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Enhancement of thermoelectric power factor of hydrothermally synthesised SrTiO$_3$ nanostructures

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Abstract

Strontium Titanate (SrTiO$_3$) nanoparticles were synthesised by varying the hydrothermal growth period as 12, 24 and 48 h. The crystal structure, morphology, functional groups and elemental composition of the prepared SrTiO$_3$ nanoparticles were studied using XRD, FESEM, Raman and XPS, respectively. XRD analysis shows that the intensity of the diffraction peaks of SrTiO$_3$ increased with growth period due to high crystallinity of the hydrothermally grown samples. From the FESEM images, it was observed that the morphology of SrTiO$_3$ was changed from spherical to cubic when the hydrothermal growth period increased from 12 to 24 h. The different modes of vibration of samples were studied using Raman spectroscopy. XPS substantiate the composition and binding states of each element in the sample. The Seebeck coefficient and electrical resistivity of the prepared SrTiO$_3$ nanostructures were measured at various temperatures by pelletizing the samples. The Seebeck coefficient of the sample gradually increased with hydrothermal growth period. The electrical resistivity of the sample relatively decreased with growth period. The power factor of the samples was calculated from the obtained Seebeck coefficient and electrical resistivity. A power factor of the sample prepared at 24 h of hydrothermal growth ($2.191 \times 10^{-4}$ W.m$^{-1}$.K$^{-2}$ at 550 K) was two order higher than that of as prepared sample ($0.012 \times 10^{-4}$ W.m$^{-1}$.K$^{-2}$ at 550 K). The experimental results revealed that the increase in hydrothermal growth period has a potential effect on the morphology. The cubic morphology with high crystalline nature facilitated the electron transport thereby thermoelectric power factor was enhanced in SrTiO$_3$ nanostructures.

Introduction

The demand for electrical energy is rapidly increasing in all perspective of life from household to office to battle field. Therefore, the need to develop smart technology is highly essential, which is feasible through nanotechnology based thermoelectric devices. Thermoelectric materials have extensively been studied because of their capability of converting waste heat energy to electricity [1–3]. For better thermoelectric performance, the figure of merit ($ZT$) of a working material needs to be $>1$ [4]. $ZT = S^2 \sigma T/\kappa$, where $S$ is the Seebeck coefficient, $\sigma$ is the electrical conductivity, $\kappa$ is the thermal conductivity of a material and $T$ is the temperature. Therefore, the thermoelectric energy conversion performance can be improved by enhancing $\sigma$ and controlling $\kappa$ of a material [5]. According to the reports, two third of energy has been lost as waste heat and therefore a hasty development of thermoelectric technology is highly important for effectively converting the waste heat into electricity. Thus, all these needs had given rises to the thermoelectric technology and creates an interest to search for cost effective novel materials for thermoelectric applications [6, 7]. Moreover, thermoelectric performance of the materials can be further improved via nanostructuring the material [8].

Recently, perovskite materials have gained great attention by researchers because of their ample applications, such as supercapacitors, solar cells, lasers, photocatalysts, photoelectrolysis and Fuel cells etc [9]. Perovskite is a
group of compounds, with the crystal structure of ABO₃, where A and B indicates cations with multi valence and O is an anion. The perovskite structure has a simple cubic structure. Among the various perovskite materials, SrTiO₃ is a significant material due to its attractive properties such as high thermal and chemical stability, high mechanical strength, low thermal expansion coefficient, high melting temperature, large non-linear optical coefficient and large dielectric constant [10]. Due to its interesting properties, SrTiO₃ has a wide spread applications in the manufacturing of thermistor, capacitor, microwave devices, photocatalyst, electro-optical device, electro-mechanical devices, RAM and FET [10–13]. Moreover, SrTiO₃ is a well-known thermoelectric material notably at high temperature, since its melting point is 2080 °C. Nanocrystals of SrTiO₃ are synthesized by various methods such as solvothermal method, chemical precipitation method, sol-gel method, microwave-assisted synthesize and hydrothermal reaction [12]. Among these methods, hydrothermal method is well-known for synthesising nanomaterials with high crystalline, high purity, and well controlled morphology [14]. Moreover, hydrothermal synthesis is a simple process which can readily be performed with starting material at low temperature without calcinations.

The effect of morphology of SrTiO₃ on its thermoelectric properties is rarely studied while the thermoelectric properties of rare earth element doped SrTiO₃ are widely reported [15–17]. The mixture of nano and meso structures of material is highly useful for enhancing the electron transport and controlling the phonon transport as the nanostructures favours for phonon scattering and the mixed structures favours for electron transport. Therefore, in the present work SrTiO₃ were synthesised with two different morphologies by varying the hydrothermal growth period and its physicochemical properties were studied using XRD, FESEM, EDAX, Raman Spectroscopy and XPS analysis. The prepared samples were pelletized using hydraulic pressing system and the pellets were sintered at high temperature. The electrical resistivity and Seebeck coefficient of the pellets were investigated as a function of temperature. The electrical resistivity of the samples decreased with hydrothermal growth period. The obtained power factor of SrTiO₃ is relatively higher for the 48 h grown sample.

Materials and methods

Chemical required

Strontium Nitrate (Sr(NO₃)₂) 99%, Sodium Hydroxide (NaOH) 98% were purchased from Alfa Aesar, India. Titantium dioxide (TiO₂) 99.97% and ethanol were procured from Sigma Aldrich, India. For purification and filtration double distilled deionised water was used throughout the synthesis.

Synthesis of SrTiO₃ nanocube

Hydrothermal method is suitable particularly for large control over the material composition [18]. SrTiO₃ nanoparticles were prepared by using Sr(NO₃)₂ and TiO₂ as precursors. 0.1 M of anatase TiO₂ was taken in 10 ml of ethanol. 0.1M of Sr(NO₃)₂ was added to 50 ml of distilled water under vigorous stirring for 15 min at 900 rpm, and then the prepared TiO₂ solution was mixed with the above solution. To stabilize the above prepared material, 0.1M of sodium hydroxide were added and stirred for 8 min. Milky white solution was obtained. Then the white solution was transferred into a 100 ml autoclave and kept in the furnace at 180 °C for different growth periods of 12 h, 24 h and 48 h [19]. For comparative analysis, Sr(NO₃)₂ nanoparticles were prepared by Co-precipitation method using the same precursors. The resulted materials were filtered and washed with ethanol for several times, until it reaches the neutral pH. Then the samples were dried in hot air oven for 6 h at 50 °C. Finally, the synthesized SrTiO₃ powder was obtained and used for further characterisation studies. The prepared samples were named as ST1 (as prepared by co-precipitation), ST2 (12 h), ST3 (24 h) and ST4 (48 h) based on the hydrothermal growth period. The hydrothermal growth of SrTiO₃ nanoparticles with various growth periods is schematically represented in figure 1.

Characterization techniques

The structural properties of the prepared SrTiO₃ samples were analyzed by XRD using Rigaku (Ultima III, Japan) x-ray diffractometer. The Raman analysis was performed using the JASCO NRS 7100 spectrophotometer, Japan. The morphology of the synthesized samples was observed by FESEM using FEI Quanta FEG 200, Scanning Electron Microscope. The EDX spectrum and elemental mappings in the samples were recorded using JSM-7001F, Scanning Electron Microscope. The chemical composition and binding states of each element of the prepared material were studied by XPS using SHIMADZU, ESCA 3400 x-ray Photoelectron Spectrophotometer.
The prepared samples were made into pellets using motorised hydraulic pressing system and the pellets were sintered at high temperature of 1450 °C for 10 h in furnace with air atmosphere. For the Seebeck Coefficient measurement, the pellets with the thickness of 3 mm and diameter of 12 mm were used (figure 2). The indigenously fabricated Seebeck coefficient measurement system was used to measure the Seebeck coefficient of the samples as a function of temperature. The pellet was placed between the heating and non heating ends of the system and measured the thermo emf for various temperature gradients. From the measured thermo power and temperature gradient, Seebeck coefficients of the sample were calculated for various temperatures.

The electrical resistivity of the pellets was measured at various temperatures. Silver paste was placed on the four corners of the pellets, for ohmic contact. The pellet with ohmic contact was placed at the sample chamber with four probes and the resistivity was measured based on Hall Effect at various temperatures using the MMR Hall Measurement system (MMR Technologies, USA).

Result and discussion

Structural analysis of SrTiO₃ in varying reaction time

The structural properties of SrTiO₃ were studied by x-ray diffraction analysis (XRD). Figure 3 shows the XRD patterns of ST1, ST2, ST3 and ST4 samples. The major peaks were observed at 2θ of 22.78, 32.32, 39.86, 46.65,
57.67 and 67.77, corresponds to the (100), (110), (100), (111), (200) and (211) planes of SrTiO3 samples. The peak at 25.3° (indicated as *) belongs to TiO2, which confirms the presence of secondary phase in the sample [19, 20]. The XRD patterns were well matched with the standard JCPDS card no 73-0661 which confirms the cubic structure of SrTiO3. Moreover, the intensity of all diffraction peaks were increased with the growth period as the intensity of peaks of ST3 sample were relatively higher than that of ST1 and ST2 samples. The high intensity of the diffraction peaks illustrated the improvement of crystallinity of the SrTiO3 crystals under hydrothermal growth condition. However, the intensity of diffraction peaks of ST4 samples was slightly decreased compared to ST3 samples which might be due to the insufficient growth unit during prolonged growth period resulted imperfect growth on the surfaces of the crystals with high surface roughness. The XRD patterns confirmed that the crystalline nature of the samples increased with hydrothermal growth period. The crystallite size of the samples was calculated using Scherrer formula

\[
D = \frac{k\lambda}{\beta \cos \theta}
\]

where,
- \( k \) = dimensionless shape factor (0.9)
- \( \lambda \) = Wavelength of x-ray
- \( \beta \) = Full width at half maximum (FWHM)
- \( \theta \) = Bragg angle

The average crystallite size for ST1, ST2, ST3 and ST4 were calculated as 19.64, 19.78, 18.78 and 18.39 nm, respectively. The crystallite size of the SrTiO3 samples decreased as the hydrothermal growth period increases.

**Raman spectroscopy of SrTiO3 nanostructures**

Raman spectra of hydrothermally synthesized ST1, ST2, ST3 and ST4 samples are shown in the figure 4. As a cubic perovskite structure, the first ordered Raman active vibration frequencies of SrTiO3 were not allowed according to the selection rule. However, very weak peaks at 244, 542, 632 and 736 cm\(^{-1}\) were observed with very low intensity and showed an excellent match associated with TO3, TO4, TO and LO4 phonon modes of SrTiO3, respectively [21]. These modes arise due to the breaking of crystal symmetry. Such modes are assigned for the following O-Sr-O bending (244 cm\(^{-1}\)), Ti–O–Ti bending mode (542 cm\(^{-1}\)) and Ti-O stretching modes (736 cm\(^{-1}\)) [22]. The presence of first order Raman lines in SrTiO3 indicates the presence of different crystal symmetry. The peak at 244 and 736 cm\(^{-1}\) are relatively stronger in ST2 and ST3 samples and these peaks are very weak in ST4 sample.

**Morphological analysis of SrTiO3 nanostructures**

Figures 5(a)–(d) shows the FESEM images of ST1, ST2, ST3 and ST4 samples. The micrographs show the mono dispersed spherical morphology of ST1 sample (figure 5(a)) with size of 200 nm, which might be due to
Figure 4. Raman Spectra of (a) ST1, (b) ST2, (c) ST3 and (d) ST4.

Figure 5. FESEM images of ST1 (a), ST2 (b), ST3 (c), and ST4 (d) samples.
simultaneous formation of large number of nucleation during the low temperature processing. The FESEM image of ST2 (figure 5(b)) is quite similar to that of ST1 which shows that at the initial stage of hydrothermal growth large number nucleation were formed and isotropically grown at all the direction which resulted spherical morphology with large number of particles. Figure 5(c) clearly shows that the spherical morphology is smoothly transformed into cubic morphology when the hydrothermal growth period is extended from 12 h to 24 h and 48 h. During the hydrothermal reaction, Sr$^{2+}$ reacted with [Ti(OH)$_6$]$_2$$^{2-}$ and formed large number of small particles within a short period of growth (figures 5(a) and (b)). When the hydrothermal growth period extends the small particles gradually grow into larger particles. When the growth proceeds, the availability of grown units decreased in the solution and thus anisotropic growth was occurred which resulted cubic morphology of the SrTiO$_3$ crystals. It is evident from the figure 5(d), when the hydrothermal growth period increased to 48 h, the growth rate may be low after the initial rapid growth which results the distortion in the cubic morphology of the crystal due to the termination of the reaction [10, 23].

The EDAX spectrum and mapping reveals the presence of Sr, Ti, and O in the sample and their distribution (figure 6). The strong Sr and Ti peaks reveals, that the atomic ratio of Sr:Ti is well coordinated with the prepared stoichiometric ratio which is seen in the inset table of figure 6. Moreover, the EDAX spectrum confirms the absence of any other impurity peaks which shows the high purity of the synthesized samples.

XPS analysis of SrTiO$_3$ nanoparticles

The binding states of each element of ST3 nanoparticles were studied by XPS analysis. The survey spectrum confirms the presence of Sr, Ti and O in the sample [24]. The core level spectrum of Sr 3d shows the doublet peaks at 133.3 and 134.7 eV belongs to Sr 3d$_{5/2}$ and Sr 3d$_{3/2}$, respectively, which confirms the Sr$^{2+}$ state in the sample (figure 7(b)). The high resolution core level spectrum of Ti 2p (figure 7(c)) shows doublet peaks at Ti 2p$_{3/2}$ and Ti 2p$_{1/2}$ with binding energies of 458.6 and 464.9 eV, respectively, which confirms the presence of Ti$^{4+}$ state in the sample [25, 26]. The core level spectrum of O 1s shows a strong peak at 530.4 eV with a shoulder peak 532.5 eV (figure 7(d)). On de-convoluting the spectrum, the two distinct peaks were observed. The peak at 530.4 eV corresponds to the lattice oxygen and the peak at 532.5 eV represents surface oxygen or un-bonded oxygen, respectively. The XPS results confirm the presence of each element and their binding states of SrTiO$_3$ nanoparticles.

Morphological analysis of SrTiO$_3$ pellets

Figures 8(a)–(c) shows the FESEM micrographs of pelletized ST1, ST3 and ST4 samples. The spherical morphology of the ST1 sample was retained in the high temperature sintered pellets. The cubic morphology of the hydrothermally prepared samples was clearly observed in the ST3 and ST4 pellets (figures 8(b), (c)). Moreover, the grains were highly inter-connected in the high temperature sintered pellets which favours electron transport in the samples.
The Seebeck coefficients of ST1, ST3 and ST4 samples were measured at various temperatures by placing the sample between the heating and non-heating ends in the indigenously fabricated Seebeck Coefficient measurement system. The variations of Seebeck coefficient of the samples are as shown in the figure 9. The negative values of Seebeck Coefficients of all the SrTiO₃ samples indicate that, the samples are n-type materials. Moreover, the Seebeck coefficient value increased with increasing temperature for all the samples. For instance, the ST1 sample exhibited the Seebeck coefficient of $-20 \mu V K^{-1}$, ST3 shows $-100 \mu V K^{-1}$ and that of ST4 was $-70 \mu V K^{-1}$ at 550 K, respectively. From the figure 9, it is obvious that the ST4 sample exhibits relatively high Seebeck coefficient compared to ST1 and ST3 at low temperature (<500 K). At all the measured range of temperatures, the Seebeck coefficient of as prepared samples are relatively low possibly due to poor crystalline nature of the samples as observed from low intensity diffraction peaks in the XRD patterns (figure 3). At the
temperature above 500 K, the Seebeck coefficient of ST3 is drastically increased compared to ST1 and ST4 samples. The high Seebeck coefficient values of ST3 and ST4 compared to ST1 is due to the morphological changes under hydrothermal conditions, which result the changes in the microstructures of pellets as evident from SEM images of the pellets (figure 8) [27, 28].

Electrical resistivity as a function of temperature in SrTiO3 nanoparticles
The electrical resistivity of ST1, ST3 and ST4 nanoparticles were measured as a function of temperature using the Hall Effect measurement system. The electrical resistivity of SrTiO3 as a function of temperature is shown in the figure 10. From the figure 10, all the samples show a metallic behaviour, as there is an increase in temperature [29]. The overall resistivity of the hydrothermal samples (ST3 and ST4) were relatively decreased compared to the sample prepared by chemical precipitation method (ST1) possibly due to high crystalline nature of the samples as evidenced from XRD (figure 3). The resistivity of ST4 samples are relatively low in the measured range of temperatures. The low resistivity of ST4 is mainly associated with the mixed morphology of spherical and cubic and its impact on grain structures in the pelletized samples. Therefore it is obvious that the increase in growth period results in low electrical resistivity of the samples [30, 31].

Calculated power factor of SrTiO3 nanoparticles
The power factor ($S^2\sigma$) was calculated using the measured electrical resistivity and Seebeck coefficient of the samples. The power factor as a function of temperature is shown in the figure 11. The power factor of the ST3
and ST4 samples increased with temperature. The ST3 sample shows relatively high power factor especially at high temperature compared to other samples. For instance, the power factor of ST3 ($2.191 \times 10^{-4} \text{Wm}^{-1} \text{K}^{-2}$) was two order increased compared to ST1 ($0.0125 \times 10^{-4} \text{Wm}^{-1} \text{K}^{-2}$) at 550 K. Furthermore, the obtained power factor for ST3 is relatively higher than that of reported values [4]. Furthermore, the ST4 shows relatively higher power factor compared to ST3 at low temperatures. The enhancement in the power factor is due to the change in the morphology from spherical to cubic thereby the microstructures of pellets and increase in the Seebeck coefficient as shown in the figures 5, 8 and 9. Therefore, hydrothermally synthesized nanostructured SrTiO$_3$ is a promising material for thermoelectric applications.

**Conclusion**

In summary, the perovskite SrTiO$_3$ nanoparticles with controlled morphologies and uniform size were successfully synthesized using hydrothermal process by varying the growth period. The growth period in autoclave played an important role in the synthesis of cubic SrTiO$_3$ nanoparticles. The structure and crystallinity of the samples were studied using XRD, which shows the high intense peaks for ST3 sample compared with other samples. The morphological studies were carried out using FESEM, which confirms the spherical and cubic morphology of the samples. The elemental composition and oxidation states of the element were confirmed using XPS studies. The electrical resistivities of hydrothermal samples (ST3 and ST4) were low compared to ST1 samples. The ST3 shows a power factor of $2.191 \times 10^{-4} \text{W.m}^{-1}.\text{K}^{-2}$ at 550 K. The experimental results demonstrated that the hydrothermally prepared nanostructured SrTiO$_3$ shows excellent thermoelectric properties.

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