

## Hydrothermal Synthesis of Superfine Strontium Titanate Particles†

QIAN QIAN<sup>1</sup>, YIN LIU<sup>1,\*</sup>, CHEN CHEN<sup>1</sup>, PEILIANG ZHANG<sup>1</sup>, BAOXIANG JIAO<sup>2</sup> and GUIHUA HOU<sup>2</sup>

<sup>1</sup>School of Materials Science and Engineering, Anhui University of Science and Technology, Huainan 232001, Anhui Province, P.R. China

<sup>2</sup>School of Materials Science and Engineering, Yancheng Institute of Technology, Yancheng 224051, Jiangsu Province, P.R. China

\*Corresponding author: E-mail: [yinliu@aust.edu.cn](mailto:yinliu@aust.edu.cn)

Published online: 10 March 2014;

AJC-14853

The superfine strontium titanate ( $\text{SrTiO}_3$ ) particles was successfully synthesized by hydrothermal method using  $\text{KTiO}(\text{C}_2\text{O}_4)_2 \cdot 3\text{H}_2\text{O}$ ,  $\text{Sr}(\text{NO}_3)_2$  as raw materials and  $\text{NaOH}$  as mineralizer. The as-prepared samples were examined by X-ray powder diffraction for crystalline phase identification and scanning electron microscopy for particles morphology. The results show that the single phase of  $\text{SrTiO}_3$  could be obtained at 200 °C for 12 h as the molar ratio of raw materials and mineralizer was 1:6. The hydrothermal temperature was the main reason which affected the crystal growth rate.

**Keywords:** Strontium titanate, Microstructure, Hydrothermal method.

### INTRODUCTION

With development of science and technology, the new multifunctional materials get more attention of people. As a kind of new typical multi-functional materials, strontium titanate ( $\text{SrTiO}_3$ ) with perovskite structure has special physical and chemical properties. Because it has high dielectric constant, low dielectric loss, high dispersion rate and well thermal stability, it could be widely used as random access memory devices, oxygen sensors actuators and grain boundary layer and high voltage capacitor materials. In addition, the band gap of strontium titanate is 3.2 eV<sup>1</sup>, which is similar to that of  $\text{TiO}_2$ , determining that it is one promising photocatalyst candidates for photocatalytic degradation of organic pollutants, photocatalytic decomposition of water to producing hydrogen and photocatalytic battery energy storage materials<sup>1-6</sup>. All these applications of strontium titanate have attracted extensive attentions on studying its fundamental theories and practical applications.

There are many methods to synthesize strontium titanate powder, including conventional solid state reaction method<sup>7-9</sup>, molten salt method<sup>10,11</sup>, sol-gel method<sup>12,13</sup>, chemical precipitation method<sup>14</sup> and hydrothermal method<sup>15</sup>. For conventional solid state reaction method, a single phase  $\text{SrTiO}_3$  could be obtained *via* prolonging the calcining time and raising the temperature above 1000 °C. The disadvantage of this approach is often associated with particle agglomeration and wrapped

impurity phase. The purity submicron powder of  $\text{SrTiO}_3$  could be synthesized by molten salt method and the reaction temperature is still high. Compared with these methods, the highly pure  $\text{SrTiO}_3$  with fine particle size and low impurity levels could be obtained by the chemical synthesis processes, such as sol-gel method, chemical precipitation method, whereas its disadvantages were complicated processing steps, high cost and high temperature. In the present work, the superfine  $\text{SrTiO}_3$  particle was successfully synthesized by hydrothermal method under 200 °C for 12 h and investigated the association between crystal growth of  $\text{SrTiO}_3$  and the processing conditions.

### EXPERIMENTAL

The superfine  $\text{SrTiO}_3$  particle was prepared by using hydrothermal method. The raw materials used in this study were  $\text{KTiO}(\text{C}_2\text{O}_4)_2 \cdot 3\text{H}_2\text{O}$ ,  $\text{Sr}(\text{NO}_3)_2$  and  $\text{NaOH}$  were used as mineralizer. All chemicals were analytical grade purity and used as received without further purification. Firstly, the raw materials were weighted according to stoichiometric proportion and mixed with mineralizer as the molar ratio of 1:(1-6) in the dried 40 mL Teflon-bladder with stirring homogeneously. Secondly, the 25 mL deionized water was added into the mixture compound with stirring 0.5 h. Then, the Teflon-bladder with mixed solution was sealed in stainless vessel. The Teflon-lined stainless autoclave was heated up from room temperature to target temperatures and thermal insulation for different time.

†Presented at The 7th International Conference on Multi-functional Materials and Applications, held on 22-24 November 2013, Anhui University of Science & Technology, Huainan, Anhui Province, P.R. China

Finally, The product was washed with deionized water to remove the impurity (such as NaOH,  $\text{KNO}_3$ ) and dried overnight at 80 °C.

The crystallinity of as-prepared samples was examined by an X-ray diffraction spectrometer (XRD, Shimadzu LabX XRD-600) using  $\text{CuK}\alpha$  radiation. The microstructure was observed through scanning electron microscopy (Hitachi, S-3000N).

## RESULTS AND DISCUSSION

**Characterization of the powders:** Fig. 1 shows the XRD patterns of  $\text{SrTiO}_3$  by using different dosage of mineralization agent. As showed in Fig. 1a, all the diffraction peaks of the as-prepared samples which were calcined at 220 °C for 72 h by controlling the molar ratio of raw materials and mineralizer as 1:1 are consistent with all  $\text{SrCO}_3$  and  $\text{TiO}_2$  (+), wherein the peaks of  $\text{TiO}_2$  are partially overlapping with the diffraction peaks of  $\text{SrCO}_3$ . Increasing the raw materials and mineralizer as 1:4 at 200 °C for 72 h, the most peaks corresponding to the characteristic peak of  $\text{SrTiO}_3$  were showed in the Fig. 1b, but there are some peaks of impurity (#:sodium titanate). When the ratio of raw materials and mineralizer was up to 1:6, all the peaks were identified with the characteristic peak  $\text{SrTiO}_3$  (JCPDS NO.35-734). There were always the purity phase of  $\text{SrTiO}_3$  by increasing the dosage of mineralizer in a shorter time.

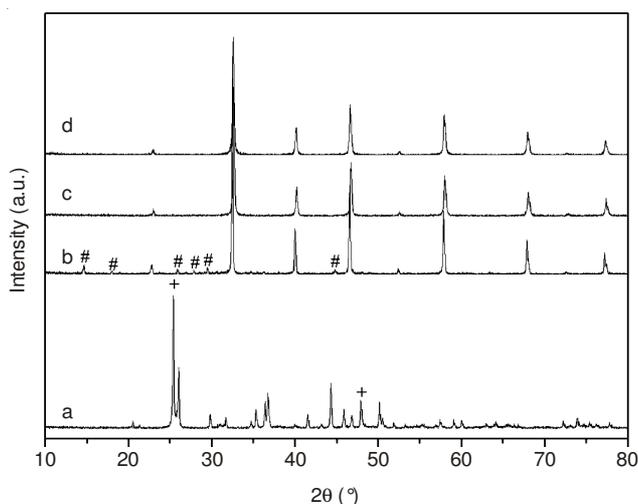


Fig. 1. XRD pattern of  $\text{SrTiO}_3$  by using different dosages of mineralizer (a: 220 °C, 72 h, 1:1, b: 200 °C, 72 h, 1:4, c: 200 °C, 72 h, 1:6, d: 200 °C, 36 h, 1:7)

Fig. 2 showed the XRD pattern of  $\text{SrTiO}_3$  synthesized by different temperatures at the condition of raw materials and mineralizer as 1:6. As showed in Fig. 2a, there are some peaks of the transitional product of  $\text{Sr}_2\text{TiO}_4$  (#). When the hydrothermal temperature was 200 °C, all the peaks of the as-prepared sample were corresponding with the characteristic peaks of the purity  $\text{SrTiO}_3$  (Fig. 2a). There were still the purity single phase  $\text{SrTiO}_3$  when the temperature was up to 220 °C.

**Microstructure of  $\text{SrTiO}_3$  powder:** Fig. 3(a-i) shows the SEM micrographs of  $\text{SrTiO}_3$  calcined at different temperatures. Fig. 3(a/d/g) showed  $\text{SrTiO}_3$  particles with hard agglomeration. It may due to superfine particles generally possessing large

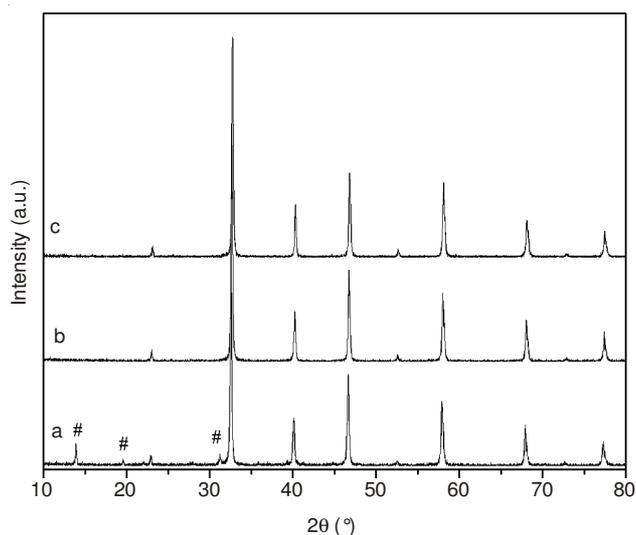


Fig. 2. XRD pattern of  $\text{SrTiO}_3$  synthesized under different temperatures (a: 180 °C, 72 h, 1:6, b: 200 °C, 72 h, 1:6, c: 220 °C, 36 h, 1:6)

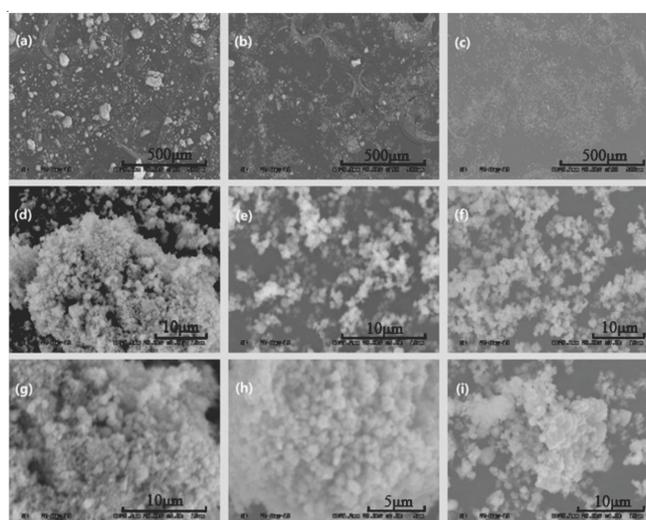


Fig. 3. SEM images of  $\text{SrTiO}_3$  prepared by different conditions (a,d,g: 180 °C/72 h/1:6, b, e, h: 200 °C/36 h/1:7, c,f,i: 220 °C/36 h/1:6)

surface and tend to aggregate into larger particles to reduce their high surface energy. The sample has slightly aggregated, which was synthesized at 200 °C as showing Fig. 3(b/e/h) and the shape of those particles was always spherical. Fig. 3(c/f/i) showed that the particles were mainly spherical one and a few cubic one (Fig. 3(i)). It may due to the crystal grown up larger with the temperature increasing.

## Conclusion

The pure phase cubic perovskite structure superfine  $\text{SrTiO}_3$  powders were synthesized by hydrothermal method using  $\text{KTiO}(\text{C}_2\text{O}_4)_2 \cdot 3\text{H}_2\text{O}$ ,  $\text{Sr}(\text{NO}_3)_2$  as raw materials and NaOH as mineralizer. The amount of mineralizing agent determines whether strontium titanate is synthesized. According to XRD, the particle size increases with the temperature increasing and extension of the time and the main impact factor is the hydrothermal temperature. The SEM observation shows that the  $\text{SrTiO}_3$  powder is uniform and regularly spheres below 200 °C, while it slowly turns to cubes when the temperature reached to 220 °C.

**ACKNOWLEDGEMENTS**

This work was supported by National Nature Science Foundation of China (51174006), Anhui Provincial Natural Science Foundation, China (1208085ME84), the Project-sponsored by SRF for ROCS, SEM) and the Opening Project of Henan Key Discipline Open Laboratory of Mining Engineering Materials (Grant No. MEM20126).

**REFERENCES**

1. C.X. Yang, T.Y. Liu, Z.J. Cheng, H.X. Gan and J.Y. Chen, *Physica B*, **407**, 844 (2012).
2. P. Blennow, K.K. Hansen, L.R. Wallenberg and M. Mogensen, *J. Eur. Ceram. Soc.*, **27**, 3609 (2007).
3. C.-A. Chang, B. Ray, D.K. Paul, D. Demydov and K.J. Klabunde, *J. Mol. Catal. Chem.*, **281**, 99 (2008).
4. H.W. Kang, S.N. Lim, D. Song and S.B. Park, *Int. J. Hydrogen Energy*, **37**, 11602 (2012).
5. F. Zou, Z. Jiang, X.Q. Qin, Y.X. Zhao, L.Y. Jiang, J.F. Zhi, T.C. Xiao and P.P. Edwards, *Chem. Commun.*, **48**, 8514 (2012).
6. B.R. Sudireddy, P. Blennow and K.A. Nielsen, *Solid State Ion.*, **216**, 44 (2012).
7. X. Li, H.L. Zhao, F. Gao, N. Chen and N.S. Xu, *Electrochem. Commun.*, **10**, 1567 (2008).
8. X. Li, H.L. Zhao, W. Shen, F. Gao, X.L. Huang, Y. Li and Z.M. Zhu, *J. Power Sources*, **166**, 47 (2007).
9. X. Li, H. Zhao, X. Zhou, N. Xu, Z. Xie and N. Chen, *Int. J. Hydrogen Energy*, **35**, 7913 (2010).
10. H.-L. Li, Z.-N. Du, G.-L. Wang and Y.-C. Zhang, *Mater. Lett.*, **64**, 431 (2010).
11. Y.-Liu, Y. Lu, M. Xu, L. Zhou and S. Shi, *Mater. Chem. Phys.*, **114**, 37 (2009).
12. H. Yu, S.X. Ouyang, S.C. Yan, Z.S. Li, T. Yu and Z.G. Zou, *J. Mater. Chem.*, **21**, 11347 (2011).
13. T. Puangpetch, T. Sreethawong, S. Yoshikawa and S. Chavadej, *J. Mol. Catal. Chem.*, **287**, 70 (2008).
14. Y.H. Chen and Y.D. Chen, *J. Hazard. Mater.*, **185**, 168 (2011).
15. A. Huang, A.D. Handoko, G.K.L. Goh, S.R. Shannigrahi and C.K. Tan, *Progr. Cryst. Growth Charact. Mater.*, **57**, 109 (2011).