Chapter - 5

Characterization of grown $\text{Bi}_2\text{S}_3$ crystals
5.1 Introduction

The field of science of crystals widely comprise of two fundamental processes namely “The crystal growth process” and “Characterization of grown crystals.” The crystal growth processes related to present research work have been represented in chapter 2. The fundamental of Characterization is going to discuss in this chapter.

Characterization refers to the broad and general process by which a materials structure and properties are probed and measured. It is a primary process in the materials science field, without which no scientific understanding of engineering materials could be ascertained. Nowadays, for acquiring quantitative and qualitative information about the composition and structure of matter, scientists and engineers have an impressive array of powerful and elegant tools. The scope of the term often differs; some definitions limit the term's use to techniques which study the microscopic structure and properties of materials, while others use the term to refer to any materials analysis process including macroscopic techniques such as mechanical testing, thermal analysis and density calculation.

While many characterization techniques have been practiced for centuries, such as basic optical microscopy, new techniques and methodologies are constantly emerging. Characterization essentially includes evaluation of the chemical composition, structure, optical, mechanical, electrical and thermal properties of the grown crystals. During growth of a single crystal, the information occurred on the process that is provided by the post growth analysis. It is always important to characterize these crystals with various angles of interests by different instruments Different types of characterization techniques are available to study the physical and chemical properties of the crystal which includes some important and complex sophisticated equipment’s. Some of them are listed below.
5.1.1 Microscopy:

Microscopy is a characterization technique which probe and map the surface and sub-surface structure of a material. These techniques can use photons, electrons, ions or physical cantilever probes to gather data about a sample's structure on a range of length scales. Some common examples of microscopy instruments include:

- Optical Microscope
- Scanning Electron Microscope (SEM)
- Transmission Electron Microscope (TEM)
- Scanning Tunneling Microscope (STM)
- Scanning probe microscopy (SPM)
- X-ray diffraction topography (XRT)

5.1.2 Spectroscopy

This group of techniques is utilized to reveal the chemical composition, composition variation, crystal structure and photoelectric properties of materials. Some common instruments include:

**Optical radiation**

- Ultraviolet-visible spectroscopy (UV-vis)
- Fourier transform infrared spectroscopy (FTIR)
- Photoluminescence (PL)

**X-ray**

- X-ray diffraction (XRD)
- Energy-dispersive X-ray spectroscopy (EDX, EDS)
- Wavelength dispersive X-ray spectroscopy (WDX, WDS)
- Electron energy loss spectroscopy (EELS)
- Auger electron spectroscopy (AES)
- X-ray Photon Correlation Spectroscopy (XPCS)
5.1.3 Macroscopic testing

Large ranges of techniques are used to characterize different macroscopic properties of materials, including:

- Mechanical testing, including tensile, compressive, torsional, creep, fatigue, toughness and hardness testing
- Differential thermal analysis (DTA)
- Thermo gravimetric analysis (TGA)
- Differential scanning calorimetry (DSC)

The methods used for characterization in this study are as follows:

1. X-ray diffraction (XRD)
2. Scanning Electron Microscope (SEM)
5.2 X-ray Diffraction (XRD)

5.2.1 Introduction

X-ray powder diffraction (XRD) is a rapid analytical technique primarily utilized for the analysis of both molecular and crystal structures, qualitative identification of various compounds, quantitative resolution of chemical species, measuring the degree of crystallinity, isomorphous substitutions, stacking faults, polymorphisms, phase transitions, particle sizes etc. The analyzed material is finely ground, homogenized, and average bulk composition is determined. XRD is an appropriate method to examine whether a resultant material has amorphous or crystalline nature. When X-rays light reflects on any crystal, it leads to form many diffraction patterns and the patterns reflect the physio-chemical characteristics of the crystal structures.

In powder specimen, diffracted beams are typically come from the sample that reflects structural physio-chemical features. Thus XRD technique can analyze structural features with other ambiguities of a wide range of materials such as on organic catalysts, superconductors, biomolecules, glasses, polymers and so on. Crystalline phases can be identified by just comparing the interplanar distance ‘d’ values obtained from XRD data with the fundamental data in Joint Committee on Powder Diffraction Standards (JCPDS). There is large number of X-ray methods for the study of the crystals. But the most useful and powerful techniques are interferometry and diffractometry. One can obtain information such as the deformation of the crystals, electronic energy density in terms of a function of width, with the application of these methods.

Since the performance of the first X-ray diffraction experiments on a single crystal in 1912, X-ray crystallography has been of major importance in natural sciences and especially in mineralogy. X-ray diffraction provided the ideal means to understand structures of minerals on an atomic scale. It thus established relationships between the crystal structure and the physical and chemical properties of the material under investigation. In other cases it related the crystal structure to the special thermo dynamical conditions under which a mineral has been formed and thus provided important information for petrology and geology.
5.2.2 Principle

In 1912, Max von Laue, discovered that crystalline substances act as three-dimensional diffraction gratings for X-ray wavelengths similar to the spacing of planes in a crystal lattice. X-ray diffraction is now a familiar technique for the study of crystal structures and atomic spacing.

X-ray diffraction is based on constructive interference of monochromatic X-rays and a crystalline sample. Monochromatic X-ray beams focused on sample material in XRD, to resolve structural information in the crystal lattice. Generally, the materials are composed of repeating uniform atomic planes which make up their crystal. The polychromatic X-rays are produced in a cathode ray tube. Filtering polychromatic X-rays through a monochromatic produces monochromatic radiation which hits onto the material atomic planes, separating the diffracted, transmitted and absorbed rays. X-rays are produced within a closed tube under vacuum atmosphere.

The most prevalent type of diffraction to X-ray crystallography is known as Bragg diffraction, which is defined as the scattering of waves from a crystalline structure. The incident X-ray radiation produces a Bragg peak if their reflections from the various planes interfered constructively. The interference is constructive, when the phase shift is a multiple of $2\lambda$, this condition can be expressed by Bragg's law,

$$n\lambda = 2d \sin \theta$$  \hspace{1cm} [1]

Where $n$ is a numeric constant known as the order of the diffracted beam, $\lambda$ is the wavelength of the beam, $d$ denotes the distance between lattice planes, and $\theta$ represents the angle of the diffracted wave.
Figure 5.1 Bragg's law

From the XRD patterns, the 2θ values obtained from the position of the Bragg peaks are used to obtain the interplanar spacing expressed in terms of Miller indices (hkl), are substituted into the above equation (1) and a number of relations which are useful for indexing and calculating the lattice parameter are obtained. By substituting the value of lattice parameter (a) and (hkl), the distance between the planes (d) may be found in the following equations:

1. Cubic system : 
   \[ d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \]

2. Orthorhombic system : 
   \[ d_{hkl} = \frac{1}{\sqrt{\frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}}} \]

3. Tetragonal system : 
   \[ d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2\left(\frac{a}{c}\right)^2}} \]
For the monoclinic and triclinic systems are most typical of a number of polymers, the corresponding formulae have more complex form. By a quantum detector (a Geiger counter or a scintillation counter) or by a photographic technique, the intensity and direction of X-rays that have been diffracted by a crystal are recorded. Various methods are used in order to elucidate the structure of crystals, which include Laue’s method, the rotating crystal method and the Debye Scherrer method (the powder technique).

5.2.3 Instrumentation

The instrument is called an X-ray diffractometer. An X-ray beam of a single wavelength is used to examine the specimens, in the diffractometer. A spectrum of diffraction intensity versus the angle between incident and diffraction beam is recorded, by continuously changing the incident angle of the X-ray beam.

The main components of diffractometer are

- **X-ray Tube**: The source of X Rays.
- **Incident-beam optics**: To condition the X-ray beam before it hits the sample
- **Goniometer**: The platform that holds and moves the sample, optics, detector, and/or tube
- **Sample holder**
- **Receiving-side optics**: To condition the X-ray beam after it has encountered the sample
- **Detector**: To count the number of X Rays scattered by the sample

The basic components of the diffractometer are shown in Fig 5.2. The ‘θ' is the angle between the X-ray source and the sample, where 2θ is the angle between the incident beam and the detector. The incident angle θ is always half of the detector angle 2θ. The basic function of a diffractometer is to detect X-ray diffraction from materials and to record the diffraction intensity as a function of the diffraction angle (2θ). The X-ray radiation generated by an X-ray tube passes through Soller slits which collimate the X-ray beam. The X-ray beam passing through the slits strikes the specimen. X-rays are diffracted by the specimen and form a convergent beam at the receiving slits before they enter a detector.
Figure 5.2 Basic components of X-ray diffractometers

The diffracted X-ray beam passes through a monochromatic filter to suppress wavelengths other than $K_a$ radiation and decrease any background radiation, before being received by the detector. The $K_a$ radiation is generated by bombarding of target surface (Cu, Fe, Cr) by accelerated electrons. Most commonly a copper target is used generating $K_a$ wavelength of 0.154 nm. Relative movements among the X-ray tube, specimen and the detector ensure the recording of diffraction intensity in a range of $2\theta$.

5.2.4 Application

X-ray powder diffraction is most broadly used for the identification of unknown crystalline materials (e.g. minerals, inorganic compounds). The major applications of XRD are discussed below.

- XRD has widespread applications in the fields of geology, materials, polymers, environmental science, chemistry, forensic science, and the pharmaceutical industry, etc.
- This method has been conventionally used for phase identification, quantitative analysis and the determination of structure imperfections.
- It has long been used in immigration for detecting and identifying censored drugs, materials and coins.
- Various types crystalline materials can be characterized by powder XRD, including organic and inorganic materials, minerals, drugs, zeolites, catalysts, metals and ceramics.
• In addition, it finds out materials identity, crystallinity, residual stress and textural features with minimum invasion.
• By properly using this technique one can yield a great deal of structural information about the material under investigation.
• One of the most important uses of the powder method is in the identification of an unknown material.
• For most applications, the amount of information which is possible to extract depends on the nature of the sample microstructure (crystallinity, structure imperfections, crystallite size and texture), the complexity of the crystal structure (number of atoms in the asymmetric unit cell and unit cell volume) and the quality of the experimental data (instrument performances and counting statistics).
• Statistical study of the relative orientations of the individual crystals of an aggregate is one of the important secondary uses of the powder method.
• Characterize thin films samples by determining lattice mismatch between film and substrate and to inferring stress and strain.
• Used in determining dislocation density and quality of the film by rocking curve measurements and measuring super lattices in multilayered epitaxial structures.
• Utilized in determining the thickness, roughness and density of the film using glancing incidence X-ray reflectivity measurements and make textural measurements, such as the orientation of grains, in a polycrystalline sample.

5.2.5 Advantages of XRD

• It is powerful and rapid (< 20 min) technique for identification of an unknown mineral.
• It is a non-destructive nature, high sensitivity and reliable.
• In most cases, it provides an unambiguous mineral determination.
• It has ability to analyze mixed phases.
• For the purpose of simultaneous and quick measurement of the positions and intensities of diffraction lines the diffractometers are advantageous.
• Operational procedure is convenient and fast speed.
• XRD units are widely available.
• Effective resolution, low maintenance cost and proper automation.
- Data interpretation is relatively straightforward.
- System is user friendly, depth profiling (glancing incident angle) and easy sample preparation.
- Easy data interpretation that could be used for both qualitative and quantitative analysis, in wide range of applications.

5.2.6 Limitations of XRD

- It has size limitations.
- Must have access to a standard reference file of inorganic compounds (d-spacings, hkl).
- It is much more accurate for measuring large crystalline structures rather than small ones.
- Requires tenths of a gram of material which must be ground into a powder.
- For mixed materials, detection limit is ~ 2% of sample.
- X-rays do not interact very strongly with lighter elements.
- For unit cell determinations, indexing of patterns for non-isometric crystal systems is complicated.
- Small structures that are present only in trace amounts will often go undetected by XRD readings, which can result in skewed results.
5.2.7 XRD analysis of grown crystals of Bi₂S₃

X-ray diffractogram of the grown crystals of Bi₂S₃ by Zone Refining as well as Gel method was recorded at SVNIT Surat with the help of Rigaku made Miniflex(1.5405 Å) with step size of 0.02°. Modular X-Ray Diffractometer (XRD) interfaced with computer system as shown in figure 5.3.

Figure 5.3 Photograph of Miniflex (50-60 Hz.) Diffractometer.

Technical specification XRD instrument used

- **Item** : Miniflex
- **X-Ray Power** : 450 W (With 1kW tube as standard)
- **Detector** : Small scintillation counter
- **Software** : peak search, qualitative, quantitative (calibration curve method, MF method), Integrated intensity calculation (Peak position, width, etc..), rock mineral software, etc
- **Goniometer operation mode** : Vertica
- **Goniometer radius** : 150 mm
- **Optical system** : Focusing method
- **Weight** : 66 kg
- **Slit** : DS : Variable Slit RS : 0.3 mm fixed
- **Dimensions** : 560 W × 244 D × 582 H mm
- **Cooling water** : small water circulating pump for dedicated use etc.
- **Power supply** : 220 V or 115 V/ 15 A
Results

The samples were finely powdered and exposed to X-ray beam of wavelength 1.5405 Å from CuKα sources of above mentioned diffractometer assembly. The observation of the intensity of diffracted beam was measured at various 2θ angles ranging from 10 ° to 80 °, where θ is the angle made by the incident beam with crystal surface.

From experiment the obtained data were recorded in Excel and raw format was processed using computer software (POWDER X/ High score Plus). The dominant peaks are indexed using standard JCPDS card number (00-006-0333) and (00-017-0320) of Bismuth Sulphide (Bi₂S₃). The lattice parameters were calculated using the data and compared with experimental values.

The diffractograms obtained from the experiments for the grown crystal for entire range of observations are presented in Fig. 5.4 (a) & (b) and 5.5 (a) & (b). The XRD pattern of the Bi₂S₃ indicated the formation of orthorhombic phase Bi₂S₃ (JCPDS No (00-006-0333) and (00-017-0320)). No other crystalline phases were detected in the product. Some selected prominent peaks for each sample are indexed using JCPDS card of Bismuth sulfide are shown in Fig. 5.4 (b) and Fig. 5.5 (b). The values of 2θ, d- spacing, and relative intensity corresponding to these prominent peaks are presented in tabular form in tables 5.1 (a) & (b) and 5.2 (a) and (b). The experimental values of these parameters are compared with standard values. Result indicates that XRD pattern and lattice parameters calculated are in good agreement with standard data.
Figure 5.4 (a) XRD graph of grown crystal of Bi$_2$S$_3$ by Gel method

Figure 5.4 (b) XRD graph of grown crystal of Bi$_2$S$_3$ by Gel method (Indexed)
For gel grown Bi$_2$S$_3$ crystals, the Powder diffraction data is shown in table 5.1 (a) & (b). The observed values are very well match with JCPDS card (06 - 0333) of Bi$_2$S$_3$. Observed peaks in diffractogram indicites Bismuth-Sulphide crystals passes Rhombus or Orthorhombic structure.

### Table 5.1 (a) Powder diffraction data for gel grown Bi$_2$S$_3$ crystals.

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Table 5.1 (b) Calculated and observed values of d and h, k, l.

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Chapter 5

Characterization of grown $\text{Bi}_2\text{S}_3$ crystals

Figure 5.5 (a) XRD graph of grown crystal of $\text{Bi}_2\text{S}_3$ by Zone Refing method

Figure 5.5 (b) XRD graph of grown crystal of $\text{Bi}_2\text{S}_3$ by Zone Refing method (indexed)
Chapter 5

Characterization of grown Bi$_2$S$_3$ crystals

The Powder diffraction data of grown Bi$_2$S$_3$ crystals by Zone Refining method is shown in table 5.2 (a) & (b). The observed values are very well match with JCPDS card (17 -0320 ) of Bi$_2$S$_3$. Observed peaks in diffractogram indicate the formation of orthorhombic phase of Bismuth-Sulphide.

**Table 5.2. (a) Powder diffraction data for zone melting grown Bi$_2$S$_3$ crystals**

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### Table 5.2 (b) Calculated and observed values of d and h, k, l.

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With the help of JCPDS data, the indexing of some major peaks in XRD pattern can be used to calculate cell dimensions of grown crystals. Using Scherrer Formula: (for Orthorhombic systems)

\[
\frac{1}{d_{hkl}^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}
\]

In Rhombus crystal structure the length of unit cells are different .but the three axis are perpendicular to each other i.e. \(a \neq b \neq c\) & \(a = \gamma = \beta = 90^\circ\).

- For crystalline parameters of \(\text{Bi}_2\text{S}_3\) crystals grown by Gel method.
- Using the values of \(d\)-value corresponding to different peaks such as \((3,0,1),(0,6,0)\) and \((0,0,2)\), cell dimensions \(a\), \(b\) and \(c\) can be evaluated.

The results are found to be

**For \(\text{Bi}_2\text{S}_3\):** \(a = 11.06\ \text{Å}, \ b = 11.33\ \text{Å} \) and \(c = 3.91\ \text{Å} \); Cell Vol. = 490.70 \(\text{Å}^3\)

- For crystalline parameters of \(\text{Bi}_2\text{S}_3\) crystals grown Zone Refining Method.
- Using the values of \(d\)-value corresponding to different peaks such as \((2,1,1),(1,2,0)\) and \((0,0,2)\), cell dimensions \(a\), \(b\) and \(c\) can be evaluated.

The results are found to be

**For \(\text{Bi}_2\text{S}_3\):** \(a = 11.19\ \text{Å}, \ b = 11.33\ \text{Å} \) and \(c = 3.98\ \text{Å} \); Cell Vol. = 504.45 \(\text{Å}^3\)

As compared to standard data of Bismuth Sulphide:

\(a = 11.14\ \text{Å}, \ b= 11.30\ \text{Å} \) and \(c= 3.98\ \text{Å} \); Cell Vol. = 501.01 \(\text{Å}^3\)

Variation in cell parameter shows that the grown crystals of \(\text{Bi}_2\text{S}_3\) are not exact single crystals but they are having crystalline behavior.
5.3 Scanning Electron Microscope (SEM)

The scanning electron microscope (SEM) utilized a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions disclose information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. Using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm), areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode.

The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions (using EDS), crystalline structure, and crystal orientations (using EBSD).

5.3.1 Fundamental principles of scanning electron Microscopy (SEM)

The signals used by a scanning electron microscope to produce an image result from interactions of the electron beam with atoms at various depths within the sample. Various types of signals are produced including secondary electrons (SE), reflected or back-scattered electrons (BSE), characteristic X-rays and light (cathodoluminescence) (CL), absorbed current (specimen current) and transmitted electrons. An electron beam passing through an evacuated column is focused by electromagnetic lenses onto the specimen surface. The beam is then restored over the specimen in synchronization with the beam of a cathode ray tube display screen. The secondary electrons are emitted from the sample surface and collected by a scintillator, the signal from which is used to modulate the brightness of the cathode ray tube. In this way the secondary electron emission from the sample is used to form an image on the digital display screen.
5.3.2 Instrumentation

The specimen has been scanned using SEM Hitachi (S-3400N) model. SEMs always have at least one detector (usually a secondary electron detector), and most have additional detectors. The specific capabilities of a particular instrument are critically dependent on which detectors it accommodates.

Essential components of all SEMs include the following:

- Electron Source ("Gun")
- Electron Lenses
- Sample Stage
- Detectors for all signals of interest
- Display / Data output devices

In addition, SEMs require a stable power supply, vacuum and cooling system, and vibration-free space and need to be housed in an area that isolates the instrument from ambient magnetic and electric fields.

5.3.3 SEM Imaging

A Scanning Electron Microscope imparts details surface information by tracing a sample in a raster pattern with an electron beam. Initially in this process an electron gun generating a beam of energetic electrons down the column and onto a series of electromagnetic lenses. These lenses are tubes, wrapped in coil and referred to as solenoids. The coils are adjusted to focus the incident electron beam onto the sample cause fluctuations in the voltage, increasing/decreasing the speed in which the electrons come in contact with the specimen surface. The SEM operator can adjust the beam to control magnification as well as determine the surface area to be scanned via computer. The beam is focused onto the stage, where a solid sample is placed. Before placed in the vacuum chamber, most samples require some preparation.

The two most commonly used prior to SEM analysis are sputter coating for non-conductive samples and dehydration of most biological specimens. In addition, all samples need to be able to handle the low pressure inside the vacuum chamber. By the acceleration rate of incident electrons, the interaction between the incident electrons and the surface of the sample is determined
which carry significant amounts of kinetic energy before focused onto the sample.

![Schematic working diagram of SEM.](image)

**Figure 5.6 Schematic working diagram of SEM.**

Energetic electrons are released from the surface of the sample, when the incident electrons come in contact with the sample. The scatter patterns made by the interaction yields information on size, shape, texture and composition of the sample. X-rays, emitted from beneath the sample surface, can provide element and mineral information. SEM produces black and white, three-dimensional images. Image magnification can be up to 10 nanometers and, although it is not as powerful as its TEM counterpart, the intense interactions that take place on the surface of the specimen provide a greater depth of view, higher-resolution and, ultimately, a more detailed surface picture.
Chapter 5

Characterization of grown Bi$_2$S$_3$ crystals

5.3.4 Applications

SEMs have a variety of applications in a number of scientific and industry-related fields, especially where characterizations of solid materials is beneficial.

- Used for topographical, morphological and compositional information.
- Scanning Electron Microscope can detect and analyze surface fractures.
- It can provide information in microstructures and examine surface contaminations.
- It reveals spatial variations in chemical compositions and provides qualitative chemical analyses and identifies crystalline structures.
- Used to generate high-resolution images of shapes of objects.
- Used to precise measurement of very small features and objects down to 50 nm in size.
- SEMs can be utilized as essential research tool in fields such as life science, biology, gemology, medical and forensic science, and metallurgy.
- SEMs have practical industrial and technological applications such as semiconductor inspection, production line of miniscule products and assembly of microchips for computers.

5.3.5 Advantages

- Most SEM's are comparatively easy to operate, with user-friendly "intuitive" interfaces.
- SEM works fast, often completing SEI, BSE and EDS analyses in less than five minutes. In addition, the technological advances in modern SEMs allow for the generation of data in digital form.
- Modern SEMs generate data in digital formats, which are highly portable.
5.3.6 Disadvantages

- SEMs are expensive, large and must be housed in an area free of any possible electric, magnetic or vibration interference.

- Maintenance involves keeping a steady voltage, currents to electromagnetic coils and circulation of cool water.

- Special training is required to operate an SEM as well as prepare samples.

- In addition, SEMs are limited to solid, inorganic samples small enough to fit inside the vacuum chamber that can handle moderate vacuum pressure.

- SEMs carry a small risk of radiation exposure associated with the electrons that scatter from beneath the sample surface.
5.3.7 SEM analysis of grown crystals (Bi$_2$S$_3$)(Gel Method)
Figure 5.7(a) SEM images of grown crystals ($\text{Bi}_2\text{S}_3$) (Gel Method)
SEM analysis of grown crystals (Bi$_2$S$_3$) Zone Refining Method
Figure 5.7(b) SEM images of grown crystals (Bi$_2$S$_3$) (Zone Refining Method)
The surface morphology of grown crystals was studied by using Scanning Electron Microscopy (SEM) and the successive photographs were taken at different magnifications. For crystal growth magnification of 1.00k, 2.00 k, 4.00k, 6.00k and 10.00k belongs to Gel method, and magnification of 250 SE, 500 SE and 1.00k belongs to zone method. All the photographs were taken at different width, for Gel ranging from 10.00 mm to 11.00 mm and for Zone Refining ranging from 8.5 mm to 9.5 mm and EHT magnification 15 kV. Figure 5.7 (a) shows the photographs of the surface morphology of Bi₂S₃ crystals grown by Gel method and Figure 5.7 (b) shows the photographs of the surface morphology of Bi₂S₃ crystals grown by Zone Refining method. SEM images of Bi₂S₃ crystals grown through using different methods revealing the differences in morphologies.

In different regions (Figure 5.7(a), Region I-V as shown in photographs) of SEM have plate like Structure. In the case of Gel grown crystals the heterogeneous nucleation process takes place. In higher magnification region V (10.00k) the shape of the particles are like rectangular plate with no attachment of micro crystals which confirms the controlled growth conditions. On other hand, in different regions (Figure 5.7(b), Region I-III as shown in photographs) of SEM have needle shape. In the case of Zone Refining grown crystals the homogeneous nucleation process takes place.

In the case of the nano synthesis of Bi₂S₃ nanoparticles, the shape and size may varied and depends on the methods used for synthesis of Bi₂S₃ nanoparticles and all grown nanoparticles of Bi₂S₃ are discussed and listed in chapter 1. The scene is also true in bulk crystals growth methods.

In our case the shape and size of the particles are very much different in the Gel and Zone Refining method. These results are very much useful for the growth of bulk crystals of Bi₂S₃ which may have wider application in a modern advanced material requirement. We had also used the theory and practically grown Bi₂S₃ nanoparticles by our colleague Nitin.M.Shah and also publishes some research paper in international repute journals.
References


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