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Influence of Nanostructure and Grain Refinement on the Thermoelectric Properties of Extruded Bulk $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ Materials

The electrical and thermal properties of extruded bulk nanostructured $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ samples were investigated in the temperature range of $\sim 77\div 300$ K and in magnetic fields up to 1.0 T. The results showed that at low temperatures the transport properties are mainly governed by charge carrier scattering at structural defects and grain boundaries. These imperfections act as effective scattering centers, reducing carrier mobility and electrical conductivity. As the grain size increases, the density of grain boundaries and the concentration of defects within the grains decrease, indicating an improvement in the material's microstructure. In addition, the concentration of ionized defects is reduced, which weakens impurity scattering and enhances charge carrier mobility.

Keywords: thermoelectric cooling, thermoelement, solid solution, extrusion, mobility, electrical conductivity.

Introduction

At present, considerable attention is being paid to issues of thermoelectric cooling using semiconductor materials. This new branch of modern technology opens a fundamentally new approach to the creation of compact devices for reducing and stabilizing temperature in local volumes. Thermoelectric cooling is the best technical solution for cooling and thermal

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stabilization of heat-generating elements in electronic and optical devices [1–5]. The best materials for thermoelement legs are solid solutions of $\text{Bi}_2\text{Te}_3\text{-Bi}_2\text{Se}_3$ (n-type) and $\text{Bi}_2\text{Te}_3\text{-Sb}_2\text{Te}_3$ (p-type) [6–17]. Due to the increasing demand for thermoelements, the development of new high-performance methods for producing thermoelement legs is a very relevant problem. Undoubtedly, the creation of nanothermoelectrics is of particular interest. At present, one of the cheapest technologies is the production of bulk nanostructured materials through mechano-activation processing in ball mills followed by hot pressing [18–23]. The figure of merit in nanostructures is influenced by the following factors: additional phonon scattering at nanograin boundaries; electron tunneling between nanostructural elements; and energy filtering of charge carriers due to the presence of potential barriers between nanograins. In this work, the influence of nanograin size on the magnitude of the kinetic coefficients in $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ materials is discussed.

Single crystals of the solid solution $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ are effective n-type thermoelectric materials and are recommended for use in energy converters operating in the temperature range of 200–300 K [24–27]. However, due to their layered structure, such materials are significantly damaged during the fabrication of thermoelement legs and during the assembly of thermoelectric energy converters based on them, which results in an extremely low yield of usable products. At the same time, the low mechanical strength of single crystals, caused by the structural features of these materials, reduces the reliability of energy converters operating under critical conditions. Thermoelectric material obtained by the extrusion method represents a fine-grained polycrystal with higher mechanical strength and thermoelectric parameters close to those of single crystals [28–32]. Thermoelectrics based on Bi-Sb-Te-Se solid solutions exhibit the highest figure of merit at relatively low temperatures. Therefore, these materials are of particular interest for improving thermoelectric efficiency (Z) through nanostructuring. Approaches to improving the efficiency of thermoelectric materials are associated with the creation of structural defects that can influence, to varying degrees, the scattering processes of charge carriers and phonons [33, 34]. In recent years, it has been suggested that Z can be increased in materials with two-dimensional and three-dimensional defects of the crystal structure, the distances between which are comparable to the mean free path of charge carriers or the wavelength of acoustic phonons responsible for heat transport. These assumptions are based on the possibility of creating conditions in which stronger scattering of thermal vibrations occurs than that of electrons and holes in regions of the material with different physical properties. In highly porous dispersed systems, the surface layers of particles can have a significant influence on Z . Dispersion leads to the formation of numerous defects on the surface of grains that make up the heterogeneous system.

There is a possibility of preferential reduction of thermal conductivity due to phonon scattering at grain boundaries, associated with changes in their phase velocity at these boundaries. At the boundaries, changes in the phase velocity of charge carriers also occur, and variations in thermoelectric efficiency are determined by the ratio of the additional scattering processes of electrons and phonons. The presence of a well-developed system of grain and subgrain boundaries in a material can lead to an increase in its efficiency if scattering at the boundaries predominantly affects either charge carriers or phonons. At present, to improve

thermoelectric efficiency, both hydrostatic (all-around) and uniaxial compression of n- and p-type crystals based on bismuth telluride are used, which leads to an increase in the figure of merit of the material due to changes in the power factor ($\alpha^2\sigma$). The problem of miniaturizing electronic coolers is becoming increasingly relevant, requiring the fabrication of thermoelement legs of such small dimensions that they become comparable to the thickness of the damaged layer formed during cutting of single-crystal thermoelectric material. This significantly reduces the actual thermoelectric efficiency of the thermoelectric energy converter. The highest thermoelectric figure of merit at relatively low temperatures (below ~ 300 K) is exhibited by samples of the $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ solid solution with grain sizes of about ~ 630 μm . Based on this, these compositions are of particular interest from the standpoint of possible enhancement of their thermoelectric properties through nanostructuring. In order to study the effect of nanostructuring on heat and charge transport in extruded samples of the $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ solid solution, the electrical properties of bulk nanostructured extruded samples were obtained and investigated in the temperature range of ~ 77 – 300 K and in magnetic fields up to 74×10^4 A/m. Both samples that were not annealed after extrusion and the same samples subjected to optimal annealing were examined.

Experimental Part

For the preparation of the investigated samples, bismuth of grade “Vi-0000”, selenium for rectifiers, and tellurium of grade “T-sCh” were used. The synthesis was carried out by direct melting of the components. The starting materials in stoichiometric proportions were placed into a quartz ampoule, pre-etched in a chromic acid solution ($\text{K}_2\text{Cr}_2\text{O}_7 + \text{H}_2\text{SO}_4$) and rinsed with distilled water. The ampoule was evacuated to a residual pressure of 10^{-3} Pa and then sealed. The synthesis was performed at a temperature of ~ 1053 K $\pm$ 5 K for 2 hours. To improve the homogeneity of the alloy, the ampoule containing the melt was subjected to rocking using a special device. The ampoule with the material was then cooled to room temperature by immersion in water.

The semiconductor material $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ was ground in a porcelain mortar, after which a fraction with a particle size of $\leq 1.6 \times 10^5$ nm was collected using a special sieve. Nanoscale powders were then obtained from this fraction using an AGO-2 ball mill. (Fig. 1). The milling process was carried out under an argon atmosphere at a rotational speed of 1820 rpm. The mass ratio of the milling balls to the powder was maintained at 20:1. The diameter of the balls was 8 mm. Metallic balls were used as the milling media. To obtain powders with different grain sizes, the milling duration was varied and set to 25, 35, and 50 min. Accordingly, variation of the milling time allowed powders with different degrees of refinement to be obtained, with grain sizes of approximately 38, 32, and 15 nm, respectively. All other milling parameters, including the milling atmosphere, rotational speed, and ball-to-powder mass ratio, were kept constant throughout the process. High-intensity mechanical milling of $\alpha\text{-Al}_2\text{O}_3$ in an AGO-2 planetary ball mill produced powders with particle sizes ranging from 18 to 40 nm, with the results obtained using different particle-size characterization methods showing good agreement [35].

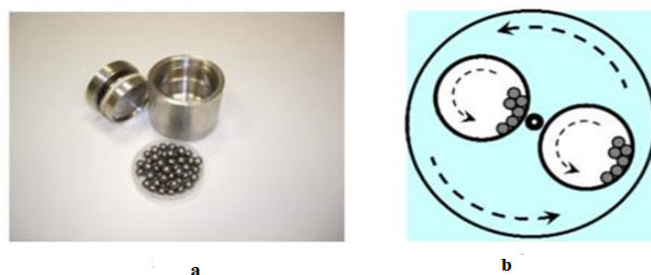


Fig. 1. Ball mill container (a) and schematic of the motion inside it (b).
The outer diameter of the cylinder in Fig. 1a is 50 mm [24]

To determine the particle sizes in the powder, an *X*-ray diffraction system XRD D8 ADVANCE (Bruker, Germany) was used, along with the TOPAS-4.2 and EVA software packages. The nanoparticles had sizes of 1.6×10^5 , 38, 30 and 15 nm. The ground semiconductor material was pressed into billets at a temperature of ~ 300 K and a pressure of 450 MPa. The diameter of the billets was approximately 30 mm. The formation of samples (thermoelement legs) was carried out using the extrusion method. The technological parameters of extrusion (temperature, pressure, draw ratio, etc.) significantly influence the material's properties and the homogeneity of their distribution, as well as the mechanism and kinetics of processes occurring during post-extrusion annealing. With an optimal combination of technological parameters, it is possible to produce rods free of macroscopic defects, possessing high thermoelectric and mechanical properties. Extrusion was performed on an MS-1000 hydraulic press, reducing the diameter from 30 mm to 6 mm (draw ratio = 25). Technological Parameters of Extrusion: $T_{ex} = 653 \pm 3$ K, $P_{ex} = 9$ t/cm². Hot pressing (extrusion) allows the production of material with high density and free of delamination cracks. Post-extrusion annealing was carried out in quartz ampoules under vacuum (10^{-3} Pa) at ~ 690 K for 5 hours.

Cylindrical samples were cut into parallelepiped specimens using an A207.40M electrical discharge machining (EDM) system. During cutting of single crystals of bismuth telluride-based solid solutions into thermoelement legs, a damaged surface layer forms, differing in structure and properties from the rest of the material, which significantly reduces their thermoelectric figure of merit. The surface layer formed during cutting, with a thickness of 10–15 μ m, was removed by electrochemical etching. The depth of the damaged layer was estimated from the dependence of etching rate on time, which differs for deformed and undeformed material. Removal of this damaged layer leads to an improvement in the properties of the investigated samples.

The etching rate of grains in a coarse-block aggregate depends on their orientation relative to the cutting plane and varies among different grains. In fine-grained polycrystalline material obtained by the Bridgman method, the etching rate is averaged over orientations. The characteristics of the damaged layer structure were studied by *X*-ray diffraction, using a stationary sample in reflection geometry on an RKSO camera with Cu anticathode radiation; the depth of the semi-absorbing layer was approximately 15 μ m. *X*-ray texture analysis was performed on nanostructured Bi₂Te_{2.7}Se_{0.3} samples with varying grain sizes. To study the

texture, the intensity of the (0006) and (1120) reflections was analyzed from three or five sections of the rods. The sections were cut at angles of 0°, 180°, 360°, 540°, 720° or 0°, 45°, 90° relative to the extrusion axis. Data collection was carried out on a diffractometer using monochromatized radiation from an Fe-anode X-ray tube, with primary beam divergence not exceeding 10' and tilts relative to the horizontal axis of the goniometer in 2° increments. For the investigated samples, tilting the working surface relative to the goniometer axis by more than 25–30° was undesirable due to strong defocusing. Corrections for defocusing during tilting were introduced based on measurements of a sample with its working surface parallel to the extrusion axis, rotated around the extrusion axis. The determination of the orientation probability density, ρ_φ from the experimental values of the angular intensity distribution was carried out using the following formula [36].

$$\rho_{(\varphi,\psi)} = \frac{2}{3} [\rho_{\varphi,\psi}]^3 \Delta\varphi\Delta\psi \sin \varphi.$$

A high-speed measuring device, CUIP (digital universal measuring instrument) with an accuracy class of 0.02 and high input resistance, was used as the recording instrument, allowing the measured quantities to be recorded with high precision. The linear dimensions of the samples were controlled using an MBS-1 microscope with an accuracy class of 0.005 mm.

Using an atomic force microscope (Multimode AFM-LN), 3D-three-dimensional topography of the surface of the studied samples (material relief) was obtained, displaying the height and depressions on its surface on a nanometer scale.

Raman spectra of bulk nanostructured samples of the $\text{Bi}_{0.85}\text{Sb}_{0.15}$ solid solution with different grain sizes were investigated using an OPTOSKY ATR 8000 spectrometer with a 532 nm excitation wavelength.

The bending strength of the obtained bulk nanostructured $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ samples was investigated using the method described in [37]. It was found that the strength of these samples is approximately three times higher than that of single-crystal samples. The electrical conductivity (σ), thermoelectric power coefficient (α), and Hall coefficient (R_H) were measured using a stationary potentiometric method, while the thermal conductivity (χ) was measured by the absolute method [38]. The electrical and thermal parameters of the investigated samples were measured along the length of the sample (rod), i.e., in the extrusion direction. The measurement error did not exceed $\pm 3\%$ for electrical parameters and $\pm 5\%$ for thermal parameters.

Results and Discussion

The analysis of the Raman spectra of nanosized $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ crystals demonstrates that the crystal grain size has a significant effect on the crystal structure, phonon dynamics, as well as the electrical and thermoelectric properties. The main peaks observed in the Raman spectra are associated with the A_{1g}^1 , A_g^2 , A_{1g}^2 optical phonon modes characteristic of the Bi_2Te_3 -type layered structure. The presence of these peaks confirms that the main crystal phase is preserved in the synthesized samples.

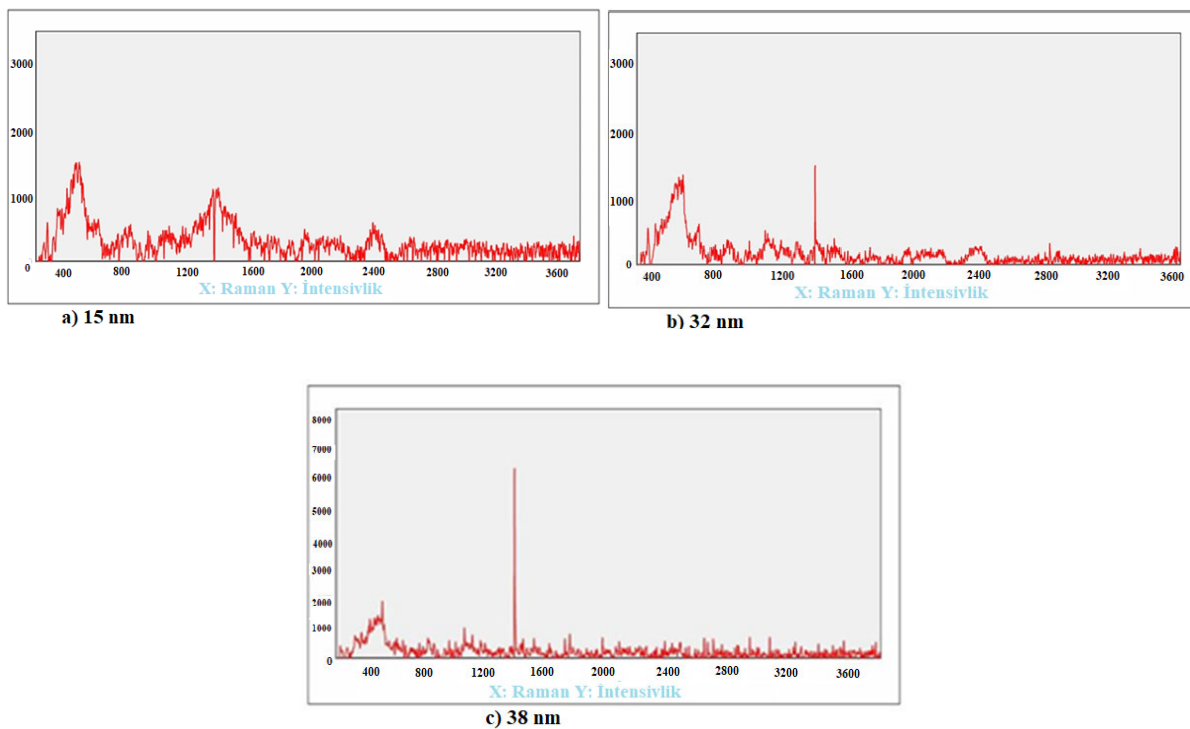


Fig. 2. Raman spectrum of samples of $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ solid solution with different grain sizes of a (15 nm), b (32 nm) and c (38 nm)

In the 15 nm sample, the Raman lines are broad and weak in intensity, which can be explained by the strong phonon confinement effect, the high surface-to-boundary ratio, and structural disorder. As the nanoscale dimensions decrease, the free propagation of phonons becomes restricted, the defect density increases, and phonon scattering intensifies. As a result, the Raman peaks broaden and the background intensity in the spectrum increases. Structural defects and scattering at grain boundaries limit the mobility of charge carriers (electrons), leading to a decrease in electrical conductivity. On the other hand, enhanced phonon scattering reduces thermal conductivity, which is considered an important advantage for nanoscale thermoelectric materials.

With the increase in size to 32 nm, the Raman peaks become sharper and more intense, indicating improved crystallinity. The relative reduction of surface effects and the growth of crystal domains enhance phonon coherence. In particular, the intense peak observed in the high-frequency region indicates that the A_{1g}^2 optical phonon mode is formed within a more stable crystal structure. The improvement of the crystalline structure reduces electron scattering and consequently increases electrical conductivity. At the same time, since nanoscale effects are still partially preserved, phonon scattering continues and thermal conductivity remains relatively low, creating favorable conditions for enhanced thermoelectric efficiency.

In the 38 nm sample, the Raman peaks exhibit maximum intensity and minimum linewidth, confirming a high degree of crystallinity. The narrowing of the peaks indicates an increase in phonon lifetime and a reduction in structural disorder within the lattice. In this sample, the phonon confinement effect weakens, and the Raman spectrum approaches the

behavior of bulk material. A more ordered crystal lattice enhances electron mobility, leading to increased electrical conductivity. However, the reduction in phonon scattering may also result in a certain increase in thermal conductivity. Nevertheless, the high electrical conductivity and carrier mobility positively influence the thermoelectric power factor ($\alpha^2\sigma$).

In addition, Se doping partially replaces Te atoms in the crystal lattice, creating local lattice distortions and affecting the frequencies of phonon modes. Since Se atoms possess a smaller atomic mass, some Raman modes may shift toward higher frequency regions. Furthermore, Se doping contributes to alloy scattering due to differences in atomic radii, atomic masses, and bonding energies, thereby enhancing local inhomogeneity within the crystal and reducing thermal conductivity. As a result, the direction of phonon propagation changes, their free transport is disrupted, heat-carrying phonons are scattered, and favorable conditions are created for improving the thermoelectric figure of merit (ZT).

The surface morphology of nanosized $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ crystals was investigated using the Atomic Force Microscopy (AFM) (Multimode AFM-LN) technique (Fig. 3). The AFM analyses revealed that significant changes occurred in the surface structure of nanocrystals with different sizes.

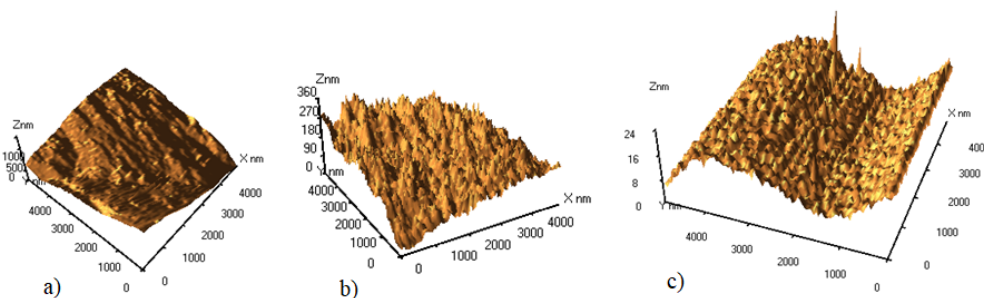


Fig. 3. 3D-three-dimensional surface topography of extruded samples of $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ solid solution with different grain sizes of 38 nm (a); 32 nm (b); 15 nm (c) using an atomic force microscope (AFM)

Figures a, b, and c represent the surface topography of samples with sizes of 15 nm, 32 nm, and 38 nm, respectively. In the AFM image of the 15 nm sample, the surface appears relatively smooth and homogeneous. The nanoparticles are densely packed, and the surface roughness is at a low level. This indicates that the crystal structure was formed in a more compact manner. For the 32 nm sample, it is observed that with the increase in grain size, protrusions and depressions on the surface become more pronounced. The increase in surface roughness is associated with the aggregation of nanoparticles and the growth of crystal grains. Compared to the previous sample, the surface morphology of this sample exhibits a rougher character. The AFM image of the 38 nm sample shows larger topographical structures and higher surface roughness. The merging of crystal grains into larger aggregates caused a non-uniform distribution on the surface. This demonstrates that the surface morphology changes significantly as the nanocrystal size increases. Overall, the AFM analysis results indicate that increasing the particle size in $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ nanocrystals leads to an increase in surface roughness and aggregation level. These changes in surface morphology may have a significant

influence on the electrical and thermoelectric properties of the material. Smaller nanocrystal sizes and a more homogeneous surface structure may facilitate carrier mobility, thereby increasing electrical conductivity. However, the growth of particle size and the increase in surface roughness enhance boundary scattering, which limits the mobility of electrons and holes and may reduce electrical conductivity. On the other hand, increased boundary scattering weakens phonon propagation, resulting in lower thermal conductivity. This is considered an important advantage for thermoelectric materials. Thus, changes in the size and surface morphology of $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ nanocrystals directly affect electrical conductivity, thermal conductivity, and consequently the thermoelectric figure of merit (ZT). Therefore, controlling nanoscale structures is of great importance for optimizing the thermoelectric parameters of the material.

The phase composition and crystal structure of the as-synthesized $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ solid solution samples with various average grain sizes (15 nm, 38 nm and 32 nm) were investigated using X-ray diffraction (XRD) technique. A comparative analysis of the diffraction patterns fundamentally demonstrates the strong dependence of the structural parameters on the size effect.

The overall profile of the diffractograms indicates that the diffraction peaks recorded for all three samples are in excellent agreement with the parameters of the rhombohedral Bi_2Te_3 - type lattice belonging to the $R3m$ space group. No impurity phases or segregations of the constituent elements (Bi, Te, Se) were detected in the matrix. This confirms the successful substitution of Te atoms by Se atoms within the Bi_2Te_3 lattice, yielding a homogeneous single-phase solid solution. The most intense diffraction peak is observed at approximately $2\theta \approx 27.5^\circ$, corresponding to the dominant crystallographic plane of the material. Furthermore, the well-resolved doublet peaks appearing around $2\theta \approx 38.2^\circ$ and 39.8° further signify the high phase purity of the structure.

As the grain size decreases from 32 nm to 15 nm, two prominent physical phenomena manifest in the profile of the diffraction peaks:

- **Peak Broadening:** Concomitant with the reduction in grain size, a noticeable increase in the full width at half maximum (FWHM) is observed for all major diffraction peaks. This phenomenon is highly characteristic of nanostructured materials and complies fully with the Scherrer formula, serving as a direct consequence of the shrinkage of coherent scattering domains (crystallite sizes) [36]:

$$D = \frac{K\lambda}{\beta \cos \theta}$$

- **Intensity Variations and Microstrain:** The relative decrease in peak intensities and the concomitant rise in the background signal for the 15 nm sample are attributed to the degradation of short-range order, an increased volume fraction of grain boundaries, and the induction of lattice microstrain originated from the extrusion process as the grain size diminishes.

From the perspective of thermoelectric applications, these structural alterations derived from the XRD analysis exert a profound influence on the phonon transport properties of the material. The reduction of crystallite size down to the nanoscale, coupled with the enhanced density of grain boundaries, induces intense sub-micron boundary scattering of heat-carrying phonons. This provides a fundamental framework for minimizing the lattice thermal

conductivity (χ_L) without detrimentally affecting the electronic transport, thereby optimizing the overall thermoelectric figure of merit (ZT) of the $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ solid solution.

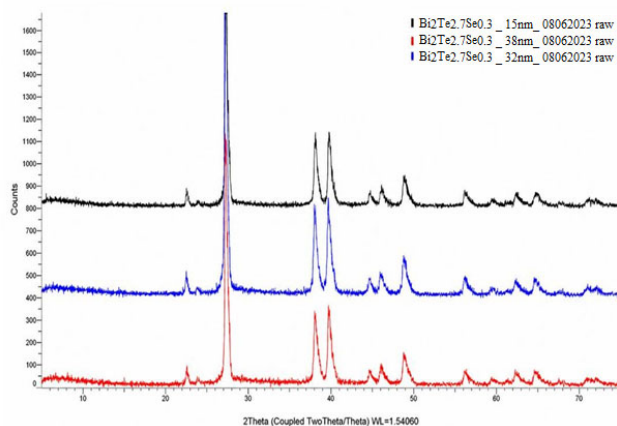


Fig. 4. X-ray diffraction (XRD) patterns of extruded $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ solid solution samples with different average grain sizes: 15nm (black line), 38 nm (red line), and 32 nm (blue line).

The obtained results are presented in Figs. 5–8 and in the table.

The figures and table 1 show the absolute values of α and R_H . It can be seen that heat treatment affects the electrical properties of the investigated samples with different grain sizes in different ways. A significant change is observed in the samples with the smallest grain size of 15 nm.

The temperature dependence of the thermoelectric power coefficient indicates that the investigated materials are not strongly degenerate. The derivative ($d\alpha/dT$) corresponds to what has been observed in single crystals of similar compositions and tends to decrease with increasing electron concentration at ~ 77 K. Nanostructuring does not change the temperature dependence of conductivity, which in the studied temperature range is described by the acoustic scattering mechanism for unannealed samples. Conductivity decreases with increasing temperature for all samples, i.e., for both annealed and unannealed samples, which is associated with changes in the drift mobility of electrons. In the high-temperature region, the coefficient β ($\beta = 1.1 \div 1.3$) in the $\sigma \sim T^\beta$ dependence is close to the data in [39, 40]. With decreasing conductivity in the samples, the $\sigma = f(T)$ dependence also decreases.

Deformation during the extrusion of polycrystalline materials based on Bi_2Te_3 leads to the formation of two types of texture in the rod: $[110] [001]$; the existence of non-textured material is also possible. The proportion and degree of perfection of the texture components depend on many factors: in composite materials, on the type and amount of the modifying additive, the base material, the extrusion temperature regime, and post-extrusion annealing. Axial $[110]$ and ring $[001]$ textures are favorable for thermoelectric properties -the basal planes of the crystals (planes with higher mobility) are aligned along the rod axis. Textures formed as a result of plastic flow of the material are determined by which slip systems are activated and by the contribution of grain boundary deformation (depending on the deformation scheme, temperature, rate, structure of the billet, and, under conditions of dynamic recrystallization, the degree of recrystallization). Analysis of deformation textures and post-deformation annealing

in tetradymites from the perspective of quantitatively evaluating the probability of alignment of the basal plane with the extrusion axis and assessing each grain's contribution to the macroscopic conductivity along the rod axis. The texture formation mechanism (basal slip, multiple slip) is also significant, as different texture components may be characterized by different substructures (and therefore different microconductivities) and varying levels of chemical homogeneity in the distribution of the material's constituent components.

Table1

Electrical and Thermal Properties of Extruded Samples of the $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ Solid Solution with Different Grain Sizes at $\sim 300\text{ K}$

Grain size	Before annealing									
	$\sigma, \Omega^{-1} \cdot \text{cm}^{-1}$	$\alpha, \mu\text{V/K}$	$R_H \times 10^{-8} \text{cm}^3/\text{C}$	$\mu, \text{cm}^2/\text{V} \cdot \text{s}$	$n, \times 10^{16}, \text{cm}^{-3}$	$\chi \times 10^2, \text{W}/(\text{cm} \cdot \text{K})$	$\chi_e \times 10^2, \text{W}/(\text{cm} \cdot \text{K})$	$\chi_L \times 10^2, \text{W}/(\text{cm} \cdot \text{K})$	$\alpha^2 \sigma, \text{mW m}^{-1} \text{K}^{-2}$	$Z \times 10^{-3}, \text{K}^{-1}$
$1.6 \times 10^5 \text{ nm}$	1145	188	0.7	802	0.89	1.88	0.0083	1.8717	4.04	2.1
38 nm	1219	186	0.29	353	0.21	1.07	0.0089	1.0611	4.22	3.9
32 nm	950	107	0.2	190	0.31	1.69	0.0069	1.6831	1.08	0.6
15 nm	208	113	0.15	31	0.41	1.16	0.0015	1.1585	0.26	0.2
After annealing										
$1.6 \times 10^5 \text{ nm}$	1141	176	0.64	730	0.97	1.67	0.0083	1.6617	3.53	2.1
38 nm	1117	75	0.54	603	0.11	1.2	0.0081	1.1919	0.62	0.5
32 nm	766	111	0.24	184	0.26	1.53	0.0056	1.5244	0.94	0.6
15 nm	255	259	0.5	128	0.12	1.06	0.0018	1.0582	1.71	1.6

For example, if tellurium complexes form inter-quintet structures, basal slip cannot lead to their destruction. In the studied samples, extrusion, where the deformation pattern is similar to uniaxial stretching, leads to the formation of axially symmetric orientations. In this case, the pole density distribution is characterized by a body of revolution with an axis parallel to the direction of the applied external force.

The specific electrical conductivity σ decreases monotonically with temperature and increases with grain size. For all the studied samples, at temperatures below $\sim 200\text{ K}$, the temperature dependences of σ and α typical of the impurity conduction region are observed (Fig. 5). The thermoelectric power coefficient (Seebeck coefficient) for all samples with nanosized grains increases with temperature for both unannealed and annealed samples. The deviation of the $\alpha(T)$ dependence from linear behavior at temperatures above $\sim 270\text{ K}$ is presumed to be associated with the onset of intrinsic conduction.

Due to the fine-grained structure of the extruded samples of the $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ solid solution, the mean free path, determined using standard formulas for a parabolic band and acoustic scattering, turns out to be smaller than the grain size. The presence of potential barriers between different grains in bulk nanostructures can lead to a strong energy dependence of the

charge carrier mean free path near the chemical potential level. As a result, this leads to carrier filtering: carriers with energies higher than the Fermi energy can overcome the grain boundaries without scattering. As a result of experimental studies and theoretical analysis, it has been established that, as thermoelectric materials, it is most advantageous to use semiconductors with high carrier mobility, large effective mass, and low lattice thermal conductivity, $Z \sim \left(\frac{\mu}{\chi_l}\right) \cdot \left(\frac{m^*}{m_0}\right)^{3/2} \cdot T^{3/2}$ where μ is the drift mobility of charge carriers; χ_l is the lattice component of thermal conductivity; m_0 is the free electron mass; m^* is the effective mass of charge carriers; and T is the absolute temperature.

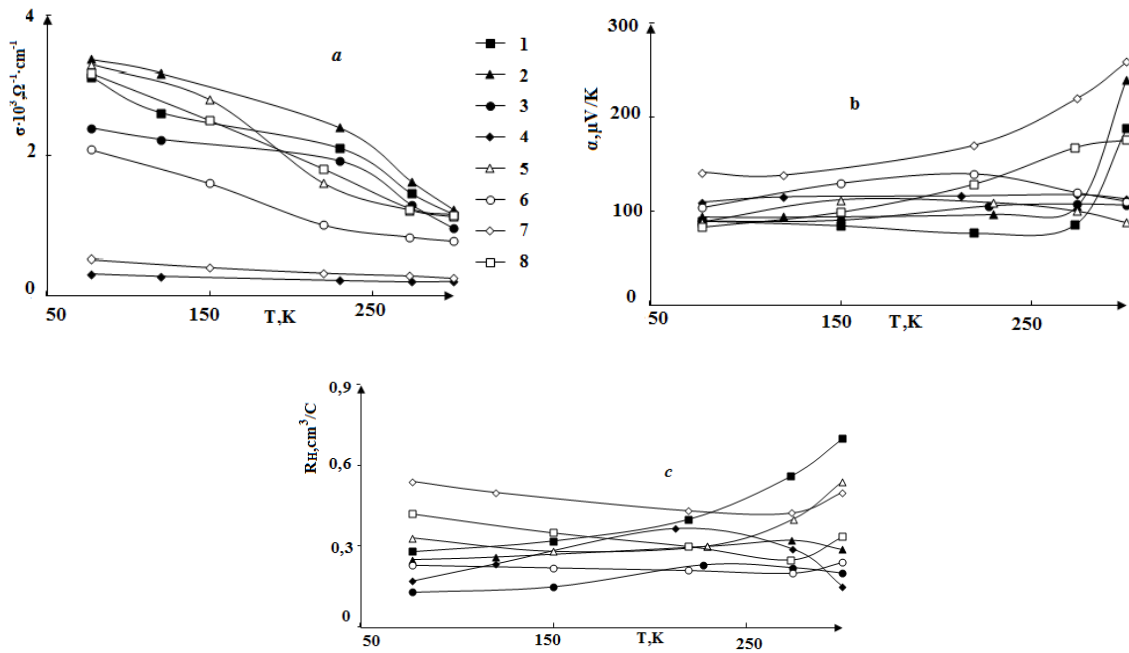


Fig. 5. Temperature dependences of the electrical conductivity σ (a), Seebeck coefficient α (b), and Hall coefficient R_H (c) for extruded samples of the solid solution $\text{Bi}_2\text{Te}_{2.7}\text{Se}_0$ with different grain sizes. The curves correspond to: 1 and 2 – samples with grain size 1.6×10^5 nm; 3 and 4 – 38 nm; 5 and 6 – 32 nm; 7 and 8 – 15 nm. Curves 1, 3, 5, and 7 represent unannealed samples, while 2, 4, 6, and 8 represent annealed samples.

The performed analysis of the dependence of R_H on the orientation of the field and the direction of current relative to the extrusion axis showed that R_H of the polycrystalline extruded sample is isotropic. The calculated Hall mobilities of charge carriers at $T = \text{const}$ correlate with the parameter $\mu_0(m^*/m_0)^{3/2}$, where μ_0 is the drift mobility of carriers in the absence of degeneracy; $m^* \sim T^s$ ($s = 0.12$ and 0.14 for Bi_2Te_3 with n- and p-type conductivity, respectively) [39, 40]. The temperature dependence of the effective mass has been experimentally confirmed. Thus, the microscopic mobility can be evaluated using the parameter $\mu_0(m^*/m_0)^{3/2}$, for the determination of which it is sufficient to measure the electrical conductivity in two directions and the Seebeck coefficient, which is isotropic within experimental error. The Hall mobility calculated from the experimental values of electrical conductivity and the Hall coefficient showed that, for all samples – both with and without heat treatment – it increases with grain

size over the entire investigated temperature range (Fig. 6). The most significant change due to heat treatment is observed in samples with the smallest grain sizes. The mobility in heat-treated samples exceeds that in untreated samples. The charge carrier mobility is determined by scattering from structural defects of deformation origin. For Bi_2Te_3 and its analogs, the parameter $r = -0.5$ is generally observed over a wide range of temperatures and concentrations [39, 40]. For thermoelectric materials, this is an important parameter,

$$\mu_0 \left(\frac{m^*}{m_0} \right)^{3/2} = \frac{\sigma_r(r+1.5)}{2l(2\pi m_0 k_0 T/h^2 \cdot F_{r+0.5}(\xi^*))},$$

where μ_0 is the drift mobility of charge carriers in the absence of degeneracy; k_0 is the Boltzmann constant; h is Planck's constant; $\xi^* = \xi/(k_0T)$ is the chemical potential of free charge carriers, measured positively upward from the bottom of the conduction band for electrons and downward from the top of the valence band for holes; and F_r values are taken from [40–42].

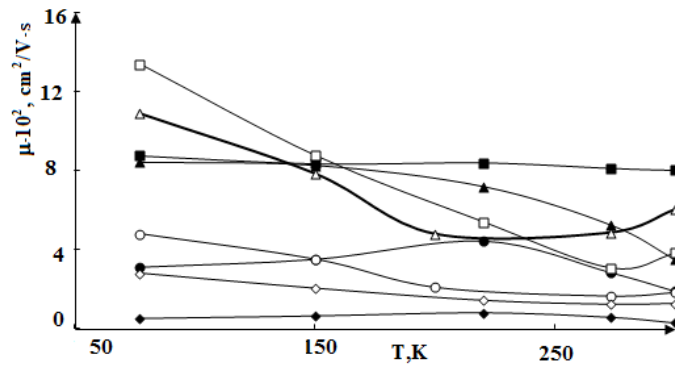


Fig. 6. Temperature dependence of the Hall mobility μ for extruded samples of the $\text{Bi}_2\text{Te}_{2.7}\text{Se}_0$ solid solution with different grain sizes. The notation is the same as in Fig. 2

The quantity $\mu_0(m^*/m_0)^{3/2}$, for a known scattering parameter in the impurity conduction region, can be calculated from experimentally determined values of two measurable parameters – electrical conductivity and the Seebeck coefficient. For degenerate samples, the Seebeck coefficient is described by the expression [39, 40],

$$|\alpha| = \frac{k_0}{e} \left\{ \frac{(2r+5)F_{r+1.5}(\xi^*)}{(2r+3)F_{r+0.5}(\xi^*)} - \xi^* \right\}.$$

The decrease in electrical conductivity with increasing temperature in all samples is associated with a reduction in the drift mobility of charge carriers, $\mu = \mu_0 T^b$. This change in mobility is due to the simultaneous scattering of carriers by both optical and acoustic deformation potentials. As the electrical conductivity decreases, the temperature dependence $\sigma = f(T)$ becomes weaker. During extrusion, the grains of the studied material change their orientation and shape relative to the direction of applied deforming forces (along the extrusion axis). At high degrees of deformation, a texture develops, which leads to the formation of a recrystallization texture and simultaneously generates various electrically active structural defects. In this case, the newly recrystallized grains acquire a preferred crystallographic orientation. The nature and degree of the texture are determined by grain size, the temperature of post-extrusion annealing, and other factors. At low annealing temperatures, recrystallization mirrors the deformation texture. At high annealing temperatures, the recrystallization texture

often differs from the deformation texture or may be absent entirely. Defects created during plastic deformation act as scattering centers for charge carriers, reducing their mobility. As the crystallite (grain) size decreases, the energy required to reorient grains simultaneously decreases, leading to an increase in the degree of texture in the sample. Samples with the smallest grains have a high concentration of charge carriers due to the high dispersion of the medium. In samples with larger grains, the energy required for grain orientation – and thus for texture formation – increases. During heat treatment, structural defects are effectively “healed,” and the structure of the studied samples is normalized. Annealing-induced normalization increases carrier mobility while simultaneously reducing the degree of texture, which can be explained by the reorientation of grains under thermal energy. The influence of grain orientation varies depending on the geometric proportions of thermoelectric elements made from these samples. The thermal resistance $(\chi - \chi_e)^{-1}$ of heterogeneous materials is higher than that of the semiconductor matrix, despite the fact that the thermal conductivity of a mechanical mixture of semiconductor and dielectric is higher than that of the semiconductor alone. This indicates that additional phonon scattering caused by the actual structure of the heterogeneous material is sufficient not only to compensate for the extra thermal conductivity of the mixture but also to increase the overall thermal resistance. The effect of nanostructuring on the thermal conductivity of extruded $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ samples has been studied (Fig. 7).

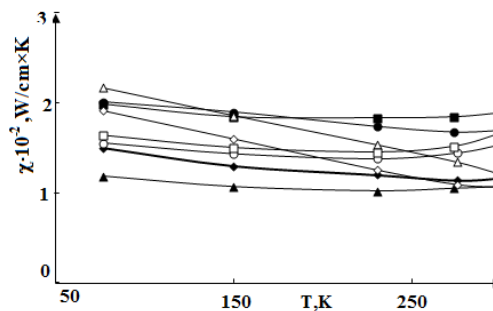


Fig. 7. Temperature dependence of the thermal conductivity of extruded samples of the $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ solid solution with different grain sizes. The notation is the same as in Fig. 1

In this analysis, the temperature dependence of the Lorenz number was not considered, which may affect the absolute values of the electronic component but does not change the character of the temperature dependence of thermal conductivity in the studied samples. The temperature dependence of thermal conductivity for $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ samples with different grain sizes that were not annealed is the same at low temperatures. With increasing temperature, the overall thermal conductivity decreases. At relatively low temperatures, the total thermal conductivity changes linearly with temperature, decreasing as temperature rises. For $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ samples that were annealed and those not thermally treated after extrusion with grain sizes of 1.6×10^5 nm, the thermal conductivity is higher at temperatures above ~ 200 K compared to samples with grain sizes of ~ 38 , 32, and 15 nm. With increasing temperature, above ~ 230 K for annealed samples with grain sizes of 38 and 15 nm, a deviation of the dependence $1/\chi_0 = f(T)$ from linearity is observed (Fig. 8).

The deviation of the dependence $1/\chi_0 = f(T)$ from linearity at high temperatures may be

caused by the involvement of additional mechanisms in thermal conductivity. The most likely of these are heat transport by bipolar diffusion (χ_{pn}) and by electromagnetic radiation-photons (χ_p).

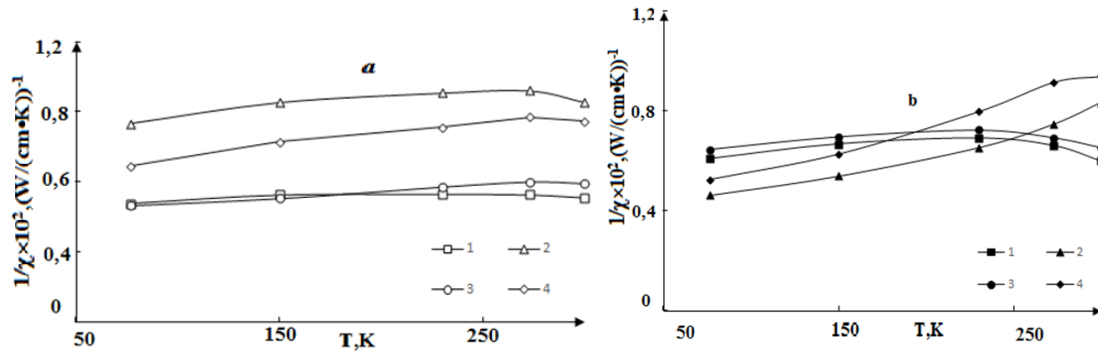


Fig. 8. Thermal resistance $1/\chi$ of extruded $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ -based samples with different grain sizes.

1 – samples with grain size 1.6×10^5 nm; 2 – 38 nm; 3 – 32 nm; 4 – 15 nm.

(a) corresponds to unannealed samples, (b) corresponds to annealed samples

The bipolar component of thermal conductivity was calculated using the formula [39].

$$\chi_{pn} = LT \frac{2\sigma_p \cdot \sigma_n}{\sigma_p + \sigma_n} \left(\frac{\varepsilon_g}{2k_0 T} + r_n + r_p + 5 \right)^2,$$

where σ_p and σ_n are the electrical conductivities due to holes and electrons, respectively; and r_p and $r_n = -1/2$ are the scattering factors for holes and electrons [41]. This formula can be transformed into

$$\chi_{pn} = \left(\frac{k_0}{e} \right)^2 T \cdot \frac{n \cdot e \cdot \mu_n}{1 + \frac{\mu_n}{\mu_p}} \left(\frac{\varepsilon_g}{k_0 T} + 4 \right)^2,$$

where n is the intrinsic charge carrier concentration, equal to $n = 2 \frac{(2\pi m^* k_0 T)^{3/2}}{h^3} e^{-\frac{\varepsilon_g}{k_0 T}}$.

Assuming $\mu_n = 2\mu_p$, knowing the estimated value of μ_n from the dependence $\mu_n = f(T)$ and m^*/m_0 , the bipolar component of thermal conductivity can be easily calculated. The thermal conductivity due to radiation was calculated using the formula: $\chi_{pn} = (16n^2 \sigma_0 T^3 d_0)/3$,

where n is the refractive index of the material, σ_0 is the Stefan–Boltzmann constant, and d_0 is the sample diameter.

At high temperatures, in the studied systems, heat transport also occurs via bipolar thermodiffusion and phonons. It is also observed that, in samples, decreasing the size of nanoparticles reduces the overall thermal conductivity (see table). In all cases, the lattice component of thermal conductivity in samples with nanosized grains (38, 32, and 15 nm) is lower than that in samples with grain sizes of 1.6×10^5 nm. In these samples, heat transfer by photons becomes significant only at higher temperatures. Thus, in bulk nanostructured samples that have not been annealed, at low temperatures, charge carriers are primarily scattered by structural defects and grain boundaries. As grain size increases, the number of grain boundaries and the concentration of defects within the grains decrease. Consequently, the concentration of carriers associated with ionized defects diminishes, leading to an increase in the charge carrier mobility in the sample. It was found that nanostructuring and heat treatment in extruded $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ samples do not change the temperature dependence of electrical conductivity, carrier mobility, or the Seebeck coefficient along the extrusion axis.

Conclusion

In bulk nanostructured $\text{Bi}_2\text{Te}_{2.7}\text{Se}_{0.3}$ samples that have not been annealed, at low temperatures, charge carriers are primarily scattered by structural defects and grain boundaries. As the grain size increases, both the number of grain boundaries and the concentration of defects within the grains decrease. Consequently, the concentration of carriers associated with ionized defects diminishes, leading to an increase in charge carrier mobility in the sample. The decrease in electrical conductivity with increasing temperature in all samples is associated with a reduction in drift mobility, $\mu = \mu_0 T^{\beta}$. This change in mobility is caused by simultaneous scattering of carriers on optical and acoustic deformation potentials. The deviation of the dependence $1/\chi = f(T)$ from linearity at high temperatures may be due to the involvement of additional mechanisms in thermal conductivity. The most likely of these are heat transport via bipolar diffusion (χ_{pn}) and electromagnetic radiation-photons (χ_p).

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Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Вплив наноструктури та подрібнення зерен на термоелектричні властивості екструдованих об'ємних матеріалів Bi₂Te_{2.7}Se_{0.3}

Електричні та теплові властивості екструдованих об'ємних наноструктурованих зразків Bi₂Te_{2.7}Se_{0.3} досліджували в діапазоні температур ~ 77 ÷ 300 К та в магнітних полях до 1.0 Тл. Результати показали, що за низьких температур транспортні властивості в основному визначаються розсіюванням носіїв заряду на структурних дефектах та межах зерен. Ці недосконалості діють як ефективні центри розсіювання, зменшуючи рухливість носіїв заряду та електропровідність. Зі збільшенням розміру зерна щільність меж зерен та концентрація дефектів усередині зерен зменшуються, що свідчить про покращення мікроструктури матеріалу. Крім того, зменшується концентрація іонізованих дефектів, що послаблює розсіювання на домішках та підвищує рухливість носіїв заряду.

Ключові слова: термоелектричне охолодження, термоелемент, твердий розчин, екструзія, рухливість, електропровідність.