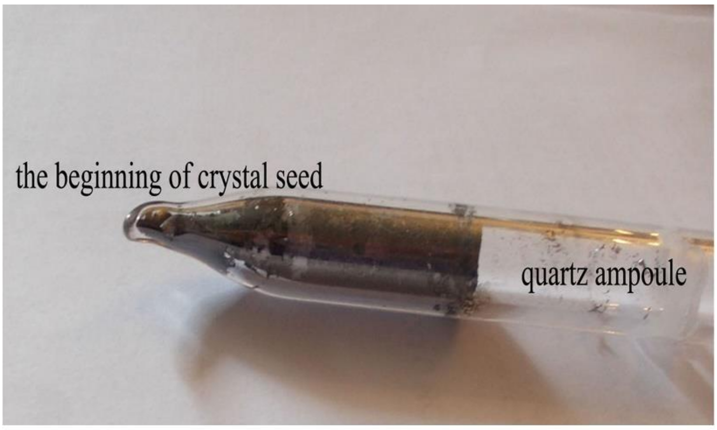
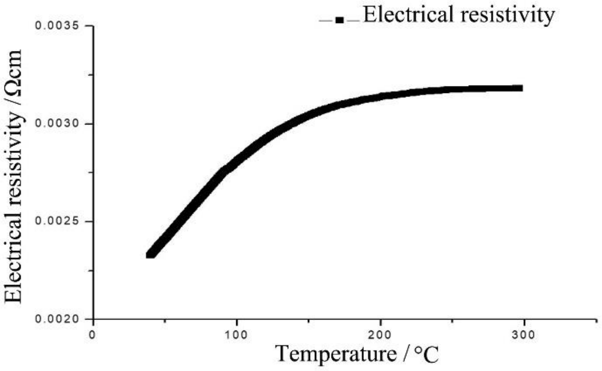
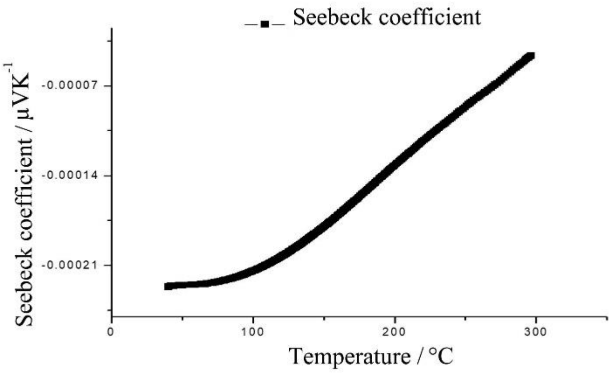


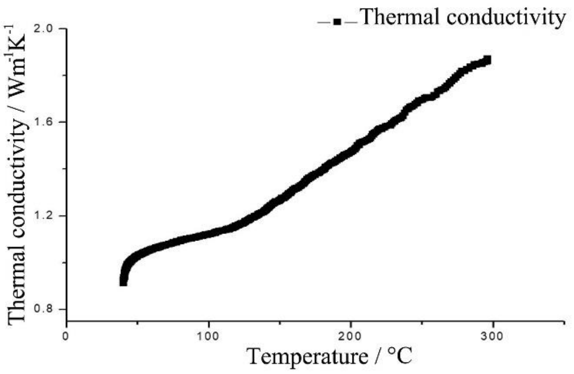
Fig. 1



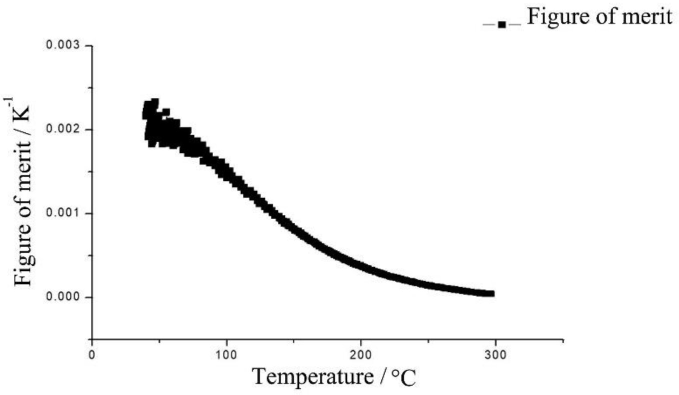
a)



b)



c)



d)

Fig. 2 a) to d)

Table I

Current intesity I [mA]	Conductivity σ [1/Ωcm]	Resistance ρ [Ωcm]	Bulk carrier concentration n_b [/cm^{3}]	Sheet carrier concentration n_S [/cm^{2}]	Mobility μ [cm^{2}/Vs]	Average Hall coefficient R_H [cm^{3}/C]
0.05	1.197x10^{2}	8.353x10^{-3}	2.252x10^{18}	2.769x10^{17}	3.319x10^{2}	2.772x10^{0}
0.1	1.744x10^{2}	5.735x10^{-3}	-4.321x10^{18}	-5.315x10^{17}	2.519x10^{2}	-1.445x10^{0}
0.5	3.957x10^{2}	2.527x10^{-3}	-4.023x10^{18}	-4.949x10^{17}	6.139x10^{2}	-1.552x10^{0}
1	3.941x10^{2}	2.538x10^{-3}	1.465x10^{19}	1.802x10^{18}	1.679x10^{2}	4.261x10^{-1}
5	4.642x10^{2}	2.154x10^{-3}	9.323x10^{18}	1.147x10^{18}	3.108x10^{2}	6.696x10^{-1}

Table II

Current intesity I [mA]	Conductivity σ [1/Ωcm]	Resistance ρ [Ωcm]	Bulk carrier concentration n_b [/cm^{3}]	Sheet carrier concentration n_S [/cm^{2}]	Mobility μ [cm^{2}/Vs]	Average Hall coefficient R_H [cm^{3}/C]
0.05	9.618x10^{1}	1.040x10^{-2}	5.937x10^{17}	1.241x10^{17}	1.011x10^{3}	1.051x10^{1}
0.1	1.833x10^{2}	5.456x10^{-3}	4.416x10^{17}	9.229x10^{16}	2.591x10^{3}	1.414x10^{1}
0.5	5.885x10^{2}	1.699x10^{-3}	7.452x10^{18}	1.557x10^{18}	4.930x10^{2}	8.377x10^{-1}
1	4.923x10^{2}	2.031x10^{-3}	-2.577x10^{19}	-5.387x10^{18}	1.192x10^{2}	-2.422x10^{-1}
5	5.250x10^{2}	1.905x10^{-3}	2.009x10^{19}	4.199x10^{18}	1.631x10^{2}	3.107x10^{-1}