CHAPTER-3

Structural studies
3.1 Introduction

The physical properties of a real crystal such as cleavage, electronic band structure, optical transparency, etc are greatly influenced due to its structure and microstructures. Therefore, crystal structure analysis is an indispensable part in the characterization of materials. The polycrystalline material is made up of crystals of small fragments in completely random manner. X-ray powder diffraction (XRD) technique is usually applied for identification of materials and their preliminary structural study. The simplicity and advantages of the X-ray powder diffraction method is that each component of the mixture produces its own characteristic pattern independent of the others. It is also possible to identify the pattern of a particular component and estimate its proportion in the mixture. From the structural analysis it is possible to know crystal system, point group, space group etc. Therefore, it is important to find out the space group symmetry and the basic crystal structure of the material in the paraelectric as well as ferroelectric phase. Though sometimes it is difficult to get complete and precise information about the crystal structure with a limited number of diffraction data, it is still possible to get preliminary structural idea of the compounds.

In the present study, preliminary structural information (i.e., basic crystal system, lattice parameters, particle size etc.) and the micro-structural study of the compounds have been carried out using the following experimental techniques:

- X-ray Diffraction (XRD)
- Scanning Electron Microscopy (SEM)

3.2 X-ray Powder Diffraction

As mentioned earlier, X-ray diffraction (XRD) technique is one of the important methods for studying structural properties (i.e., cell parameters, space group, particle size etc.) of single crystal, polycrystalline and thick/thin films. Some advantages of the X-ray diffraction method can be described as follows:

(i) The powder diffraction pattern is the characteristic of a substance.
(ii) Each substance in a mixture produces its own diffraction pattern independent of the others.

(iii) It describes the state of chemical combination of elements in the material.

(iv) The method is competent in developing quantitative and qualitative analysis of a substance.

As described in the previous chapter the accurate determination of interplanar spacing (d) of a plane (h k l) and lattice parameters (a, b, c, α, β and γ) provides an important basis in understanding various properties of a material.

### 3.2.1 Experimental

The X-ray diffraction patterns of all the compounds have been recorded at room temperature (25°C) using X-ray powder diffractometer ((D8ADV, Bruker, Germany) with CuKα radiation (λ = 1.5405 Å) and in a wide range Bragg angle (2θ ≤ 2θ ≤ 80°) at a scanning rate of 3°/min. The powder for X-ray analysis was prepared by reducing the particle size to 2-5 μm by grinding the ceramic powder in an agate and mortar. Klug and Alexander [203] reports the size of the crystallites is of great importance in X-ray analysis. They showed that the size of the crystallites composing the powder must fall within the acceptable limits or range so that we can get a sharp and smooth diffraction pattern. They have found that when the coarsest fraction of powder is less than 5 μm, the mean deviation in intensity is 1.2 %, whereas for the coarsest fraction ranging from 15 to 50 μm an average intensity deviation of 18 % occurs. If the crystalline size is not allowed to exceed 5 μm, the micro-absorption and particle orientation effects can be neglected. The fine powder was packed uniformly into a slotted-glass slide using the free falling technique described by National Bureau of Standards monograph [221] in order to avoid preferred orientation and induced packing. Ideally, the powder should be mounted in such a way that no foreign material is exposed to the X-ray beam. The operating voltage and current of the X-ray tube were 30 kV and 15 mA respectively. The divergence of the X-ray beam was limited to 3 to 5° in the vertical direction. For recording the intensity distribution of the profiles, the calibration and the accuracy of the adjustment of the diffractometer were checked with the help of a standard Si powder specimen provided with the instrument.
3.2.2 Results and discussion

The room temperature XRD patterns of all the proposed compounds have been shown in Figs.3.1-3.6. The diffraction patterns, consisting of a large number of sharp and single diffraction peaks of the materials, are different from those of the ingredients of the prepared compounds. The nature of the peaks exhibits better homogeneity and crystallization, and thus confirms the formation of single-phase new compounds [203]. The interplanar spacing (d) of the plane was calculated from Bragg angle (2θ) and was indexed in different crystal systems and unit cell configurations using a standard computer program package “POWDMULT” [222]. The lattice parameters (a, b, c, α, β and γ) have been estimated from the peaks positions of the diffractograms. The observed values of a, b and c have been refined using least-squares fit of the observed positions (d_{hkl} interplanar spacing) of the peaks so that \( \sum \Delta d = \sum (d_{obs} - d_{cal}) \) was found to be minimum. In the present investigation of samples the estimated standard deviation is found to be in the range of + 0.0004 to 0.0040 Å. This shows a good agreement between the observed and calculated values of interplanar spacing, and hence confirms the correctness of the chosen crystal system and unit cell parameters. However, with very limited powder diffraction data it is not possible to determine the space group uniquely.

Further, the scattered crystallite or particle size (P) of the compounds was calculated using the broadening of some widely spread (over Bragg angles) strong and medium reflections in the Scherrer’s equation: 

\[
P_{hkli} = \frac{K\lambda}{\beta_{1/2}\cos\theta_{hkli}}\]

[201], where K (constant) = 0.89, \( \lambda = 1.5405\text{Å} \) and \( \beta_{1/2} \) = full width at half maximum (in radians). As the powder sample was used to get XRD pattern, contributions of strain and other effects in the broadening of XRD peaks and crystallite size calculation have been ignored. The refined lattice parameters and average value of particle size are in given in Table 3.7.
(i) \( \text{Ba(Bi}_{0.5}\text{Nb}_{0.5})\text{O}_{3} \)

![XRD pattern of \( \text{Ba(Bi}_{0.5}\text{Nb}_{0.5})\text{O}_{3} \)](image)

**Fig. 3.1** XRD pattern of \( \text{Ba(Bi}_{0.5}\text{Nb}_{0.5})\text{O}_{3} \)

**Table 3.1** Comparison of observed and calculated \( d \) values (in Å) of \( \text{Ba(Bi}_{0.5}\text{Nb}_{0.5})\text{O}_{3} \) with relative intensity \( I/I_0 \).

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Figure 3.1 shows the XRD pattern of \( \text{Ba(Bi}_{0.5}\text{Nb}_{0.5})\text{O}_{3} \) at room temperature. The diffraction peaks of the compound were indexed the in different crystal systems and unit cell configurations using a standard computer program package “POWD” [222]. A monoclinic unit cell was selected on the basis of good agreement between observed and calculated inter planar spacing \( d \) (i.e., \( \sum \Delta d = d_{\text{obs}} - d_{\text{cal}} \) = minimum). The lattice parameters of the selected unit cell were refined using the least-squares sub-routine of the standard computer program package. The refined lattice parameters are; \( a = 6.432 \) (4) Å, \( b = 3.037(4) \) Å, \( c = 4.546(4) \) Å, \( \beta = 109.430 \) and \( V=83.74 \) Å³. Using refined
lattice parameters, each peak was indexed and inter-planar spacing (d) of reflection planes of the compound was calculated, and compared with its observed value (Table 3.1). It is found that refined unit cell parameters with proposed crystal structure (monoclinic) which is reported by us [223] and space group (P2/m) of the sample are very much consistent with the reported earlier [160,161]. The average value of P was found to be 143 nm [201].

(ii) $\text{Ba(Bi}_{0.5}\text{V}_{0.5}\text{)}\text{O}_3$

![Fig. 3.2 XRD pattern of Ba(Bi$_{0.5}$V$_{0.5}$)O$_3$](image)

**Table 3.2** Comparison of observed and calculated d values (in Å) of Ba(Bi$_{0.5}$V$_{0.5}$)O$_3$ with relative intensity $I/I_o$.

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The X-ray diffraction (XRD) patterns of Ba(Bi\textsubscript{0.5}V\textsubscript{0.5})O\textsubscript{3} recorded at room temperature on powder sample is shown in Fig.3.2. The average value of particle size (P) of the above compound is found to be 24 nm [201]. A monoclinic unit cell was selected on the basis of good agreement between observed and calculated inter planar spacing d (i.e., \( \sum \Delta d = d_{\text{obs}} - d_{\text{cal}} = \text{minimum} \)). The lattice parameters of the selected unit cell were refined using the least-squares sub-routine of the standard computer program package ‘POWD’ [222]. The refined lattice parameters are; \( a = 4.2085(20) \, \text{Å} \), \( b = 3.0246(20) \, \text{Å} \), \( c = 3.0361(20) \, \text{Å} \), \( \beta = 91.29^0 \) and \( V=38.64 \, \text{Å}^3 \)[224]. Each peak of XRD pattern was indexed using refined lattice parameters and inter-planar spacing (d) of reflection planes of the compound was calculated, and compared with its observed value (Table 3.2). There are a few small spikes which cannot be indexed in any crystal system. It suggests that these may be part of background or unknown secondary phase related to impurities of ingredients.

(iii) Ba(Bi\textsubscript{0.5}Ta\textsubscript{0.5})O\textsubscript{3}

![XRD pattern of Ba(Bi\textsubscript{0.5}Ta\textsubscript{0.5})O\textsubscript{3}](image)

**Fig. 3.3** XRD pattern of Ba(Bi\textsubscript{0.5}Ta\textsubscript{0.5})O\textsubscript{3}
Table 3.3 Comparison of observed and calculated d values (in Å) of Ba(Bi$_{0.5}$Ta$_{0.5}$)O$_3$ with relative intensity I/I$_0$.

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Figure 3.3 shows the room temperature XRD pattern of calcined powder of the compound Ba(Bi$_{0.5}$Ta$_{0.5}$)O$_3$. The angular position (2θ) and intensity of peaks of the diffraction pattern of the sample are different from that of ingredients suggesting the formation of a new single-phase compound. Indexing of all peaks of the XRD pattern was carried out by taking 2θ and intensity value of each peak using a standard computer program package "POWD"[222]. A monoclinic unit cell was selected based on good agreement between observed and calculated interplanar spacing d (i.e., $\sum \Delta d = d_{obs} - d_{cal} = \text{minimum}$) which is very much consistent with the reported earlier [225]. The lattice parameters of the selected unit cell were refined using the least-squares sub-routine of the standard computer program package. The refined lattice parameters are $a = 4.2771(40) Å$, $b = 3.0327(40) Å$, $c = 4.2793(40) Å$, $\beta = 90.18^0$ and $V = 55.51 Å^3$. Each peak was indexed again by using refined lattice parameters, and inter-planar spacing (d) of reflection planes of the compound was calculated, and compared with its observed value (Table 3.3) [226]. However, again with very limited powder diffraction data, it is not possible to determine the space group uniquely. Using broadening of a few selected reflections, coherently scattered crystallite size (P) of the compound was calculated using Scherrer equation [201]. The average value of P was found to be 50nm.
(iv) \( \text{Sr(} \text{Bi}_{0.5}\text{Nb}_{0.5}\text{)}\text{O}_3 \)

![XRD pattern of \( \text{Sr(} \text{Bi}_{0.5}\text{Nb}_{0.5}\text{)}\text{O}_3 \)](image)

**Table 3.4** Comparison of observed and calculated d values (in Å) of \( \text{Sr(} \text{Bi}_{0.5}\text{Nb}_{0.5}\text{)}\text{O}_3 \) with relative intensity \( I/I_0 \).

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Fig. 3.4 shows the room temperature XRD pattern of \( \text{Sr(} \text{Bi}_{0.5}\text{Nb}_{0.5}\text{)}\text{O}_3 \) calcined powder. The well-resolved and sharp peaks of \( \text{Sr(} \text{Bi}_{0.5}\text{Nb}_{0.5}\text{)}\text{O}_3 \) show that the material is
highly crystallized. The diffraction peaks of the compound were indexed in different crystal systems and unit cell configurations using a standard computer program package ‘POWD’ [222]. On the basis of the best agreement between observed and calculated inter planar spacing d (i.e., $\sum \Delta d = d_{\text{obs}} - d_{\text{cal}} = \text{minimum}$), a suitable unit cell of the compound was selected in the orthorhombic crystal system which is reported by us [227]. The lattice parameters of the selected unit cell were refined using the least-squares sub-routine of the above standard computer program package. The refined lattice parameters are; a = 12.7295(11) Å, b = 4.2994(11) Å, c = 6.2887(11) Å, and V=344.18 Å$^3$ with error in the parenthesis. Each peak of XRD pattern was indexed using refined lattice parameters and inter-planar spacing (d) of planes (Table 3.4). Further, the scattered crystallite or particle size (P) of the compound was calculated using the broadening of some widely spread (over Bragg angles) strong and medium reflections in the Scherrer’s equation [201]. The average value of P is found to be 294 nm. This large value of the particle is very much consistent with the nature of reflection profile.

(v) $\text{Sr(Bi}_{0.5}\text{V}_{0.5})\text{O}_3$

![Fig. 3.5 XRD pattern of Sr(Bi$_{0.5}$V$_{0.5}$)O$_3$](image)
Table 3.5 Comparison of observed and calculated d values (in Å) of Sr(Bi_{0.5}V_{0.5})O_3 with relative intensity I/I_o.

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The X-ray diffraction pattern of Sr(Bi_{0.5}V_{0.5})O_3 recorded at room temperature on the powder sample is shown in Fig. 3.5. The diffraction peaks of the compound were indexed in different crystal systems and unit cell configurations using a standard computer program package ‘POWD’ [222]. A monoclinic unit cell was selected on the basis of good agreement between observed and calculated inter planar spacing d (i.e., \(\sum \Delta d = d_{obs} - d_{cal} = \text{minimum}\)). The refined lattice parameters are; a = 4.818(6) Å, b = 4.167(6) Å, c = 7.331(6) Å, \(\beta = 114.88^0\) and V=133.52 Å³ with error in the parenthesis [228]. Using refined lattice parameters, each peak was indexed and inter-planar spacing (d) of reflection planes of the compound was calculated, and compared with its observed value (Table 3.5). The best agreement between the observed and the calculated interplanar spacings (d) and Bragg angles was found for the monoclinic crystal structure. The average value of P was found to be 136 nm.
(vi) \( \text{Sr(Bi}_{0.5}\text{Ta}_{0.5})\text{O}_3 \)

![XRD pattern of \( \text{Sr(Bi}_{0.5}\text{Ta}_{0.5})\text{O}_3 \)](image)

**Fig. 3.6** XRD pattern of \( \text{Sr(Bi}_{0.5}\text{Ta}_{0.5})\text{O}_3 \)

**Table 3.6** Comparison of observed and calculated \( d \) values (in Å) of \( \text{Sr(Bi}_{0.5}\text{Ta}_{0.5})\text{O}_3 \) with relative intensity \( I/I_0 \).

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</table>

The X-ray diffraction (XRD) pattern of \( \text{Sr(Bi}_{0.5}\text{Ta}_{0.5})\text{O}_3 \) recorded at room temperature on powder sample is shown in Fig.3.6. The diffraction pattern consisting of number of sharp and single diffraction peaks of the material is different from those of the ingredients of the prepared compound. It exhibits better homogeneity and crystallization, and thus confirms the formation of single-phase of new compound. The well-resolved
and sharp peaks of \( \text{Sr(Bi}_{0.5} \text{Ta}_{0.5})\text{O}_3 \) show that the material is highly crystallized. Generally, a good agreement between observed (obs.) and calculated (cal.) interplanar spacing (d) was found in orthorhombic crystal system and a selected unit cell. The selected lattice parameters of a unit cell were refined using least-squares refinement subroutine of POWD. The observed and calculated values of d of each reflection are compared in Table 3.6. The average value of P is found to be 292 nm. The least-squares refined unit cell parameters are; \( a = 6.4669(11) \) Å, \( b = 8.1639(11) \) Å, \( c = 4.2528(11) \) Å, and \( V=224.53 \text{ Å}^3 \) (the number indicated in parenthesis is estimated as standard deviation of unit cell parameters).

**Table 3.7** Comparison of cell parameters (in Å), volume (V in \text{ Å}^3), average particle size (P in nm), average grain size (G in \text{ μm}) and \( \beta \) (in °) for monoclinic structure of the compounds. The estimated standard deviations in cell parameters are given in parenthesis.

<table>
<thead>
<tr>
<th>Compounds</th>
<th>structure</th>
<th>a (Å)</th>
<th>b (Å)</th>
<th>c (Å)</th>
<th>Volume (Å³)</th>
<th>P (nm)</th>
<th>G (μm)</th>
<th>( \beta ) (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \text{Ba(Bi}<em>{0.5} \text{Nb}</em>{0.5})\text{O}_3 )</td>
<td>monoclinic</td>
<td>6.432(4)</td>
<td>3.037(4)</td>
<td>4.546(4)</td>
<td>83.74</td>
<td>143</td>
<td>2-12</td>
<td>109.43</td>
</tr>
<tr>
<td>( \text{Ba(Bi}<em>{0.5} \text{V}</em>{0.5})\text{O}_3 )</td>
<td>monoclinic</td>
<td>4.2085(20)</td>
<td>3.0246(20)</td>
<td>3.0361(20)</td>
<td>38.64</td>
<td>24</td>
<td>2-10</td>
<td>91.29</td>
</tr>
<tr>
<td>( \text{Ba(Bi}<em>{0.5} \text{Ta}</em>{0.5})\text{O}_3 )</td>
<td>monoclinic</td>
<td>4.2771(40)</td>
<td>3.0327(40)</td>
<td>4.2793(40)</td>
<td>55.51</td>
<td>50</td>
<td>2-6</td>
<td>90.18</td>
</tr>
<tr>
<td>( \text{Sr(Bi}<em>{0.5} \text{Nb}</em>{0.5})\text{O}_3 )</td>
<td>orthorhombic</td>
<td>12.7295(11)</td>
<td>4.2994(11)</td>
<td>6.2887(11)</td>
<td>344.18</td>
<td>294</td>
<td>1-3</td>
<td>------</td>
</tr>
<tr>
<td>( \text{Sr(Bi}<em>{0.5} \text{V}</em>{0.5})\text{O}_3 )</td>
<td>monoclinic</td>
<td>4.818(6) Å</td>
<td>4.167(6)</td>
<td>7.331(6)</td>
<td>133.52</td>
<td>136</td>
<td>1-4</td>
<td>114.88</td>
</tr>
<tr>
<td>( \text{Sr(Bi}<em>{0.5} \text{Ta}</em>{0.5})\text{O}_3 )</td>
<td>orthorhombic</td>
<td>6.4669(11)</td>
<td>8.1639(11)</td>
<td>4.2528(11)</td>
<td>224.53</td>
<td>292</td>
<td>2-8</td>
<td>------</td>
</tr>
</tbody>
</table>

It is found (from Table 3.7) that The diffraction patterns, consisting of a large number of sharp and single diffraction peaks of the materials, are different from those of the ingredients of the prepared compounds. The nature of the peaks exhibits better homogeneity and crystallization, and thus confirms the formation of single-phase new compounds. The cell parameters are slightly changed with change in B	extsuperscript{2-} ions. The average value of the particle size also changes significantly with change of B	extsuperscript{2-} ions and lies between 24-294 nm. Among the proposed compounds \( \text{Sr(Bi}_{0.5} \text{Nb}_{0.5})\text{O}_3 \) and \( \text{Sr(Bi}_{0.5} \text{Ta}_{0.5})\text{O}_3 \) exhibit orthorhombic crystal structure and others exhibit monoclinic crystal structure.
3.3 Scanning Electron Microscopy

The scanning electron microscopy (SEM) is a powerful tool to characterize microstructures of the samples. The high magnification attainable combined with a large depth of field makes SEM an outstanding diagnostic system for micro-fabrication. SEM makes use of point to point scanning of the solid surface and produces a clear image of specimens, which can be visible with a naked eye and provides information about their size which lies in the range of μm. It uses a beam of highly energetic electrons to examine objects on a very fine scale. The following information can be obtained from SEM micrographs:

- **Topography**: The surface features of an object or “how it looks”, its texture: direct relation between these features and materials properties (hardness, reflectivity, etc)
- **Morphology**: The shape and size of the particles making up the object; direct relation between the structure and materials properties (ductility, strength, reactivity, etc)
- **Composition**: The elements and the compounds that the object is composed of and the relative amounts of them: direct relationship between composition and materials properties (melting point, reactivity, hardness, etc)
- **Crystallography information**: How the atoms are arranged in the object, direct relation between these arrangements and materials properties (conductivity, electrical properties, strength, etc).

### 3.3.1 Experimental

Samples for micro-structural study were prepared as follows. First the surfaces of the pellets were polished with a very fine emery paper, and then these polished faces were electroded with high quality gold paste using a vacuum coating technique. These gold-coated samples were used for SEM analysis. The microstructures of gold-coated pellets were recorded by JEOL JSM-5800 and HITACHI S3400N scanning electron microscope. It is a versatile electron microscope with a magnetic deflection field just above the objective of the microscope. The final reducing lens causes the electron to scan the surface of the object in the two mutually perpendicular directions. The secondary electrons emitted by the object constitute the signal current. They are drawn by collecting electrode and after amplification it is made to modulate the brightness of the viewing tube whose screen was scanned in synchronization with the specimen. The magnification of the image was equal to the ratio of scanning area on the screen and the selected area of
the specimen. The image contrast was varied according to shape and composition of the specimen. The basic features of the instrument that are used in the present investigation are as follows:

(i) Resolution: As small as 3.5 nm. (ii) Magnification: X 18 to X 300 000

(iii) Accelerating voltage: 0.3 to 30 kV

(iv) Specimen movements: X movement: 125 mm, Y movement: 100 mm Z movement: 43 mm, Tilt: -10 to +90°, Rotation: 360° endless

(v) Maximum specimen size: 8-inch dia. can be mounted, 7-inch dia can be observed with full coverage by specimen rotation

- Working pressure: $10^{-4}$ Pa order

### 3.3.2 Results and discussion

The surface morphology of the investigated compounds was recorded at room temperature using scanning electron microscope (SEM) technique. The microstructures of the sintered pellets show that the grain growth process is more or less completed during sintering and no secondary re-crystallization has been taken place. Figs. 3.7 - 3.12 show the scanning electron micrographs of proposed compounds at different magnification.

(i) $\text{Ba(Bi}_{0.5}\text{Nb}_{0.5})\text{O}_3$

![SEM micrograph of $\text{Ba(Bi}_{0.5}\text{Nb}_{0.5})\text{O}_3$](image.png)

Fig. 3.7 SEM micrograph of $\text{Ba(Bi}_{0.5}\text{Nb}_{0.5})\text{O}_3$
The SEM micrograph of sintered pellet of Ba(Bi$_{0.5}$Nb$_{0.5}$)O$_3$ at room temperature is shown in Fig. 3.7. The SEM micrograph shows the polycrystalline natures of microstructure having highly distinctive and compact grain distributions where the grains of different sizes and shapes are in-homogeneously distributed throughout the sample surface. The grains are overall dense and closely packed with a few scattered pores with a certain degree of porosity in the sample. The grain size lies between 2-12 µm [223].

(ii) \[ \text{Ba(Bi}_{0.5}\text{V}_{0.5}\text{)}\text{O}_3 \]

The SEM micrograph of sintered pellet of Ba(Bi$_{0.5}$V$_{0.5}$)O$_3$ at room temperature is shown in Figure 3.8. The SEM micrograph shows the polycrystalline nature of microstructure where the grains of different sizes and shapes are in-homogeneously distributed throughout the sample surface. This clearly indicated the purity of chemical composition of compound. It is observed from the SEM micrograph that the grains of different shapes having unequal sizes 2–20 µm distributed throughout the sample [224]. In solids, sintering process, grain growth and stresses play important role in determining many physical properties of a material. Though the stresses are independent of grain size spontaneous cracking occurs predominantly in grains which are larger in size due to the reason that the reduction in the internal strain energy is proportional to the cube of the particle size.
(iii) \( \text{Ba(Bi}_{0.5}\text{Ta}_{0.5})\text{O}_3 \)

The SEM micrograph of sintered pellet of \( \text{Ba(Bi}_{0.5}\text{Ta}_{0.5})\text{O}_3 \) at room temperature is shown in Fig.3.9. The micrographs of these compounds indicate the existence of polycrystalline nature of the sample comprising of grains of different type/size appear to be distributed in homogeneously throughout the sample surface. It is observed from the SEM micrograph that the grains of different shapes having unequal sizes 2–6 \( \mu m \) distributed throughout the sample apart from very few agglomerations of grains[226].

(iv) \( \text{Sr(Bi}_{0.5}\text{Nb}_{0.5})\text{O}_3 \)

Fig.3.9 SEM micrograph of \( \text{Ba(Bi}_{0.5}\text{Ta}_{0.5})\text{O}_3 \)

Fig.3.10 SEM micrograph of \( \text{Sr(Bi}_{0.5}\text{Nb}_{0.5})\text{O}_3 \)
Chapter 3

The SEM micrograph of sintered pellet of \( \text{Sr(Bi}_{0.5}\text{V}_{0.5})\text{O}_3 \) at room temperature is shown in Fig.3.10. The SEM micrograph shows the polycrystalline nature of the sample. Though grains of different sizes and shapes are in-homogeneously-distributed throughout the sample surface, they are closely packed with grain size of 1 to 3\( \mu \text{m} \) [227]. The grains are overall dense and closely packed. This clearly indicated the purity of chemical composition of compound.

\[ \text{(v) } \text{Sr(Bi}_{0.5}\text{V}_{0.5})\text{O}_3 \]

Fig.3.11 SEM micrograph of \( \text{Sr(Bi}_{0.5}\text{V}_{0.5})\text{O}_3 \)

The SEM micrograph of sintered pellet of \( \text{Sr(Bi}_{0.5}\text{V}_{0.5})\text{O}_3 \) at room temperature is shown in figure 3.11. The SEM micrograph shows the polycrystalline natures of microstructure where the grains of different sizes and shapes are in-homogeneously distributed throughout the sample surface. It is observed that the grains are uniformly distributed on the sample surface, and also in the bulk. The presence of voids of irregular dimension indicates that the pellet samples have certain degree of porosity. Cross-sectional micrographs (not shown here) of all the samples were found similar to those of plane surface. This confirms the uniform distribution of grains throughout the bulk sample. The grain size lies in the range of 1-4 \( \mu \text{m} \) [228].
(vi) \( \text{Sr(Bi}_{0.5}\text{Ta}_{0.5})\text{O}_3 \)

Fig. 3.12 SEM micrograph of \( \text{Sr(Bi}_{0.5}\text{Ta}_{0.5})\text{O}_3 \)

Fig. 3.12 shows the SEM micrograph of the sintered pellets of \( \text{Sr(Bi}_{0.5}\text{Ta}_{0.5})\text{O}_3 \) recorded at room temperature. The micrograph shows a group of small sized grains homogeneously distributed throughout the surface of the sample. In spite of sintering at optimized high temperature some voids of irregular shape and dimension are still observed. It is observed from the SEM micrograph that the grains of different shapes having unequal sizes 2–8 µm distributed throughout the sample. The large sized grains are agglomeration of many small ones.

It is found from the SEM micrographs that the grain growth is dependent on substitution of different B” ions, but no specific relation has been observed with average grain size. The grains and pores are distributed more or less uniformly throughout the surface of the samples. The grain size (diameter) has been measured by linear intercept method [229] and the range of grain sizes of all the samples is given in Table 3.7.
3.4 Conclusion

On the basis of the structural and microstructural studies it can finally be concluded that:

- all the studied compounds were found to be single-phase at room temperature.
- among the proposed compounds \( \text{Sr}(\text{Bi}_{0.5}\text{Nb}_{0.5})\text{O}_3 \) and \( \text{Sr}(\text{Bi}_{0.5}\text{Ta}_{0.5})\text{O}_3 \) exhibit orthorhombic crystal structure and others exhibit monoclinic crystal structure.
- the grains were found to be elongated and densely packed over the entire sample surface with minimum voids/pores.
- the density of the sintered pellets was found to be in the range of 85-93% of the theoretical density.