

# Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> Synthesis with High Specific Surface Area and Single Phase

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## Abstract

We have investigated a novel Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> synthesis with high specific surface area, high crystallization and single phase and its mechanism. The method was performed with a solid phase synthesis by using CH<sub>3</sub>COOLi·2H<sub>2</sub>O and anatase TiO<sub>2</sub> via Li<sub>2</sub>TiO<sub>3</sub> as an intermediate in pre-sintering at 500°C and sintering at 750°C. This result showed specific surface area of 12 m<sup>2</sup>/g and single phase-Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> by applying the high specific surface anatase TiO<sub>2</sub> as a precursor.

## Keywords

Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub>, Solid Phase Synthesis, Specific Surface Area, SEM, TEM

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## 1. Introduction

A negative electrode in lithium ion secondary battery has applied C<sub>6</sub> graphite to intercalate lithium ions. The graphite expands by overcharge to intercalate them more than stoichiometry. To solve the expansion, materials with keeping a crystal structure have been developed [1]. Hence, Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> consisting of a spinel structure has been expected for not changing the structure or size by intercalating the lithium ion [2] [3]. Furthermore, it is superior to the graphite in stability of charge capacitance under a high temperature or fast charge-discharge cycle [4]-[6]. On the other hand, an electrical conductivity is low like an insulator, and effects of a carbon doping or metal ions by adding V<sup>5+</sup>, Mn<sup>4+</sup>, Fe<sup>3+</sup>, Mg<sup>2+</sup> and Ag<sup>+</sup> have been investigated for the higher conductivity [7]-[14]. Especially the carbon doping has been available, and attempted by a sol-gel or spray-dry method in a liquid phase [15]-[17].

Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> synthesis methods have been known as solid phase, sol-gel, hydrothermal or co-precipitation method. The solid phase method has surpassed others in industrialization to be easy and controllable for the synthe-

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sis, while it is difficult to obtain homogeneous grain size. The small grain size needs lower sintering temperature, but the higher crystallization requires the higher sintering temperature. Furthermore, the solid phase method has been studied for the improvement in these points [18] [19].

$\text{Li}_4\text{Ti}_5\text{O}_{12}$  for the negative electrode requires high crystallization, single phase, small grain size and high specific surface area to perform an excellent property. Especially, the high specific surface area is important to intercalate many lithium ions into the crystal efficiently. We have investigated the synthesis method for the high specific surface area with  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  of the completely single phase by the solid phase reaction. In this study, we attempted the synthesis by applying anatase  $\text{TiO}_2$  of the different specific surface area via a pre-sintering.

## 2. Experimental

$\text{Li}_4\text{Ti}_5\text{O}_{12}$  synthesis was carried out by using  $\text{CH}_3\text{COOLi}\cdot 2\text{H}_2\text{O}$  (Wako Pure Chemical Industries, Ltd.) as a Li source that was melted with low temperature at about  $300^\circ\text{C}$ , and 30 or  $50\text{ m}^2/\text{g}$  anatase  $\text{TiO}_2$  (Toho Titanium Co., Ltd.) as a Ti source. The first synthesis process was mixed with the  $\text{CH}_3\text{COOLi}\cdot 2\text{H}_2\text{O}$  and  $\text{TiO}_2$  with Li:Ti = 4:5 by ball milling (Fritsch, Pulversitte 7) at 1 h, rotating speed at 320 rpm and orbital speed at 110 rpm in agate mortar and balls. The mixed powder was pre-sintering at  $500^\circ\text{C}$  with  $10^\circ\text{C}/\text{min}$ , 1 h in air, and an intermediate was formed. Furthermore, the intermediate was mixed by the ball milling for rotating speed at 320 rpm and orbital speed at 110 rpm at 1 h similarly, and was sintered at  $700^\circ\text{C}$  and  $750^\circ\text{C}$  with  $10^\circ\text{C}/\text{min}$  at 1 h in air.

The obtained powder was identified by XRD (Rigaku Corp., Rint 2000) at scanning step 0.02 deg and scanning speed 5 deg/min by  $\text{CuK}\alpha$ , and was also measured by BET specific surface area (Shimadzu Corp., Flow-Sorb III 2305) at 0.1 g sample, gas flow rate  $80\text{ cm}^3/\text{min}$  in  $\text{N}_2$  and current 50 mA, after degassing the sample with heating at  $160^\circ\text{C}$  at 2 h. Particle size of the obtained sample was observed by FE-SEM (Hitachi, Ltd., S-4200), and crystalline estimation tried with TEM (JEOL Ltd., JEM-2100).

## 3. Results and Discussion

### 3.1. XRD Measurements

We yielded the intermediate by pre-sintering a mixture of  $\text{CH}_3\text{COOLi}\cdot 2\text{H}_2\text{O}$  and anatase  $\text{TiO}_2$  at  $500^\circ\text{C}$ , and showed XRD patterns as (a); a green line in **Figure 1(A)** and **Figure 1(B)** by applying the  $30\text{ m}^2/\text{g}$  and  $50\text{ m}^2/\text{g}$  anatase  $\text{TiO}_2$  respectively. The intermediate was identified with unreacted  $\text{TiO}_2$  and  $\text{Li}_2\text{TiO}_3$  known as a sub-phase of  $\text{Li}_4\text{Ti}_5\text{O}_{12}$ .  $\text{Li}_2\text{TiO}_3$  has been understood by a phase diagram of  $\text{Li}_2\text{O}\text{-TiO}_2$  among  $\text{TiO}_2$ ,  $\text{Li}_2\text{TiO}_3$  and  $\text{Li}_4\text{Ti}_5\text{O}_{12}$ , which shows an accurate mixed rate at Li and Ti atoms [20]. A Ti site of  $\text{Li}_2\text{TiO}_3$  and  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  is common in center of an octahedron, while a Li site exists in a tetrahedron in the case of  $\text{Li}_2\text{TiO}_3$ .  $\text{Li}_2\text{TiO}_3$  show inactivity as the electrode not to charge and discharge Li ions [21].

To obtain  $\text{Li}_4\text{Ti}_5\text{O}_{12}$ , the intermediate was sintered at  $700^\circ\text{C}$ , and the identification was shown as (b); a red line in the XRD patterns, which obtained main  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  peaks and slight peaks of  $\text{Li}_2\text{TiO}_3$ , and was not able to confirm the unreacted  $\text{TiO}_2$ . This result is not enough to obtain a single phase  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  at the sintering temperature although all unreacted  $\text{TiO}_2$  is consumed. Then, effect of the specific surface area by applying the  $30\text{ m}^2/\text{g}$  or  $50\text{ m}^2/\text{g}$  anatase  $\text{TiO}_2$  was not confirmed in **Figure 1(A)** and **Figure 1(B)**. In the sintering temperature at  $750^\circ\text{C}$ , the single phase  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  was obtained by applying either specific surface area of the anatase  $\text{TiO}_2$  as shown in (c); a blue line in the patterns. On the other hand, the peak intensity was strong by applying the anatase  $\text{TiO}_2$  of  $50\text{ m}^2/\text{g}$  than that of  $30\text{ m}^2/\text{g}$ , which would be superior to the crystallization.

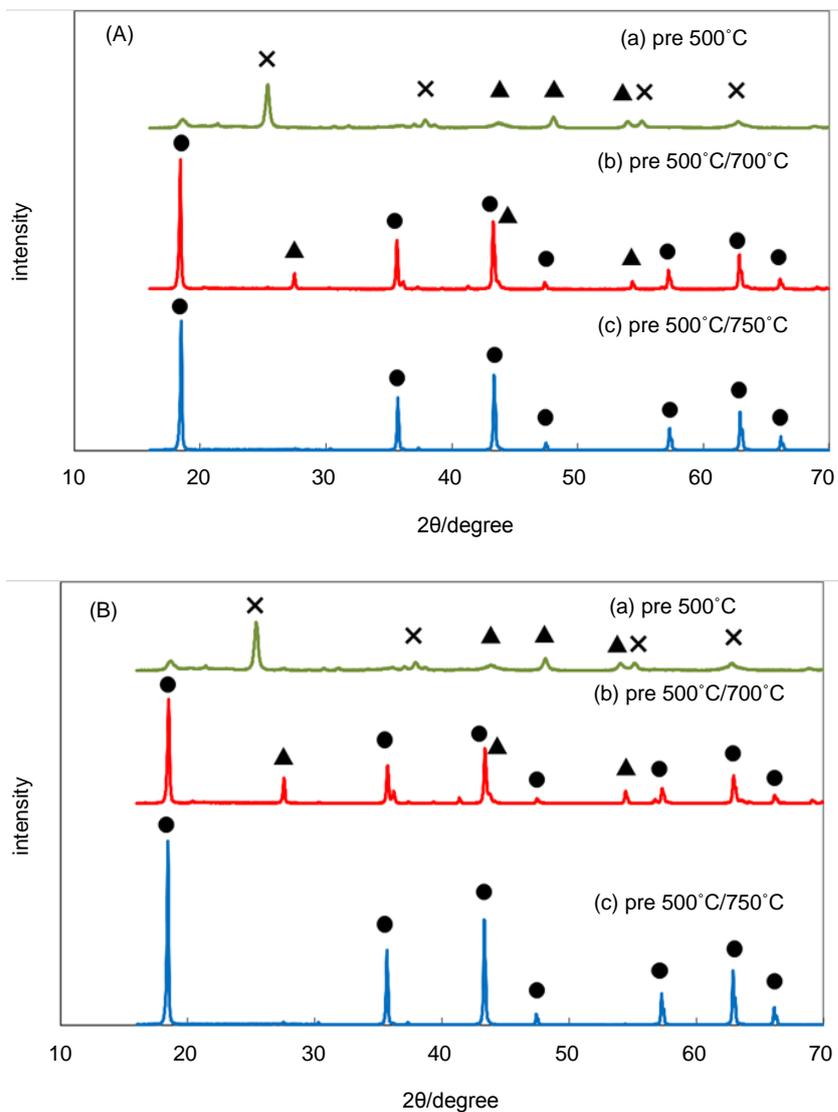
Now, to estimate the synthesized rate of the single phase  $\text{Li}_4\text{Ti}_5\text{O}_{12}$ , we defined a single phase rate in a following equation.

$$\text{Single phase rate (\%)} = [S(\text{Li}_4\text{Ti}_5\text{O}_{12}) / \{S(\text{Li}_2\text{TiO}_3) + S(\text{Li}_4\text{Ti}_5\text{O}_{12})\}] \times 100$$

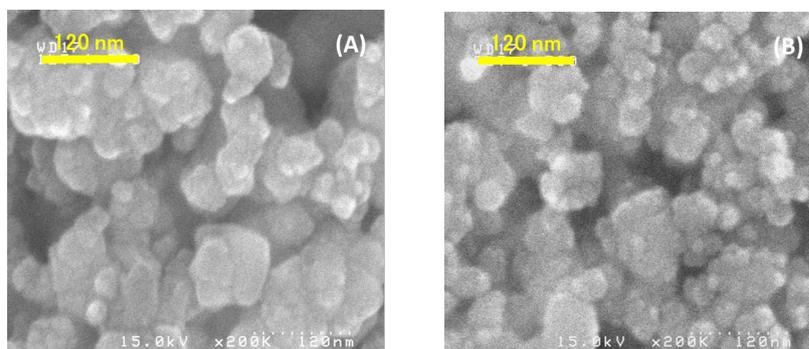
Here, “S” shows area of the peaks. The value was nearly same in 86% in **Figure 1(A)** and 89% in **Figure 1(B)** at  $700^\circ\text{C}$ , and was 100% at  $750^\circ\text{C}$  in any cases. Consequently, the effect of the specific surface area in the anatase  $\text{TiO}_2$  does not be indicated by the single phase rate, while the sintering temperature gave the great effect.

### 3.2. SEM Observation

The intermediate was produced by the pre-sintering the mixture of  $\text{CH}_3\text{COOLi}\cdot 2\text{H}_2\text{O}$  and anatase  $\text{TiO}_2$  at  $500^\circ\text{C}$ , and was showed in **Figure 2(A)**;  $30\text{ m}^2/\text{g}$  and **Figure 2(B)**;  $50\text{ m}^2/\text{g}$  anatase  $\text{TiO}_2$ . These results showed two



**Figure 1.** XRD patterns of (A) applying the (30 m<sup>2</sup>/g); and (B) 50 (m<sup>2</sup>/g) anatase TiO<sub>2</sub> respectively, addition to (a) pre-sintering temperature at 500 (°C); (b) sintering temperature at 700 (°C); and (c) at 750 (°C) after the pre-sintering. The mark shows ●: Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub>; ▲: Li<sub>2</sub>TiO<sub>3</sub>; and ✕: TiO<sub>2</sub>.



**Figure 2.** SEM images of the intermediate synthesized by applying (A) 30 (m<sup>2</sup>/g); (B) 50 (m<sup>2</sup>/g) anatase TiO<sub>2</sub> after pre-sintering at 500 (°C).

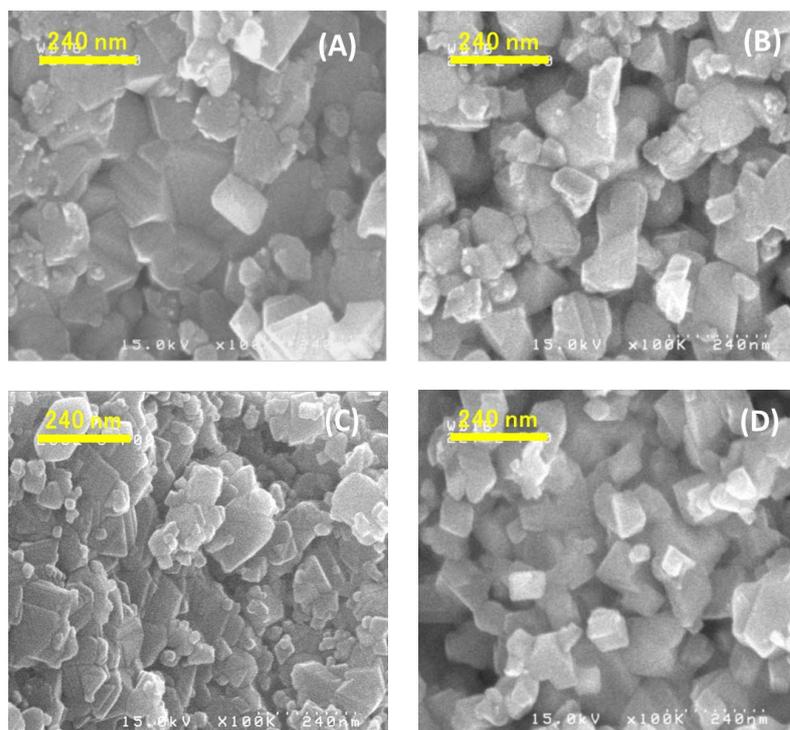
grain species to differ from contrast, which will indicate the unreacted  $\text{TiO}_2$  and  $\text{Li}_2\text{TiO}_3$  as shown in the XRD pattern. The intermediate would consist of the heterogeneous parts by them. The average grain size of the intermediate was different from 60 nm at **Figure 2(A)** and 30 nm at **Figure 2(B)**, and the specific surface area was about  $26.6 \text{ m}^2/\text{g}$  and  $33.0 \text{ m}^2/\text{g}$  respectively. The specific surface area of the applying anatase  $\text{TiO}_2$  will give the effect to the average grain size and specific surface area of the intermediate.

**Figure 3** showed homogeneous grains without almost the contrast by the sintering at  $700^\circ\text{C}$  or  $750^\circ\text{C}$ , especially the heterogeneous parts was not observed at  $750^\circ\text{C}$  at all. In fact, the single phases rate is 100%, which would support acquisition of the single phase  $\text{Li}_4\text{Ti}_5\text{O}_{12}$ . While the average grain size trended for greater with the high temperature about **Figure 3(A)**; 101 nm, **Figure 3(B)**; 83 nm, **Figure 3(C)**; 124 nm and **Figure 3(D)**; 87 nm, it will show no much change by applying the  $50 \text{ m}^2/\text{g}$  anatase  $\text{TiO}_2$ . Similarly, the specific surface area was about (a);  $8.3 \text{ m}^2/\text{g}$ , (b);  $14 \text{ m}^2/\text{g}$ , (c);  $5.1 \text{ m}^2/\text{g}$  and (d):  $12 \text{ m}^2/\text{g}$ , hence the single phase  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  was obtained with high specific surface area by the  $50 \text{ m}^2/\text{g}$  anatase  $\text{TiO}_2$  at sintering temperature of  $750^\circ\text{C}$ . Furthermore, the crystallization will be higher by the grain of a horned shape.

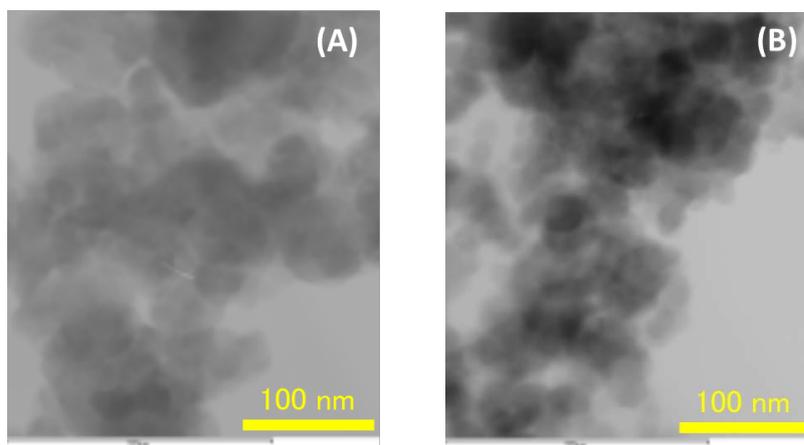
### 3.3. TEM Observation

**Figure 4** shows TEM images at pre-sintering temperature of  $500^\circ\text{C}$ . The minute  $\text{TiO}_2$  particles will encircle the  $\text{Li}_2\text{TiO}_3$  at the  $50 \text{ m}^2/\text{g}$  anatase  $\text{TiO}_2$  specially, which may take advantage of the  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  synthesis by the following sintering. These images did not indicate a clear morphology like a facet in this pre-sintering, and would show a very low crystallization.

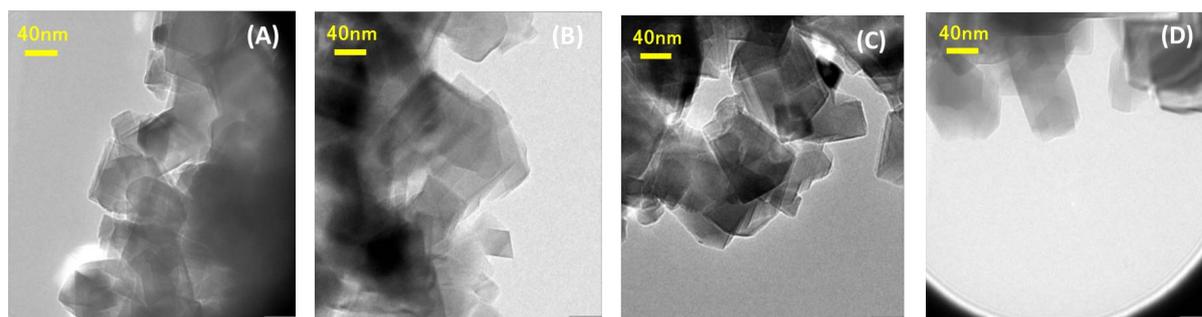
TEM images show the samples by the sintering temperature of  $700^\circ\text{C}$  or  $750^\circ\text{C}$  in **Figure 5**. The  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  particles would be the higher crystallization to form at an angular grain which included the facets in the morphology, while the sintering temperature and the specific surface area of the anatase  $\text{TiO}_2$  would hardly give the effect for the synthesized  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  not to be obtained about the clear differences. The morphology have been reported about the synthesis by using nano-particle or nano-wire  $\text{TiO}_2$ , which have described that the nano-particle or nano-wire  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  was obtained on keeping the  $\text{TiO}_2$  morphology like a mold [22] [23]. This knowledge will suggest the synthesis mechanism for the solid phase synthesis of  $\text{Li}_4\text{Ti}_5\text{O}_{12}$ , which the (002) face of  $\text{Li}_2\text{TiO}_3$



**Figure 3.** SEM images of the synthesized  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  which is (A)  $700^\circ\text{C}$ , by the  $30 \text{ (m}^2/\text{g)}$ ; (B)  $700^\circ\text{C}$ , by the  $50 \text{ (m}^2/\text{g)}$ ; (C)  $750^\circ\text{C}$ , by the  $30 \text{ (m}^2/\text{g)}$ ; (D)  $750^\circ\text{C}$ , by the  $50 \text{ (m}^2/\text{g)}$  anatase  $\text{TiO}_2$ .



**Figure 4.** TEM images of the intermediate synthesized by applying (A) 30 (m<sup>2</sup>/g); (B) 50 (m<sup>2</sup>/g) anatase TiO<sub>2</sub> after pre-sintering at 500 (°C).



**Figure 5.** TEM images of the synthesized Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> which is (A) 700 (°C), by the 30 (m<sup>2</sup>/g); (B) 700 (°C), by the 50 (m<sup>2</sup>/g); (C) 750 (°C), by the 30 (m<sup>2</sup>/g), (D); 750 (°C), by the 50 (m<sup>2</sup>/g) anatase TiO<sub>2</sub>.

plays a role of the mold in the spinel structure. These may show that the mobility of titanium ions is improved to close to the (002) Li<sub>2</sub>TiO<sub>3</sub> and the (111) Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub>.

#### 4. Conclusion

We attempted the single phase Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> synthesis with the higher specific surface area by using CH<sub>3</sub>COOLi·2H<sub>2</sub>O as a Li source that was melted with low temperature at about 300 °C, and 30 or 50 m<sup>2</sup>/g anatase TiO<sub>2</sub> as a Ti source. The synthesis method obtained Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> by the sintering at 700 °C or 750 °C via Li<sub>2</sub>TiO<sub>3</sub> as the intermediate in pre-sintering at 500 °C. Then, the single phase Li<sub>4</sub>Ti<sub>5</sub>O<sub>12</sub> was obtained with 12 m<sup>2</sup>/g specific surface area and the higher crystallization at 750 °C. The mechanism may give a suggestion as the improvement of mobility for the titanium ions.

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