^{60}Co Gamma Irradiation and Annealing Effects on Transport Properties of Antimony Telluride Platelets Grown by Physical Vapor Deposition

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Abstract Physical vapor deposition method was employed to deposit antimony telluride (Sb_2Te_3) crystals in a dual-zone furnace. The microstructure, surface topography and composition of samples were characterized using X-ray diffraction, atomic force and scanning electron microscopy. Seebeck coefficient ($S \perp c$), electrical conductivity ($\sigma \perp c$) as well as power factor (PF) were enhanced for pure Sb_2Te_3 samples upon annealing, and the samples annealed at 473 K exhibited the highest PF of $3.16 \times 10^{-3} \text{ W m}^{-1} \text{ K}^{-2}$ with an enhancement of 22% in the figure of merit (Z). When the delivered dose of ^{60}Co gamma radiation was increased from 0 to 30 kGy in the stoichiometric crystals, $\sigma \perp c$ decreased due to the decrease in mobility. As a result of the increase in S , PF and Z improved by 12.11 and 13.7%, respectively, in the 30 kGy gamma-irradiated crystals. Both R_H ($B \parallel c$) and $S \perp c$ were positive, suggesting that the prepared Sb_2Te_3 crystals retained the p-type semiconductivity after these treatments.

KEY WORDS: Antimony telluride; Physical vapor deposition; Irradiation; Annealing; Conductivity; Seebeck coefficient

1 Introduction

Thermoelectric materials are promising in solid-state refrigeration and power generation devices that are designed efficiently to convert waste heat into electricity [1]. They need to be good electrical conductors and thermal insulators with high thermopower [2, 3]. However, the interrelated transport properties such as Seebeck coefficient, electrical conductivity and thermal conductivity make it difficult to improve one of them without affecting the others [4]. Antimony telluride is an important V–VI layer type semiconductor with tetradymite structure [5], and their alloys are well suited for realizing high figure of merit

(Z) due to their low band gaps, complex crystal structures and the presence of heavy elements [6, 7]. Crystals of Sb_2Te_3 have a very high concentration of holes because of the presence of large number of charged point defects. Traditionally, Sb_2Te_3 crystals were prepared by unidirectional crystal growth methods such as Bridgman technology [8], modified Bridgman method [9], zone melting [10] and Czochralski technique [11]. Although the resultant crystalline material exhibits reasonably good thermoelectric properties, the thermal stress-induced deformations and damages on the cleaved surfaces while cutting and polishing result with conventional bulk crystal growth processes. Physical vapor deposition (PVD) is an attractive technique since the composition and deposition rate can be adjusted by varying the zone temperatures. This method produces crystals with high crystallinity, improved physical and transport properties, where the as-grown platelets are suitable for application in microdevices. Irradiation with gamma rays causes structural defects leading to density change and modifies the physical properties of the

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materials through which they penetrate depending on the absorbed quantum of radiation [12]. Since defects can be created in thermoelectric devices when they are subjected to radiation environment, the influence of high-energy photons on the structure, chemical composition and the thermoelectric properties are very significant while working with these devices. Bahabri and Shoroog [13] proved that the degree of crystallinity increased in gamma-irradiated Bi_2Te_3 thin films. Abdel et al. [14] studied the physical properties of irradiated chalcogenides. According to Fang et al. [15], annealing has either a favorable or adverse effect on the improvement of conductivity and thermopower. Ibrahim et al. [7] investigated thermoelectric properties of annealed Sb_2Te_3 thin films as well as melt-quenched crystals. However, the effect of annealing and the research of ^{60}Co gamma-irradiated Sb_2Te_3 crystals prepared by PVD have not been reported so far. Therefore, the aim of this study is to investigate the effect of ^{60}Co gamma irradiation and annealing on the structure, composition and transport properties of Sb_2Te_3 crystals grown by PVD method.

2 Materials and Methods

Antimony telluride ingots were prepared by melting a mixture of antimony beads (size 1–2 mm, 99.999% purity) and tellurium granules (diameter 300 μm , 99.999% purity) procured from Sigma-Aldrich, India. The materials were powdered and sealed in an evacuated quartz tube after weighing out in appropriate atomic ratios (38.88 wt% antimony, 61.12 wt% tellurium). The raw materials were melted at 700 °C in a furnace for about 48 h and slowly cooled to room temperature. The synthesized polycrystalline ingot obtained was powdered and kept for growth in a dual-zone furnace, as described by Thankamma and Kunjomana [16]. From the obtained crystals, samples labeled 1–4 were subjected to gamma irradiation by a source of ^{60}Co having a rate of 3.0 kGy h^{-1} and 5–7 were annealed at 375, 475 and 575 K for 1 h in a vacuum chamber at a pressure of 1.33×10^{-3} Pa. The dose of gamma radiation ranged from 0 to 30 kGy, and the time of exposure varied from 1–6 h in order to attain the required magnitude.

The crystal structure and lattice parameters of irradiated and annealed samples were deduced from X-ray powder diffraction data collected using a BRUKER D8 diffractometer, operating at 40 kV and 50 mA employing nickel filtered CuK_α radiations ($\lambda = 1.5418 \text{ \AA}$). Surface morphology of the antimony telluride crystals was observed using a field-emission scanning electron microscope (FUJITSU DX 2100) operating at 20 kV. The energy-dispersive analysis by X-rays (EDAX) was performed to

calculate the ratio of constituent elements in the grown crystals. The atomic force microscope (NanoSurfAFM) was employed to investigate the topographical features of the samples. The Hall effect measurements were obtained at room temperature (300 K) by applying a magnetic field in the range $\pm 0.5 \text{ T}$ and an electrical current in the range $\pm 10 \text{ mA}$. The carrier concentration was calculated from the Hall coefficient assuming single carrier model. The Hall coefficient was evaluated using the formula

$$R_H = (V/(IB)) \times t, \quad (1)$$

where V is the Hall voltage, I is the current through the crystal, B is the magnetic field, and t is the sample thickness. The carrier concentration and mobility were calculated, applying the relations

$$p = (1/(R_H e)), \quad (2)$$

and

$$\mu = R_H \sigma, \quad (3)$$

where e is the electronic charge and ρ is the resistivity. The Seebeck coefficient S was calculated from the linear gradient $\Delta V/\Delta T$ of the measured thermoelectromotive force and temperature difference. The thermo-emf developed across the sample was measured using Keithley 195 digital multimeter, and the electrical conductivity (σ) measurements were carried out with van der Pauw technique where the heater was energized by a Keithley 228 current source. The power factor is given by the formula

$$\text{PF} = S^2 \sigma, \quad (4)$$

where σ is the conductivity in S/m.

Thermal conductivity (κ) and figure of merit (Z) were calculated by the formulae

$$\kappa = \alpha C_p \rho \text{ and } Z = S^2 \sigma / \kappa, \quad (5)$$

where α is thermal diffusivity, C_p is the specific heat capacity, and ρ is the density. To compute the values of C_p and α , differential scanning calorimetry (Perkin-Elmer YJ-STRC) and laser flash technique (Netzsch LFA-457) were used.

3 Results and Discussion

3.1 Composition, Morphology and Micro-structure

Figure 1 shows the grown platelets of dimensions $8 \text{ mm} \times 6 \text{ mm} \times 2 \text{ mm}$, when temperature difference $\Delta T = (T_S - T_G)$ was kept at 120 °C as described by Reshmi et al. [17]. The temperature of the source zone $T_S = 700 \text{ °C}$ and that of growth zone $T_G = 580 \text{ °C}$. T_G

Fig. 1 Antimony telluride platelets grown by PVD method

was set higher than the melting point of Te (449.5 °C) in order to avoid its excess deposition leading to tellurium-rich samples.

The results of elemental analysis on these crystals after the gamma ray exposure and annealing are given in Table 1. Figure 2 depicts the EDAX pattern of crystals annealed at 473 K, which exhibited the highest power factor with Sb/Te atomic ratio of 40.76:59.24. The atomic ratio Sb/Te of virgin samples was close to 2:3 and hence was stoichiometric, whereas a slight deficiency of tellurium was observed in samples irradiated for 30 kGy dose and those annealed at 473 and 573 K, which can be attributed to the evaporation of tellurium atoms. During exposure to high energy, the materials decompose and the final composition of the crystal changes due to the different vapor pressure of the elements. The tellurium evaporates faster than antimony, thereby showing difference in composition. Such variations in Sb_2Te_3 compounds due to tellurium evaporation were presented by Fan et al. [18].

Table 1 Elemental atomic ratio of the grown Sb_2Te_3 crystals

Sample no.	Gamma dose	Annealing temperature	Sb/Te ratio
1	15 kGy	–	40.19:59.81
2	20 kGy	–	40.19:59.81
3	25 kGy	–	40.25:59.75
4	30 kGy	–	40.79:59.21
5	–	373 K	40.39:59.61
6	–	473 K	40.76:59.24
7	–	573 K	40.98:59.02

Fig. 2 EDAX profile of Sb_2Te_3 crystals annealed at 473 K which yielded the highest power factor of $3.16 \times 10^{-3} \text{ W m}^{-1} \text{ K}^{-2}$

The SEM images shown in Fig. 3c and d correspond to the consequent developments due to irradiation dose of 25 and 30 kGy, which exhibit few defects, whereas in Fig. 3a and b no significant effects were noted in the surface features of the samples irradiated with 0–20 kGy. Depending upon the energy, gamma ray photons interact with matter in three different ways, i.e., photoelectric effect, Compton scattering and pair production. For ^{60}Co , Compton effect dominates where the momentum of incident photon is shared between the inelastically scattered photon and the ejected electron. The incident radiation has sufficient energy to knock out an atom from its position leading to modifications in crystal structure [19]. The surface of sample in Fig. 3e annealed at 373 K was found to have circular and oval-shaped dislocation loops. The condensation of excess vacancies is expected to form as loops in the cleavage planes of the $\text{Te}^1\text{--Te}^1$ layers in the antimony telluride sequence: $\dots\text{Te}^1, \text{Sb}, \text{Te}^2, \text{Sb--Te}^1\dots\text{Te}^1, \text{Sb}, \text{Te}^2, \text{Sb--Te}^1\dots$. Such loops have been observed on melt-quenched crystals of antimony telluride [6]. Figure 3f shows that the above-mentioned loops are annealed out at higher temperatures. The power XRD spectra of gamma-irradiated and annealed Sb_2Te_3 crystals are shown in Fig. 4a and b. The crystal structure is confirmed as rhombohedral, exhibiting D_{3d}^5 space group symmetry which is in good agreement with that in Ref. [20]. The intensity of the Sb_2Te_3 peaks (Fig. 4a) showed slight enhancement in intensity with an increase in annealing temperature, indicating that the degree of crystallinity of the crystals increased. The sharp and well-defined diffraction peaks as seen in Fig. 4a reveal the periodicity of the grown crystals analogous to the results reported by Fang et al. [15]. At higher temperature, recovery and recrystallization reduced the residual stress which improved the diffraction intensity of (015), (006) and (1010) peaks, respectively.

The lattice parameters of the 30 kGy irradiated sample (Fig. 4b) moderately increased ($a = b = 0.4262 \text{ nm}$, $c = 3.0353 \text{ nm}$) in comparison with the pure Sb_2Te_3

Fig. 3 SEM images of unirradiated Sb_2Te_3 crystals **a**; 20 kGy **b**; 25 kGy **c**; 30 kGy **d** gamma ray-irradiated Sb_2Te_3 crystals; Sb_2Te_3 crystals annealed at 373 K **e**; 473 K **f**

Fig. 4 Powder XRD patterns of as-deposited and annealed (473 K, 573 K) Sb_2Te_3 crystals **a**; Gamma-irradiated (0, 25, 30 kGy) Sb_2Te_3 crystals **b**

crystals ($a = b = 0.4257$ nm, $c = 3.0264$ nm). Thus, in addition to peak broadening, the positions of peaks show a slight shift to lower angles indicating lattice expansion after irradiation. These deviations are attributed to the uniform and non-uniform tensile strain experienced by the crystal during irradiation [21]. As a consequence of the strain created at right angles to the reflecting planes due to incident photons, the corresponding diffraction lines shift to lower angles and the interplanar spacing becomes greater than the initial value.

3.2 Effects of Annealing on Transport Properties and Power Factor

Measurements of the Hall mobility μ_H ($B \parallel c$), carrier concentration, Seebeck coefficient ($S \perp c$) and electrical conductivity ($\sigma \perp c$) are illustrated in Fig. 5a and b for various annealing temperatures.

It was observed that both R_H ($B \parallel c$) and ($S \perp c$) are positive indicating p-type conductivity, whereas the carrier concentration decreased and the carrier mobility increased on increasing the temperature. At the elevated annealing temperature, annihilation of some of the defects as well as vacancies improves the periodicity of crystal structure and the carriers drifting through the lattice undergo coherent scattering during atomic collisions allowing the charge carriers to drift without hindrances raising their mobility. In addition, the increase in grain size decreases the density of grain boundaries and enhances the mobility [22].

The intrinsic defects that occur in antimony telluride are called antisite defects which are characterized by antimony/tellurium entering the lattice by the replacement of tellurium/antimony atoms. These are specified by the notation Sb_{Te} , which means that an antimony atom is placed at a Te site, whereas in Te_{Sb} a tellurium atom is placed at an Sb site. Loss of tellurium reduces the Te_{Sb} native antisite defects which in turn decreases the carrier concentration. The compositional analysis as shown in Table 1 also

supports these observations, and such tellurium deficiencies were recorded by Fan et al. [18].

The electrical conductivity was enhanced due to two competing effects: the decrease in carrier concentration and an increase in carrier mobility. Carrier mobility (μ), conductivity (σ) and carrier concentration (p) are related by

$$\sigma = pe\mu. \quad (6)$$

Since μ increases at a rate higher than the decrease of p , σ increases with rise in annealing temperature.

In the present vapor-grown samples, the computed values of scattering parameter γ obtained from Fig. 5a are found to be fairly consistent, which showed negligible dependence in the density of the grain boundaries. In addition, the decreased carrier density n_c of annealed samples as evident in Fig. 5a contributed an enhance in the value of S in accordance with $S = \gamma - \ln n_c$ [23].

3.3 Effect of Gamma Irradiation on Transport Properties and Power Factor

Figure 6a and b reflect the transport coefficients of the gamma-irradiated samples, which exhibited p-type nature for all the doses.

For specimens 4 and 5 with radiations above 25 kGy, the mobility falls off rapidly and carrier concentration was slightly decreased. According to the irradiation theory developed by Arshak and Korostynska [24], when an incident energetic gamma radiation collides with a lattice atom in a solid material, the latter may receive sufficient energy to be displaced to an interstitial site, thus creating an interstitial–vacancy pair. Moreover, if the energy is large enough, subsequent collisions may produce secondary and tertiary displaced atoms. Consequently, a large number of lattice defects can result from the collision between gamma radiation and the lattice atom. In addition to vibrations of the lattice, any deviation from the perfect periodicity and imperfections in structure will scatter charge carriers due to

Fig. 5 Variations of mobility and carrier concentration **a**; Seebeck coefficient and conductivity **b**; power factor **c** with annealing temperature of Sb₂Te₃ crystals

Fig. 6 Dependences of mobility and carrier concentration **a**; Seebeck coefficient and conductivity **b**; power factor **c** on irradiation dose

the microscopic deformation of crystals reducing the mobility greatly [25]. According to the Frenkel defect energy level model of James and Lark-Horovitz [26], the redistribution of electrons from interstitials atoms over the available localized states leads to carrier scattering.

The surface roughness scattering arising from deviations of the lattice plays an important role in reducing carrier mobility at room temperature. In the case of irradiated thin Sb₂Te₃ platelets, enhanced carrier scattering may occur due to higher surface roughness which in turn limit the mobility of carriers. Figure 7 represents the AFM profile of the strained and unstrained (virgin) samples in which the rms value of roughness (R_{rms}) was found to increase from 0.952 to 0.987 nm in the irradiated crystals. Lattice strain (distortion) caused by the dislocations is clearly seen from the AFM image, and analysis of the pattern showed that the interplanar spacing d_{110} is increased from 2.1302 to 2.1548 Å, which indicates the presence of an expansive strain. For degenerate semiconductors, Seebeck coefficient is given by

$$S = \frac{8k_B^2 m^* T \pi^2}{3eh^2} \left(\frac{\pi}{3p} \right)^{2/3}, \quad (7)$$

where p is the carrier concentration and m^* is the effective mass of the carrier [27]. The deposited Sb₂Te₃ crystals of stoichiometric composition possess a conductivity of

40×10^4 S/m and Seebeck coefficient of 80 μV/K, which were slightly higher values than those reported for melt-grown crystals [20]. Upon irradiation, conductivity and carrier concentration decreased, whereas Seebeck coefficient showed an enhancement. Scattering limits the mobility considerably and the space charge region created by charged defects resulted in decrease of conductivity. Radiation-induced crystal defects produced by atomic displacements cause decreases in majority carrier density and carrier mobility [28].

3.4 Power Factor, Thermal Conductivity and Figure of Merit

Under a given temperature difference, the ability of a material to produce useful electrical power is quantified by its power factor (PF). The PF was calculated by the measured Seebeck coefficient and electrical conductivity (Figs. 5c and 6c). The as-deposited antimony telluride crystals have the value of $2.56 \times 10^{-3} \text{ W m}^{-1} \text{ K}^{-2}$ for PF. The largest value of power factor was obtained for the stoichiometric crystals annealed at 473 K ($3.16 \times 10^{-3} \text{ W m}^{-1} \text{ K}^{-2}$). Thus, after the annealing treatment, the PF was increased by 23.44%. At higher temperature ($T > 373$ K), annealing leads to lower resistivity in comparison with pure Sb₂Te₃ which was due to rapidly rising hole mobility. Significant increase in the latter

Fig. 7 AFM images **a** Distorted lattice, **b** virgin sample, **c** strained sample after irradiation with 30 kGy gamma ray dose

Fig. 8 Variations of thermal conductivity **a**; figure of merit **b** with annealing temperature

Fig. 9 Variations of thermal conductivity **a** and figure of merit **b** with gamma ray dose

can be associated with progressive recrystallisation, dislocation elimination as well as annealing out of defects. Upon irradiation by gamma rays, the samples tend to be imperfect and the creation of defects inhibits the movement of charge carriers causing their resistivity to be enhanced. The cumulative effect of decrease in conductivity and increase in Seebeck coefficient made the samples exhibits a hike of PF by 12.11%. According to Drude model,

$$\mu = e\tau/m^*, \quad (8)$$

where τ is the momentum relaxation time. Irradiation causes a marked reduction in carrier life time and relaxation time. In addition to strain changes, various scattering mechanisms affect the momentum relaxation time and effective mass contributing to decrease in carrier mobility. The anomalous lattice expansion due to the displacement of atoms and the formation of point defects can also be attributed to the diminishing mobility.

The total thermal conductivity κ_T and figure of merit Z of the samples are demonstrated in Figs. 8 and 9. The variations in κ_T can be attributed to the changes in

electrical conductivity σ as the total thermal conductivity of a semiconductor can be expressed as

$$\kappa_T = \kappa_L + \kappa_e, \quad (9)$$

where κ_L is the lattice and κ_e is the electronic thermal conductivity. The electronic component depends on the electrical conductivity according to Wiedemann–Franz ratio,

$$\kappa_e = L\sigma T, \quad (10)$$

where L is the Lorentz number and T is the absolute temperature. The figure of merit is enhanced by 22 and 13.7% after annealing and irradiation, respectively. Even though the higher temperature above 573 K may lead to deterioration of the starting composition, the annealing treatment was testified to be an efficient method over irradiation to improve the transport properties of Sb₂Te₃. In order to optimize the power factor, the treatment was performed for different time intervals. It was found that the annealing temperature is a key factor and time of annealing had an insignificant effect on power factor as well as figure of

merit. Thus, gamma ray irradiation and annealing have positive effect on the figure of merit of the PVD-deposited crystals.

4 Conclusions

In this work, stoichiometric platelets of antimony telluride (Sb_2Te_3) were grown by physical vapor deposition method (PVD). Gamma ray irradiations of samples were done by a source of ^{60}Co , and the annealing effect was studied at 373 K, 473 K and 573 K, respectively. The crystals exhibited good crystalline nature and corresponded to single phase, possessing rhombohedral D_{3d}^5 space group. SEM images indicated that increasing the radiation dose beyond 25 kGy led to the formation of defects. In the post-irradiated samples, various types of scattering reduced the carrier mobility and conductivity, whereas the Seebeck coefficient ($S_{\perp c}$) was improved by 10.62%, thereby enhancing the power factor by 12.11% and figure of merit by 13.7%. Upon annealing, the Seebeck coefficient ($S_{\perp c}$) and electrical conductivity ($\sigma_{\perp c}$) were increased and a 22% rise in the figure of merit was obtained at 473 K. Thus, compared with gamma ray irradiation, annealing has significant effect on the power factor and figure of merit of vapor-deposited Sb_2Te_3 crystals, which is beneficial for thermoelectric device applications.

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