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Analysis Lattice Constant of Ceramic SrTiO₃ Doped Cuprum(II) Acetate Cube Structure by Cramer-Cohen Method

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ABSTRACT

Analysis of SrTiO₃ ceramics with the doping Cuprum(II) acetate (Cu(CH₃COO)₂), the variation in the concentration of doping used 0%; 0,5%; and 1% using the solid state reaction method was successful. The annealing process is carried out starting from room temperature then raised to an annealing temperature of 850 °C with a temperature increase of 1,67 °C / minute and held constant for 8 hours. Next proceed with the cooling process until it returns to room temperature for 13 hours. Analysis of XRD data using the Cramer-Cohen method yielded a lattice parameter of 3,909 Å; 3,906 Å; and 3,905 Å, a cubic crystal structure.

INTRODUCTION

The last ten years ferroelectric materials are loved by many people, in contrast to the 60s to 70s which had few enthusiasts. Ferroelectric materials are unique materials because they have several properties in the form of ferrelectricity, conductivity and semiconductivity [1]. One example of a ferroelectric material is Strontium titanate (SrTiO₃). SrTiO₃ has a cubic perovskite structure and is one of the metal oxide materials. SrTiO₃ itself is widely used because it has unique chemical and physical properties [2]. Strontium titanate is one part of the perovskite compound material and has a cubic structure at room temperature. The detailed cubic structure consists of a small titanium atom at the center, a small strontium atom at each corner of the cube, and oxygen at each side (wall) of the cube [3]. There are several kinds of properties possessed by SrTiO₃, namely superconductivity, paraelectricity, and photocatalyst [4]. The concentration of doping in this study was varied into 3 variations, namely 0%; 0,5%; and 1%. Imprisonment was carried out to determine the crystal properties of Strontium titanate (SrTiO₃). Crystal properties tests are performed using XRD (X-ray Diffraction).

The process of integrating SrTiO₃ and Cu(CH₃COO)₂ uses the Solid State Reaction method and continues with the annealing stage. Solid State Reaction is a method of mixing materials by grinding them until they are homogeneous, which is then followed by compaction [5]. Grinding in this study will use mortar and paste. SrTiO₃ and Cu(CH₃COO)₂ powder that have been mixed will be compacted through the annealing process. The annealing process is carried out with high temperature followed by

cooling to room temperature and after the annealing process will proceed with XRD (X-ray Diffraction) characterization.

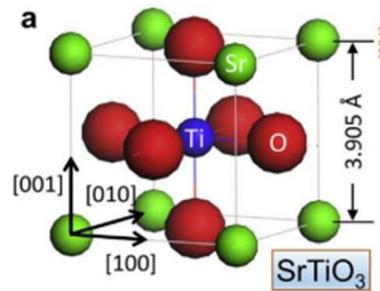


Fig 1. Crystal structure of material SrTiO₃ [6]

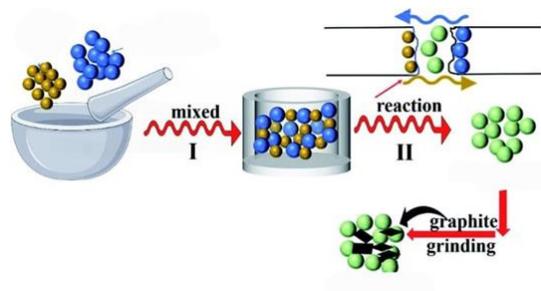


Fig 2. Solid state reaction [7]

Figure 1 shows the crystal structure of SrTiO₃ and Strontium titanate materials are widely used because they are one of the semiconductor materials that have good conductivity. In addition to its good conductivity, Strontium titanate material is also an environmentally friendly material and a more affordable price [8].

The addition of other elements to a material is called doping [9]. The doping used in this research is Cu(CH₃COO)₂. Cuprum is usually found in marine environments [10]. Doping that have been added result in some effect on the lattice structure of the material based on the ionic radius. Doping with a larger ion radius will cause the peak to shift to the left at a lower angle than the peak without the doping, while if the ion radius of the doping is smaller the peak will shift to the right with a lower angle than the peak without the doping [10].

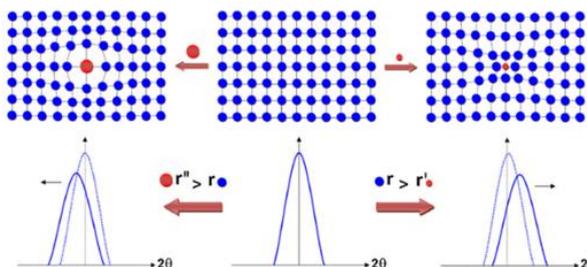


Fig 3. Effect of the size of the ion radius of the doping [10]

Figure 3 The effect of the size of the ion radius of the doping where the addition of ingredients in this study used Cuprum(II) acetate.

METHOD

Research is carried out by preparing all materials. The main ingredients used are Strontium titanate and Cuprum(II) acetate. The sample was varied with a doping concentration of 0%; 0,5%; and 1%. The mass of each sample used was set at 1,2 grams which was weighed using a digital scale. Making each sample by mixing the ingredients into one mortar which is then crushed or mixed using pestle until evenly distributed for 2 hours. The mashed material is then transferred in a small saucer and then closed. Table 1 shows the composition of the sample making material.

Table 1. The composition of the sample making material

Doping variation (%)	Strontium titanate (g)	Titanium oxide (g)	Cuprum(II) acetat (g)
0%	1,2	0	0
0,5%	1,1940	0,0060	0,0060
1%	1,1880	0,0120	0,0120

The next stage that must be done is the annealing stage. The annealing process in this research was carried out starting from the room temperature stage and rose gradually until it reached a temperature of 850 ° C. The temperature increase to reach a constant temperature in this annealing process is 1,67°C/min. The time required to reach a constant temperature is ± 8 hours, after which it will be held for 8 hours, then it will be cooled from 850°C to room temperature for ± 13 hours. This process of cooling to room temperature will be done manually. Apart from being used for compaction, annealing is also used to adjust optical parameters and constants [11] [12]. Figure 5 shows the annealing process which includes the heating and cooling stages.

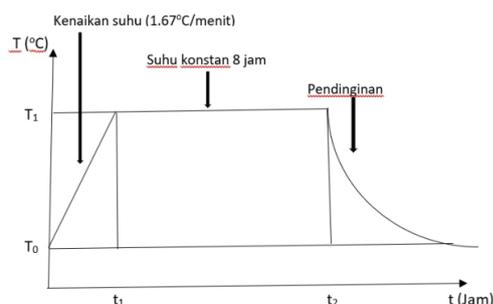


Fig 4. Annealing stage [13]

After the annealing process, the characterization process will continue. The test carried out is XRD (*X-ray Diffraction*). Analysis of a crystalline solid material can be done using XRD (*X-Ray Diffraction*) characterization [14]. XRD characterization is one of the oldest characterization methods. This technique is used to identify the crystalline phase in the material by determining the parameters of the lattice structure as well as obtaining the particle size. The X-rays produced in this tool come from the interaction between the external electron beam and electrons in the atomic shell. When the X-ray beam interacts with a material, it is possible that 3 things will occur, both absorption (absorption), diffraction (scattering), and fluorescence (re-transmitting X-rays with lower energy) [15] [16]. It is this characterization that will determine the shape of a crystal or material without doping and which uses doping.

RESULTS AND DISCUSSIONS

Testing of crystal properties using the Cramer-Cohen method is by XRD data analysis. This XRD data is used to determine the lattice parameters and crystal structure. The resulting XRD data will be

analyzed to produce a graph of the relationship between intensity and angle 2θ . The angle range of 2θ used in the XRD characterization process is 10° to 80° with a step of 0,02.

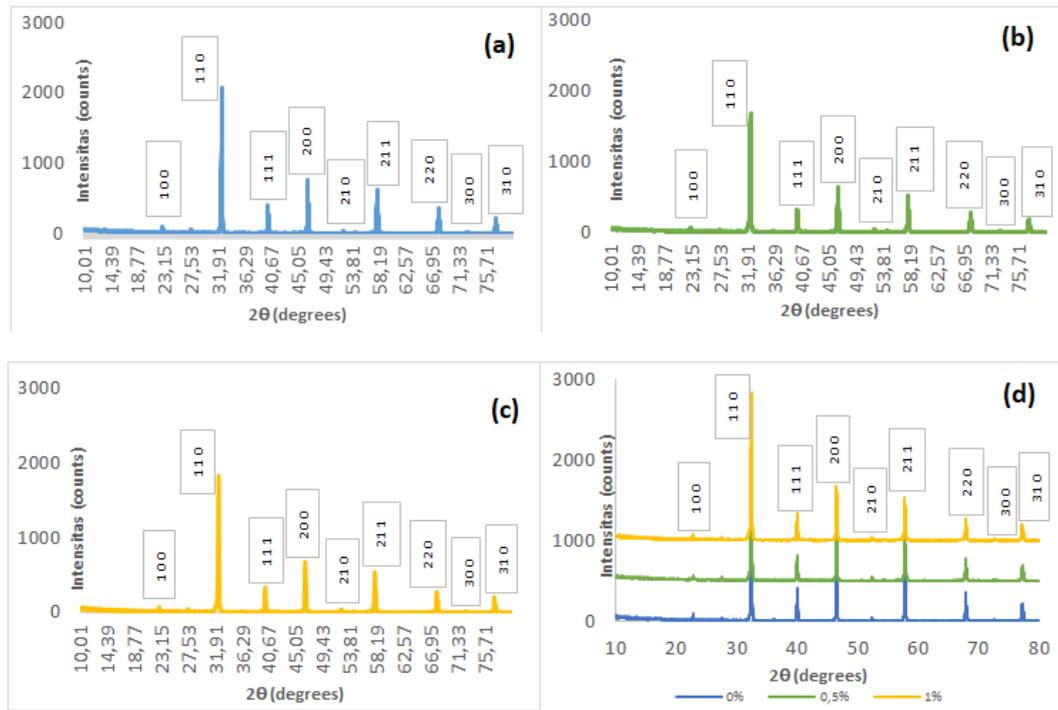


Fig 5. Relation of 2θ with $SrTiO_3$ (a) without doping, (b) with 0,5% doping, (c) with 1% doping, (d) comparison of graphs a,b, and c

Figure 5 shows that the Relation of 2θ with $SrTiO_3$ with and without doping $Cu(CH_3COO)_2$. The pattern of peaks formed can be determined miller index value (hkl), it is this value that can be used to determine the value of the lattice parameters of a crystal. The lattice parameters in this research were determined using the Cramer-Cohen method by equalizing the diffraction peaks obtained from characterization data with ICDD $SrTiO_3$ data to determine the hkl value. The hkl value obtained is used to calculate the grid parameters followed by equalizing the data with the grid parameter value data contained in ICDD data which aims to determine the effect of $Cu(CH_3COO)_2$ doping.

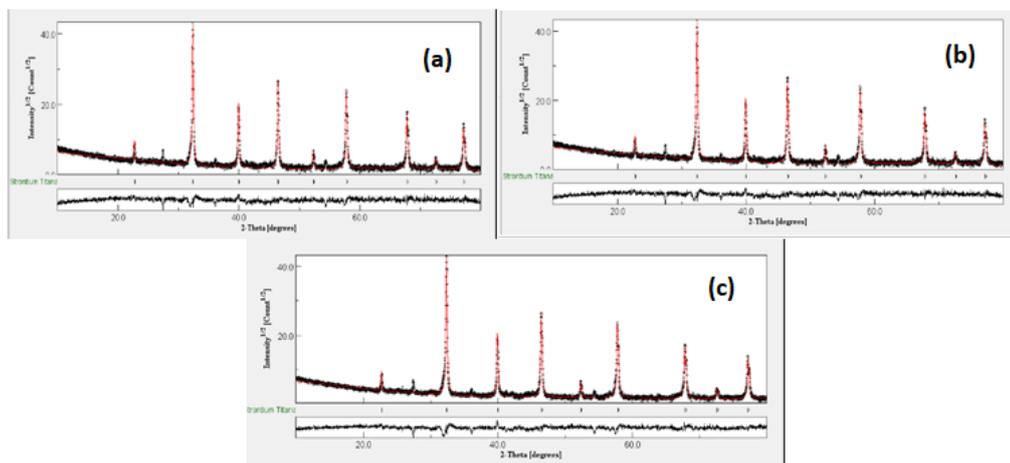


Fig 6. $SrTiO_3$ ceramic diffraction pattern using MAUD (a) without doping, (b) with 0,5% doping, (c) with 1% doping

Furthermore, the resulting XRD data will also be processed using MAUD software. MAUD software is used to determine the crystal size in each phase of SrTiO₃ using equations (1), (2), (3):

$$\Sigma\alpha\sin^2\theta = C\Sigma\alpha^2 + B\Sigma\alpha\gamma + A\Sigma\alpha\delta \quad (1)$$

$$\Sigma\gamma\sin^2\theta = C\Sigma\alpha\gamma + B\Sigma\gamma^2 + A\Sigma\gamma\delta \quad (2)$$

$$\Sigma\delta\sin^2\theta = C\Sigma\alpha\delta + B\Sigma\gamma\delta + A\Sigma\delta^2 \quad (3)$$

Table 2. Parameter of SrTiO₃ ceramic lattice doping Cu(CH₃COO)₂ (Cramer-Cohen and MAUD)

Doping variation (%)	Cramer-Cohen lattice parameters	ICDD literature (Å)	MAUD lattice parameters
	a	A	A
SrTiO ₃ with doping	3,909	3,905	3,906
SrTiO ₃ + Cu(CH ₃ COO) ₂ 0,5%	3,906	-	3,906
SrTiO ₃ + Cu(CH ₃ COO) ₂ 1%	3,905	-	3,905

Table 2 shows the parameters of cubic structured SrTiO₃ ceramic lattice doping Cu(CH₃COO)₂. Based on the data of table 2 above, the parameters of the SrTiO₃ ceramic lattice experience several conditions. The parameters of the SrTiO₃ ceramic lattice by the Cramer-Cohen method decreased when the sample was added with the doping Cu(CH₃COO)₂. This can occur because the radius of the Cu²⁺ ion is smaller than that of the Sr²⁺ host so that the shape of the structure will contract. The results of calculations using the Cramer-Cohen method are close to the values of lattice parameters in the ICDD literature. The grid parameters produced by the MAUD software were also close to the ICDD literature values and decreased, but there was no significant difference in the samples carried out. This can happen because of the small concentration of drugs and the small difference between the two, in addition to the possibility of MAUD software in the iteration process that does not run smoothly.

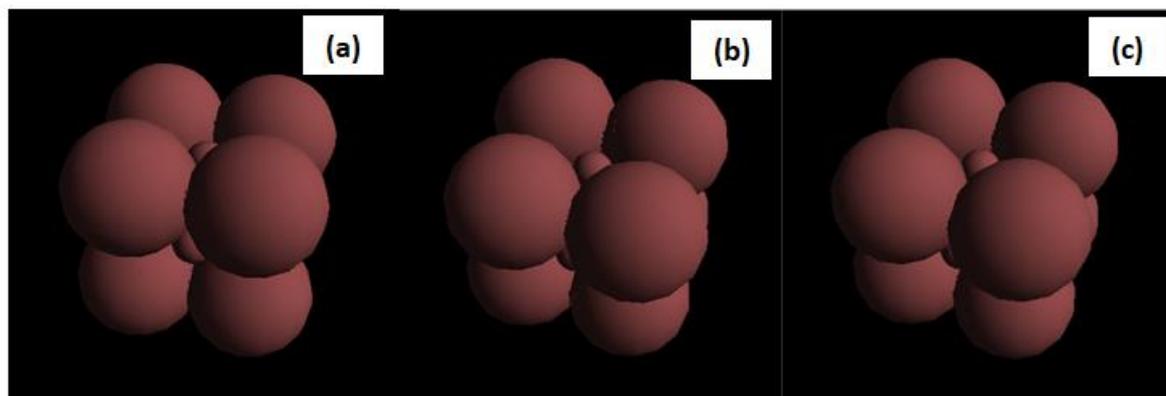


Fig 7. SrTiO₃ ceramic structure using MAUD (a) without doping, (b) with 0,5% doping, (c) with 1% doping

Figure 7 shows the shape of the ceramic structure SrTiO₃ with the doping Cu(CH₃COO)₂ displayed by MAUD software corresponding to Shi et al [6] i.e. it has a cubic structure.

CONCLUSION AND SUGGESTION

Analysis of crystalline properties of ceramic SrTiO₃ doping Cu(CH₃COO)₂ with doping variation 0%; 0,5%; And 1% it can be concluded that the process of making ceramics can be done by the solid state

reaction method. The addition of $\text{Cu}(\text{CH}_3\text{COO})_2$ doping results in lattice parameter values that shrink as the concentration of the doping increases, this happens because the size of the ionic radius of Sr^{2+} is larger than the ionic radius of Cu^{2+} . The results of XRD analysis show the structure of SrTiO_3 in cubic form.

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