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Structural Analysis of CaLaAlO_4 Powder via Solid State Method

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Abstract: We report here a single phased Calcium Lanthanum Aluminate (CLA) tetragonal perovskite type structure polycrystalline successfully prepared by solid state reaction method at different calcination temperatures. The phase purity and structural analysis investigated by PXRD. The PXRD pattern of CLA calcination at 1200 °C well matched with tetragonal perovskite structure. We calculated the average crystalline size 201 nm from debye-scherrer and 197 nm from William-Hall method. The morphology studies were analysed by SEM and the approximate particle size 160 nm. With this analysis we confirmed that the CaLaAlO_4 powder is single phased tetragonal structure calcination at 1200 °C. The CaLaAlO_4 polycrystalline material was first time prepared by solid-state reaction method.

Key words: solid-state reaction, Debye-Scherrer, W-H method, CaLaAlO_4

I. INTRODUCTION

Recently, a significant research on tetragonal perovskite type structured oxide materials have been investigated widely. Calcium Lanthanum Aluminate (CLA) has a tetragonal K_2NiF_4 structure and belongs to a family of compounds with the general formula XYZO_4 , where X is an alkaline earth cation, Y is La, Sc, or a trivalent rare-earth element and Z is Al, Ga or a transition metal ion. Its crystal structure consists of interleaved layers with a perovskite and rock-salt structure. With the formula of XYZO_4 , they have chemical stability and non-toxic, and can be used as basis material for lasers and phosphors. XYZO_4 are auspicious materials for electronic, optical and structural utilizations and they provide benefits of polarised cross sections and high lifetimes correlated to other oxide ceramics.

This ceramic material extensively used as host material for luminescent phosphors, solid lasers. They are also used for significant substrates for an epitaxial growth of films being piezoelectrics, ferroelectrics and elevated temperature superconductors [1-4]. Perovskite type structured XYZO_4 is effective material for laser or optical associated utilizations. The XYZO_4 perovskite with a giant transparency and large energy band gap, they have large surface area and catalytic activity due to this it can also used as catalyst for oxidative coupling of methane [5-6].

Yang Zhang et al., has reported the synthesis of a CaYAlO_4 at 1400 °C for 6h. by solid-state reaction method [7]. Myung Sub Kim et al., synthesized CaYAlO_4 by sol-gel method calcination at 1000 °C for 5h [8]. Yang Zhang et al., synthesized CaGdAlO_4 by high temperature solid-state reaction method at 1200 °C for 5h [9]. Xiang Nan Chen et al., synthesized SrLaAlO_4 by co-precipitation method with pH=9 calcination at 1000 °C for 5 h [10]. Xiaoyan Fu et al., prepared SrLaGaO_4 at 1300 °C for 4 h by solid state reaction method [11]. A. Novoselov et al., prepared the CaLaAlO_4 single crystal by the Czochralski method [12]. From the literature and best of our knowledge yet no one prepared the CaLaAlO_4 polycrystalline with tetragonal perovskite type structure material. Above all mentioned methods are previously reported methods for preparation of XYZO_4 materials, we were adopted solid state reaction method from the above literature for preparing CaLaAlO_4 .

The solid state reaction method is good method for preparing single phase homogeneity, smaller particle size and uniform morphology for commercial production. Herein, solid state reaction method has been suggested for synthesizing nearly mono-disperse CaLaAlO_4 polycrystalline material.

In this present work we demonstrate the synthesis of pure CaLaAlO_4 single phase poly crystalline materials by solid state reaction method in muffle furnace at high temperature and investigated their structural and morphological properties.

II. EXPERIMENTAL DETAILS

The CaLaAlO_4 polycrystalline powder was prepared by a conventional solid state reaction technique at high temperature. The raw materials CaCO_3 (Analytical Reagent, A.R.), La_2O_3 (99.9 %), Al_2O_3 (A.R.) and Eu_2O_3 (99.9 %) were used as an initial compounds. A stoichiometric quantity of an initial compounds were homogeneously assimilated by an agate mortar and grinded with a pestle for about 30 min. the homogeneous mixture was put into a porcelain crucible and calcination at 1000 °C, 1100 °C and 1200 °C for 5 h.

III. CHARACTERIZATION TECHNIQUES

The crystal structure and phase formation of the CaLaAlO₄ was determined using of powder X-ray diffraction (PXRD) with 10⁰ min⁻¹ scanning rate. The 2θ range in between 20⁰ to 80⁰ with Cu-Kα radiation (λ=1.5405Å) with a Bruker D8 advance PXRD. The exterior morphological nature and particle size of the material were analyzed using Philips XL30 ESEM and JEOL JSM840 electron microscope.

IV. RESULTS AND DISCUSSION

Fig.1. shows the PXRD pattern of CLA calcination at 1000 °C, 1100 °C and 1200 °C for 5 h. Calcination at 1000 °C and 1100 °C for 5 h PXRD pattern not well matched with CLA diffraction data and these patterns consists of multi phases of CLA. The above results were due to uncompleted oxide reactions of initial compounds. Calcination at 1200 °C for 5 h powder shows diffraction peaks were well matched with JCPDS: 24-0206 and the mixed phase peaks were disappeared at this temperature and it indicates the pure crystallinity of the material and without any supplementary peaks and this promise pure tetragonal phase of CLA.

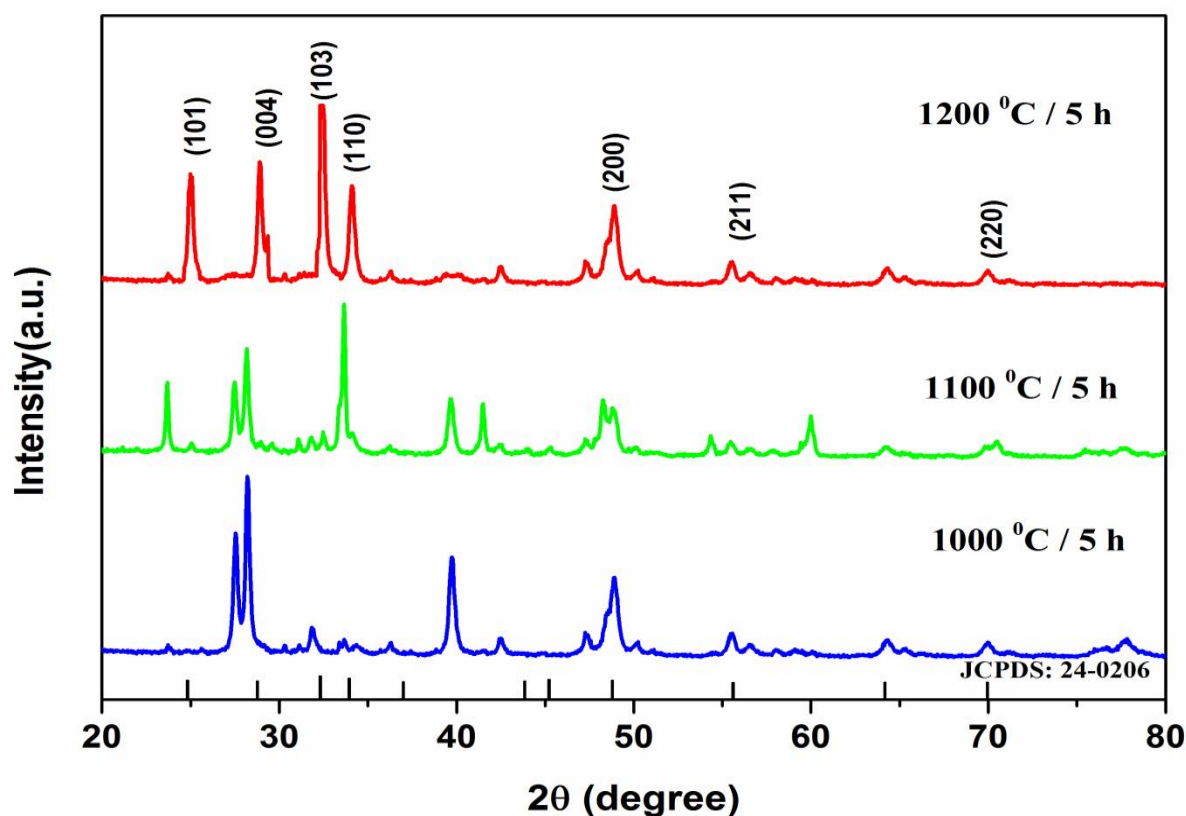


Fig. 1. PXRD pattern of CLA calcination at different temperatures.

The space group I4/mmm (139) and its lattice parameter values are a=b=3.716 Å, c=12.33 Å and α=β=γ= 90°. The average crystallite size (D) is calculated from Debye-Scherrer formula given in Eq.1 [13].

$$D_{hkl} = k\lambda / [\beta(2\theta) \cos \theta] \quad (1)$$

Where k is spherical particles constant (0.89), λ is the X-ray wavelength (1.5405Å), θ and β are the diffraction angle and full-width at half maximum (FWHM, in radian) of an observed peak, respectively. The strongest five peaks (1 0 1) at 2θ=2.9497⁰, (0 0 4) at 2θ=28.9097⁰, (1 0 3) at 2θ=32.5122⁰, (1 1 0) at 2θ=34.12308⁰ and (2 0 0) at 2θ=48.9147⁰ were used to determine the average crystallite size (D) of the CLA calcination at 1200 °C for 5 h sample. The approximate mean crystallite size is close to 201 nm.

The strain induced in powders due to crystal imperfection and distortion was calculated using the formula:

$$\epsilon = \frac{\beta_{hkl}}{4 \tan \theta} \tag{2}$$

From Equations 1 and 2, it was confirmed that the peak width from crystallite size varies as $1/\cos\theta$ and the strain varies as $\tan\theta$. Assuming that the particle size and strain contributions to line broadening are independent to each other and both have a Cauchy-like profile, the observed line breadth is simply the sum of Equations 1 and 2.

$$\beta_{hkl} = \frac{k\lambda}{D \cos\theta} + 4\epsilon \tan \theta \tag{3}$$

By rearranging the above equation, we get

$$\beta_{hkl} \cos\theta = \frac{k\lambda}{D} + 4\epsilon \sin \theta \tag{4}$$

The above equations are W-H equations [14]. A plot is drawn with $4\epsilon \sin\theta$ along the x-axis and $\beta_{hkl} \cos\theta$ along the y-axis for as-prepared CLA calcination 1200°C as shown in Figure 2. From the linear fit to the data, the crystalline size was estimated from the y-intercept, and the strain ϵ , from the slope of the fitting.

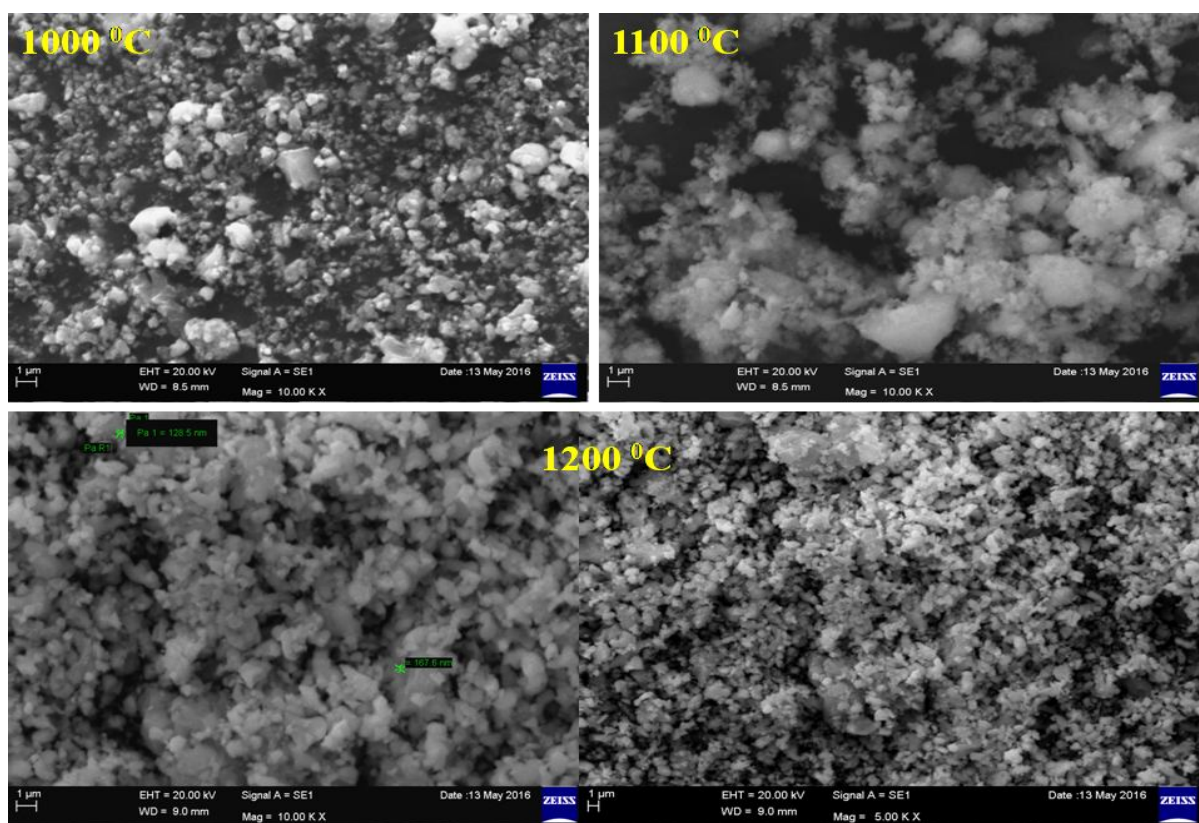


Fig.2. Plot of $\beta_{hkl} \cos\theta$ vs $4\epsilon \sin\theta$ of CLA calcination at 1200°C for 5 h.

Equation 4 represents the uniform deformation model (UDM), where the strain was assumed to be uniform in all crystallographic directions, thus considering the isotropic nature of the crystal, where the material properties are independent of the direction along which they are measured. The uniform deformation model for CLA is shown in fig.2. The calculated crystalline size from W-H plot method was 197 nm and the slope value is negative $2.01878\text{E-}4$ due to compressive strain. The average crystalline size is smaller in W-H method compare to Debye-Scherrer, this is due to compressive strain is considered in the W-H method.

Fig.3. Shows the SEM micrographs of CLA calcination at 1000°C , 1100°C and 1200°C for 5 h. The CLA calcination at 1000°C appeared the non-uniform morphology and agglomerated particle nature of the material. Calcination at 1100°C for 5 h. CLA sample micrographs were indicates the non-uniformly morphology nature but some better uniformity nature observed compared to previous one. Calcination at 1200°C for 5 h. CLA micrographs were well uniformly dense morphology nature observed and it appears non-agglomerated spherical like particle nature and the particle size is approximately 160 nm.

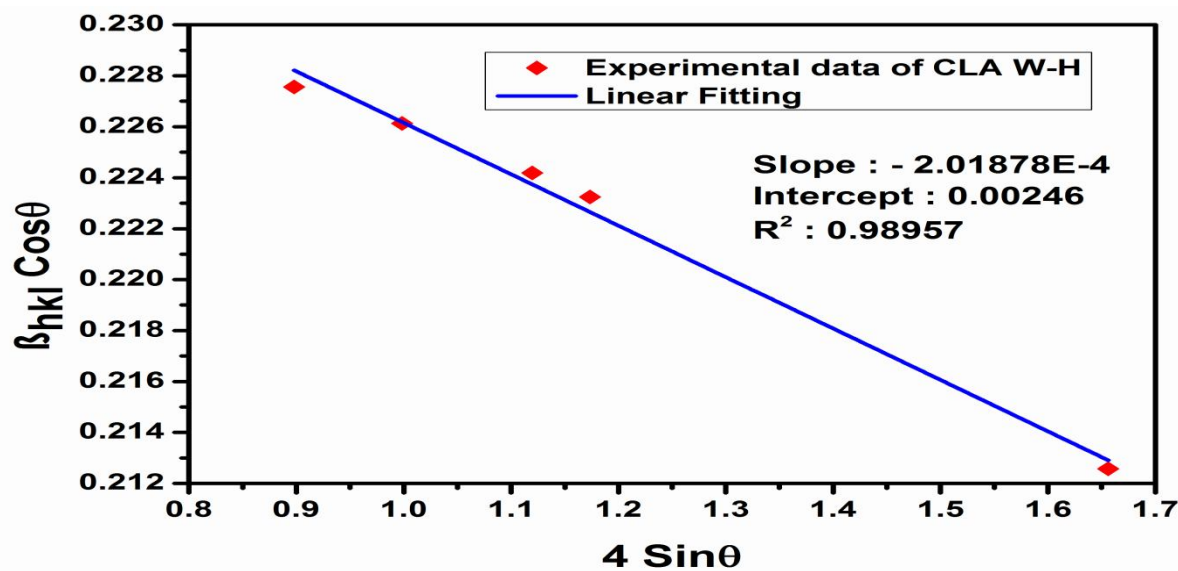


Fig.3. SEM micrographs of CLA calcination at different temperatures.

From these SEM micrographs calcination at 1200 °C for 5 h. of CLA samples have better uniform spherical like morphology compared to remaining samples and it supports the PXRD characterization results.

V. CONCLUSION

In summary, the single phased CaLaAlO₄ were auspiciously prepared by conventional solid-state reaction method calcination at different temperatures. PXRD patterns confirmed that, well crystallinity and pure tetragonal phase perovskite with rock-salt structured CaLaAlO₄ calcination at 1200 °C for 5 h. The average crystalline size 201 nm is calculated by using a Debye-Scherrer method. The average crystallinity size 197 nm and the compressive strain -2.01878E-4 calculated from William-Hall method. The closely packed particles with uniform agglomerated particles and uniform spherical like shaped particles nature were confirmed by SEM pictures calcination at 1100 °C and 1200 °C respectively. The approximate particle size 160 nm clarify from SEM micrographs calcination at 1200 °C. The CaLaAlO₄ tetragonal phase perovskite with rock-salt structured polycrystalline material were first time prepared by solid state reaction method.

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