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OPTIMIZATION OF A VACUUM THERMAL EVAPORATION SYSTEM FOR THE DEPOSITION OF Bi–Sb–Te THIN FILMS

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In this study, thin films based on bismuth and antimony chalcogenides (Bi_2Te_3–Sb_2Te_3) were synthesized using an optimized thermal evaporation vacuum system, and their morphological characteristics were thoroughly investigated. Atomic force microscopy (AFM) results revealed nanoscale granularity on the film surface (in the range of 50–150 nm) and a distinct stepped morphology. Longitudinal profile analyses indicated that the height variations lie within the range of 0.790–0.798 μm. Scanning electron microscopy (SEM) observations confirmed the formation of grains, larger particles, and surface voids, indicating a dual-level morphological structure. Such a structure is critically important for enhancing thermoelectric efficiency by intensifying phonon scattering, thereby reducing thermal conductivity while preserving electron transport properties. The findings demonstrate that Bi_2Te_3–Sb_2Te_3-based thin films possess high scientific and practical potential as thermoelectric materials.
Keywords: *Thin Film; Morphology; Atomic Force Microscopy; Electron Microscopy; Thermoelectric Efficiency; Nanogranularity; Stepped Structure*
PACS: 68.55.-a; 81.15.-z; 78.67.Pt; 73.50.Lw; 72.15.Jf

1. INTRODUCTION

In recent years, thin films of bismuth and antimony chalcogenides have attracted significant scientific and practical attention as thermoelectric materials [1,2]. These materials are distinguished by their high efficiency in heat-to-electric energy conversion, as well as by their stability and environmental friendliness [3]. A key indicator of thermoelectric efficiency is the figure of merit, ZT, and its enhancement requires a strategic approach that reduces thermal conductivity while maintaining electrical conductivity [4]. From this perspective, composite thin films of Bi_2Te_3 and Sb_2Te_3 are promising candidates [5].

The technological processes employed in thin-film synthesis directly determine the structural and morphological characteristics of the thin films [1,3]. The granular structure of the films, their step-like growth features, and porosity significantly influence the mechanisms of phonon propagation [2,4]. Consequently, these effects suppress thermal transport, thereby enabling enhanced thermoelectric efficiency. Furthermore, it has been established that surface morphology plays a crucial role in defining electron transport pathways, influencing recombination processes, and shaping the material's optical and electrical properties [5].

Vacuum technologies used in thin-film fabrication, particularly optimized thermal evaporation methods, enable the production of high-quality films under highly controlled conditions [3,4]. Processes carried out in vacuum systems eliminate external contaminants, thereby facilitating the formation of structurally pure materials. As a result, the synthesized films exhibit stable morphological and crystallographic properties, significantly enhancing their functional performance.

The aim of this work is to optimize a vacuum thermal evaporation system for the deposition of Bi_2Te_3–Sb_2Te_3 thin films and to analyze the influence of key technological parameters on the resulting film morphology.

To achieve this aim, the effects of vacuum level, evaporation conditions, and condensation processes on the surface morphology of the deposited films were systematically investigated. In this study, thin films based on Bi_2Te_3–Sb_2Te_3 were synthesized using a specially designed vacuum thermal evaporation system, and their morphological characteristics were comprehensively examined using atomic force microscopy (AFM) and scanning electron microscopy (SEM). The results confirmed that the films possess a dual-level morphological structure, which may significantly enhance thermoelectric efficiency.

2. EXPERIMENTAL SETUP AND DEPOSITION CONDITIONS

Thin films with a stoichiometric composition of 25% Bi_2Te_3 – 75% Sb_2Te_3 were deposited by the thermal evaporation method under vacuum using a UVN-71 P2 vacuum coating system. Prior to deposition, the vacuum chamber was evacuated to a base pressure of 1.33×10^{-3} Pa, ensuring minimized contamination during the film growth process.

The evaporation was carried out from a resistively heated crucible maintained at a temperature of 610 ± 10 °C, while the substrate temperature was fixed at 90 °C. The distance between the evaporation source and the substrate was optimized and set to 7 sm, providing uniform material flux across the substrate surface. The deposition duration was 8 minutes, and the total mass of the evaporated source material was 70 mg.

Polyethylene terephthalate (PET) substrates were used for thin-film deposition. The substrate thickness ranged from 50 to 125 μm, allowing sufficient mechanical stability during the evaporation process. As a result of the selected deposition parameters, uniform thin films with an average thickness of 0.790 μm were obtained.

The chosen vacuum level, evaporation temperature, source–substrate distance, and deposition time were optimized to ensure stable condensation conditions and reproducible film growth. These parameters played a crucial role in

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determining the morphological uniformity of the deposited Bi_2Te_3–Sb_2Te_3 thin films, which were subsequently analyzed using atomic force microscopy (AFM) and scanning electron microscopy (SEM).

3. DESCRIPTION OF THE OPTIMIZED THERMAL EVAPORATION VACUUM SYSTEM

Vacuum technology plays a key role in the fabrication of semiconductor devices, as certain technological processes require high-vacuum (low-pressure) conditions.

The technological equipment used for the production of semiconductor devices typically consists of two main parts:

1. The working chamber, which generally includes heaters, electrical contacts, evaporators for material deposition, substrates, thermocouples, and other components. This chamber (often referred to as the “bell jar”) can be raised or lowered by means of a hydraulic lifting mechanism.
2. The vacuum system, which evacuates air from the working chamber and comprises a rotary mechanical pump, valves, and pipelines (tubing that connects the pump to the chamber).

Even trace amounts of residual impurities inside the working chamber can contaminate the pure material being deposited. Such contamination can, in turn, adversely affect the electrical characteristics of the final device by introducing external or internal defects [6].

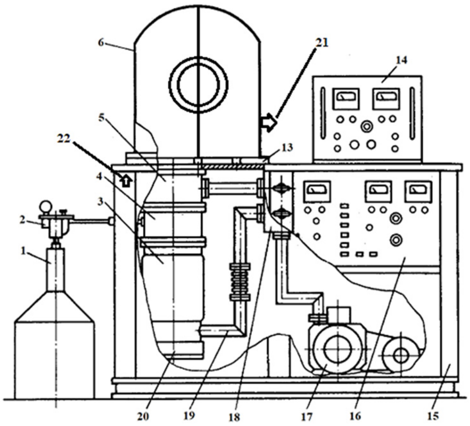

Figure 1. Schematic diagram of the main model of the UVN 71 P2 apparatus

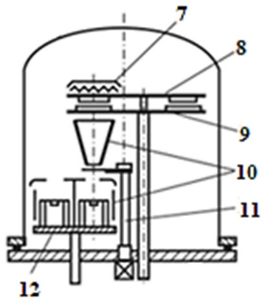

Figure 2. Internal technological configuration of the chamber

In this work, the UVN 71 P2 vacuum system is presented. It is designed for resistive coating (resistive thermal evaporation) to form thin films under vacuum conditions. The device consists of a vacuum generation system, a bell jar (chamber cover), and an electronic control unit. The vacuum generation system includes a forevacuum–diffusion pump that evacuates excess pressure and ensures system balance. The schematic elements of the vacuum coating system for thin films include the following: a thermally insulated dewar vessel for storing liquid nitrogen or helium (1), an automatic unit for supplying liquid nitrogen or helium in a continuous or controlled amount (2), a vapor–oil (diffusion) pump for achieving high vacuum (3), a condensation unit for vapors and gases using nitrogen or helium (4), a mechanical barrier that isolates the vacuum chamber from the external environment (5), the main chamber where the deposition process takes place (6), a heating system to raise substrates to the required temperature (7), a substrate rotation mechanism (8), a rotating mask holder used during the coating process (9), a shield (10), a baffle (11), a carousel with evaporators and individual evaporating sources (12), a rubber gasket ensuring chamber hermeticity (13), a vacuum gauge (vacuummeter) (14), a welded steel frame serving as the primary structural body of the apparatus (15), a control panel (16), a rotary mechanical pump used for initial vacuum generation (17), a valve block (18), tubing connecting the pump and the chamber (19), an electric heater for the vapor–oil (diffusion) pump (20), an air inlet valve or handle (21), a hydraulic switch used to raise and lower the bell jar cover (22), [7].

In such a system, the forevacuum pump is intended to evacuate air through an appropriate manifold until a pressure of approximately 1.33×10^{-1} Pa is achieved in the main chamber. Within the working chamber itself, the pressure typically ranges from (6.67×10^{-1} to 1.33) Pa. Simultaneously, the diffusion pump operates to maintain a steady high-vacuum level in the active volume of the system, with a pressure of approximately 1.33×10^{-4} Pa [8].

4. MORPHOLOGY OF BISMUTH AND ANTIMONY CHALCOGENIDE-BASED THIN FILMS OBTAINED USING A THERMAL EVAPORATION VACUUM SYSTEM

The thermoelectric performance of Bi_2Te_3–Sb_2Te_3-based thin films is commonly evaluated using the dimensionless figure of merit, ZT, defined as

$$ZT = \frac{S^2 \sigma T}{\kappa} \tag{1}$$

where S is the Seebeck coefficient, σ is the electrical conductivity, T is the absolute temperature, and κ is the total thermal conductivity. Since the thermal conductivity is strongly influenced by phonon scattering at grain boundaries and surface features, the morphology of thin films plays a crucial role in determining their thermoelectric efficiency. Therefore, analysis of surface morphology provides important insights into potential enhancement of ZT in Bi_2Te_3–Sb_2Te_3 thin films.

To investigate the surface topography of Bi_2Te_3–Sb_2Te_3 thin films, atomic force microscopy (AFM) was performed using a Solver-NEXT SPM 9700HT (Shimadzu).

The AFM image acquired over a 5.0 × 5.0 μm scan area reveals the surface morphology of the sample with nanometer-scale resolution (Figure 3).

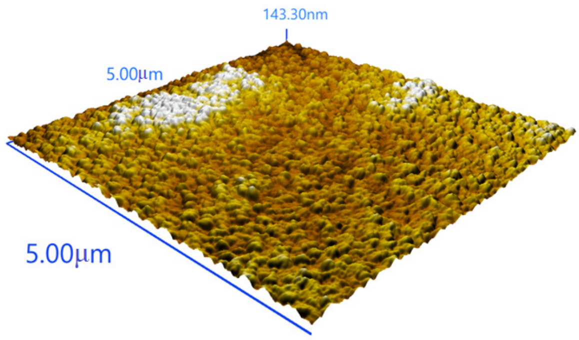

Figure 3. 3D topographic image of a Bi_2Te_3–Sb_2Te_3-based thin film obtained over a 5.00 × 5.00 μm scan area

According to the measurement results, the key surface parameters were determined as follows: average roughness (Ra) – 15.595 nm, root mean square roughness (Rq) – 20.091 nm, maximum peak height (Rp) – 79.030 nm, maximum valley depth (Rv) – 63.621 nm, total height difference (Rz) – 142.651 nm, and ISO-standardized ten-point height difference (Rzjis) – 70.534 nm. These values confirm that the sample's surface topography is well developed at the nanometer scale.

As observed in the AFM image, the surface is composed of grains with sizes distributed in the range of approximately 50–150 nm. Such a structure is attributed to the multidirectional growth of crystallites and irregularities arising during the nucleation process. The elevated Rz value indicates pronounced peaks and deep valleys in the surface topography. This morphological feature is typically observed in thin films synthesized by vacuum evaporation or chemical vapor-phase deposition methods (PVD, CVD).

The closeness of the Ra and Rq values (Ra = 15.6 nm, Rq = 20.1 nm) indicates that the statistical surface irregularities are of a random nature. Such surface morphology can significantly influence the material's functional properties. For instance, in photovoltaic elements, a nanostructured surface enhances light scattering, thereby improving absorption efficiency. In thermoelectric films, grain boundaries can limit phonon propagation, which reduces thermal conductivity and, consequently, enhances thermoelectric performance.

In addition, the significant difference between the Rp and Rv values indicates an uneven distribution of crystallites across the surface. This may directly affect the electrical properties of the material, particularly the charge carrier recombination rate. Recombination processes occurring at crystallite boundaries are among the key factors limiting the efficiency of devices fabricated on the basis of the film. Therefore, surface statistical roughness parameters are directly related not only to morphological characteristics but also to the electronic and optical properties of the material.

Overall, the AFM analysis results confirm the formation of a highly developed nanostructured surface morphology on the sample. Such a structure can potentially enhance the functional capabilities of the material; however, it may also give rise to certain adverse effects, such as an increased rate of recombination processes.

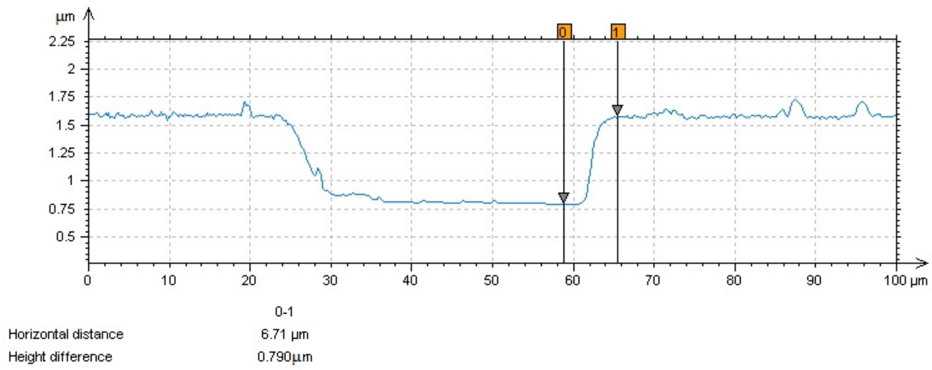

Figure 4. Longitudinal profile of the surface relief of the synthesized film obtained using Atomic Force Microscopy (AFM)

In the profile section presented in Figure 4, a sharp height variation was observed in the range of 60–70 μm. According to the measurement results, the horizontal distance was 6.71 μm, while the height difference amounted to 0.790 μm. These indicators confirm the formation of a step-like structure on the surface relief. Such a morphology typically arises due to the growth of crystallites in different directions and at varying rates. The development of a stepped morphology may lead to the distinct manifestation of grain boundaries and can promote anisotropic surface evolution.

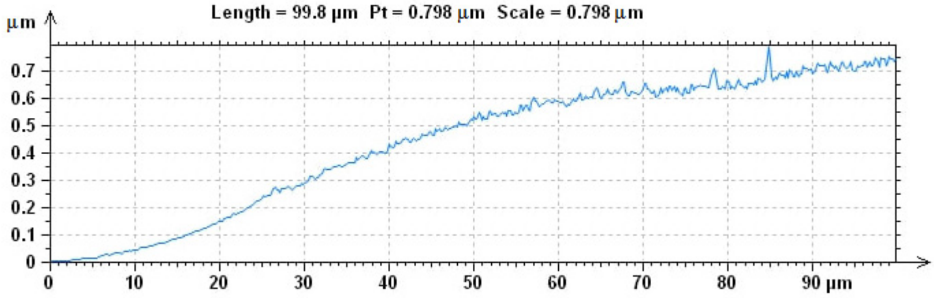

Figure 5. Longitudinal surface profile of the synthesized film obtained using AFM in the range of 0–99.8 μm

The longitudinal profile shown in Figure 5 demonstrates a gradual variation in surface topography over the range of 0–99.8 μm, with a total height difference of 0.798 μm. The overall shape of the profile reflects a global surface inclination, which may be attributed to the initial relief of the substrate or to a directionally controlled growth process during the deposition. Additionally, local oscillations observed within the 0.6–0.7 μm range indicate the presence of nanometer-scale granular morphology.

Based on the analysis, a dual-scale morphological structure was observed in the Bi_2Te_3–Sb_2Te_3-based thin film:

1. Step-like growth at the microscale – indicates the stepwise evolution of crystals and their anisotropic nature.
2. Granularity at the nanoscale – associated with statistical surface roughness as revealed by AFM images; this may enhance phonon scattering and thus potentially reduce thermal conductivity.

AFM (Atomic Force Microscopy) observations provided additional quantitative parameters that further elucidate the morphological characteristics of the material:

- Surface height variation: Maximum height differences in the range of 0.790–0.798 μm indicate the formation of a step-like relief during the film growth process.
- Statistical surface roughness: The calculated *Ra* (average roughness) values fall within the range of approximately 15–20 nm, which is attributed to the influence of nanoscale grains.
- Grain height: Local fluctuations indicate grain heights within the 50–150 nm range, which is further confirmed by high-resolution AFM imaging.
- Overall morphology: A complex relief structure is observed, resulting from the combination of microscale step-like growth and nanoscale granular morphology.

This dual-scale morphology is of strategic importance for thermoelectric materials. On the one hand, electron transport processes are largely preserved; on the other hand, phonon propagation is suppressed. This can reduce thermal conductivity and contribute to an increase in the material's thermoelectric efficiency, expressed by the figure of merit ZT. Moreover, the step-like surface structure may shorten electron scattering paths, potentially inducing anisotropy in electrical conductivity.

Therefore, at the subsequent stages, the obtained morphological parameters were compared with data obtained by other methods, including Scanning Electron Microscopy (SEM), in order to draw comprehensive conclusions.

The surface topography of the resulting Bi_2Te_3–Sb_2Te_3 thin film was examined using Scanning Electron Microscopy (SEM) with a JSM-IT200 system (JEOL).

The acquired images clearly revealed the complex surface morphology of the material, including its granular structure, pores, and surface defects.

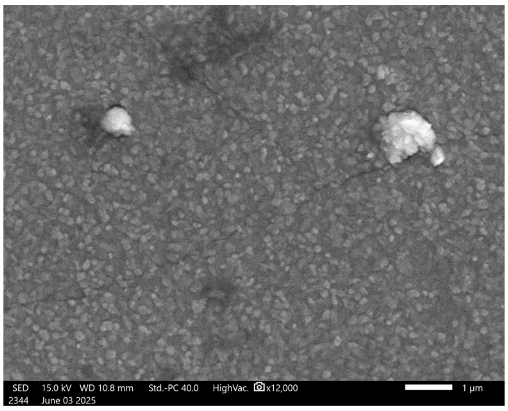

Figure 6. SEM image of the surface of synthesized Bi_2Te_3–Sb_2Te_3 thin films (magnification ×12,000)

In the SEM image presented in Figure 6 (magnification ×12,000, scale bar 1 μm), a clearly defined granular surface morphology is observed. The surface grains are uniformly distributed, and their sizes range from approximately 50 to 150 nm. In some regions, larger particles and surface protrusions can be seen. Such morphological features may result from the non-uniform growth of crystallites or the formation of localized agglomerations during the synthesis process. The high density of grain boundaries is expected to limit phonon transport paths, thereby reducing thermal conductivity. At the same time, its effect on electron transport may be relatively minor, which is considered a favorable factor for enhancing thermoelectric performance.

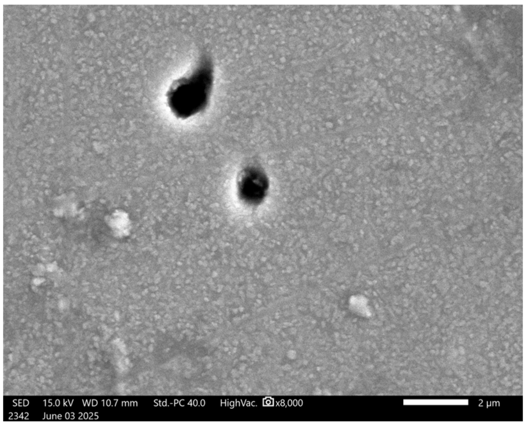

Figure 7. SEM image of the surface morphology of the synthesized Bi_2Te_3–Sb_2Te_3 thin films (magnification ×8,000)

Figure 7. The SEM image (magnification ×8,000, scale bar 2 μm) shows the formation of pores on the surface of the thin film. The pore diameters range from several hundred nanometers up to a few micrometers. The appearance of such porosity is typically associated with the evaporation rate during the synthesis process, the substrate temperature, or stoichiometric imbalances in the material composition. The presence of pore structures has a dual effect: on the one hand, it can enhance thermoelectric performance by reducing thermal conductivity; on the other hand, an excessive number of pores may compromise the mechanical stability of the material.

SEM observations revealed two predominant morphological features in the Bi_2Te_3–Sb_2Te_3-based thin films:

1. Nanometer-scale granularity — grains with sizes ranging from 50 to 150 nm are almost uniformly distributed across the entire surface. This promotes enhanced phonon scattering while preserving electron transport properties.
2. Micrometer-scale pores and coarse particles — some local defects formed during the film growth process may contribute to the reduction of thermal conductivity. However, if left uncontrolled, such defects could potentially compromise the mechanical integrity of the films.

These observations offer important implications for thermoelectric materials. Grain boundaries and pore structures can serve to reduce heat transport, thereby enhancing the thermoelectric figure of merit (ZT). At the same time, it is advisable to optimize synthesis parameters to preserve nanoscale granularity while minimizing the formation of large pores.

The SEM results confirm that the surface morphology of Bi_2Te_3–Sb_2Te_3-based thin films exhibits a nanogranular texture combined with a microporous structure. This complex topography plays a crucial role in improving thermoelectric performance.

5. CONCLUSIONS

The conducted studies demonstrated the formation of a dual-scale morphological structure in Bi_2Te_3–Sb_2Te_3-based thin films. Parameters identified through atomic force microscopy (AFM) confirmed the presence of nanoscale granularity on the film surface. The grain sizes ranged from 50 to 150 nm, and their presence is expected to enhance phonon scattering, thereby reducing thermal conductivity.

Longitudinal surface profile analyses revealed a step-like morphology with height variations of 0.790-0.798 μm. Scanning electron microscopy (SEM) revealed grains, coarse particles, and surface pores. Although the grains were distributed almost uniformly, local agglomerations and pores were also observed in certain regions.

Such a complex morphological structure, on the one hand, ensures efficient phonon scattering while preserving electron transport properties; on the other hand, it enhances thermoelectric performance by limiting thermal conductivity. However, excessive porosity may negatively affect the mechanical stability of the material.

In general, the coexistence of nanogranular and step-like relief observed in Bi_2Te_3–Sb_2Te_3-based thin films is of critical importance for thermoelectric materials. This morphology can be further optimized to improve functional properties by selecting appropriate synthesis conditions in future studies.

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ОПТИМІЗАЦІЯ ВАКУУМНОЇ ТЕРМІЧНОЇ ВИПАРУВАЛЬНОЇ СИСТЕМИ ДЛЯ ОСАДЖЕННЯ ТОНКИХ ПЛІВОК Bi–Sb–Te

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У цьому дослідженні тонкі плівки на основі халькогенідів вісмуту та сурми (Bi_2Te_3–Sb_2Te_3) були синтезовані за допомогою оптимізованої вакуумної системи термічного випаровування, а їхні морфологічні характеристики були ретельно досліджені. Результати атомно-силової мікроскопії (АСМ) виявили нанорозмірну зернистість на поверхні плівки (в діапазоні 50–150 нм) та чітку ступінчасту морфологію. Аналіз поздовжнього профілю показав, що варіації висоти знаходяться в діапазоні 0,790-0,798 мкм. Спостереження скануючої електронної мікроскопії (СЕМ) підтвердили утворення зерен, більших частинок та поверхневих пустот, що вказує на дворівневу морфологічну структуру. Така структура є критично важливою для підвищення термоелектричної ефективності шляхом посилення розсіювання фононів, тим самим зменшуючи теплопровідність, зберігаючи при цьому властивості електронного транспорту. Результати дослідження демонструють, що тонкі плівки на основі Bi_2Te_3–Sb_2Te_3 мають високий науковий та практичний потенціал як термоелектричні матеріали.

Ключові слова: *тонка плівка; морфологія; атомно-силова мікроскопія; електронна мікроскопія; термоелектрична ефективність; ногогранулярність; ступінчаста структура*