SOLID STATE REACTION SYNTHESIS AND ELECTROCHEMICAL CHARACTERIZATION OF Na₂Ti₃O₇ AS ANODE MATERIAL FOR SODIUM ION BATTERIES

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Abstract

In this work, we were successfully synthesized Na₂Ti₃O₇ material by solid state reaction method which used NaOH and TiO₂ as the precursors. Structural and morphological characterization of synthesized materials were confirmed by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The XRD results indicated that, the Na₂Ti₃O₇ material with single crystal structure is formed at a reaction temperature of 900°C. The electrochemical performance of Na₂Ti₃O₇ material is evaluated by performing galvanostatic charge-discharge (GCD) analysis on the CR2032 cells at different rates. The Na₂Ti₃O₇ material delivered a specific capacity of 189 mAh/g at 0.1C within the potential range from 0 to 2.5 V. The results suggest that the Na₂Ti₃O₇ is a promising material as anode electrode for sodium ion batteries.

Keywords: Na₂Ti₃O₇ anode; sodium ion battery; Rietveld refinement; solid state reaction synthesis.

1. Introduction

Though sodium ion batteries (SIBs) were researched in tandem with lithium ion batteries (LIBs) from the late 1970s and early 1980s, its inferior performance in comparison with LIBs was the main reason for the decline of the SIBs [1]. Recently, research on SIBs has been gaining interest due to the explosive growth in the exploitation and use of renewable energy and especially, the rapid development of electric vehicles that have raised concerns about the shortage of lithium in the near future [2]. Among anode materials for SIBs that have been studied, Na₂Ti₃O₇ attracts attention due to its high capacity and low working potential. It has reported that the theoretical capacity of

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Na₂Ti₃O₇ can reach 354 mAh/g [3, 4]. In fact, several groups have announced that the experimental capacity of the material can exceed 200 mAh/g [4], which is higher than most intercalation anode materials [5]. In addition, its working potential of about 0.3 V is not only beneficial for the voltage of the SIBs but also helps to avoid the plating of Na metal on the electrode surface that takes place at around 0 V vs Na/Na⁺ [6]. Despite these advantages, a serious problem that hinders the applicability of Na₂Ti₃O₇ is its short cycle life. Generally, this issue is often attributed to the volumetric expansion and the formation of the solid electrolyte interphase (SEI) layer on the surface of the material during charge/discharge. A volumetric expansion of about 30% during charge/discharge has estimated for Na₂Ti₃O₇ by in-situ X-ray diffraction analyses [7]. The formation of the SEI layer on the surface of the Na₂Ti₃O₇ anode has also investigated [8]. However, so far only studies so far have assessed the prolonged degradation of the anode during cycle test [9].

Several methods have been used to synthesize $Na_2Ti_3O_7$ materials including solid state reaction method [10, 11], sol-gel method [12, 13], hydrothermal method [14, 15], spray-drying method [16], sonochemical method [17], etc. Among them, the solid state reaction method was widely used due to its simplicity and the well crystallization of the product. However, while most authors used Na_2CO_3 and TiO_2 as the precursors for the $Na_2Ti_3O_7$ synthesis [18-20], very few groups were interested in the solid state reaction of NaOH and TiO_2 .

In this work, $Na_2Ti_3O_7$ was synthesized by solid state reaction method which used NaOH and TiO₂ as the precursors. The effects of the calcination temperature on the morphology and the composition of the products were investigated. Electrochemical performance of the $Na_2Ti_3O_7$ anode was studied based on the galvanostatic charge/discharge measurements. By studying the transformation of the voltage profiles during cycling test, the degradation behaviour of the anode was also mentioned.

2. Experiment

Na₂Ti₃O₇ materials have synthesized by solid state reaction method. The mixture consisted of 4.792 g of anatase TiO₂ (analytical grade, Sigma Aldrich) and 1.6 g NaOH (analytical grade, Sigma Aldrich) were ball milled in acetone at a speed of 200 rpm for 4h. The received slurry was then dried at 120°C for 4h. Finally, the mixture was calcined in air at 700, 800 and 900°C for 16h to obtain the materials.

The morphology of the materials was observed by Hitachi S-4800 FESEM. The X-ray powder diffraction analyses were performed by Siemens D5000 X-ray diffractometer using CuK α radiation at room temperature. The 2-theta angle of diffraction (2 θ) was scanned from 5° to 70° at a step of 0.03°/sec. The Rietveld refinement of X-ray diffraction data was conducted by the FullProf software package [21]. The electrochemical

characteristics of Na₂Ti₃O₇ material is evaluated by performing galvanostatic chargedischarge analysis on the CR2032 cells. Galvanostatic charge-discharge analysis was measured by a battery test system (AcuTech Systems, BAT-750B) at room temperature in the potential range of 0-2.5 V (vs. Na/Na⁺) at 0.1C (10 mA/g), 0.2C (18 mA/g), 1C (80 mA/g) and 5C (400 mA/g).

For the preparation of Na₂Ti₃O₇ electrodes, 0.5 gram of slurry consisted of the Na₂Ti₃O₇ material, carbon black and polyvinylidene fluoride (PVDF) at a mass ratio of 70:20:10 was casted onto a copper foil, and then dried at 80°C in a vacuum oven for 12 h. The resulted tape was rolling pressed to increase the density and cut into 16 mm diameter circles for using as the positive electrode, the mass loading of materials was about 8-12 mg. Half-cells (CR2032) were fabricated in a dry glove box, including the Na₂Ti₃O₇ electrodes as the working electrodes, polyethylene as the separator, and sodium metal foils as counter electrodes. The liquid electrolyte was 1 M NaClO₄ in ethylene carbonate (EC), ethyl methyl carbonate (EMC), and dimethyl carbonate (DMC) (1:1:1 volume ratio).

3. Results and Discussion

The structure of the synthesized materials was investigated by XRD analyses. Fig. 1a showed the X-ray diffraction patterns of the materials that were synthesized at different calcination temperatures. As seen in the figure, three components including Na₂Ti₃O₇ (space group P21/m - JCPDS 72-0148), Na₂Ti₆O₁₃ (space group C2/m - JCPDS 73-1398) and Na₂Ti₇O₁₅ (space group C2/m [22]) have indexed in the XRD patterns of the samples synthesized at temperatures of 700-900°C.



Fig. 1. (a) X-ray powder diffraction patterns of the materials that were synthesized at different calcination temperatures (the calculated powder diffraction patterns of $Na_2Ti_7O_{15}$, $Na_2Ti_6O_{13}$ and $Na_2Ti_3O_7$ were also inserted); (b) X-ray diffraction and Rietveld refinement patterns of the $Na_2Ti_3O_7$ that was synthesized at 900°C. 18

The crystal structures of these phases were illustrated in Fig. 2. No fingerprints of the TiO₂ precursor were detected on the diffraction patterns. Table 1 shows the detail of the product compositions which were calculated by Rietveld refinement method. The calculated results revealed that Na₂Ti₃O₇ accounts for about 31 wt% of the sample composition at 700°C. When the temperature was raised to 800°C, about 85.5 wt% of the Na₂Ti₇O₁₃ phase was observed in the synthetic material. At 900°C, diffraction pattern showed the presence of only Na₂Ti₃O₇, which also confirmed by the overlab between X-ray diffraction and Rietveld refinement patterns of the Na₂Ti₃O₇ that was synthesized at 900°C (Fig. 1b).



Fig. 2. Illustrations of the crystal structures of Na₂Ti₇O₁₅, Na₂Ti₆O₁₃ and Na₂Ti₃O₇.

The process of changing from a phase with a low sodium concentration at 700°C (Na₂Ti₇O₁₅, Na₂Ti₆O₁₃) to Na₂Ti₃O₇ phase at 900°C showed that Na⁺ ions were continuosly intercalated into the solid phases to form higher sodium concentration phase at higher temperature. The phase transformation from Na₂Ti₆O₁₃ to Na₂Ti₃O₇ due to the intercalation of Na⁺ ion into TiO₂ in a NaOH solution was observed during hydrothermal synthesis of the materials [7]. It also should be noted that though Na₂Ti₃O₇ and Na₂Ti₆O₁₃ phases have often mentioned, Na₂Ti₇O₁₅ is rarely found in the products of sodium titanium oxides which were synthesized by solid state reaction method. The detail of refinement structure of Na₂Ti₃O₇ is shown in the Table 2. The calculated results are good agreement with the literature data [23].

Table 1. Structura	l parameters as re	fined based on	X-ray powder diff	raction data for the
NaNi _{0.5} Ti _{0.5} O ₂ 1	naterial that was .	synthesized with	th the pre-milling ti	ime of 16 hours.

	Components (% wt.)		
Annealing temperatures	Na ₂ Ti ₇ O ₁₅	Na ₂ Ti ₆ O ₁₃	Na ₂ Ti ₃ O ₇
700°C	31.4 %	37.6 %	31 %
800°C	-	14.5 %	85.5 %
900°C	-	-	100 %

Space group	<i>P21/m</i> (11)
a; b; c (Å)	0.9134; 0.3803 ; 0.8571
β (°)	101.603
Na1 (<i>x</i> , <i>y</i> , <i>z</i>)	(0.5932, 1/4, 0.6957)
Na2 (<i>x</i> , <i>y</i> , <i>z</i>)	(0.1535, 1/4, 0.4950)
Ti1 (<i>x</i> , <i>y</i> , <i>z</i>)	(0.9837, 1/4, 0.1432)
Ti2 (x, y, z)	(0.6767, 1/4, 0.2466)
Ti3 (<i>x</i> , <i>y</i> , <i>z</i>)	(0.2793, 1/4, 0.0309)
O1 (<i>x</i> , <i>y</i> , <i>z</i>)	(0.1823, 1/4, 0.2120)
O2 (x, y, z)	(0.4657, 1/4, 0.1416)
O3 (<i>x</i> , <i>y</i> , <i>z</i>)	(0.6464, 1/4, 0.4472)
O4 (<i>x</i> , <i>y</i> , <i>z</i>)	(0.9054, 1/4, 0.3108)
O5 (x, y, z)	(0.7512, 1/4, 0.0084)
O6 (<i>x</i> , <i>y</i> , <i>z</i>)	(0.3175, 1/4, 0.8007)
O7 (x, y, z)	(0.0401, 1/4, 0.9132)

Table 2. Structural parameters as refined based on X-ray powder diffraction data for the $Na_2Ti_3O_7$ material that was synthesized at 900°C.

The SEM images of the Na₂Ti₃O₇ powders that were calcined at different temperatures were showed in Figures of 3a, 3b and 3c, respectively. As seen in the Fig. 3a, the material that was calcined at 700°C had poor crystallinity. The SEM image showed clusters of granules of less than 100 nm in size that stick together in unclear shapes. When the calcination temperature was raised to 800°C, the small granules grew into larger grains with clearer grain boundaries. At the temperature of 900°C, the grains size increased sharply up to 1 μ m. Based on the comparison of SEM images, it seems that only the grains grow, while the shape of the clusters is almost unchanged at different reaction temperatures. This observation is consistent with the prediction above that Na⁺ ions intercalated into TiO₂ during the reaction to form Na₂Ti₃O₇.

The electrochemical characteristics of the Na₂Ti₃O₇ material that were synthesized at 900°C were shown in Fig. 4. As seen in Fig. 4a, the first, second and third discharge capacities of the electrode at a rate of 0.1C (10 mA/g) were 189.5, 107.4 and 94.7 mAh/g, respectively. At the first cycle of charge and discharge, the discharge profile showed three voltage plateaus D₁, D₂ and D₃ respectively at 0.79, 0.18 and 0.11 V, and the charge profile showed two voltage plateaus C₁ and C₂ respectively at 0.94 and 0.42V. There voltage plateaus were later confirmed by three reduction peaks R₁, R₂, R₃ and two oxidation peaks O₁, O₂ in the plot of differential capacity in Fig. 4c. At subsequent cycles, only four plateaus which correspond to the reduction peaks of R₁, R₂ and the oxidation peaks of O₁, O₂ were observed in the profiles of charge-discharge. Origin of these redox peaks was mentioned in the literature by many authors. Most of 20

them agreed that the redox couples of R_1/O_1 peaks can be assigned for the intercalation /de-intercalation processes of Na^+ ions into/out of black carbon in the cathodes [3].



Fig. 3. SEM images of the materials that were synthesized at 700 (a), 800 (b), and 900°C (c).



Fig. 4. Electrochemical characteristic of the Na₂Ti₃O₇ electrode:

(a) Profiles of voltage versus capacity for the electrode during three initial cycles,

- (b) Cycling behavior and coulombic efficiency of the electrode,
- (c) Differential capacity curves of the electrode at the three initial cycles,
- (d) Rate capability of the electrode.

However, the redox reactions related to the R_2/O_2 peaks were in controversial. J. M. Tarascon et al., via in-situ XRD analyses, claimed that the R_2/O_2 peaks originated from the transformations of $Na_2Ti_3O_7/Na_4Ti_3O_7$ phases [3]. X. D. Guo et al., based on in-situ synchrotron XRD results, proved that these redox peaks were related to the phase transformations of layered $Na_2Ti_3O_7$ and tunnel $NaTi_{1.25}O_3$ [7]. About reduction peak R₃, J. M. Tarascon et al. suggested that it might be assigned to the formation of $Na_{16}Ti_{10}O_{28}$ phase at the end of the discharge [3]. The cycling performance of the $Na_2Ti_3O_7$ cathode was presented in Fig. 4b. The capacity of the cathode decreased rapidly with only ca. 32% capacity compared to the second cycle remained after 100 cycles of charge-discharge. As seen in Fig. 4d, the cathode also exhibited a poor rate capability with 34 mAh/g and 4.25 mAh/g remained at the rates of 1C and 5C, respectively. However, it can be seen that the cathode seems to have better cycling performance at higher charge-discharge rates.



Fig. 5. (a) Profiles of voltage versus state of charge/discharge for the electrode at the 2nd, 3rd, 4th, 5th and 6th cycles of charge/discharge; (b) Profiles of voltage versus state of charge/discharge for the electrode at the 10th, 20th, 30th, 40th and 50th cycles of charge/discharge.

The voltage decay behavior was investigated to study the degradation of the Na₂Ti₃O₇ cathode. The profiles of voltage versus state of charge/discharge for the electrode were shown in Fig. 5. In the first cycle, the voltage variation is negligible indicating a relatively stable electrode. The potential increase of the discharge profiles in the first cycle shows that the Na⁺ ions intercalation process is easier with each cycle. However, the charge profiles have unstable change. In particular, from the 4th cycles onwards, the charge profiles started to be interference. This instability may be related to the volumetric shrinkage in the Na⁺ ions de-intercalation. The charge-discharge profiles of the 10th cycle to 50th cycle is shown in Fig. 5b. It can be seen clearly that, the charge voltage increases more strongly than the discharge potential, which is due to the structural 22

disruption of the material. These observations indicated different voltage decay characteristics during charge and discharge.

4. Conclusion

The Na₂Ti₃O₇ material was successfully synthesized by a solid state reaction method which used NaOH and TiO₂ as the precursors. The product compositions depend on the reaction temperature. The calculated results revealed that Na₂Ti₃O₇ accounts for about 31 wt% of the sample composition at 700°C. When the temperature was raised to 800°C, the component Na₂Ti₃O₇ phase increases up to 85.5 wt% in the synthetic material and single phase Na₂Ti₃O₇ is formed at 900°C. The Na₂Ti₃O₇ material delivered a specific capacity of 189 mAh/g at 0.1C within the potential range from 0 to 2.5 V. However, the capacity of the Na₂Ti₃O₇ material decreased rapidly after 100 cycles of charge-discharge, only remained 32% capacity compared to the second cycle.

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PHẢN ỨNG TỔNG HỢP TRẠNG THÁI RẮN VÀ TÍNH CHẤT ĐIỆN HÓA CỦA Na₂Ti₃O₁₇ LÀM VẬT LIỆU CỰC DƯƠNG CHO ẮC QUY NATRI-ION

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Tóm tắt: Trong nghiên cứu này, chúng tôi đã tổng hợp thành công vật liệu Na₂Ti₃O₇ bằng phương pháp phản ứng ở trạng thái rắn có sử dụng tiền chất là NaOH và TiO₂. Đặc điểm cấu trúc và hình thái của vật liệu tổng hợp được xác nhận bằng nhiễu xạ tia X (XRD) và kính hiển vi điện tử quét (SEM). Kết quả XRD chỉ ra rằng, vật liệu Na₂Ti₃O₇ với cấu trúc đơn tinh thể được tạo thành tại nhiệt độ phản ứng là 900°C. Hiệu suất điện hóa của vật liệu Na₂Ti₃O₇ được đánh giá bằng cách thực hiện phép đo nạp - xả dòng không đổi trên các ắc quy cúc áo CR2032 tại các tốc độ nạp xả khác nhau. Vật liệu Na₂Ti₃O₇ có công suất riêng là 189 mAh/g tại tốc độ nạp - xả 0,1C trong khoảng điện thế từ 0 đến 2,5 V. Kết quả cho thấy rằng, Na₂Ti₃O₇ là một vật liệu đầy hứa hẹn làm điện cực anot cho ắc quy ion natri.

Từ khóa: Cực dương Na₂Ti₃O₇; ắc quy natri-ion; phân tích Rietveld; phản ứng pha rắn.

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