Thermal conductivity and microstructure of Bi-Sb alloys

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Abstract

Four Bi-Sb alloys with compositions $Bi_{79.6}Sb_{20.4}$, $Bi_{56.9}Sb_{43.1}$, $Bi_{39.8}Sb_{60.2}$, $Bi_{18.6}Sb_{81.4}$ have been investigated regarding the microstructures and thermal properties. The microstructure was examined by scanning electron microscopy with energy-dispersive X-ray spectrometry. The light flash method was applied to determine thermal diffusivity and to obtain thermal conductivity in the temperature range 25 to 150 °C, while the indirect Archimedean method was used for determination of densities of the investigated Bi-Sb alloys. The obtained results have shown that the density of the studied alloys decreased monotonically with increasing the antimony content. On the other hand, the specific heat capacity of Bi-Sb alloys increased with the increase in the antimony content as well as with increasing the temperature. Thermal diffusivity of the alloys increased slightly with increasing the temperature. Thermal conductivities of the examined Bi-Sb alloys were determined to be in the range of 3.8 to 7 W m^{-1} K^{-1}, which is lower than thermal conductivities of pure bismuth and antimony. The results obtained in this work represent a contribution to better knowledge of the thermal properties of Bi-Sb alloys, which are of key importance for determining the possibility of their practical application.

Keywords: Bi–Sb system; thermal properties; SEM-EDS; light flash method.

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1. INTRODUCTION

The metal bismuth (Bi) and metalloid antimony (Sb) belong to the group 5A of the periodic table of elements, with the A7 rhombohedral structure. Bismuth is the least toxic heavy metal, but it has poor mechanical and thermal properties and because of that, it is rarely used as a structural material [1,2]. Bismuth alloys are being considered for use as Pb-free high-temperature [3] and low-temperature [4-6] solders due to their suitable melting interval. Antimony found its application as an alloying element in lead alloys (for solder, bearings, cable sheathing, battery grids, and ammunition) and in tin alloys. Antimony is also used as a component of III-V semiconductors such as InSb, AlSb, and GaSb [7].

The Bi-Sb phase diagram is characterized by the existence of a continuous solid solution phase (Bi, Sb) [8]. By adding antimony to bismuth, it is possible to control the structure of the energy bands and the transport properties of the obtained alloys [9,10]. Depending on the antimony content, these alloys can exhibit semimetal behavior (molar ratio of antimony $x_{Sb} < 0.07$), or semiconductor behavior ($0.07 < x_{Sb} < 0.22$), which is interesting from the scientific point of view. For higher antimony contents, the solid solution adopts a semimetal behavior again [11,12]. Because of this, Bi-Sb alloys with less than 20 at.% Sb have the potential for efficient thermoelectric conversion and are of interest from the practical point of view.

Many studies have been performed to investigate the possibility of using Bi-Sb alloys in thermoelectric and thermomagnetic cooling devices [13,14]. These alloys remain the best choice for n type leg of thermocouples used in thermoelectric cooling devices operating at temperatures below 200 K [11].

Thermal and electrical properties of polycrystalline $Bi_{1-x}Sb_x$ (x = 0.10, 0.12 and 0.15) semiconductor alloys were investigated in literature [11]. Electrical resistance, thermoelectric power and thermal conductivity were measured in the temperature range 300 to 500 K. It was found that the thermal conductivity of Bi–Sb alloys is lower than the thermal

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conductivity of pure bismuth, due to electron scattering because of point defects created by the addition of Sb (when Bi is doped with Sb, it undergoes a semi-metal-semiconductor transition). In this process, additional electron scattering centers and a higher concentration of pores are formed.

$Bi_{1-x}Sb_x$ (x = 0.08 to 0.17) alloys obtained by melting pure elements and subsequent quenching and annealing were investigated regarding semiconductor and thermoelectric properties by measuring the Hall coefficient, electrical resistance and Seebeck coefficient in the temperature range from 20 to 300 K for hardened and annealed samples [10]. Literature values of thermal conductivity for several Bi-Sb alloys of different compositions are given in a compilation of values of thermophysical quantities of pure elements and alloys [15].

In the present study, as a contribution toward a more complete understanding of thermal transport properties of Bi-Sb alloys, four of these alloys with compositions $Bi_{79.6}Sb_{20.4}$, $Bi_{56.9}Sb_{43.1}$, $Bi_{39.8}Sb_{60.2}$, $Bi_{18.6}Sb_{81.4}$ were experimentally examined. Thermal diffusivity and thermal conductivity were obtained in wide composition and temperature ranges using the light flash technique. In addition, microstructure was analyzed by scanning electron microscopy with energy-dispersive X-ray spectrometry (SEM-EDS).

2. EXPERIMENTAL

The alloys were prepared by melting pure Bi (99.99 %, Alfa Aesar, Germany) and Sb (99.999 %, Alfa Aesar, Germany) in evacuated quartz tubes. The samples were re-melted several times to improve homogeneity. The prepared alloys were annealed at 240 °C for one week and after that slowly cooled within the furnace.

A scanning electron microscope (VEGA 3LMU, Tescan, Czech Republic) with the energy-dispersive X-ray spectrometer (X-act, Oxford Instruments, UK) was used to analyze the microstructure and chemical composition of the samples. SEM-EDS analysis was performed with an accelerating voltage of 20 kV. An EDS area and point analysis was used to determine the overall composition of the alloys as well as the compositions of coexisting phases within each alloy. The samples were prepared using a traditional metallographic procedure that included wet grinding, polishing with alumina slurry (granulation 0.3 μm), etching with a diluted solution of ferric chloride in water, and after that cleaning in an ultrasonic bath. Backscattered electron mode (BSE) SEM microstructure images were captured on the surfaces of the prepared samples.

The light flash method was applied by using a TA Instruments compact benchtop apparatus (Discovery Xenon Flash DXF-500, TA Instruments, USA) to measure thermal diffusivity and specific heat capacity in the range 25 to 150 °C. More information on the fundamental theoretical concepts and practical procedures underlying the used flash method can be found in literature [16-19]. The Bi-Sb alloy samples were pressed into round disks (12.7 mm in diameter and 2 mm thick, with two ground plane parallel end-faces). The prepared disc-shaped samples were then placed in the vacuum furnace of the DXF-500 instrument and heated to the desired temperatures (25, 50, 100 and 150 °C) at a constant heating rate (10 °C min^{-1}). The reported results are the average values obtained from three repeated measurements.

Specific heat capacity values were calculated by using the CALPHAD method [20], and densities of the investigated alloys at room temperature were determined by using the indirect Archimedean method [21] with distilled water (ρ = 0.99679 kg m^{-3}) and an electronic instrument (Mettler Toledo, Switzerland).

Thermal conductivity of the alloys was subsequently calculated using the experimentally determined and calculated values of thermal diffusivity, density, and specific heat capacity, following the equation [16]:

$$\lambda = \alpha\rho C_p \quad (1)$$

where λ represents thermal conductivity, α is the measured thermal diffusivity, ρ is the density, and C_p is the calculated specific heat capacity of the studied alloys.

3. RESULTS AND DISCUSSION

3. 1. Microstructure observation

SEM micrographs, illustrating microstructures of the investigated alloys $Bi_{79.6}Sb_{20.4}$, $Bi_{56.9}Sb_{43.1}$, $Bi_{39.8}Sb_{60.2}$, $Bi_{18.6}Sb_{81.4}$ under different magnifications, are given in Figures 1-4.

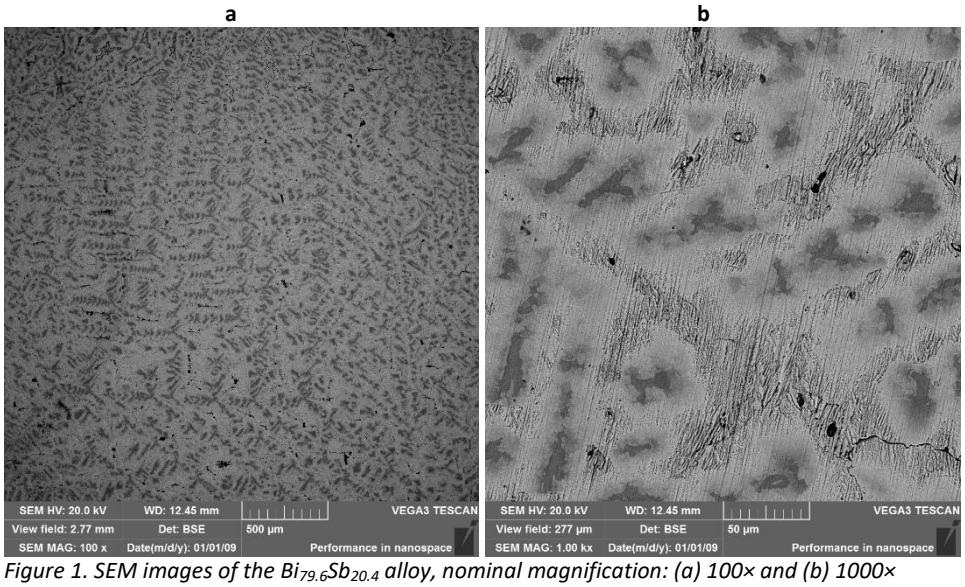

Figure 1. SEM images of the $Bi_{79.6}Sb_{20.4}$ alloy, nominal magnification: (a) 100× and (b) 1000×

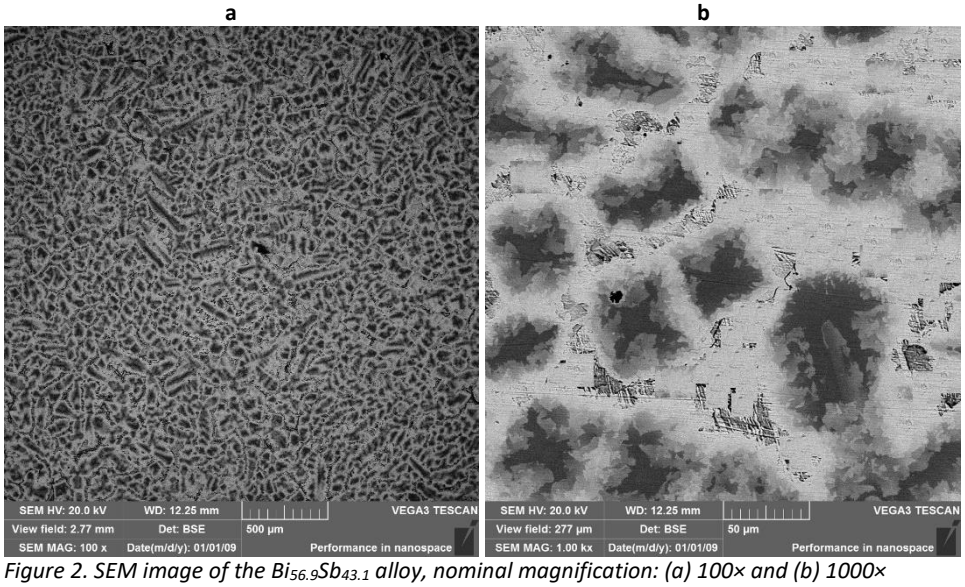

Figure 2. SEM image of the $Bi_{56.9}Sb_{43.1}$ alloy, nominal magnification: (a) 100× and (b) 1000×

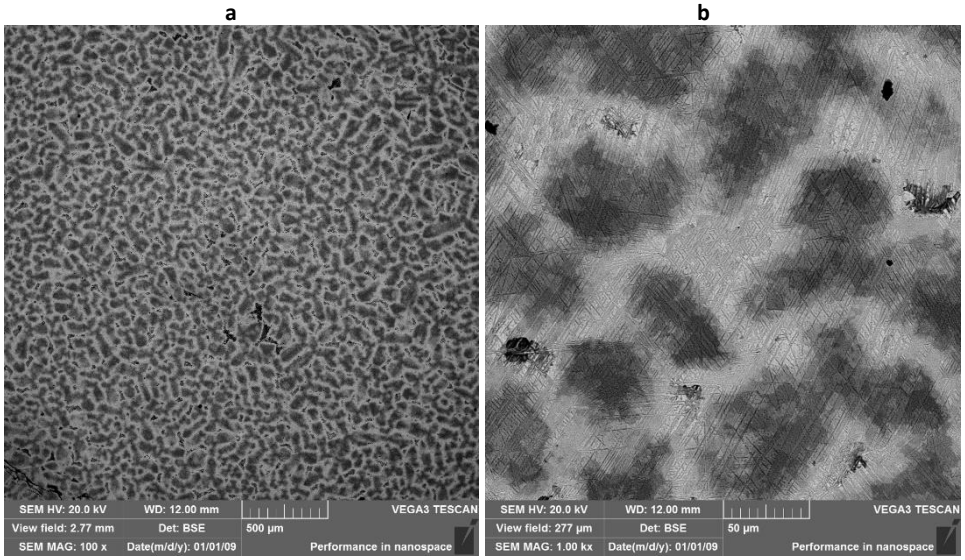

Figure 3. SEM image of the $Bi_{39.8}Sb_{60.2}$ alloy, nominal magnification: (a) 100× and (b) 1000×

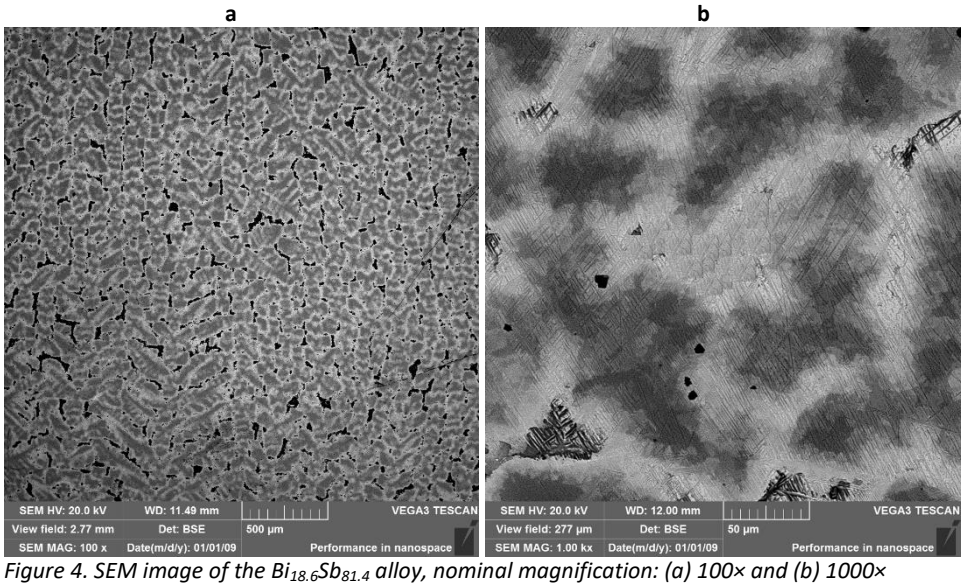

Figure 4. SEM image of the $Bi_{18.6}Sb_{81.4}$ alloy, nominal magnification: (a) 100× and (b) 1000×

Alloys have a highly homogeneous macrostructure that can be seen throughout the samples (Figs. 1-4). However, the microstructure of all four investigated alloys shows inhomogeneity due to coring on solidification. Coring is generally observed in alloys with a noticeable difference between liquidus and solidus temperatures. The liquidus and solidus lines are separated from each other in the Bi–Sb phase diagram resulting in segregation (chemical inhomogeneity) in the melt-grown crystals [10]. The center of each grain, which is the first part to freeze, is rich in the high-melting element (*e.g.* Sb for this Bi–Sb system), whereas the concentration of the low-melting element increases from the central part of the grain to the grain boundary. This is known as a cored structure. Segregation is characterized by an unequal distribution of elements and formation of concentration gradients within the grains.

In order to remove chemical inhomogeneity as the result of segregation, it is necessary to carry out a long-term homogenization annealing. Obviously, annealing at 240 °C, which is temperature just below solidus temperature in the case of the Bi-Sb alloys studied, in the duration of 1 week was not sufficient to fully eliminate segregation.

By the EDS analysis of surfaces of the samples, the overall composition of the investigated Bi-Sb alloys was determined. The obtained values show that there are minor deviations from the planned compositions (20, 40, 60, and 80 % Sb) of the alloys that occurred during the preparation of the samples. The EDS analyses of the grains confirmed the occurrence of crystal segregation. The inner areas of the grains that have a darker shade in the SEM images are areas richer in antimony, while the outer, lighter parts of the grains are richer in bismuth, as given in Table 1.

Elemental SEM-EDS mapping was applied to study the elemental distribution within the studied Bi-Sb alloys. The example of the EDS elemental map for the $Bi_{56.9}Sb_{43.1}$ alloy is given in Figure 5.

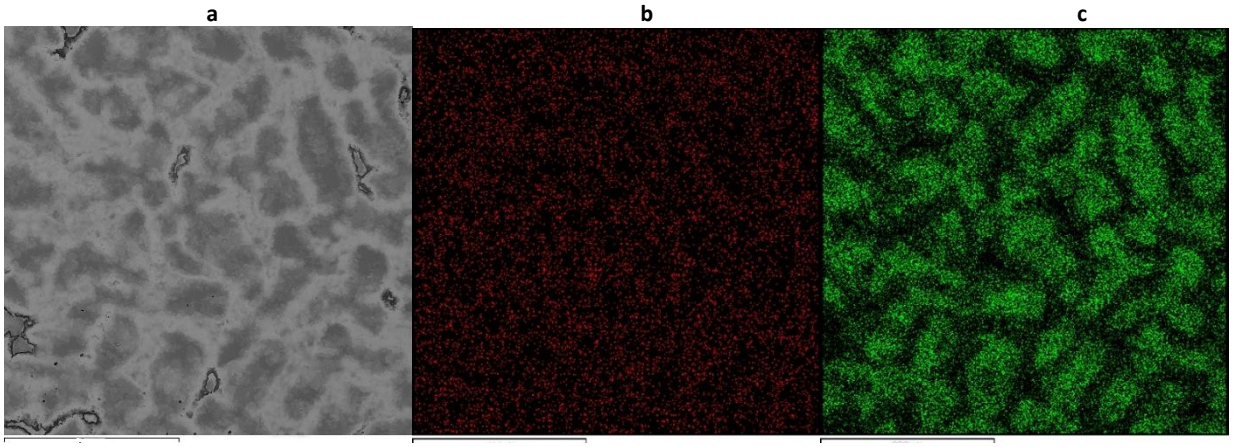

Figure 5. Elemental SEM-EDS mapping for the $Bi_{56.9}Sb_{43.1}$ alloy: (a) microstructure, (b) Bi distribution, (c) Sb distribution (scale bar = 300 µm)

It can be seen that the green contrast for Sb distribution (Fig. 5c) shows high intensity within the regions that correspond to the darker regions on the image of alloy microstructure (Fig. 5a). This analysis confirms that Sb is mainly concentrated in the central parts of the grains exhibiting dark shade on SEM image. On the other hand, Bi is mainly concentrated in the brighter areas shown in the SEM image.

Table 1. The results of the SEM-EDS analysis of the investigated alloys

Overall Sb content, at.%	Microstructure	Identified phase	Region on SEM image	Content, at.%	
				Bi	Sb
20.4	Cored grains of (Bi,Sb) solid solution	(Bi,Sb) solution	dark	49.1	50.9
			gray	60.3	39.7
			bright	84.2	15.8
43.1	Cored grains of (Bi,Sb) solid solution	(Bi,Sb) solution	dark	20.2	79.8
			gray	40.4	59.6
			bright	72.5	27.5
60.2	Cored grains of (Bi,Sb) solid solution	(Bi,Sb) solution	dark	16.2	83.8
			gray	28.7	71.3
			bright	67.2	32.8
81.4	Cored grains of (Bi,Sb) solid solution	(Bi,Sb) solution	dark	5.9	94.1
			gray	26.1	73.9
			bright	40.8	59.2

3. 2. Thermal conductivity of Bi-Sb alloys

Literature values of thermophysical properties of pure Bi and Sb are presented in Table 2.

Table 2. Thermophysical properties of pure Bi and Sb [7]

Metal	Melting point, °C	Latent heat of fusion, J g^{-1}	Density, g cm^{-3} *	Specific heat capacity*, J g^{-1} K^{-1}	Thermal conductivity, W m^{-1} K^{-1}*
Bi	271.4	53.976	9.808	0.122	8.2
Sb	630.7	163.17	6.697	0.207	24.3

*at 25 °C

Bismuth has one of the lowest thermal conductivity among all metals (8.2 W m^{-1} K^{-1} at 25 °C). Antimony has significantly higher values of melting temperature, latent heat of fusion, specific heat capacity and thermal conductivity compared to bismuth, while bismuth has a higher density than antimony.

Table 3 shows the values of specific heat capacity, thermal diffusivity and thermal conductivity for the examined Bi-Sb alloys in the temperature range 25 to 150 °C. Thermal diffusivities were measured by the flash method, while thermal conductivities were calculated using the density and specific heat capacity data of the investigated materials (Eq. 1).

Firstly, alloy densities at room temperature were measured using the buoyancy method based on Archimedes' principle with an estimated uncertainty of ± 1 % [21]. It was assumed that the densities of the alloys are approximately constant in the narrow temperature range studied. Figure 6 shows density values for the investigated Bi-Sb alloys, as well as density values for pure bismuth and antimony at room temperature.

It can be seen that the densities of Bi-Sb alloys lie between the values of densities for pure bismuth and antimony and that the density of the alloys decreases monotonically with increasing the antimony content.

The CALPHAD method [20] and the thermodynamic COST 531 lead-free solder database [22] were used to calculate the specific heat capacities of the alloys under consideration. The calculated specific heat capacity values were checked for accuracy by comparison with the corresponding measured values obtained at room temperature using the light flash technique (pure bismuth and antimony were used as reference materials).

Table 3. Specific heat capacity, thermal diffusivity, and thermal conductivity of the investigated Bi–Sb alloys in the temperature range 25-150 °C

Sb content, at.%	Temperature, °C	Calculated specific heat capacity, J g^{-1} K^{-1}	Thermal diffusivity, mm^2 s^{-1}	Thermal conductivity, W m^{-1} K^{-1}
20.4	25	0.133	3.46±0.10	4.2±0.3
	50	0.134	3.64±0.11	4.4±0.3
	100	0.137	3.85±0.12	4.7±0.3
	150	0.140	3.99±0.12	5.0±0.3
43.1	25	0.148	4.76±0.14	5.9±0.4
	50	0.149	5.00±0.15	6.3±0.4
	100	0.152	5.27±0.16	6.7±0.4
	150	0.155	5.40±0.16	7.0±0.4
60.2	25	0.162	4.01±0.12	5.1±0.3
	50	0.163	4.07±0.12	5.2±0.3
	100	0.166	4.17±0.13	5.4±0.3
	150	0.169	4.24±0.13	5.6±0.3
81.4	25	0.183	2.90±0.09	3.8±0.2
	50	0.185	2.96±0.09	3.9±0.2
	100	0.187	3.05±0.09	4.1±0.2
	150	0.190	3.12±0.09	4.2±0.3

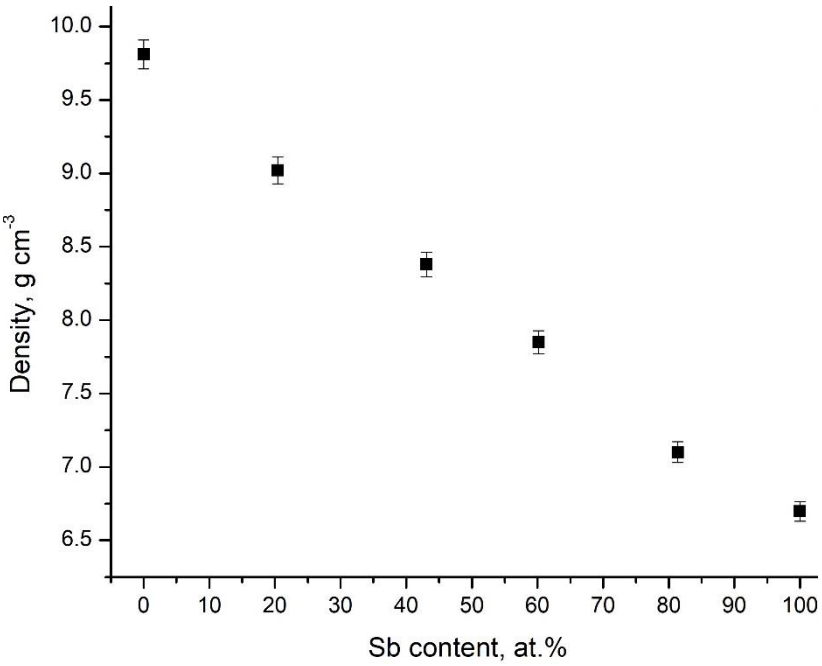

Figure 6. Densities of Bi-Sb alloys depending on composition at 25 °C (data are average of n=3)

Based on the results shown in Table 3, results it can be concluded that the specific heat capacity increases with the increase in antimony content in the alloys. Also, with the increase in temperature, there is an increase in the specific heat capacity of all tested alloys.

Figure 7 shows a comparison of calculated and experimentally determined specific heat capacities of Bi-Sb alloys at 25 °C.

Based on the presented results it can be noted that the specific heat capacity of Bi-Sb alloys increases with increasing the Sb content and temperature. The calculated values of specific heat capacity for $Bi_{79.6}Sb_{20.4}$ and $Bi_{18.6}Sb_{81.4}$ alloys are in very good agreement with the values obtained by the flash method. In the case of $Bi_{56.9}Sb_{43.1}$ and $Bi_{39.8}Sb_{60.2}$ alloys, slightly larger deviations are visible between the calculated values and those obtained by the flash method. Determination of specific heat capacity using the flash method requires the use of a reference material of known specific heat capacity.

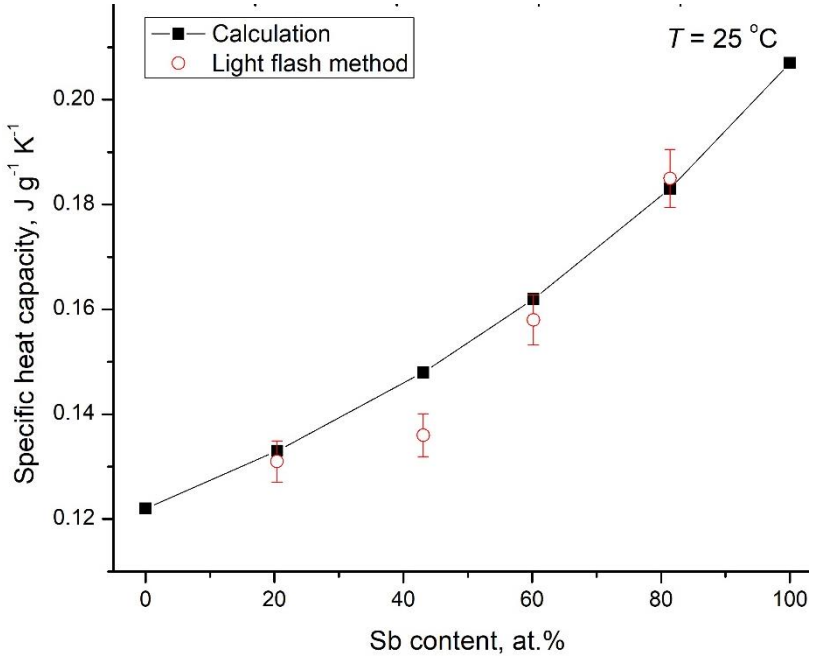

Figure 7. Comparison of calculated and experimentally determined specific heat capacities of Bi-Sb alloys at 25 °C (data are average of n=3)

In the present study, pure bismuth was used as a reference material for measuring the specific heat capacity of $Bi_{79.6}Sb_{20.4}$ and $Bi_{56.9}Sb_{43.1}$ alloys with high bismuth contents. To determine the specific heat capacity of antimony-based alloys $Bi_{39.8}Sb_{60.2}$ and $Bi_{18.6}Sb_{81.4}$, pure antimony was used as a reference material. The reason for the better agreement between the calculated and experimental values of the specific heat capacity for the $Bi_{79.6}Sb_{20.4}$ and $Bi_{18.6}Sb_{81.4}$ alloys can be explained by their greater similarity in terms of specific heat capacity with the reference materials that were used. This leads to the conclusion that coupling of a reference and sample with similar thermal properties might increase the accuracy of the specific heat capacity measurement.

Figure 8 shows the dependence of the experimentally determined thermal diffusivities of the examined Bi – Sb alloys on temperature.

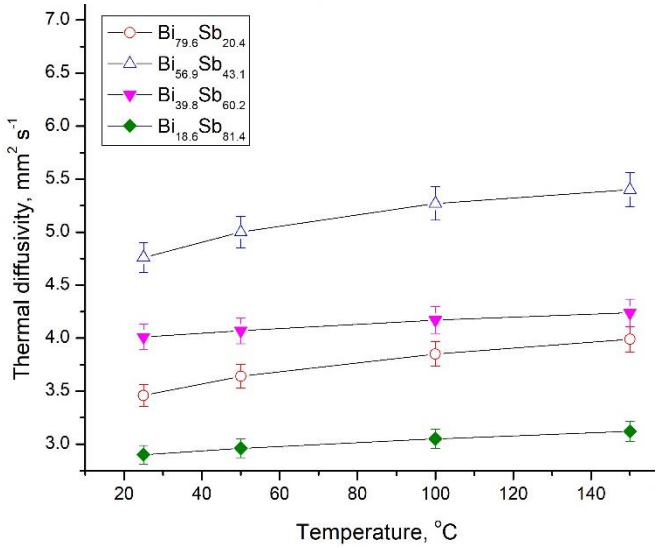

Figure 8. Temperature dependences of the experimentally determined thermal diffusivities of the examined Bi-Sb alloys (data are average of n=3)

It can be observed that with the increase in temperature there is a slight increase in thermal diffusivity.

Based on the results in Table 3, it can be concluded that the thermal conductivity values of the tested Bi-Sb alloys are low and are approximately in the range of 3.8 to 7 W m^{-1} K^{-1}, which is lower than the thermal conductivity of pure bismuth. With an increase in temperature, there is a slight increase in thermal conductivity.

Figure 9 shows a comparison of the obtained thermal conductivity values for the tested Bi-Sb alloys at 25 °C with literature values [11,15].

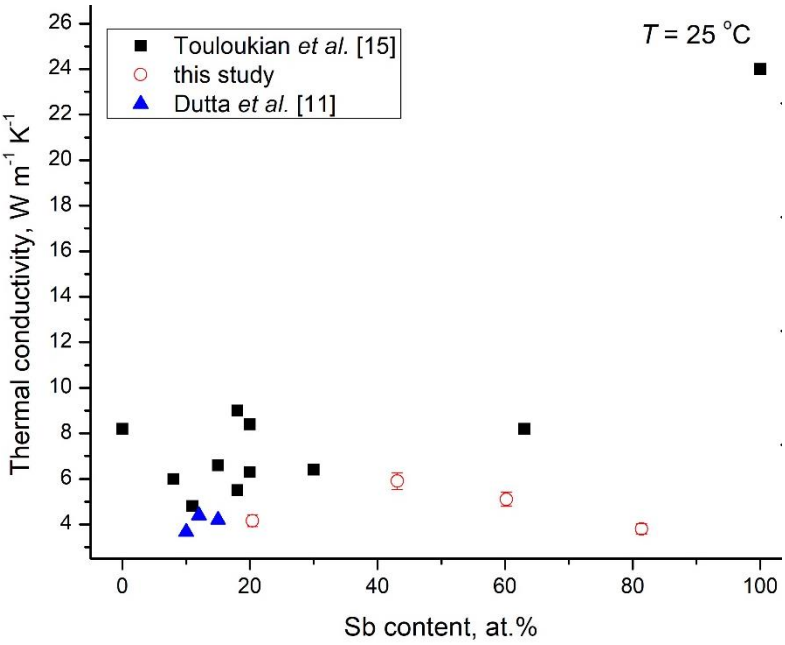

Figure 9. The obtained values of thermal conductivity for the tested Bi-Sb alloys at 25 °C in this study compared to literature values

Based on the literature values, it can be noticed that both studies [11] and [15] generally reported values that did not exceed 10 W m^{-1} K^{-1}, while the investigated alloys generally had higher amounts of Bi. The thermal conductivity values obtained in this work are in relatively good agreement with these literature values. It can be concluded that Bi-Sb alloys have very low thermal conductivities, which are lower than that of pure antimony and significantly lower than the thermal conductivity of pure bismuth.

4. CONCLUSION

Based on the performed experiments and the analysis of the obtained results, the following can be concluded:

a. The microstructure of all examined alloys is similar and consists of large grains of (Bi, Sb) solid solution, indicating that Bi and Sb form a continuous series of solid solutions. The microstructure is inhomogeneous. The reason for this is crystal segregation that appeared during crystal growth during cooling. Annealing at 240 °C for 1 week was not sufficient to remove crystal segregations.
b. Densities of Bi-Sb alloys lie between the values of the densities for pure bismuth and antimony, decreasing monotonically with increasing the antimony content.
c. Thermal diffusivity of Bi-Sb alloys increases slightly with increasing temperature.
d. The specific heat capacity of Bi-Sb alloys increases with increasing the Sb content and with increasing temperature. The values obtained at 25 °C were compared with the values obtained by the flash method and a good agreement was found.
e. With an increase in temperature, there is a slight increase in thermal conductivity. The thermal conductivities of the examined Bi-Sb alloys are low and are in the range of 3.8 to 7 W m^{-1} K^{-1}, which is lower than the thermal conductivity of pure bismuth. Alloys with intermediate antimony content had slightly higher values of thermal diffusivity and thermal conductivity than alloys with low and high antimony contents.

The results presented in this paper are essential for a better understanding of the thermal behavior of the investigated Bi-Sb alloys under different temperature conditions.

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Toplotna provodljivost i mikrostruktura Bi-Sb legura

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Izvod

Ispitivani su mikrostruktura i termičke osobine Bi-Sb legura sastava $Bi_{79.6}Sb_{20.4}$, $Bi_{56.9}Sb_{43.1}$, $Bi_{39.8}Sb_{60.2}$, $Bi_{18.6}Sb_{81.4}$. Za ispitivanje mikrostrukture pripremljenih legura korišćena je skenirajuća elektronska mikroskopija sa energetsko disperzivnom rendgenskom spektrometrijom. Svetlosno-impulsna metoda je primenjena za merenje toplotne difuzivnosti i za određivanje toplotne provodljivosti. Za određivanje gustine Bi-Sb legura korišćena je indirektna Arhimedova metoda. Dobijeni rezultati pokazuju da se gustina proučavanih legura monotono snižava sa povećanjem sadržaja antimona. Specifični toplotni kapacitet Bi-Sb legura raste sa povećanjem sadržaja Sb i sa povećanjem temperature. Toplotna difuzivnost ispitivanih legura određena je u temperaturnom intervalu od 25 do 150 °C. Toplotna difuzivnost Bi-Sb legura blago raste sa povećanjem temperature. Toplotne provodljivosti ispitivanih Bi-Sb legura su u rasponu od 3,8 do 7 W m^{-1} K^{-1}, što je niže od toplotnih provodljivosti čistog bizmuta i antimona. Rezultati dobijeni u ovom radu predstavljaju doprinos boljem poznavanju toplotnih osobina Bi-Sb legura, koje su od ključnog značaja za određivanje mogućnosti njihove praktične primene.

Ključne reči: Bi-Sb sistem; termičke osobine; SEM-EDS; metoda svetlosnog bljeska