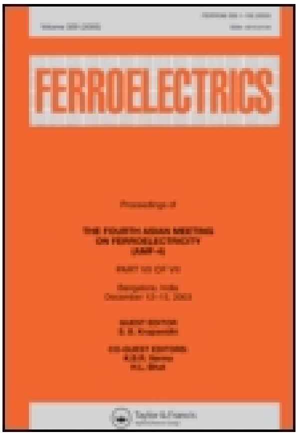

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The Effect of Ba/Sr Ratio on Electrical and Optical Properties of $Ba_xSr_{(1-x)}TiO_3$ (x = 0.25; 0.35; 0.45; 0.55) Thin Film Semiconductor

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The Effect of Ba/Sr Ratio on Electrical and Optical Properties of $Ba_xSr_{(1-x)}TiO_3$ (x = 0.25; 0.35; 0.45; 0.55) Thin Film Semiconductor

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Ferroelectric $Ba_xSr_{(1-x)}TiO_3$ thin film semiconductors with Ba/Sr ratio deposited on silicon using chemical solution deposition (CSD) method have been investigated, followed by annealing at 850°C for 15 hours. Observations by I-V meter, LCR meter, and oscilloscope were employed to characterize the electrical properties of the thin films and the observation of fourier transform spectroscopy (FTIR) and particle size analyzer (PSA) to characterize the optical properties. The results showed that the dielectric constant was given around 2–18. Moreover the obtained films were found to be resistor because the I-V graph of each sample was ohmic either in dark or bright environment. The increase of BST mol fraction at dark environment is proportional to the increase of the curve slope. While at bright environment gives the highest curve slope for BST with fraction x = 0.45. Based on electrical conductivity of thin films, we conclude that the thin films are semiconductor. Moreover, functional group and particle size of sample were analyzed using FTIR and PSA analyzer. The augmentation of Ba would decrease the transmittance band of OH^- and increase the transmittance band of C-O because the radius of Ba is higher than Sr. The particle distribution size of BST 0.45 is 134.93 nm smaller than BST 0.25 which gives 186.26 nm, BST 0.35 gives value of 467.86 nm and BST 0.55 is 407.49 nm.

Keyword $Ba_xSr_{(1-x)}TiO_3$; thin films; electrical properties; optical properties; semiconductor.

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Introduction

Nowadays, synthesis and characterization of nanomaterials have been focused by researchers because of their novel chemical and physical properties arise from the large surface-volume ratios and also the quantum size effect, compared with those of bulk counterparts[1]. One of them is ferroelectric thin films which have found wide applications in many electronic devices[2]. Ferroelectric materials are of great technological importance as promising candidates in a wide range of applications, due to their unique properties of having two polarization states. Applications of ferroelectric devices include dynamic random access memories (DRAM) [3–8], nonvolatile ferroelectric memories [3], microwave tunable phase shifters [6, 9, 10], electro-optical modulators and second-harmonic generators [3]. Similarly Barium Strontium Titanate ($Ba_{1-x}Sr_x$ TiO_3, BST) ferroelectric materials have attracted considerable attentions due to their chemical stability, high permittivity, high tunability and low dielectric losses[1, 2, 11, 12]. BST possesses a wide range of relative dielectric permittivity varying from a few hundred to thousands depending on Ba/Sr ratio, grain size, and temperature [9, 13].

A variety of techniques have been employed to manufacture BST thin films, e.g.: Metal-organic decomposition (MOD), reactive sputtering, sol-gel, metalorganic chemical vapor deposition (MOCVD), atomic layer deposition (ALD), RF magnetron sputtering, ion beam assisted deposition (IBAD) and pulsed laser deposition (PLD) [14–40]. Among those methods, PLD [41] and sol gel or chemical solution deposition (CSD) method are mostly used by researchers because of some advantages, such as the relatively low processing temperature, composition homogeneity, precise control of composition, and large area deposition[5, 6, 42–51]. Despite of that, CSD also offers advantages like stoichiometry control, homogeneity, low sintering temperature, and most importantly low capital cost. These advantages are suitable for scientific research and development.

In this paper we reported the effect of different Ba/Sr ratio of BST thin film on silicon substrate prepared by CSD method on the electrical and optical properties of BST thin films.

Experimental Methods

In this work, BST thin films on platinized silicon substrates were produced by chemical solution deposition (CSD) due to its benefits when dealing with these materials and its easy procedures. In the processing of thin films by CSD, the goal is to optimize film properties for specific applications. To effectively meet this goal, a fundamental understanding of substrate, solution chemistry, and thermal processing effect on the structural evolution of the as-deposited film to the crystalline ceramic state is required [24].

The equipment used in this work are the scales Sartorius Model BL 6100, a set of reactor spin coating, mortar, pipette, measuring cups, glass cups, test tubes, plastic tweezers, scissors, stopwatch, spatula, rubber gloves, masks, glass beaker, lamp, IC 741, resistors, potentiometers, mikrovoltmeter, multimeter, battery, wires, bread board, power supply (GW INSTEK), oscilloscope (TEKTRONIX TDS 1002), FTIR Spectroscopy ABB MB300, and VASCO PSA114102.

The materials used in this research are Barium Acetate [$Ba(CH_3COO)_2$, [99%], Strontium Acetate [$Sr(CH_3COO)_2$, 99%], Titanium Isopropoxide [$Ti(C_{12}O_4H_{28})$, 99.999%], solvent 2- methoxy-ethanol [$H_3COCH_2CH_2OH$, 99%], substrate Si (100) p-type, aquades, HF (Fluoride Acid), preparate glass and aluminum foil.

This research consists of two stages. The first covers the preparation of p-type Si substrate, BST solution making, thin film growing, annealing processing, and contact installing on thin film prototype. The second stage includes electrical and optical properties characterization of BST thin film semiconductor which consists of I-V measurement, dielectric constant, and conductivity. FTIR and PSA characterization were used to obtain functional group and particle size of $Ba_xSr_{(1-x)}TiO_3$ thin films with x = 0.25, 0.35, 0.45 and 0.55.

In the first step we prepared substrate of thin film silicon p-type, where the silicon was cut in size of 8 mm × 8 mm, which then washed successively with acetone, DI Water, Methanol, HF and DI water mixture, and DI Water. Washing was conducted to clean the impurities which may be found in the layer of silicon films. The next step was the solution manufactured of BST. In this study $Ba_xSr_{1-x}TiO_3$ is prepared by chemical solution deposition method (CSD). All materials used are in the form of fine powder, such as barium acetate [Ba $(CH_3COOH)_2$, 99%], strontium acetate [Sr $(CH_3COOH)_2$, 99%)], titanium isopropoxide [$Ti(C_{12}O_4H_{28})$, 99%], and 5 ml 2-metoksietanol [$H_3COOCH_2CH_2OH$, 99%] as a solvent. Where we mixed them in ultrasonic with model Branson 2210 for 1 hour. Mass of each material for any mole fraction is given in Table 1.

The precursor was then deposited on the p-type silicon substrate (100) by dripping it in the middle of the substrate. Deposition process was carried out using spin - coating which rotated at 3000 rpm for 30 seconds, and repeated 3 times. After the deposition process, the next step is annealing. This process was done using the furnace model Vulcan^{TM}3–130. For each substrate annealing process was conducted at a temperature of 850°C, starting from room temperature with increase rate 1.67°C/min until annealing temperature of 850°C, then the temperature was maintained for 15 hours. The last stage is the reduction of furnace temperature (furnace cooling) to obtain room temperature, followed by weighing the mass of these thin films.

The final step was the lead generation measurement approximately 2 mm × 2 mm in the BST layer using silver paste as an adhesive, which previously shot with the use of silver in a vacuum chamber. The process of firing was done in order to metallized silicon substrate BST thin films.

Characterization of BST thin film thickness was carried out using the volumetric method.. Characterization of the dielectric constant was carried out by measuring the capacitance value of the charging and discharging time graph on the oscilloscope which

Table 1
The mass of each mole fraction BST using CSD method

Material	Mol Fraction	Mass (g)
Ba $(CH_3COOH)_2$	0.25	0.3193
	0.35	0.4470
	0.45	0.5747
	0.55	0.7024
Sr $(CH_3COOH)_2$	0.75	0.7714
	0.65	0.6686
	0.55	0.5657
	0.45	0.4628
$Ti(C_{12}O_4H_{28})$	1	1.4211

Table 2
Electric Conductivity of $Ba_xSr_{1-x}TiO_3$ thin films

	Electric Conductivity (S/cm)			
Voltage (Volt)	$Ba_{0.25}Sr_{0.75}TiO_3$	$Ba_{0.35}Sr_{0.65}TiO_3$	$Ba_{0.45}Sr_{0.55}TiO_3$	$Ba_{0.55}Sr_{0.45}TiO_3$
1	5.4×10^{-8}	1.6×10^{-6}	5.8×10^{-7}	4.864×10^{-8}
2	2.7×10^{-8}	1.5×10^{-6}	2.9×10^{-7}	4.846×10^{-8}
3	2.4×10^{-8}	1.5×10^{-6}	1.4×10^{-7}	4.842×10^{-8}
4	2.4×10^{-9}	1.5×10^{-6}	1.4×10^{-7}	4.845×10^{-8}
5	4×10^{-8}	1.5×10^{-6}	1.4×10^{-7}	4.956×10^{-8}

displayed using the RC circuit, where the resistor being used is 10 kOhm and the frequency of the voltage provided at 10 kHz with a variation in voltage between 1–13 volts with increase of 3 volt each. Furthermore the characterization of current-voltage diode was done by I-V meter. Moreover, characterization of electrical conductivity was carried out by measuring electrical conductance using LCR-meter. Conductance measurements of these thin films were done at frequency of 10 KHz, and voltage variation between 1–5 volts.

Analysis of the molecular functional group or compound was carried out using FTIR, in which the functional groups of molecules or compounds are identified by the specific absorption peaks at specific wave number, and to determine the particle size and distribution of BST thin films samples were done by PSA (particles Size Analyzer). Particle size distribution can be determined through the resulting image, which expressed in the radius size of spherical particles. A summary of preparation and washing process of substrate silicon (100) p-type, sample fabrication and characterization are given in Fig. 1 and Fig. 2.

Result and Discussion

The result showed that the alteration of Ba ratio would give effect on the electrical and optical properties of BST thin films either on dielectric constant, I-V measurement, electrical conductivity, FTIR spectra, or particle size. By using volumetric method, we obtained the thickness of thin film (d) at range of $0.34 - 1.34\ \mu m$. This thickness value is important in calculating dielectric constant and electrical conductivity. The augmentation of Ba would decrease the dielectric of thin film which caused by the decrease of its resistance. Characterization of dielectric constant was performed by setting an R-C circuit which shown in Fig. 3, where we can calculate the charging time and the discharging time of the voltage. The charging time is given when t = RC. Where then we get the capacitance value. The dielectric constant could be found by using $= \varepsilon\varepsilon_0 \frac{A}{d}$.

One of the benefits of BST thin films are their usage in capacitor. The capacitor properties arise as a result of the appearance of depletion layer at p-n junction. The depletion layer arises because of the diffusion of electron and hole to the lower concentration region and performs recombination at each other. Holes in p-type have higher concentration than holes in n-type, so that they diffuse from p-type to n-type. The same process takes place on the electron which goes from the n-type to the p-type. But this will not happen forever, because when the hole leaves the p-type region and disappeared by recombination it will leaves a negative acceptor behind at the p-type which form a negative charge. The same

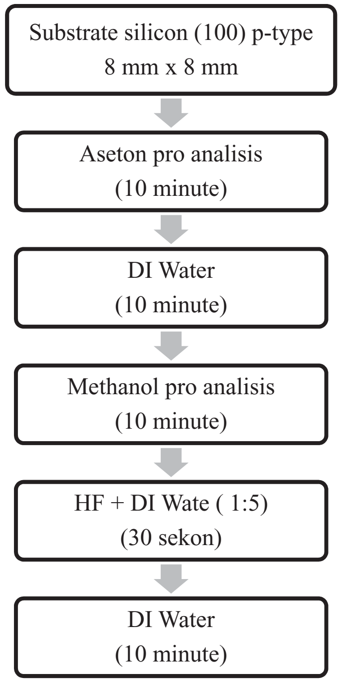

Figure 1. The preparation and washing process of the p-type substrate silicon (100).

ways goes to the electron in the n-type. This creates an electricfield that provides a force opposing the continued exchange of charge carriers.

The measurement gave the dielectric constant value of BST with variation of Ba concentration, and by using voltage variation we could find the relationship between dielectric constant and voltage which given by Fig. 4. Figure 4 showed that on concentration variation of $Ba_{0.25}Sr_{0.75}TiO_3$ gives constant dielectric value for each voltage variation, it shows that the dielectric value of BST thin films is tend to be isotropic, while it is not the case for another film concentration. The fluctuation of dielectric constant was caused by inhomogeneity of thin films surface.

By using CSD method, the dielectric constant was obtained around 2–18. The values are different if compared with hydrothermal method at high temperature, where the dielectric constant is around 583–1733 [52], and 220–520 in sol-gel method [53]. The dielectric constant values of BST thin films by using CSD method have smaller values if compared with other method, with resistor-like of I-V graph which is given by Fig. 5 and 6.

The I-V measurement in voltage range of −1,5 – 1,5 volt showed that all of the BST thin films are ohmic either in dark environment or bright environment, where the curve slope at dark environment are higher than the bright one, which means that by increasing the light intensity would decrease the conductivity value. The augmentation of BST mol fraction at dark environment is proportional to the rise of the curve slope. While at bright environment gives the highest curve slope for BST with fraction $x = 0.45$ (Fig. 5 and 6). These Ohmic arise from the usage of 850°C annealing temperature, in accordance with the report of Jian-Zhong Lou et al [54]. On his paper, Jian-Zhong Lou reported that BST with the fraction $x = 0.6$ shows ohmic at low voltage when annealed at 700 – 850°C, and gives Schottky emission behavior at 850°C [54]. Moreover, the augmentation of Ba would

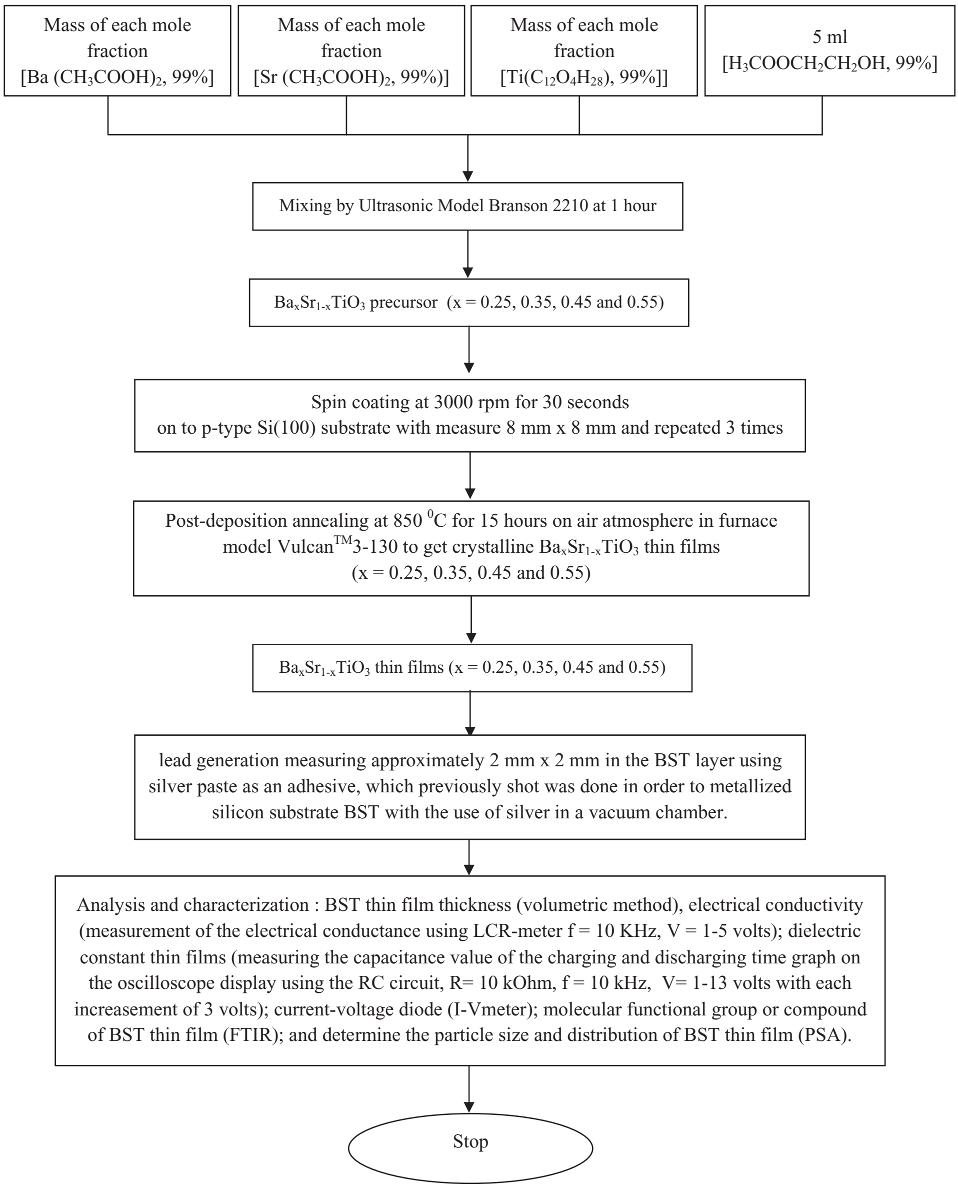

Figure 2. The flow diagram of Step by Step Research.

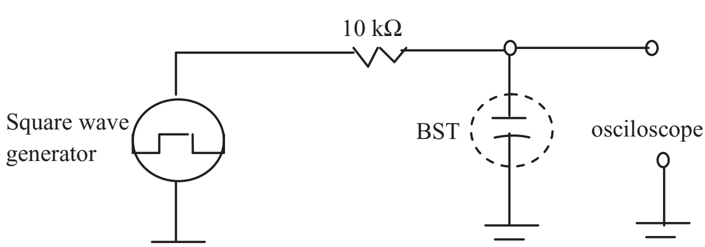

Figure 3. RC circuit to measure the thin films capacitance.

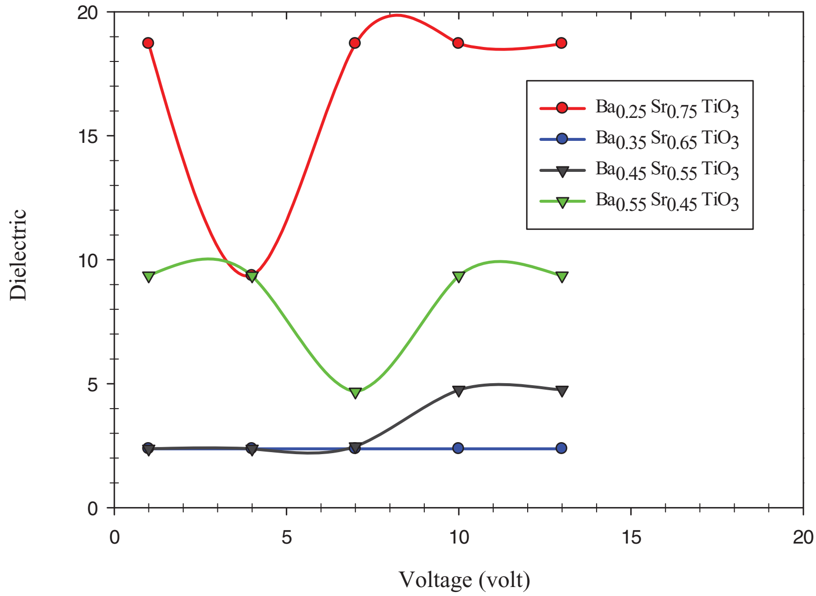

Figure 4. Relationship between dielectric constant and voltage of BST (x = 0.25, 0.35, 0.45, and 0.55) (Figure available in color online).

decrease the resistance value, where the highest resistance value is given by x = 0.25 which shown by the flat line at Fig. 5 and 6.

The augmentation of Ba fraction would decrease the BST transmittance band particularly OH^- transmittance band and increase C-O transmittance band. It was caused by larger

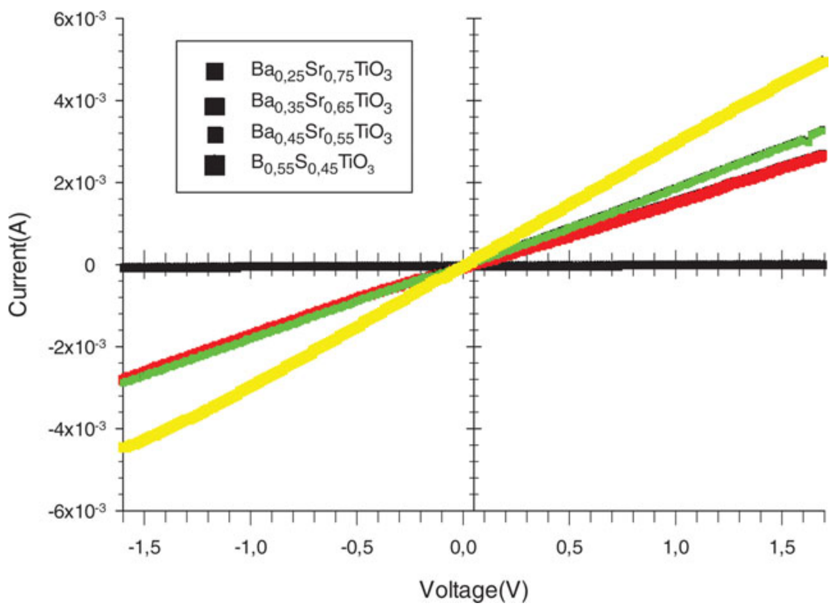

Figure 5. I-V Graph of BST in the dark environment.

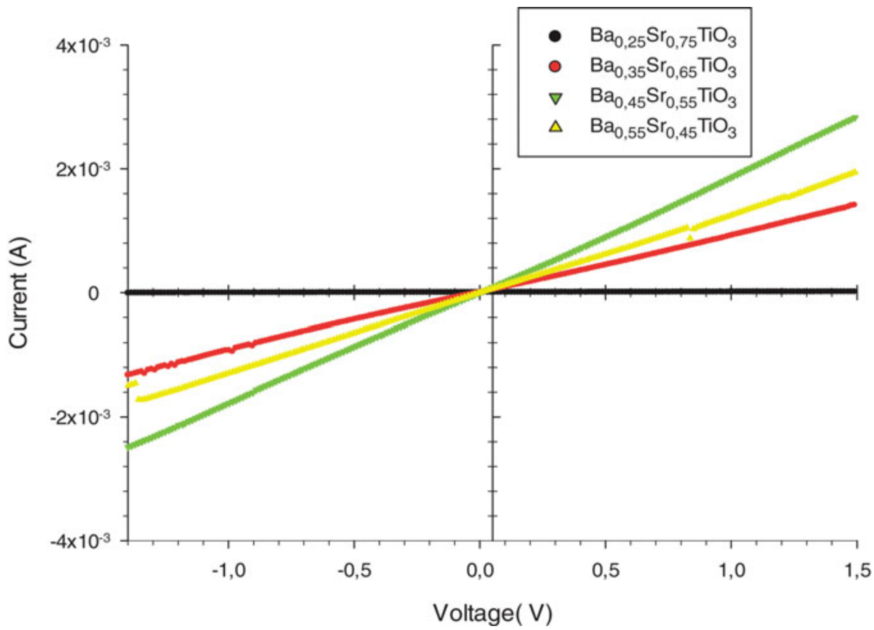

Figure 6. I – V Graph of BST in the bright environment.

radius of Ba compared with Sr. FTIR spectra are given by Fig. 7, where it showed that the transmittance band at 3749 cm^{-1}, 3502 cm^{-1}, and 3479 cm^{-1} can be interpreted as a weak OH^- stretching. The water residue and the hydroxyl group can be detected inside the sample because the sample growth and the heat treatment can eliminate them. The

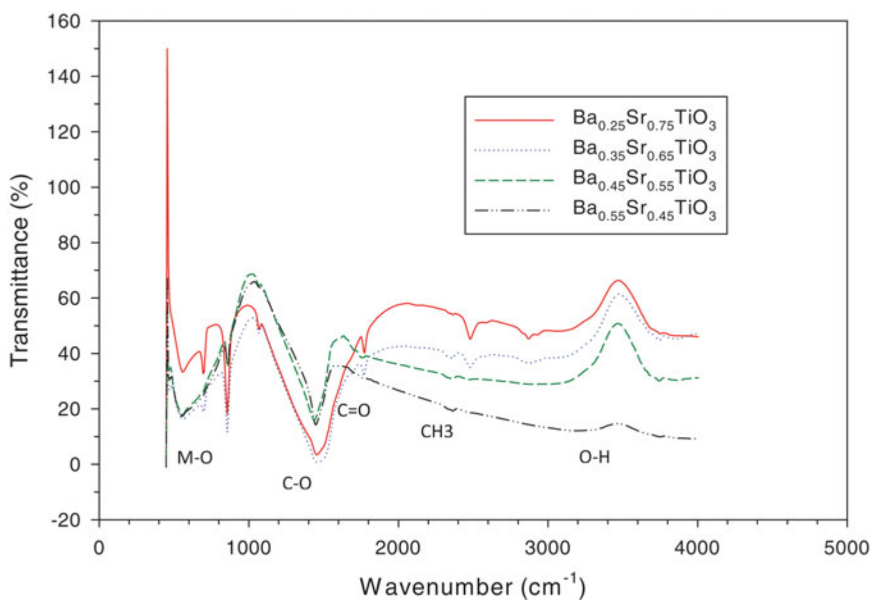

Figure 7. FTIR spectrum of BST (x = 0.25, 0.35, 0.45, and 0.55) (Figure available in color online).

absorbance band at wavenumber 2893 cm^{-1}, 2869 cm^{-1}, 2491 cm^{-1}, 2484 cm^{-1}, 2376 cm^{-1}, and 2368 cm^{-1} give a weak methyl CH_3 stretching, and the wavenumber of 1779 cm^{-1}, and 1758 cm^{-1} give a weak ester C=O stretching. The wavenumber at 1488 cm^{-1}, 1458 cm^{-1}, and 1439 cm^{-1} give a strong CO vibration as a result of a huge carbonate residue from the environment. The absorbance band at 864 cm^{-1}, 856 cm^{-1}, 848 cm^{-1}, 702 cm^{-1}, 563 cm^{-1}, 555 cm^{-1} and 540 cm^{-1} comes from deformation mode (M-O) [55, 56].

Moreover, the particle sizes of BST thin films were analyzed using PSA by using Pade-Laplace method to represent the distribution of the particle sizes shown in Fig. 8 and 9. These distribution is a narrow one, therefore each of the value are not significant. DV10 values for each concentration are particle diameter at size 10% "smoother" than specific point. Furthermore DV50 is a particle diameter at size 50% "smoother" than specific point, and DV90 is for 90% "smoother" than specific point [1]. According to the data, the particle for each concentration have a symmetric shape because the size distribution of volume diameter for each particle size distribution (10%, 50%, and 90%) in every concentration give the same value. Meanwhile the particle distribution size of BST 0.45 is 134.93 nm smaller than BST 0.25 which gives 186.26 nm. BST 0.35 gives value of 467.86 nm and BST 0.55 is 407.49 nm.

Besides FTIR spectra, the difference of Ba and Sr radius would also influence the alteration of conductivity value, where the highest and the lowest values were given by BST of $x = 0.35$ and $x = 0.25$ respectively. The measurement of thin films conductivity was done by measuring their conductance values with LCR meter. The conductance value of BST thin films was influenced by their crystal sizes [57]. After the results have been taken, the values were inserted into the equation $G = \frac{\sigma A}{d}$.

The measurement was done for each fraction of thin films, where voltage variations were performed in 1 V, 2V, 3V, 4V, and 5V [58]. The conductivity values from the measurement are shown at Fig. 10. This figure showed that the highest conductivity value

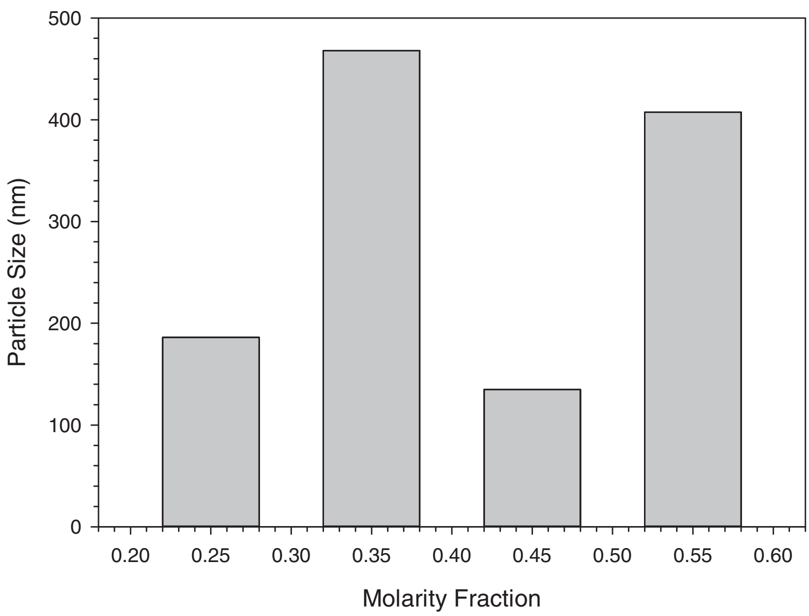

Figure 8. The particle size of BST.

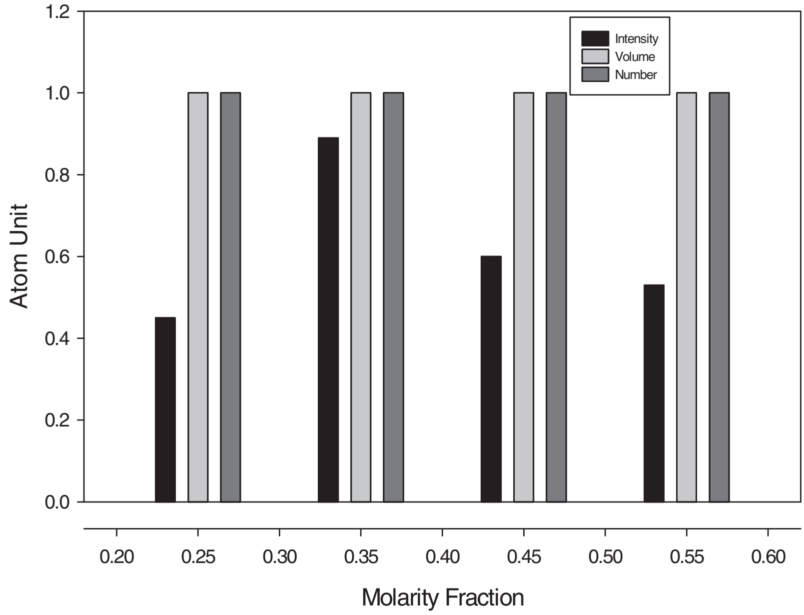

Figure 9. Size distribution of BST by intensity, volume, and number.

is given by the thin films with concentration $Ba_{0.35}Sr_{0.65}TiO_3$, where its highest value is found at 1.6×10^{-6} S/cm with given voltage at 1 V and the lowest conductivity is given by $Ba_{0.25}Sr_{0.75}TiO_3$ with the value 2.4×10^{-9} S/cm and given voltage 4 V. It also shows that the voltage variation input on LCR meter is not affect the conductivity value which

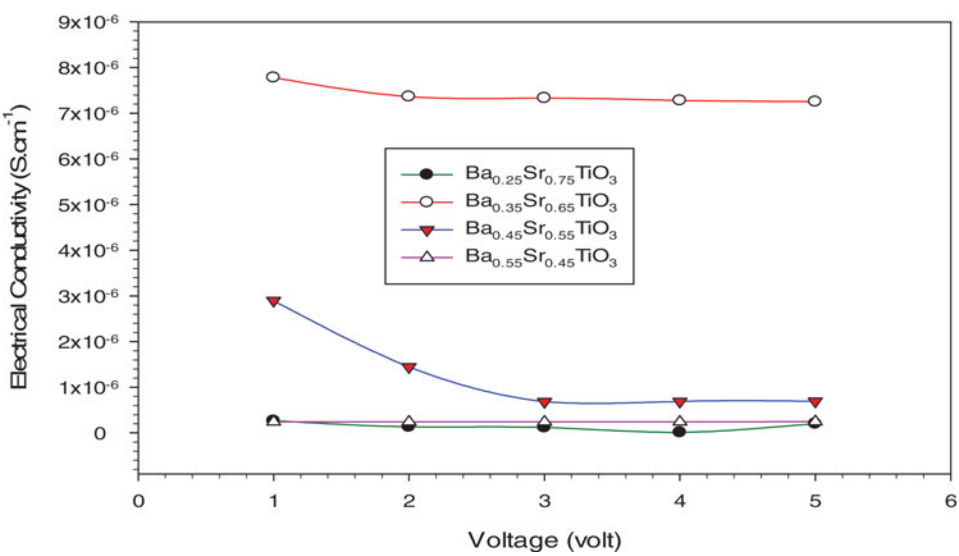

Figure 10. Electrical conductivity value of thin films for each concentration (Figure available in color online).

is shown by the graph shape which relatively flat, some exception is given by thin films with concentration $Ba_{0.45}Sr_{0.55}TiO_3$ which shows some reduction at the beginning of the voltage variation. The order of electrical conductivity is given at range $10^3 - 10^{-9}$ S/cm which considered as semiconductor material as reported by Kwok [59].

Conclusion

The addition of Ba on BST thin films semiconductor with ohmic gave alteration on dielectric constant, electrical conductivity, transmittance band, and particle size. This is probably arise because of the radius differences between Ba and Sr and the inhomogeneity on thin film surface.

Acknowledgments

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