Effects of Sintering Temperature and Holding Times on the Microstructure and Chemical Bond of Strontium Titanate (SrTiO₃)

D N Hikmah, D K Sandi, F Nurosyid, Y Iriani*

Physics Department, Faculty of Mathematics and Sciences, Universitas Sebelas Maret, Indonesia

*Email: yofent_iriani@staff.uns.ac.id

Abstract. Strontium Titanate (SrTiO₃) is one attractive material studied. In this study, SrTiO₃ has been fabricated via the co-precipitation technique. The samples were sintered at 800°C and 900°C with holding times of 2 h and 4 h for each temperature. The purposes of this study were to synthesize SrTiO₃ material using co-precipitation technique and to observe the microstructure and chemical bonds of the SrTiO₃ as the variations of the sintering temperatures and holding times. According to the X-Ray Diffraction (XRD) results, the sintering temperatures and holding times influenced the intensity values and peak broadening. The alteration in both parameters consequently changed the crystallite size and lattice strain of the SrTiO₃ material. Furthermore, the Fourier Transform Infrared (FTIR) results validated the SrTiO₃ material by existence of Sr–Ti–O chemical bonds. Also, the absorption peaks of O–H, C–H, and C=O chemical bonds in the SrTiO₃ declined due to the higher temperature and longer holding time demonstrating impurities declined. Therefore, according to this study, the sintering temperature of 900°C and the holding time of 4 h was the best parameter for fabricating SrTiO₃ powder.

1. Introduction

Strontium Titanate (SrTiO₃) is an ABO₃ cubic perovskite structure with Pm3m space group and lattice of \(a = b = c = 3.9046\) Å [1]. It possesses an energy band of 3.2 eV with a density of 5.18 g/cm³ at ambient temperature [2,3]. SrTiO₃ is a perovskite material with semiconductor properties attributed to titanium oxide and enhanced by the strontium addition [4,5]. As a result, SrTiO₃ is used in the manufacture of electronic components. The use of strontium titanate for the manufacture of electronic components such as optical sensors, RAM, and transistors just received consideration in the early 2000s [6]. Besides, SrTiO₃ is non-toxic, economical, chemically and photochemically stable making it suitable for water splitting [1,2]. Recently, the demand for strontium titanate has been increasing rapidly. Therefore, the manufacture of SrTiO₃ has received much attention for researchers.

Commonly, the are several techniques used to synthesize SrTiO₃ material those are hydrothermal [1,5], solvothermal [7], sol-gel [4,8], and co-precipitation [9-11]. The co-precipitation procedure is low-price and relatively easy which results in homogenously smaller particles as compared to other techniques [12]. It mixes two or more different solids or liquids by stirring continuously to produce a precipitate [10]. SrTiO₃ fabrication with this technique has not been widely carried out, so studies on this still need to be conducted and developed. Hence, the goals of this study are to synthesize SrTiO₃...
material using co-precipitation technique by varying the sintering temperatures and holding times and to investigate the effects of those variations on the crystallite structure and chemical bonds of SrTiO$_3$.

2. Method

SrTiO$_3$ was synthesized using the co-precipitation technique. The starting materials involved Strontium nitrate (Sr(NO$_3$)$_2$), Oxalic Acid Dihydrate (C$_2$H$_2$O$_4$·2H$_2$O) (Merck ≥ 99%), Titanium Tetrabutoxide (Ti(OCH$_2$CH$_2$CH$_2$CH$_3$)$_4$) (Sigma Aldrich, 97%), Isopropanol (IPA). First, Oxalic Acid Dihydrate was dissolved in IPA named Solution 1. Next, Titanium Tetrabutoxide was also dissolved into IPA called Solution 2. Solution 1 and 2 were then mixed producing Solution 3. Afterward, Sr(NO$_3$)$_2$ was dissolved in IPA and mixed into Solution 3. The mixed solutions were continuously stirred until producing a homogeneous solution. The solution was titrated using distilled water while stirring using a magnetic stirrer on the titrated solution. After that, the titrated solution was precipitated for 24 h at room temperature. The precipitated solution was washed with ethanol and distilled water twice. After washing, the solution was hydrolysed using an oven for 10 h yielding a dry precipitate. The resulting precipitate was milled to obtain homogeneous powder. The samples were sintered at temperatures of 800°C and 900°C with holding times of 2 h and 4 h for each temperature. The sample powder was tested via XRD to observe the crystal structure and FTIR to determine the chemical bonds. The crystal size of the sample was estimated using modified Scherrer equation as Equation 1 [13]. By plotting $\ln \beta$ vs $\ln \frac{1}{\cos \theta}$ as x-axis and y-axis, the value of $\ln \frac{k\lambda}{\beta} \cos \theta$ as x-axis and y-axis, the value of $\ln \frac{k\lambda}{\beta}$ could be taken as the intercept of the plot. Thus, the crystal size could be attained.

$$D = \frac{k\lambda}{\beta \cos \theta}$$

$$\ln \beta = \ln \frac{k\lambda}{\beta} + \ln \frac{1}{\cos \theta}$$

3. Results and Discussion

Figure 1 depicts the XRD results of SrTiO$_3$ with sintering temperatures of 800°C and 900°C, respectively, in the terms of diffraction peaks and 2θ angles. After the peaks were compared with the ICDD database, it is known that the peaks are belong to SrTiO$_3$ phase matched with ICDD number #86-0178. However, other peaks marked (*) are apparent confirming impurities in the synthesized SrTiO$_3$ powder. The impurities are SrCO$_3$ based on the ICDD database #221437. Nonetheless, the impurity intensities decline from 800°C to 900°C and from 2 h to 4 h. Carbon and Hydrogen compounds can react with the main components of Strontium and Titanium to form unwanted compounds/ impurities such as SrCO$_3$, in which these compounds can be diminished by increasing sintering temperature and/or holding time. This denotes that with the higher sintering temperature and longer holding time, the reaction process for the formation of SrTiO$_3$ material is more perfect, leaving less impurities.
Figure 1. The XRD Peaks of SrTiO$_3$ Powder at (left) 800°C for Holding Times of 2 h and 4 h, and (right) 900°C for Holding Times of 2 h and 4 h

Table 1. Intensity of the 3 Highest Peaks of SrTiO$_3$ at Various Temperature and Holding Time

| Samples | Intensity | 2 h | 4 h |
|---------|-----------|-----|-----|
| Temperature 800°C Plane (100) | 718 | 718 |
| Plane (200) | 266 | 273 |
| Plane (211) | 187 | 202 |
| Temperature 900°C Plane (100) | 1061 | 1112 |
| Plane (200) | 396 | 419 |
| Plane (211) | 257 | 267 |

Figure 2. (100) peaks broadening of SrTiO$_3$ with variation of sintering temperatures and holding times

According to the XRD peaks, the variation of the sintering temperatures and holding times does not change the phase of SrTiO$_3$. It is showed by the absence of the angles shift on the diffraction peaks of
each varied sample However, the change is apparent on the intensity of the diffraction peaks as shown in Table 1 and Figure 2. Table 1 exhibits the intensity values of the three highest peaks of the XRD patterns for SrTiO$_3$ with variations in temperature and holding time. It reveals that the higher sintering temperatures and longer holding time induce the higher intensity which indicates the better crystallinity of the samples. Besides, the temperature and holding times also affect the peak broadening as presented in Figure 2. The peak broadening indicates a change in the crystallite size and lattice strain of the SrTiO$_3$ materials exhibited by Figure 3. As the longer holding time, the crystallite size at 900°C expands while at 800°C it slightly shrinks. The lattice strain at 800°C decreases from 0.0835 to 0.0615 for the holding times of 2 h and 4 h, respectively. Meanwhile, at 900°C, it increases from 0.0872 to 0.0977 for the holding times of 2 h and 4 h, respectively.

**Figure 3.** Crystallite size and Lattice strain of SrTiO$_3$ with variation of sintering temperatures and holding times

**Figure 4.** FTIR spectra of SrTiO$_3$ with variation of sintering temperatures and holding times

Figure 5 displays the FTIR spectra of SrTiO$_3$ with variation of sintering temperatures and holding times on the absorption band of 400 – 4000 cm$^{-1}$. The wavenumber of 557 cm$^{-1}$ is included in the stretching vibration of the Sr-Ti-O bond [6]. In the wavenumbers 859.32 – 860.29 cm$^{-1}$; 2488.28 – 2490.21 cm$^{-1}$; and 3420.9 – 3422.83 cm$^{-1}$ exhibit O-H molecules. The absorption peak of 1771.69 – 1772.66 cm$^{-1}$ is due to the stretching vibration of C=O from the COOH group [5]. Based on
Figure 5, the sintering temperatures and holding times did not affect the wavenumber of each molecule significantly, but the transmittances or absorption values. It reveals that absorption peaks of O-H, C=O, and C-H reduce at higher temperature and longer holding time. Those molecules are the cause of the impurities production. Thus, it is in line with the XRD results that the higher temperature and longer holding time can reduce the impurities.

4. Conclusions
SrTiO$_3$ material has been synthesized using co-precipitation technique with variation of the sintering temperatures and holding times. According to the XRD results, the sintering temperatures and holding times did not affect the XRD peaks position but the intensity values and peak broadening. The change in both parameters could modify the crystallite size and lattice strain of the material. Moreover, based on the FTOR results, the all sample confirmed the SrTiO$_3$ material with presence of Sr-Ti-O chemical bonds. Besides, the absorption peaks of O-H, C-H, and C=O chemical bonds in the SrTiO$_3$ decreased as the higher temperature and longer holding time indicating impurities declined. Thus, in this study, the best parameter for fabricating SrTiO$_3$ powder was using the sintering temperature of 900$^\circ$C for 4 h.

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