Gamma Irradiation Effects in $\text{InBi}_{0.8}\text{Te}_{0.2}$ Crystals Grown by Horizontal Directional Freezing

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Abstract The high-energy gamma-ray irradiation treatment using Co-60 isotope offers the possibility of engineering mechanical and optoelectronic properties of $\text{InBi}_{0.8}\text{Te}_{0.2}$ crystals. Tellurium-doped indium bismuthide (InBi) crystals were prepared by horizontal directional freezing technique. Dose-dependent modifications in structure, composition and surface topographical features have been analyzed by X-ray powder diffraction, X-ray energy-dispersive analysis, transmission electron and atomic force microscopy, respectively. Dielectric constant and dielectric loss were found to increase with the cumulative dose of radiation, and a shift in the ferroelectric transition temperature (T_c) from 405 to 410 K was observed for 25 kGy. Upon irradiation, there is an enhancement in microhardness (H_V), yield stress (σ_y) and stiffness constant (C_{11}). The optical transmittance was decreased by 12.45%, resulting in a reduction in the optical band gap from 0.210 eV to 0.198 eV. These results indicate the suitability of $\text{InBi}_{0.8}\text{Sb}_{0.2}$ crystals for low-wavelength infrared applications.

KEY WORDS: Crystal growth; Dielectric properties; Optical properties; Hardness; Irradiation

1 Introduction

$\text{A}^{\text{III}}\text{B}^{\text{V}}$ compounds are a potential group of semiconductors having a direct band gap, enhanced carrier mobility and fine-tunable optical properties. They have a wide range of applications from simple light-emitting diodes [1] to high-speed optical, microwave communication devices and long-wavelength infrared detectors [2]. Unlike other III–V compounds, relatively less consideration has been given to InBi due to its semimetallic nature [3]. According to Binnie [4], InBi belongs to the space group P4/nmm with axial ratio $c/a = 0.995$ (lattice parameters, $c = 0.4773$ nm and $a = 0.5000$ nm). InBi has a low band gap (E_g) of -1.5 eV,

and varying the alloying percentage of InBi with InSb could change this value to 0.18 eV [5]. However, this approach poses difficulties due to the limitation of the solubility of InBi in InSb [6]. Shah et al. [7] have evaluated E_g as a function of concentration of Se in InBi single crystals. Therefore, an attempt has been made to tune the band gap of InBi doped with tellurium by irradiation so as to improve their semiconducting properties. In addition, defects created in crystals irradiated with gamma rays are of particular interest, as they influence the mechanical and optoelectronic properties of semiconductors. Applications of electronic devices and detectors require a well-established understanding about the radiation response. Investigations on the surface and bulk properties of electron-irradiated indium phosphide single crystals were carried out by Didyk et al. [8]. The process of formation of antistructural defects in tin-doped InP crystals exposed to gamma radiation has been studied by Rashidova [9]. Effect of γ irradiation on the parameters of localized states in the Sn-doped InSe single crystals was also reported [10]. However, to the best of our knowledge, significant data on

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mechanical, electrical and optical properties of gamma-ray-irradiated $\text{InBi}_{1-x}\text{Te}_x$ crystals have rarely been reported. One of the important properties of any device material is the mechanical strength represented by its hardness. Microhardness measurements can be used for investigating elastic, plastic and fracture properties of a crystal. The Vickers microhardness measurements on the annealed samples were found to be in the same range as those of cold-worked ones in the analysis of zone-melted (ZM) $\text{InBi}_{0.85}\text{Sb}_{0.15}$ crystals [11]. Though the increasing concentration of indium was found to enhance the hardness of $\text{In}_x\text{Bi}_{2-x}\text{Te}_3$, it was observed that the compound converts from p - to n -type with x in the range of 0–0.3 [12]. Jani et al. [13] have investigated the impurity hardening of tellurium-doped InBi crystals prepared by ZM where the truly constant hardness range could not be explored. Generally, InBi and its related compounds are prepared by crystal growth methods such as vertical Bridgman, Czochralski [3], syringe pulling [7] and zone-melting [13] techniques. But the grown ingots generally have the disadvantage of poor crystallinity and meager strength. In view of these considerations, directional freezing approach was pursued by the authors to grow tellurium-doped $\text{InBi}_{1-x}\text{Te}_x$ ($x = 0, 0.1$ and 0.2) crystals with improved structural perfection and mechanical properties. Among the various compositions, $\text{InBi}_{0.8}\text{Te}_{0.2}$ crystals irradiated with 25 kGy exhibited maximum dielectric constant, dielectric loss and electrical conductivity. Hence, the results obtained on these samples were discussed and presented in this work.

2 Experimental

$\text{InBi}_{1-x}\text{Te}_x$ crystals were grown in an indigenously developed directional freezing setup consisting of a two-zone heating furnace as described by Ajayakumar and Kunjomana [14]. The stoichiometric proportions of the elements indium, bismuth and tellurium, of 99.999% purity, were sealed in a quartz ampoule under a pressure of 10^{-4} Pa. The ampoule was placed in a muffle furnace and rocked continuously for mixing the constituents at a temperature 500 °C for 24 h. The ingot was cooled to room temperature, powdered and transferred to another quartz ampoule of dimensions 10 mm bore and 10 cm length for crystal growth. Systematic trials have been carried out to obtain good-quality stoichiometric crystals under a growth period of 48 h and temperature gradient of 6 °C/cm. The crystals were cleaved at liquid nitrogen temperature to minimize deformation and subjected to gamma irradiation process at Microtrol Sterilization Services Pvt. Ltd, Bangalore, using Co-60 radiation source. The samples were irradiated from 0 to 30 kGy at room temperature under identical conditions,

and the doses were accumulated by sequential exposure. The diffraction pattern of the grown crystals was studied using a Philips X'pert diffractometer with monochromatic CuK_α radiation ($\lambda = 0.15418$ nm) as the X-ray source. EDAX system attached to a scanning electron microscope (SEM, FUJITSU DX 2100) was used for elemental analysis. The transmission electron microscope (JOEL 200Cx) and the atomic force microscope (NanoSurfAFM) were employed to study the surface features of the samples at high resolution. The mechanical properties were evaluated by hardness testing using a MVH-1 tester fitted with Vickers diamond pyramidal indenter. Microhardness measurements were taken on freshly cleaved samples at room temperature using (5–100) g load and a dwell time of 10 s. The diagonals of the indented impression were observed using the microscope set at a magnification of $\times 400$, and the length of diagonals for various loads was measured using the micrometer eyepiece. The crystal size was much larger than the indentation size, which helped the elimination of the boundary effects.

The powdered material was mixed with KBr for pelletization to disk-shaped samples, and the optical transmission spectra were obtained using a Fourier transform infrared spectroscope (ThermoNicolet 200) in the wavenumber range, 400–4000 cm^{-1} . The samples were subjected to dielectric measurements using an impedance analyzer (AGILENT 4284A) in the frequency range 1 kHz–1 MHz. Crystals of area ~ 34 mm² and thickness 1.5 mm were used for this purpose. The opposite faces of the sample were coated with silver paste in order to obtain good ohmic contact.

3 Results and Discussion

3.1 Structural, Topographical and Compositional Analysis

Figure 1 shows the XRD profiles of pristine- and gamma-irradiated $\text{InBi}_{0.8}\text{Te}_{0.2}$ crystals, where the sharp peaks confirm good degree of crystallinity. The samples exhibited single-phase solid solution with a tetragonal structure having $P4/nmm$ space group. The post-irradiated XRD profiles for dose 25 kGy reveal slight peak shift and intensity changes. Generally, when the melt grown crystals are exposed to high-energy radiations, significant changes occur in the structural, electrical, mechanical and optical properties of the crystals. But in the directionally solidified $\text{InBi}_{0.8}\text{Te}_{0.2}$ crystals, the Co-60 irradiation could induce only point imperfections, thereby retaining the tetragonal structure. Hence, the lattice parameters, c/a ratio and volume of the unit cell were found to be fairly constant as shown in Table 1. However, the defects with strain fields

Fig. 1 X-ray diffractograms of pristine- and gamma-ray-irradiated $\text{InBi}_{0.8}\text{Te}_{0.2}$ crystals

will locally deform the lattice and affect the Bragg condition $2d\sin\theta = n\lambda$. As a result, the intensity of the diffracted beam was diminished for 25 kGy dose (Fig. 1) as explained by Bowen et al. [15]. The slight nonlinear variation in the value of lattice parameters can be attributed to the random distributions of point defects at high dosage. The fluctuations in the values listed in Table 1 are because of the irregular strain field created by the lattice distortion via the irradiation process.

The transmission electron microscopic images (Fig. 2a, b) illustrate the appearance of defects of the irradiated samples at 25 kGy. The primary interaction between energetic gamma radiation and semiconductors results in

the loss of energy of the electrons, creating electron–hole pairs. The sequence of energy-sharing collisions constitutes a displacement cascade, which could lead to structural modifications at high doses [16].

Upon irradiation, the production of defects and dislocations that caused alterations in the surface roughness and the rms roughness was found to increase from 0.834 to 0.838 nm in the AFM topography scans. The 3D profile consisting of hill and valley regions exhibited crystal structure with preferred orientations (Fig. 3).

Scanning electron microscopy (SEM) coupled with energy-dispersive X-ray analysis estimated the composition of indium, bismuth and tellurium in the pristine and irradiated crystals. Within the limits of experimental error, the analytical data on the elemental contents of these elements are found agreeable with their corresponding expected values of 37.33, 54.36 and 8.31 at% (In, Bi and Te) for all the samples. The EDAX spectrum corresponding to pristine- and 25 kGy-irradiated crystals is shown in Fig. 4.

3.2 Electrical Properties

The bonded electrons coupled with free charges in a material interact with external forces such as electric field, magnetic field and mechanical stress or temperature, producing dielectric properties. The information about electric field distribution and charge transport mechanism can be obtained by a study of dielectric response in the crystal.

The parameters connected with this process are the variation of real and imaginary dielectric constant and the dielectric loss ($\epsilon'' = \tan\delta$).

The complex dielectric constant (ϵ) of a material is expressed as

$$\epsilon = \epsilon_1 - i\epsilon_2, \quad (1)$$

where (ϵ_1) is the real part of ϵ due to polarization and (ϵ_2), the imaginary part representing the energy dissipation which is given by

$$\epsilon_2 = \epsilon_1 \tan\delta, \quad (2)$$

The frequency (1 kHz–1 MHz) dependence of the dielectric constant (ϵ_1) and loss (ϵ'') at 300 K was

Table 1 The lattice parameters of pristine and irradiated crystal

Dose (kGy)	$a = b$ (10^{-1} nm)	c (10^{-1} nm)	c/a	Volume (10^{-3} nm ³)	Crystal structure
0	4.9892	4.7823	0.9585	119.04	Tetragonal
10	4.9892	4.7823	0.9585	119.04	Tetragonal
15	4.9892	4.7824	0.9585	119.04	Tetragonal
20	4.9890	4.7825	0.9586	119.03	Tetragonal
25	4.9899	4.7820	0.9583	119.06	Tetragonal
30	4.9892	4.7824	0.9585	119.04	Tetragonal

Fig. 2 TEM micrographs exhibiting **a** strained region (direct magnification 68000 $\times$), **b** defects (direct magnification 4100 $\times$) in the gamma-ray-irradiated $\text{InBi}_{0.8}\text{Te}_{0.2}$ crystals

Fig. 3 AFM images of 2D profile **a** of scan dimensions 1 $\mu\text{m} \times 1 \mu\text{m}$ demonstrating strained regions in the crystal lattice and 3D profile **b** (2.54 nm $\times$ 2.54 nm $\times$ 1.95 nm) exhibiting growth hillocks

investigated for pristine- and gamma-irradiated $\text{InBi}_{0.8}\text{Te}_{0.2}$ samples.

The dielectric constant (ϵ_1) was calculated using the formula

$$\epsilon_1 = Ct/(\epsilon_0 A), \quad (3)$$

where C is the capacitance (F), t the thickness (m), A the cross-sectional area (m^2) of the sample and ϵ_0 is the permittivity of free space having a value of 8.854×10^{-12} F/m. It was observed that ϵ_1 and ϵ'' increase with increasing

concentration of tellurium and gamma doses (0–25 kGy), and for doses above 25 kGy there was a decrease in the dielectric parameters for all the samples.

The rise in dielectric constant and dielectric loss with the cumulative dose of radiation (Fig. 5a, b) can be attributed to the various defects in the lattice as illustrated in transmission electron microscopic images. With the increase in irradiation dose up to 25 kGy, polarization due to space-charge region created by ionization tends to rise the dielectric constant and loss. As reported by Kaur et al.

Fig. 4 SEM-EDAX spectra of pristine **a** and 25 kGy **b** irradiated $\text{InBi}_{0.8}\text{Te}_{0.2}$ crystals

Fig. 5 Plots of frequency versus dielectric constant **a** and frequency versus dielectric loss **b** for $\text{InBi}_{0.8}\text{Te}_{0.2}$ crystals irradiated with gamma radiation

[17], the redistribution and formation of stable defects of at higher gamma doses prevent the movement of ions as well as the effect of polarization, thereby decreasing ϵ_1 and ϵ'' above 25 kGy dose. Dielectric constant decreased with increasing frequency because the dipoles cannot reorient themselves fast enough with the applied field.

Irradiation develops disorder and distortions in the lattice, contributing to an increase in the space-charge polarization. When the field frequency is very small, the electric dipoles orient in the field direction, leading to the manifestation of high values of ϵ_1 and ϵ'' . At high frequency, the dipoles take more time to align in the direction of the field decreasing orientational polarization which in turn reduces the values of the dielectric constant and dielectric loss [18]. Frequency response of dielectric loss also exhibited a similar trend as illustrated in Fig. 5b.

Figure 6a, b presents the temperature dependence of the dielectric constant and loss of 25 kGy gamma-irradiated $\text{InBi}_{0.8}\text{Te}_{0.2}$ crystals at various frequencies in the

investigated range (300–423 K). High-energy photons traversing through the material create electron–hole pairs which build up electrode polarization at interfaces within the sample contributing to the increase in ϵ_1 and ϵ'' [20]. Figure 6a shows the characteristic feature of a ferroelectric phase, and the transition temperature T_c was found to increase from 405 to 410 K in the irradiated sample. The shift of T_c could be related to strain and internal bias field, caused by radiation-induced charges trapped in grain boundaries [19].

The AC conductivity (σ_{ac}) exhibits nearly invariant behavior at low frequency (Fig. 7), and in the region of high frequency, the conductivity decreases in accordance with the variations of ϵ_1 and $\tan \delta$ as per the relation

$$\sigma_{ac} = \omega \epsilon_1 \epsilon_0 \tan \delta, \quad (4)$$

where ω is the angular frequency of the applied field.

The electrical resistivity (ρ) of the crystals is sensitive to energy of ionizing radiation and the applied dose level. For the cumulative doses of 25 kGy, ρ decreased from

Fig. 6 Plots of temperature versus dielectric constant **a** and temperature versus dielectric loss **b** of $\text{InBi}_{0.8}\text{Te}_{0.2}$ crystals subjected to 25 kGy gamma-ray dose for various frequencies

Fig. 7 Variations of AC conductivity with temperature for 25 kGy gamma-ray-irradiated $\text{InBi}_{0.8}\text{Te}_{0.8}$ crystals at various frequencies

4.5×10^{-4} to $3.94 \times 10^{-4} \Omega \text{ cm}$ on account of an increase in carrier density due to electron-hole pairs created by irradiation.

3.3 Mechanical Properties

Hardness is the property of a material that enables it to resist plastic deformation by an indenter of well-defined geometry and resistance against lattice destruction. The Vickers hardness (H_V) is the ratio of the load applied to the surface area of the indentation which is given by

$$H_V = 2P \sin(\theta/2)/d^2, \quad (5)$$

where H_V is the Vickers microhardness in kg/mm^2 , P is the applied load (kg), d is the mean diagonal of the indentation (mm), and θ is the angle between opposite faces of the diagonal indenter (136°). For statistical reliability,

Fig. 8 Variations of microhardness (H_V) with the applied load (P)

systematic trials have been carried out for each load and the average hardness values were obtained as a function of the applied load within an experimental error of $\pm 2\%$. Figure 8 illustrates the variation of microhardness with an applied load for pure and gamma-irradiated samples.

The Vickers microhardness number (H_V) was found to increase with indenting load and saturates at higher loads. Dislocations are created locally in the region of indentation, and the lattice experiences increased frictional stress to the free movement of the defects which resulted in an increase in hardness [20]. Lattice strains developed in crystals when irradiated with high-energy photons result in the formation of various types of imperfections and defects which leads to an increase in H_V [21]. The rise in hardness with the radiation dose can be attributed to work hardening on account of an enhancement in the dislocation

interactions. The load dependence of H_V can be explained quantitatively using Kick's [22] law for pyramidal indenter.

$$P = ad^n, \quad (6)$$

where P is the applied indentation load, a is the material constant, d is the average diagonal length of the indented impressions, and n is the strain hardening index. The value of n obtained from the plot of $\log P$ versus $\log d$ was found to be >2 , indicating a reverse indentation size effect (ISE), where H_V increases with increasing load [23].

Yield stress of a material is defined as the stress applied to the material at which permanent or plastic deformation starts to occur, while the material is loaded. Yield stress was evaluated using the equation [24]

$$\sigma_y = \left(\frac{H_V}{2.9}\right) [1 - (n - 2)] \left\{ \frac{12.5(n - 2)}{[1 - (n - 2)]} \right\}^{n-2}. \quad (7)$$

The first-order elastic stiffness constant (C_{11}) was computed for the crystals using Wooster's empirical relation [25]

$$\log C_{11} = \frac{7}{4} \log H_V, \quad (8)$$

The calculated values of H_V , n , σ_y and C_{11} are listed in Table 2. The microhardness obtained in the present study is found to be higher than the values grown by zone-melting

Table 2 Hardness parameters

Sample	H_V (kg/mm ²)	n	σ_y (kg/mm ²)	C_{11} (kg/mm ²)
InBi _{0.8} Te _{0.2} (pure)	15.4	2.47	9.322	119.7
InBi _{0.8} Te _{0.2} (irradiated)	16.8	2.62	14.25	141.2

method [11]. As revealed in TEM analysis, the point defects created in the lattice resist the deformation of the crystal surface due to indentation which enhances the microhardness. The values of a and n can be found out from the plot of $\log P$ versus $\log d$ (Fig. 9). The slope of this straight line gives the value of constant n , and the intercept on $\log P$ axis gives the value of $\log a$.

3.4 Optical Studies

Optical transmission measurement was used to evaluate the band gap of the pure and irradiated crystals. The transmittance (T) was used to compute the absorption coefficient (α) according to the relation

$$\alpha = 2.3036 \log(1/T)/t, \quad (9)$$

where t is the thickness of the sample.

Incident photon energy $h\gamma$, the optical energy gap E_g and α are connected by the quadratic Tauc's equation

$$\alpha(h\gamma) = B(h\gamma - E_g)^n/h\gamma, \quad (10)$$

B is a constant that depends on electronic transition probability and the exponent n , on the type of electronic transition ($n = 2$ for indirect and $n = 0.5$ for direct). By taking an intercept at $(\alpha h\gamma)^2 = 0$, the values of E_g have been evaluated from the variation curves of $(\alpha h\gamma)^2$ with $h\gamma$ (Fig. 10).

From the transmittance spectra (Fig. 11) obtained for the pristine- and gamma-irradiated crystals, it was observed that the average transmittance (T) decreased by about 12.45% for the irradiation dose of 25 kGy.

A slight decrease in the value of band gap from 0.210 to 0.198 eV was observed due to irradiation. An optical band gap of 0.232 eV has been reported for the InBi_{0.85}Se_{0.15}

Fig. 9 Plots of $\log P$ versus $\log d$ of pure InBi_{0.8}Te_{0.2} crystals **a** and InBi_{0.8}Te_{0.2} crystals irradiated with 25 kGy gamma-ray dose **b**

Fig. 10 Plots of $(\alpha h\nu)^2$ versus photon energy ($h\nu$) of the as-grown and irradiated $\text{InBi}_{0.8}\text{Te}_{0.2}$ crystals

Fig. 11 Transmittance spectra of pristine- and gamma-ray-irradiated $\text{InBi}_{0.8}\text{Te}_{0.2}$ crystals

crystals grown by syringe pulling method [7]. The decrease in the optical band gap can be understood on the basis of density of state model proposed by Mott et al. [26], where defects created on account of irradiation are responsible for the presence of localized states and modification in the band gap.

4 Conclusions

The effects of Co-60 gamma-ray exposure (0–30 kGy) on the structure, composition, dielectric parameters, microhardness and optical transmittance of $\text{InBi}_{0.8}\text{Te}_{0.2}$ crystals prepared by directional freezing were investigated in the present work. The X-ray powder diffraction pattern revealed that the crystals exhibited good crystalline nature

and correspond to single phase, possessing tetragonal structure with space group $P4/nmm$. EDAX analysis confirmed the stoichiometric and homogeneous composition of the grown crystals. The transmission electron microscopic images showed the evidence of strained lattice in the irradiated samples. Upon irradiation, the RMS roughness increased from 0.834 to 0.838 nm according to the AFM topography scan. There was a rise in dielectric constant and dielectric loss with the accumulated gamma dose and the Curie temperature was elevated from 405 to 410 K. The mechanical parameters are improved after irradiation treatment, and the values of microhardness (H_v), yield stress (σ_y) and stiffness constant (C_{11}) obtained are 16.8, 14.25 and 141.2 kg/mm², respectively. The average transmittance (T) decreased by 12.45% for the irradiation dose of 25 kGy leading to a reduction in band gap from 0.210 to 0.198 eV. The inferences from these investigations suggest the possibility of the use of $\text{InBi}_{0.8}\text{Sb}_{0.2}$ crystals for low-wavelength infrared applications.

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