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Optimization of Rotational Speed in the Growing of the Thin Film Using Sol Gel Method which is Prepared with Spin Coating

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Abstract

We investigated the effects of the rotational speed in the spin coating process for growing the BZT thin film. In this process, the rotational speed is taken to be 2000 rpm, 3000 rpm and 4000 rpm while the number of layer is kept constant in five layers. We also discuss the effect of the layers number in the growing of the BZT thin films. For this purpose, we take the layers number to 5, 10 and 15 layers and take the constant rotational speed of 4000 rpm. In order to characterize the formed BZT thin film, the composition, crystal structure and morphological tests are performed. We found that the increasing of the rotational speed then the deposited material in the Si substrate is decreased. As a result, the X-ray intensity in certain orientation is also decreased which is indicates that the probability to form the crystal in a certain orientation is influenced by deposited material in the Si substrate. The measured grains size of the BZT thin film almost similar for the three rotational speeds. However, in the variation of layer numbers, the grain size is increasing as the increasing of layers number.

Keywords: Rotational speed; Spin coating; Crystal structur; Grain size

Introduction

Barium titanate is a material having a ferroelectric property and has a crystal structure of perovskite (ABO_3). It is usually used as material for the capacitor since it has high dielectric constant [1-4]. The perovskite structure of $BaTiO_3$ has an oxygen ion (O^{2-}) in the diagonal of cell unit , Titanium ion(Ti^{4+}) in the diagonal space of the cell unit and Barium ion (Ba^{2+}) at the top end in each sides of the cell unit [5].

Barium zirconium titanate (BZT) obtained by changing the ions in the place of B with Zr in the $BaTiO_3$ compound of the perovskite structure of ABO_3. It might be done since the ion of Zr^{4+} have a size of 0.087 nm which is larger than the Ti^{4+} ion, i.e., 0.068 nm. BZT become a prominent material for barium strontium titanate (BST) in the fabrication of ceramic base capacitor since chemically the Zr^{4+} is more stable than Ti^{4+} [6,7]. The addition of Zr in the $BaTiO_3$ compound reduce the dielectric loss of barium zirconium titanate (BZT). The application of BZT for microwave technology attracts the attention of the researchers since it has high dielectric constant, low dielectric loss and large tunability [8].

There are several methods for the growing of the BZT thin film such as pulse laser deposition [9,10], sputtering [11] and sol gel method [12-16]. In this paper, we present our results of the growing of BZT thin films using the sol gel method which is prepared with spin coating process. In the spin coating process, the physical parameter which is important in order to yield a good thin film is the rotational speed. Thus, it is necessary to optimize this parameter in order to know the physical characteristic of the BZT thin film. For this purpose, one did several tests, i.e. composition test, crystal structure test and morphological test. The effect of the number of the thin film will also be discussed.

Experiment

In this research, we use barium acetate, zirconium isopropanol and titanium isopropoxide with the pure of 99%, 80%, and 80%, respectively. As a solution, acetic acid and etylene glycol are used. And wafer silicon is used as substrate. Composition of BZT was $BaZr_{0,2}Ti_{0,8}O_3$. The BZT thin film which is growing using sol gel method is prepared with spin coater of CHEMAT technology. The rotational speed of this equipment can be set until 6000 rpm. There are three fundamental things that should be taking care in the sol gel method, i.e., the preparation of the solution (chemical process), the growing the thin film with spin coating method and thermal process (annealing). On fabrication of BZT solution, barium acetate and acetic acid was mixed by magnetic stirrer. And then were added successively into the mixture i.e. titanium isopropoxide, zirconium isopropanol and ethylene glycol while were stirred. After that, the solution was heated by hot plate. The BZT solution is made with turbidity of 0.5 M. In the spin coating process, we varied spin speed in the value of 2000 rpm, 3000 rpm, and 4000 rpm, respectively while the number of layers is taken to be five layers. The number of thin layer is also varied with constant rotational speed of 4000 rpm during 30 seconds. In the annealing process, the temperature is set to 800°C for 3 hours. The growth BZT thin film in the Si substrate is then characterized with XRF (X-ray fluoresence) of JEOL to know its composition, XRD (X-ray diffraction) of Bruker D8 Advance with λ Cu = 1,547 Å to find out the crystal structure and SEM (Scanning Electron Microscopy) of Philips to know the morphological structure.

Results and Discussions

Composition test

The composition test of the composited material of the BZT, which are barium, zirconium and titanium, is done using the X-Ray Fluorescence. Our results shown that composite material of the BZT are deposited in the Si substrate and it is measured in the percent weight (wt%). These results are presented in tables 1 and 2.

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From table 1, it is clear that by increasing the rotational speed then the percent of weight of the composited material of the BZT is decreased. This is because large part of the solution is throwing away from the substrate. In the rotational speed of 4000 rpm, the increasing the thin layer numbers makes the composite material is increased as shown in table 2.

Crystal structure test

Crystal structure test of the composited BZT thin film is done using the XRD. The resulted diffraction pattern of the BZT thin film is presented in figures 1 and 2 for variation of rotational speed with five layer numbers and for variation of layer numbers with constant rotational speed of 4000 rpm, respectively. There are several peaks appears and after it fitted with ICDD database PDF#36-0019, it is clear that those patterns are for BZT, zirconium titanium oxide and Si substrate.

From figure 1, we see that if the rotational speed is increase then the X-ray intensity is decrease. It is happen since the large part of the solution of the BZT is throwing away from the substrate where it cannot avoid the centrifugal force of the rotational motion. And if the BZT thin film deposited in the substrate is increase then the probability to form a crystal in certain orientation is large. This can be seen also from the results of composition test in table 1. In our experiment, we also did the measurement for the rotational speed of 1000 rpm. However, the solution is forming a gel in the substrate. And when the substrate is heated, the thin film is crack and there is no thin film of BZT is formed. The same situation is also found for the speed of 5000 rpm, where the thin film formed is too thin and indeed no thin film formation.

In figure 2, we see that the increasing the number of the layer then the X-ray intensity in a certain orientation is also increased. This indicates that if the number of layers in the Si substrate is increase then the percent weight of the compound materials of BZT is also increased. This can also be seen in the composition test results in table 2. This fact is also indicating that the probability to form a crystal with certain orientation is large.

Morphological test

The resulted photos from SEM for the BZT thin film are given in figures 3 and 4. Figure 3a shows the results for rotational speed of 2000 rpm. And the results for the 3000 rpm and 4000 rpm are shown in figures 3b and 3c, respectively. The number of layers is keep fixed at 5 layers. From figure 3, we can see the sharp and homogenous grain with a sharp border between them. In the rotational speed of 2000 rpm and 3000 rpm, the grain size is found to be 140 nm. While for the rotational speed of 4000 rpm is around 170 nm. The figure is taken with the zoom of 60.000 times. This probably was caused by increasingly little BZT thin film which has been deposited on substrate. More active grain movement on diffusion process cause more grains diffused become more big grain.

Material / Rotational Speed	Si (wt%)	Ba (wt%)	Zr (wt%)	Ti (wt%)
2000 rpm	80.1517	14.0457	0.0380	5.7646
3000 rpm	83.1131	12.1608	0.0324	4.6937
4000 rpm	84.8643	10.7140	0.0250	4.3967

Table 1: Composition test of BZT thin film in various rotational speeds with five layers.

Material / Number of layers	Si (wt%)	Ba (wt%)	Zr (wt%)	Ti (wt%)
5 layers	84.8643	10.7140	0.0250	4.3967
10 layers	61.0206	27.2290	0.1131	11.6372
15 layers	40.4091	40.8123	0.1370	18.6416

Table 2: Composition test of BZT thin film in various layers number for rotational speed of 4000 rpm.

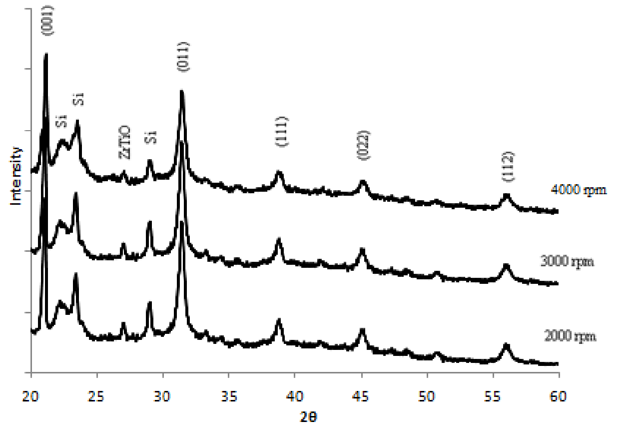

Figure 1: Diffraction pattern of BZT thin film in various rotational speeds.

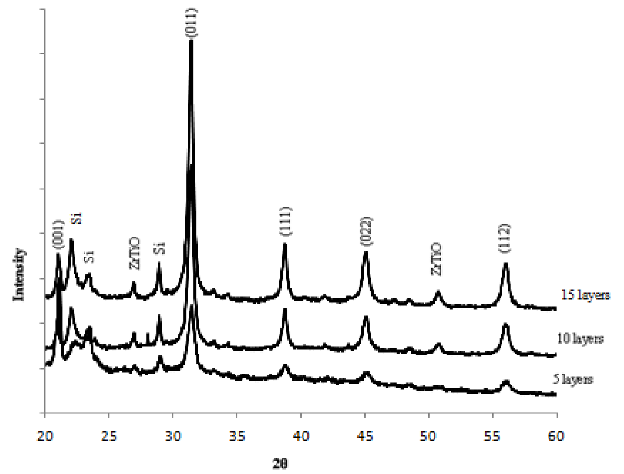

Figure 2: Diffraction pattern of BZT thin film in various layer numbers.

Figure 4a shows the photos for the five layer numbers of the BZT thin films. And for the 10 and 15 layers are given in figures 4b and 4c, respectively. The rotational speed is kept constant at 4000 rpm. From figures 4a and 4b, we can see the grain clearly. But it is not the case for the 15 layers. The growth of the grain is caused by the diffusion process in the annealing process as well as in the adding of the layer numbers. In the annealing process, the diffusion is taking place in the vertical direction. And in the process of adding the number of layers, it is in the horizontal direction. The grain size of the BZT thin film with five layers is around 170 nm. And for BZT thin film with 10 layers is 400 nm. However, for the thin films with 15 layers, the border between the grains is not clear and it makes difficult to measure the grain size. But it is larger than the size of thin film with 5 and 10 layers.

Conclusion

We have investigated the effects of the rotational speed in the

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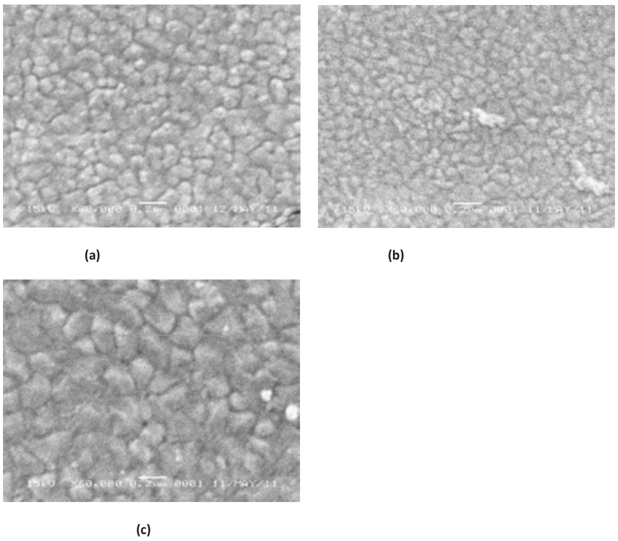

Figure 3: The resulted photo of SEM for the BZT thin films for the rotational speed of. (a) 2000 rpm (b) 3000 rpm (c) 4000 rpm.

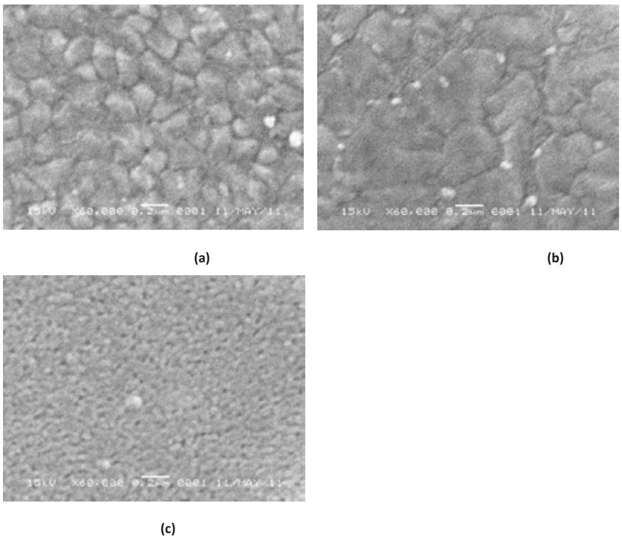

Figure 4: The resulted photo of SEM for the BZT thin film with .
(a) 5 layers (b) 10 layers (c) 15 layers.

spin coating process for the growing of the BZT thin film. It is found with the increasing of the rotational speed, the deposited material in the Si substrate is decreased. As a result, the X-ray intensity in certain orientation is also decreased. It indicates that the probability to form the crystal in a certain orientation is influenced by deposited material in the Si substrate. The measured grains size of the BZT thin film almost similar for the three rotational speeds used in this experiment. However, in the variation of layer numbers, the grain size is not similar but it is increasing as the increasing of layers number. This is caused by the diffusion process happen between the grain in the horizontal and the vertical directions.

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